

ISSN 2313-5891 (Online)
ISSN 2304-974X (Print)

Ukrainian Food Journal

Volume 5, Issue 1
2016

Київ

2016

Київ

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Editorial office address:

National University
of Food Technologies
Volodymyrska str., 68
Ukraine, Kyiv 01601

Адреса редакції:

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харчових технологій
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Scientific Council of the National
University of Food Technologies
recommends the journal for printing.
Minutes № 11, 24.03.2016

Рекомендовано вченою радою
Національного університету
харчових технологій.
Протокол № 11 від 24.03.2016 р.

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Topic covered by the journal include:

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Studies must be novel, have a clear connection to food science, and be of general interest to the international scientific community.

Ukrainian Food Journal is indexed by scientometric databases:

Index Copernicus (2012)

EBSCO (2013)

Google Scholar (2013)

UlrichsWeb (2013)

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CABI full text (2014)

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Universal Impact Factor (2014)

Directory of Open Access scholarly Resources (ROAD) (2014)

European Reference Index for the Humanities and the Social Sciences (ERIH PLUS) (2014)

Directory of Open Access Journals (DOAJ) (2015)

InfoBase Index (2015)

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Effect of rape seeds microwave pretreatment on the composition and antioxidative properties of press rape oil

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Abstract

Keywords:

Microwave
Pretreatment
Rape
Oil
Oxidative
Stability

Introduction The influence of microwave pretreatment of rape seeds on the pressing oil yield, acid and peroxide values of oil, fatty acid composition of oil, phosphorus-containing substances, tocopherols and carotene content in oil as well as oil oxidation stability were studied in this work.

Materials and methods Peroxide and acid values of oils were determined according to procedures given by IUPAC, total phosphorus and carotenes content – by spectrophotometric methods, fatty acid composition and tocopherols content of oils – by chromatographic methods. The induction periods of oil oxidation were calculated from the curve of oxidation in the presence of 2,2-azo-bis-isobutyronitrile.

Results and discussion The advantages of microwave heating are very high rates of temperature increase and as a consequence the high rates of the moisture decrease. We have shown that decreasing of rape seed moisture from 13.0 to 7.2 % had run during 10 and 30 min under microwave and conventional heating respectively. It was shown that pressing oil yield after microwave pretreatment of rape seeds increased by 16-90 %. The final seed moisture after pretreatment was the main factor that determines the pressing oil yield. The oil yield for the seeds with the same moisture after microwave and conventional pretreatment was higher in a case of microwave pretreatment by 16 %.

Data obtained have shown that used microwave heating had no effect on the fatty acid composition of rape oil. But oil from the rape seeds after microwave pretreatment had lower acid and peroxide values, higher phosphorus, tocopherols and carotenes content. Increasing of oxidative stability of oil sample after seed microwave pretreatment was confirmed by rising of induction period time of oil oxidation, initiated by 2,2-azo-bis-isobutyronitrile. Induction period of control sample oil was equal to 27 min. and for sample oil from microwave pretreated seeds it was three time longer and exceed 90 min.

Conclusions Microwave pretreatment of rape seeds can be used for increase of rape seeds pressing affectivity and improving of oil biological value and oxidation stability.

Article history:

Received 14.01.2016

Received in revised
form 19.02.2016

Accepted 24.03.2016

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Introduction

The main advantage of microwave heating is its easy and quick penetration inside products and as result their temperature increases very rapidly. At the same time the energy of microwave frequency waves are strongly absorbed by water molecules. Thus the higher moisture of product the higher temperature increase under microwave heating. The high temperature of materials under microwave heating induces the high pressure inside the cells, rupture of cell wall and releasing the cell components [1].

Microwave heating is widely used for sterilization, pasteurization, cooking and drying the product. It is also used for intensification of extraction of different substances for food and other application.

The development of microwave assisted extractions was first reported by Ganzler *et al.* [2] and Ganzler and Salgo [3] as a sample preparation method for chromatography. Subsequently this method was proposed to use for extraction of different compounds from plant and animal materials, such as essential oil [4-7], phenolics with maximal antioxidant activities [8, 9] and other bioactive compounds from plant materials [10, 11].

Microwave pretreatment of raw material is predominately used for edible oil recovery from oil seed [12]. Valentova *et al.* [13] have shown that yield of cold-pressed rape oil increased with increasing doses of microwave irradiation up to about 40 % of seed mass (w/w) while the amount of oil, obtained from untreated flakes, was about 33% (w/w). The degree of lipid oxidation was significantly lower after microwave treatment, total phosphorus contents were higher in the oils from treated seeds. At the same time Terigar *et al.* [14] have not detected the effect of microwave on the phospholipid content in oil. Azadmard-Damirchi *et al.* [15] have shown that microwave pretreatment of rape seed can increase the oil yield (by 10%), phytosterols (by 15%), tocopherols (by 55%) of the pressing oil and increased oxidative stability to 8 h. In our previous study we have shown the influence of microwave pretreatment on the yield of press oil from soybean, sunflower, walnut and pumpkin seed [16].

Moreno *et al.* [17] have detected the increase of extraction efficiency in Soxhlet-hexane extraction coupled after microwave pretreatment of avocado. Terigar *et al.*, [14] have proposed pilot-scale continuous microwave-assisted extraction system for soybean and rice bran oil.

On the other hand there are contrary data about influence of microwave roasting of sunflower seed on the oil quality [18]. Using extraction of oil by n-hexane the authors have revealed that microwave roasting decreased the oil content of the seeds significantly. Analysis of the extracted oils demonstrated a significant increase in free fatty acids (FFA) content, peroxide, p-Anizidine and saponification values, density, diene, conjugated triene and color values for roasting periods of 10 and 15 min. Such significant increase of saponification value is very doubtful on the basis of the fatty acids composition changes since the main changes concerned only the oleic and linoleic acids ratio but not the content of fatty acids of different molecular weight. The iodine values and the amounts of tocopherol constituents of the oils were remarkably decreased. Microwave heating resulted in increase of oleic acid content by 16–42% and decrease of linoleic acid content by 17–19%, but palmitic and stearic acid contents were not affected significantly.

It is obviously that such substantial changes of linoleic acid content could be only the result of its oxidation and these data indicate about acceleration of oil oxidation as a result of microwave roasting.

The influence of microwave roasting on fatty acid content was reported for other oils [19, 20].

However Yoshida *et al.* [21], have reported no significant ($P > 0.05$) changes in the FA composition within 12 min of roasting in sunflower seed oil. Yen [22], Yoshida and Kojimoto [19], Kim *et al.* [23] have not found differences in FA composition of sesame seed oils and of rice germ oil treated at different roasting temperatures and time.

Thus the question about the role of microwave heating in oil seed processing and its influence on the oil quality is still open. In our opinion parameters of microwave heating are very important for oil quality, notably the temperature of heating and seed moisture in the case of seed pretreatment. In this study we have investigated the influence of microwave pretreatment conditions on the yield of press rape oil and its composition.

Materials and methods

Materials. Seeds of the winter rape plant (*Brassica napus*, low content of glucosinolates and zero erucic acid), obtained from local market were used in this research. Moisture and oil content of seeds were 8.5 % and 46.4 % (of dry substances), respectively.

Proximate analysis. Moisture content was determined by drying of samples to stable mass. Determination of oil content in seeds and cakes was carried out by extraction in a Soxhlet extractor on a water bath (75–80°C) for 16–18 h with *n*-hexane (b. p. 68°C). The mass of extracted oil was calculated by weighing of seed residues after extraction.

Microwave pretreatment of rape seeds. 500 g of rape seed were moistened to moisture content 11–13 % by water vapour and then were heated in domestic microwave or in electric oven. Final seed moisture varied from 3 to 10 %. The frequency of microwave was 2450 MHz and capacity varied from 100 to 300 W. The temperature of microwave heating varied from 85 to 105 °C and conventional heating from 100 to 105 °C, respectively.

Seed pressings. Pressings of the seeds were performed after heating using the laboratory screw press at 55–60°C operating temperature. Oil yield was calculated as difference between oil content in seeds and oil residuals in cake after pressing.

Quality parameters of pressed oils evaluation. Peroxide value (PV) and acid value were determined according to procedures given by IUPAC (2.501 and 2.201, respectively) [24]. Total phosphorus content was determined by a spectrophotometric method measuring absorbance of yellow molybdenumvanadiophosphoric acid at $\lambda = 400$ nm using dry ashing and magnesium oxide as an ashing aid.

Tocopherols content determination. Content of tocopherol homologue was determined by high-performance liquid chromatography of unsaponifiable substances. For this purpose 5 g of oil was saponificated at 85–90 °C in the presence of 15 ml methanol, 10 ml of 10 % water solution of ascorbic acid and 4 ml of 50 % potassium hydroxide solution during 30 min. Unsaponifiable matters were extracted thrice by diethyl ether. Extract was properly washed by distilled water and dried by incubation with sodium sulphate during 30 min. Ether was evaporated on rotor evaporator at 40...50°C and residual was diluted in 10 ml of methanol. Obtained solutions were analyzed on Hewlett Packard liquid chromatograph model HP 1100 with reversed-phase column Hypersil MOS (200 mm x 2.1 mm). Detection was done with fluorescence (wave length of: excitation – 295 nm, absorption – 330 nm) and diode matrix detectors. The column was held at constant temperature by a water jacket at 40 °C. The eluant was composed of acetonitril/water (70:80), the eluation velocity was 0.4 ml/min. The standard solutions of α -tocopherolacetate (Supelco) were used for calibration.

Total carotenes content determination. Total carotenes content was determined by a spectrophotometric method. Solution of oil in hexane (1:9) was used for absorbance measurement at $\lambda=451$ nm. Total carotenes content (g/100 ml) was calculated using the following equation:

$$C=10A/10\cdot256$$

where A correspond to the absorbance of oil solution at 451 nm and cuvette thickness equal to 10 mm and 256 is the specific absorption coefficient of β,β carotene at 451 nm [25].

Fatty acid composition of oils. Fatty acid composition was determined by gas-liquid chromatography of fatty acid methyl esters. They were prepared by IUPAC standard method 2.301 [24] and analyzed on Hewlett Packard gas chromatograph model HP 6890 with capillary column HP-88 (88%-cyanopropyl aryl-polysiloxane, 100m x 0.25 mm x 0.25 μ m film thickness (Agilent Technologies). The temperature of injector was 280 °C, and pf detector – 290 °C. The column temperature program of heating rate was from 60 to 230 °C. The rate of carrier gas was 1.2 ml/min. Identification of the fatty acids was performed by comparison of the retention times with standards mixture of fatty acid methyl esters (37 Component FAME Mix, Supelco).

Oxidative stability of rape oil. The oxidative stability of rape oil was estimated as changes of peroxide value during 12 h exposition of oil in thermostat at 70°C. The aliquots of oil were taken for PV determination every 2 h.

Induction periods of oil oxidation were calculated from the curve of oxidation, initiated by 2,2-azo-bis-isobutyronitrile (AIBN). Oxidation curves at 80 °C were measured as volume of absorbed oxygen. For oxidative reaction 2 g of oil mixed with 3 ml xylene and 0.3 ml of 0.1 mol/L solution of oxidation initiator 2,2-azo-bis-isobutyronitrile (AIBN) in xylene. Reaction mixture was oxygen purged during 1 min and thermostated at 80 °C during 10 min before measurements. Oxidation curves were plotted in coordinates: heating time (t, min) – height of absorbed oxygen column (H, mm).

Statistical analysis. Samples were analyzed in triplicate. Statistical analysis was performed using Microsoft Excel 2007 (Microsoft, City of Redmond, USA). The results were reported as mean \pm SD. Differences were considered to be significant at validity of $\alpha=0.95$.

Results and discussion

Microwave heating effects on the yield of press oil

In our previous work [16] we have shown that microwave pretreatment of oil seeds had resulted in the increase of pressing oil yield. The main parameter affected pressing oil yield was moisture of seeds before pressing. Data, obtained in this work, had shown that highest oil yield was achieved when seed moisture was about 3.0 % (Table 1). About 92 % of oil was recovered from seeds. The advantages of microwave heating are very high rates of temperature increase and as a consequence the high rates of the moisture decrease. We have shown that decreasing of seed moisture from 13.0 to 7.2 % had run during 10 and 30 min under microwave and conventional heating respectively. The oil yield for the seeds with the same moisture after microwave and conventional pretreatment was higher in a case of microwave pretreatment by 16 %. Obviously in a case of microwave heating the changes of seeds microstructure are involved also as contributing factor to oil yield [26].

Table 1
Oil yield as a function of parameters of rape seed pretreatment and pressing

Seed moisture before heating, %	Microwave capacity, W/200 g of seeds	Temperature of seeds, °C	Time of treatment, min	Final moisture of seeds, %	Oil yield, % from seed mass
13.0±0.3	300	105±3	10	7.2±0.2	29±3
13.0±0.3	100	80±2	10	10.0±0.3	25±2
11.0±0.2	300	100±2	10	3.0±0.2	38±1
11.0±0.2	100	84±3	10	7.2±0.3	29±2
13.0±0.4	-	100±2	10	12.0±0.3	20±4
13.0±0.3	-	100±2	30	7.2±0.2	25±3

Chemical composition of press rape oil

According to our data, rape oil, obtained after microwave pretreatment, had lower content of free fatty acids and considerably lower mean of peroxide value (Table 2), possibly due to result of a short-time microwave heating, durations of heating in order to reach the same seeds moisture were 10 min and 30 min in microwave and electric oven respectively. On the other hand, it is possible that microwave heating inactivates lipases and of oxidative enzymes, such as peroxidases and lipoxygenases, more effectively comparing with conventional heating.

Table 2
Characteristics of Rapeseed Oil from Seeds after heat pretreatment

Characteristic	Pretreatment heating	
	conventional	microwave
Pressing oil yield, %	25±1.5	38±1.8
Peroxide value (meq O kg ⁻¹ of oil)	1.8±0.2	0.4±0.1
Acid value (mg KOH g ⁻¹ of oil)	2.2±0.1	1.5±0.05
Total phosphorus content (mg kg ⁻¹ of oil)	27.7±1.3	31.8±1.5
Tocopherol content (mg·100 g ⁻¹ of oil):		
α tocopherol	12.3±0.10	13.3±0.15
β tocopherol	20.5±0.2	21.1±0.15
Total carotines content, %	0.003±0.001	0.047±0.005

We have obtained very low total phosphorus content in all oil samples, but still it was higher (about 15 %) in oil after microwave heating. Since neutral lipid fractions are extracted from seeds under pressing at first and such as phospholipids are polar lipids, obviously the higher oil yield the more polar lipid fractions content in oil.

The tocopherol homologues content was somewhat higher in the sample of rape oil from microwave treated seeds. The α-tocopherol content increase was 8 % to control samples. It is known that this homologue has lowest optimum of antioxidative activity that

is 10-25 mg/100 g of oil [27]. Thus it can cause the high oxidative stability of rape oil even at low concentration.

Analysis of fatty acid composition of obtained rape oil samples are shown in Table 3. We have not detected significant changes of fatty acid content in oil after microwave heating, neither decreasing linoleic acid content nor increasing oleic acid content as it was shown by [18-20]. We suppose that considerable changes in FA composition [18] could accompany very strong destruction of oil, that is not evident from other oil characteristics (FFA content, PV value) shown in those works. Our data have shown that “soft” conditions of microwave heating do not cause nor considerable changes in FA composition nor creation of trans isomers of fatty acids.

Effect of microwave pretreatment on oxidative stability of rape oil

Changes of peroxide value of rape oil from seeds, treated in electric and microwave oven, under oxidation condition are given on Fig. 1. It is evident that oil from seeds after conventional heating had very poor oxidative stability, its oxidation have started from the very beginning of oil heating and the rate of peroxide accumulation was very high. At the same time the rate of peroxide formation in oil from seeds after microwave heating was increasing slowly during first 4 hours and then peroxide content was constant until 10 h. At the end of observation the rate of peroxide formation declined in both oil samples possibly as result of peroxide dissipation to second products of oxidation.

Increasing of oxidative stability of oil sample after microwave pretreatment was confirmed also by rising of induction period time of oil oxidation, initiated by 2,2-azo-bis-isobutyronitrile. Induction periods of oil oxidation were calculated from the curve of oxidation (Fig. 2). Induction period of control sample oil was equal to 27 min. and for sample oil from microwave pretreated seeds it was three time longer and exceed 90 min.

Table 3

Fatty acid composition of rape oils

Fatty acid	Fatty acid, % from the common content	
	Oil after microwave pretreatment	Oil after conventional heating
C 16:0	4,50 ± 0,15	4,49 ± 0,15
cis-9-C 16:1	0,23 ± 0,05	0,21 ± 0,05
C 18:0	1,66 ± 0,10	1,67 ± 0,10
cis-9-C 18:1	59,27 ± 0,20	59,63 ± 0,20
cis-11-C 18:1	4,94 ± 0,15	4,76 ± 0,15
cis, cis-9,12-C 18:2	19,29 ± 0,20	19,08 ± 0,20
C 20:0	0,55 ± 0,05	0,54 ± 0,05
cis,cis,cis-9,12,15-C 18:3	7,68 ± 0,20	7,80 ± 0,20
cis-11-C 20:1	1,19 ± 0,10	1,17 ± 0,10
cis-13-C 22:1	0,10 ± 0,05	0,09 ± 0,05
C 24:0	0,12 ± 0,05	0,11 ± 0,05
cis-15-C 24:1	0,13 ± 0,05	0,13 ± 0,05

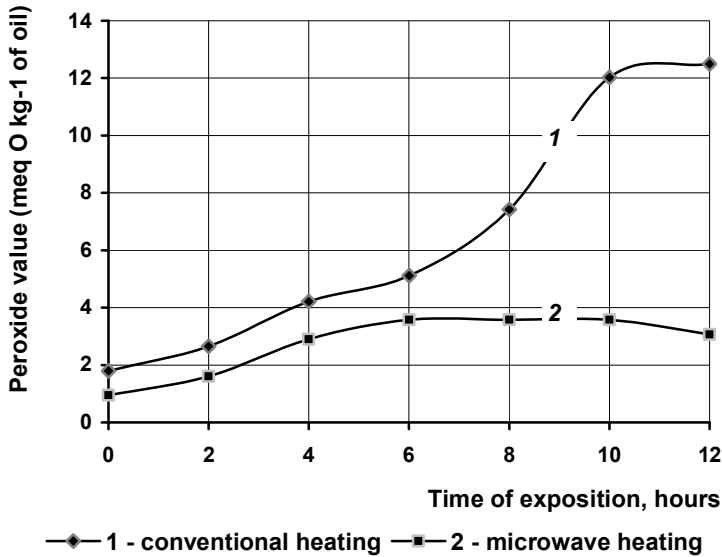


Fig. 1. Oxidation stability of rape oil after conventional and microwave pretreatment of seeds

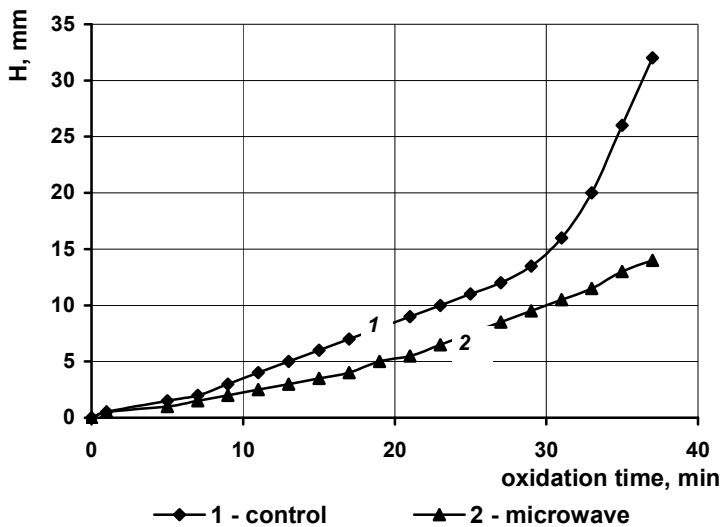


Fig. 2. Curves of rape oil oxidation, initiated by 2,2-azo-bis-isobutyronitrile

Conclusions

In this study we have shown that main parameters that influence the yield of press rape oil are seed moisture, which in turn depends on the temperature and the time of seed heating. The time which is necessary for achievement of proper seed moisture is considerably lower in case of microwave heating in comparison with conventional heating.

On the other hand since water has very high dielectric constant the higher initial seeds moisture the more effective heating.

Thus parameters that determine the effect of microwave heating on seed moisture are: microwave power, pretreatment duration as well as initial seed moisture. In addition, the influence of microwave heating on the oil yield is evidently the result of creation of proper inner microstructure of seed due to quick water heating and evaporation that results in creation of porous microstructure. Such microstructure is favorable for oil releasing.

Increasing of oil yield after microwave pretreatment is accompanied by increased content of polar lipids (phospholipids, carotenes and tocopherols) in oil. Microwave pretreatment of seeds did not influence the fatty acid composition of rape oil. Rape oil from the seeds after microwave pretreatment had low peroxide value and considerably higher oxidative stability.

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Effect of heat treatment with antioxidants on oxygen radical scavenging during storage of bell pepper fruits

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Abstract

Keywords:

Storage
Pepper
Antioxidant
Treatment

Article history:

Received 19.12.2015
Received in revised form
20.02.2016
Accepted 24.03.2016

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Introduction. Despite known effectiveness of heat preconditioning and antioxidant treatment for decreasing of oxidative damage induced by cooling, their combined effect on fruits of sweet bell pepper was never studied before.

Materials and methods. Fruits of sweet bell pepper Nikita F1 and Hercules F1, which were preconditioned with warm composition of antioxidants, were stored in $7 \pm 0,5$ °C. Content of malondialdehyde (MDA) was determined by thiobarbituric method. Superoxide dismutase (SOD) activity was determined by estimation of its ability to inhibit the reaction of auto-oxidation of adrenaline in alkaline medium with a modification in the stage of raw materials preparation. Activity of peroxidase (PO) and catalase (CAT) was determined by titration of undecomposed rest of hydrogen peroxide.

Results and discussion. Without using additional methods of prevention of chilling injury after 24 days third part of fruit is damaged. In pepper, treated with heat and composition of antioxidants, chilling injuries are seen only on 21st day independently from hybrid. Chilling injuries decrease in 3,9...4,5 times in comparison with untreated fruits. During the storage of pepper in temperature conditions above the cold-sensitivity threshold content of MDA increases constantly. Heat treatment with antioxidants changes a dynamic of MDA in the sweet bell pepper fruit. Till the 12th day of study level of lipid peroxidation remains stable in treated samples. Then, on each stage of storage, level of MDA raises by 5...15% depending on hybrid. On the 18th day (loss of commercial quality of control samples) level of MDA in the studied samples is lower in 1,7...2 times. Heat preconditioning with antioxidants decreases speed of SOD deactivation by 25% and CAT by 30...50%. Activity of PO during storage of pepper decreases till the moment of commercial quality loss and then raises. In studied fruits growth of PO activity starts 12 days after such in the control group. Between the activity of studied enzymes and content of MDA strong reverse correlations were found ($r = -0,81... \approx -1$) that testifies to the antioxidant functions of these enzymes.

Conclusions. Combination of heat preconditioning and antioxidant treatment for preparation of pepper to storage increases effectiveness of functioning of the system aimed on the reactive oxygen species utilization, which allows to minimize chilling injury.

Introduction

Fruit and vegetables during the periods of treatment, storage and sale can be potentially affected by the numerous negative factors (low temperature, storage condition, mechanical damage), which can lead to the oxidative stress. Oxidative stress occurs when partly reduced reactive oxygen species (ROS), such as singlet oxygen ($^1\text{O}_2$), superoxide anion (O_2^-), hydrogen peroxide (H_2O_2), hydroxyl (OH^\cdot) and peroxynitrite (ONOO^-) are overproduced, which leads to the failure of the organism ability to maintain a cell redox homeostasis [1]. Duration of the ROS affect on tissues is determined by antioxidant system, which is a set of cell, tissue and organism defense mechanisms that are aimed on the homeostasis maintenance. Endogenous antioxidants allow preserving low constant level of products of lipid peroxidation, thus preventing illnesses in the postharvest period [2].

Antioxidant system of plant tissues consists of non-enzyme (low-molecular) and enzyme (high-molecular) antioxidants [3]. Three enzymes are mainly responsible for the defense of the organism from oxidative damage: superoxide dismutase (SOD), catalase (CAT), peroxidase (PO) [4].

Superoxide dismutase plays a central part in the protection from oxidative stress in all aerobic organisms [5]. Dismutation of superoxide radicals is SOD function. Hydrogen peroxide is a result of superoxide anion dismutation. That is why the group of enzymes, which utilize hydrogen peroxide, are a necessary element of antioxidant defense of plants. Catalase and peroxidase are such enzymes and they act in a cell as a second defense line.

Due to the interruption of synthesis processes of substances, which are needed for the normal metabolism, system of the antioxidant control over ROS generation is acting properly limited time only. Level of ROS increases dramatically when the irreversible aging processes develop [6, 7]. Mechanisms of antioxidant protection exhaust, which leads to the number of metabolic disorders and cell death. Therefore utilization of ROS excess during the storage of fruit and vegetables is a key to preserve the quality of production.

Together with decreasing of storage temperature, the intensity of respiration decreases, production of ethylene and weight losses are reduced [8, 9]. This allows extension of shelf life of fruit and vegetables. Still for many species of horticultural crops low temperature is harmful as leading to the oxidative damage. Despite powerful endogenous antioxidant system [10], sweet bell pepper fruits are quite sensitive to the influence of cold. Level of sensitivity depends on variety: hot species of *Capsicum annuum* can withstand temperature decrease till 5 °C, although "paprika" fruits immediately react on the temperature decreasing below 7 °C with physiological disorders [11,12].

For protection of fruits from oxidative damage that was induced by cooling in industry heat treatment is often used [13]. Positive influence of heating procedures is connected with formation and protective action of heat shock proteins, which play a part in the regulation of ROS formation and protection of cell compartments from oxidative stress. Temperature preconditioning also decrease sensitivity of sweet bell pepper fruits to cold [14, 15]. Group of scientists from Israel investigated the influence of temperature preconditioning on pepper storing in the temperature diapason from 22 to 55 °C [14]. They have found, that temperature conditioning above 44,5 °C significantly reduce damage of fruits from cooling.

Another effective method to reduce oxidative damage is using of direct approach – antioxidants [16]. Combination of direct and indirect approaches can lead to the synergic effect of system stress resistance. However, analysis of antioxidant defense functioning in sweet bell pepper fruits during the heat treatment with antioxidants was newer studied, that is why area of research is of a great interest.

Materials and methods

Fruits of pepper hybrids Hercules F1 and Nikita F1, grown in open field conditions in agribusiness of Melitopol district of Zaporizhzhska region, were investigated. For storage fruits of technical stage of ripening were taken (fruits coloured in the main colour to 80-90%), uniform in size also. Fruits were placed in the prepared solutions of biologically active substances with a temperature of 45 °C for 15min. Complex composition with bactericide and antioxidant activity was used. It is based on the approved for usage in food antioxidants: butylhydroxitoluol (ionol), lecithine and water extract of horseradish root [Priss, O. P., Prokudina, T. F., Zhukova, V. F. Antioxidant composition for the treatment of fruit vegetables before storage [Antyoksydantna kompozytsiya dlya obrobky plodovykh ovochiv pered zberihannyam]. Pat. 59733 Ukraine, IPC A 23 7/14, 2011].

After drying fruits were put in boxes, lined with polyethylene wrap and stored at $7 \pm 0,5$ °C and relative humidity $95 \pm 1\%$. Control group consists of not treated fruits. Development of chilling injury (CI) was evaluated after storage under mentioned conditions and transferring for 1-day storage of pepper under the room temperature (21 ± 2 °C). That was done five times (20 pepper fruits in each group). Level of chilling injury during the pepper storage was evaluated with a subjective scale from 0 to 3 points and expressed through the chilling injury index [14].

The content of malondialdehyde (MDA) was determined by the thiobarbituric method [Musienko, M. M. et al. (2001) *Spectrophotometric methods in the practice of physiology, biochemistry and ecology of plants* [Spektrofotometrychni metody v praktytsi fiziolohiyi, biokhimiyyi ta ekolohiyi roslyn]]. SOD activity was determined by estimation of its ability to inhibit the reaction of auto-oxidation of adrenaline in alkaline medium [Sirota T.V. (2000), *A method for determining the antioxidant activity of superoxide dismutase and chemical compounds* [Sposob opredeleniya antioksidantnoy aktivnosti superoksiddismutazy i himicheskikh soedinenij], Russian Federation Patent 2144674] (method was modified in the stage of preparation of raw materials for research). For the measurement of SOD activity to 0,5 g of plant material 5 ml of phosphate buffer pH=7,8 was added and substance was triturated in a mortar with glass (on ice). Next, homogenate was transferred to the centrifuge tubes with 0,3 ml of chloroform and 0,6 ml of alcohol and centrifuged at 8000 rpm. 20 minutes. For spectrophotometric measurements supernatant was used. SOD activity was expressed in conventional units (CU), which show the percentage of inhibition of adrenaline auto-oxidation. Catalase activity was determined by titration of the undecomposed rest of hydrogen peroxide with sodium thiosulfate [Hrytsayenko, Z.M. et al. (2003) *Methods of biological and agrochemical research plants and soils* [Metody biolohichnykh ta ahrokhimichnykh doslidzhen roslyn i gruntiv]]. Determination of peroxidase activity was conducted by titration of undecomposed rest of hydrogen peroxide in the reaction of pyrocatechol oxidation [Zemljanuhin, A. A. (1985) *Small workshop on Biochemistry* [Malyj praktikum po biohimi]].

Results and discussion

First signs of chilling injury in the control group were noticed on the 15th day of storage in pepper fruits from both hybrids. Without additional treatment aimed on prevention from chilling injury third part of fruits become damaged after 24th day of storage. Figure 1 shows that combination of heat and antioxidant treatment significantly induces tolerance to cold.

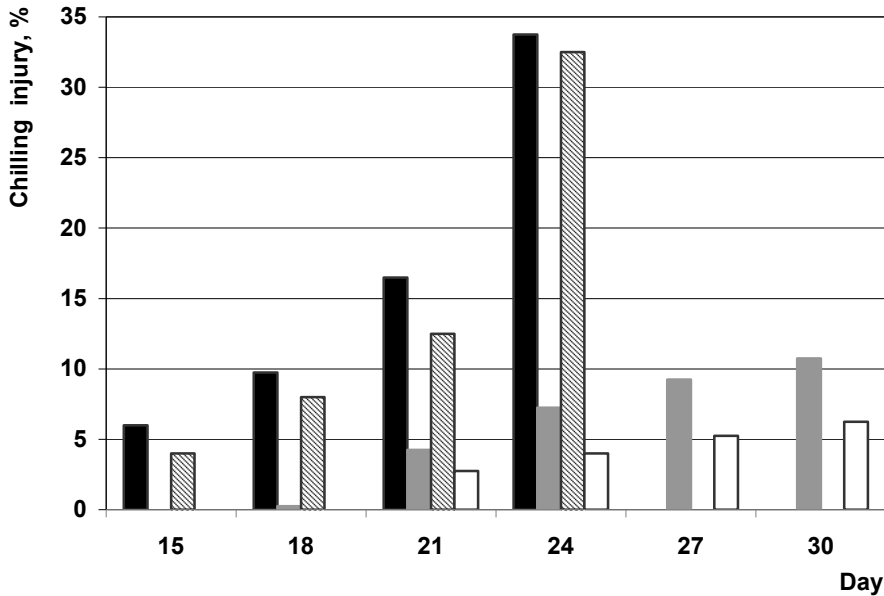


Fig. 1. Chilling injury in sweet bell pepper:

- - Nikita hybrid, without treatment
- - Temperature preconditioning with antioxidants of Nikita hybrid fruits
- ▨ - Temperature preconditioning with antioxidants of Hercules hybrid fruits
- - Hercules hybrid, without treatment

In the fruits of sweet bell pepper that was treated with the composition of antioxidants cold injury is seen only after 21st day independently from the studied hybrid. Cold-induced damage decreases in 3,9...4,5 times in comparison to the fruits without any treatment. After 30 days of storage chilling injury of fruits, which were preconditioned with temperature and antioxidants, was on the level of 10%.

The degree of severity of chilling injury increases with the increasing of storage life. However, heat treatment with antioxidants leads to reduction of chilling injury index (Fig. 2).

The severity of chilling injury symptoms in studied fruits decreases in 8,8...13,2 times comparing to the fruits without treatment.

Increasing of malondialdehyde content is a direct indication of cell oxidative damage, and such a rising is natural during aging of fruit tissues. During the storage of pepper in the temperature conditions above the cold-sensitivity threshold content of MDA increases constantly [17, 18]. Same dynamics can be seen in our study (Fig.3). As You can see in Fig. 3, there is a significant difference in a background MDA content depending on the variety of pepper. Speed of lipid peroxidation processes in Nikita pepper is higher comparative to such in Hercules. That is why after storage content of MDA is same in both hybrids.

First six days of shelf life content of MDA in the control group of fruits has only a tendency to rise (some years studies show statistically insignificant difference). On twelfth day of storage content of MDA grows in 1,6 times in Hercules hybrid fruits and in 2,2 times in Nikita hybrid. This is an evidence of intensification of free radical processes in this stage of storage.

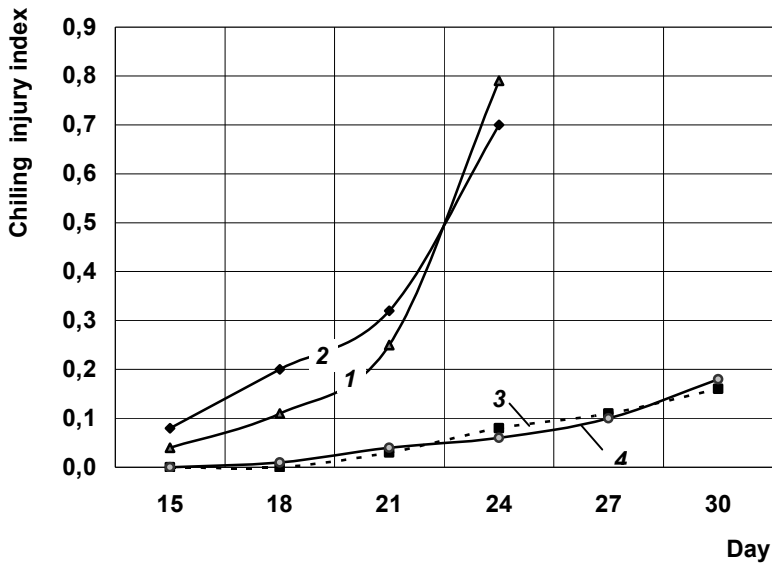


Fig. 2. Index of chilling injury:
1 - fruits of Nikita hybrid without treatment;
2 - fruits of Hercules hybrid without treatment;
3 - temperature preconditioning with antioxidants of Nikita hybrid fruits;
4 - temperature preconditioning with antioxidants of Hercules hybrid fruits.

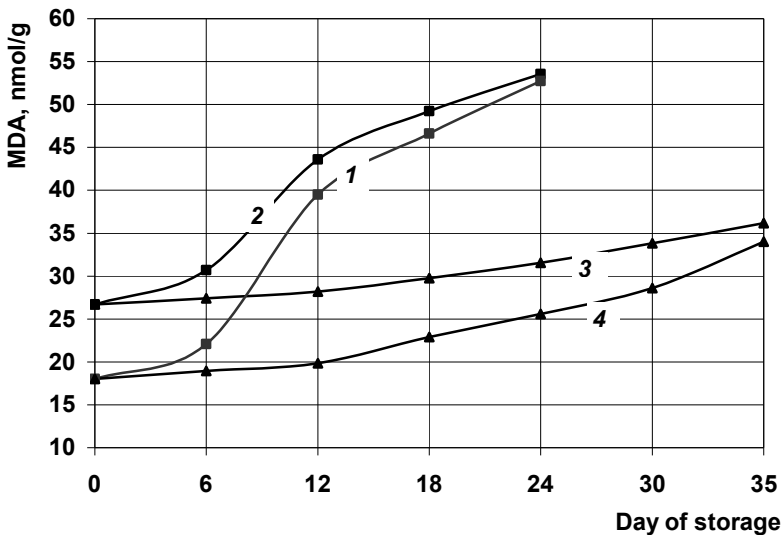


Fig. 3. MDA content in bell peppers during storage:
1 - fruits of Nikita hybrid without treatment;
2 - fruits of Hercules hybrid without treatment;
3 - temperature preconditioning with antioxidants of Nikita hybrid fruits;
4 - temperature preconditioning with antioxidants of Hercules hybrid fruits.

To the end of storage content of lipid peroxidation products in the control samples raises twice in Hercules hybrid and in 2,9 times in Nikita fruits. Differences in the speed of lipid peroxidation collection in different hybrids can be explained with lower antioxidant status of Nikita pepper.

As it is shown in Fig. 3, heat preconditioning combined with antioxidants changes the dynamic of MDA in sweet bell pepper fruit. Till 12th day of storage level of lipid peroxidation products stays almost stable in both hybrids. Then, on each stage of storage, level of MDA raises by 5...6% in Hercules hybrid and by 12...15% in Nikita hybrid. On the 18th day (loss of commercial quality of control samples) level of MDA in the studied samples is lower in 1,7 times for Hercules hybrid and twice for Nikita hybrid in average. That confirms an explanation that exogenous antioxidants inhibit processes of lipid peroxidation.

Development of lipid peroxidation and aging of pepper during storage is considered to be connected with decreasing of antioxidant enzymes (SOD, CAT and PO) activity [17]. Activity of SOD rises till pepper reaches full-red colour and decreases during over maturation [19]. Dynamic of SOD activity during pepper storage also differs in dependence on cultivar sensitivity to cold. Tolerant to cooling cultivars demonstrate increasing of SOD activity during decreasing of such in sensitive [20]. As far as studied hybrids have similar sensitivity to cooling, it is natural that dynamic of SOD activity during storage in both hybrids is same (Fig. 4).

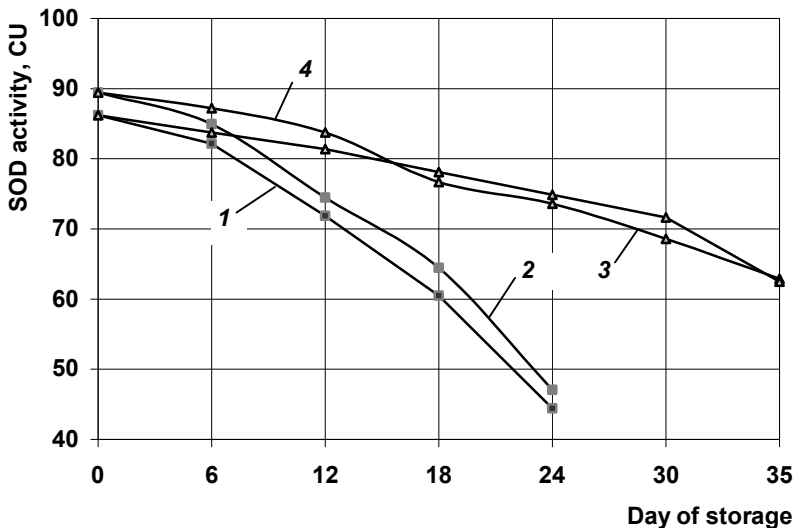


Fig. 4. SOD activity in bell peppers during storage:

- 1 - fruits of Nikita hybrid without treatment; 2 - fruits of Hercules hybrid without treatment; 3 - temperature preconditioning with antioxidants of Nikita hybrid fruits; 4 - temperature preconditioning with antioxidants of Hercules hybrid fruits.**

Activity of enzyme constantly decreases in control and studied samples of both hybrids. Heat preconditioning with antioxidants significantly decreases speed of SOD deactivation in comparison to the control group. Control samples in 18 days of storage loose 40% of enzyme activity in average. Slowdown of SOD activity in preconditioned fruits is seen only after 35 days of shelf life.

In studied groups probable decreasing of activity of SOD happens only after 12th day of storage. Partly such stabilization of SOD activity can be explained by heat preconditioning, which induces SOD activity directly after conditioning [21]. On 18th day of storage activity of SOD in fruits that were preconditioned with heat and antioxidants is higher by 25% in average in comparison with a control group. In the end of storage of studied fruits (30 days) activity of this enzyme stays on the same level that is in a control samples on 18th day.

These results testify to induction of SOD activity with heat preconditioning with antioxidants which enlarges its ability to dismutate highly toxic superoxide anions and prevents oxidative damage of cells during longer shelf time.

Activity of superoxide dismutase has high reverse correlation to the content of malondialdehyde in control and studied groups that testifies to ability of antioxidant defense system to self regulation and confirms high antioxidant status of fruits (table 1).

Activity of catalase during ripening and storage of pepper decreases independently from stage of maturation [22]. Same results were received in this study (Fig. 5).

Table 1
Correlation between SOD activity and content of MDA in sweet bell pepper fruits during shelf life

Year	Hercules		Nikita	
	Without treatment	Temperature preconditioning with antioxidants	Without treatment	Temperature preconditioning with antioxidants
2009	-0,90	-0,97	-0,95	-0,96
2010	≈-1,00	-0,97	-0,90	-0,96
2011	-0,93	≈-1,00	-0,99	-0,97

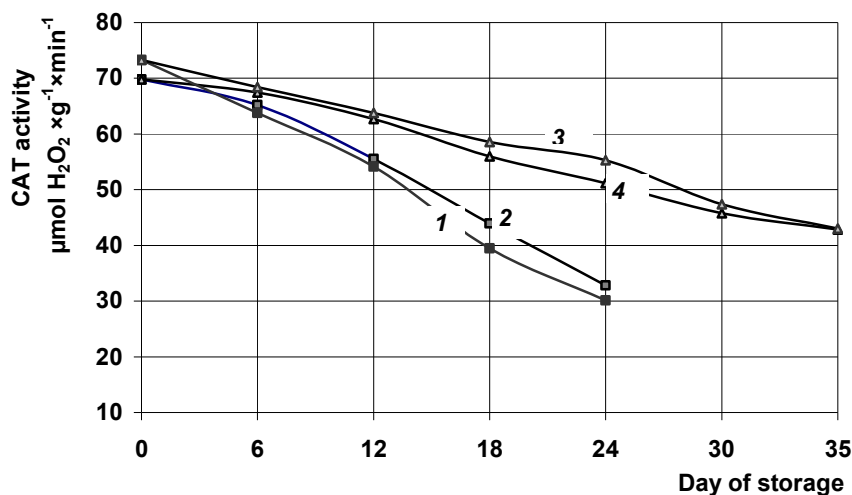


Fig. 5. CAT activity in bell peppers during storage:
 1 - fruits of Nikita hybrid without treatment;
 2 - fruits of Hercules hybrid without treatment;
 3 - temperature preconditioning with antioxidants of Nikita hybrid fruits;
 4 - temperature preconditioning with antioxidants of Hercules hybrid fruits.

Although values of CAT activity in studied hybrids differ statistically, these differences smoothen during storage. In the end of study catalase activity in control groups was lower than beginning values in 2,1 times for Hercules hybrid fruits and in 2,4 times for Nikita hybrid.

Heat preconditioning with antioxidants does not change a character of dynamic of catalase activity in pepper fruits. On the other hand, slow down of enzyme deactivation speed is noticeable. On the 18th day of shelf life catalase activity in treated peppers of Nikita hybrid is higher than in control in 1,5 times. For Hercules hybrid this difference is 1,3 times. Catalase activity in studied variants to the end of storage is higher than in control group on 18th day by 5...20%, where value depends on the hybrid of pepper. Persistence of catalase activity on the high level in studied fruits preserves pepper tissues from peroxidative damage during longer period of storage.

During all years of study control and studied samples had strong direct correlation between CAT and SOD activities and reverse correlations with MDA (table 2) that testifies to normal functioning of antioxidant defense system.

Table 2

Correlation between activity of CAT, SOD and content of MDA in sweet bell pepper fruits during shelf life

Year	Hercules				Nikita			
	Without treatment		Temperature preconditioning with antioxidants		Without treatment		Temperature preconditioning with antioxidants	
	SOD	MDA	SOD	MDA	SOD	MDA	SOD	MDA
2009	0,97	-0,96	0,99	-0,97	≈1,00	-0,97	0,99	-0,96
2010	0,99	-0,97	0,99	-0,97	0,98	-0,96	0,94	-0,99
2011	0,98	-0,96	0,97	-0,97	0,98	-0,98	0,97	-0,97

Peroxidase activity during storage of pepper can increase during shelf life of fruits in green stage of maturity and decrease during the storage of fruits, which have reached main colour more than by 80% [23, 24]. Activity of this enzyme has a big part in defense from low-temperature stress, as far as gvyakol peroxidase activates with damage from cold temperature [24]. Character of graphic of peroxidase activity change during storage of different hybrids is similar (Fig. 6).

Peroxidase activity is slowly decreasing till 12th day, after that speed of enzyme inactivation grows twice and starting from 18th day activity of peroxidase increases. Similar character of pepper peroxidase dynamic in temperature around 8 °C is described also in other works [17]. It is obvious that induction of peroxidase in the moment, when fruit lose their commodity quality and exhaust abilities of antioxidant defense mechanisms, are connected with growth of amount of unutilized H₂O₂. Another reason of peroxidase activity growth in pepper is a development of physiological disorders and microbiological illnesses of fruit [25].

Heat treatment with antioxidants changes a bit the dynamic of peroxidase activity in fruit of sweet bell pepper. Stable decreasing of peroxidase activity can be noticed till the 30th day. On the other hand, speed of activity deactivation is lower. Activity of enzyme in studied fruits of both hybrids on the 18th day is twice higher in comparison to the control. That allows to prolong maintenance of hydrogen peroxide concentration on the stationary

level. Increasing of PO activity after 30th day of storage coincides with the loss of commercial quality of pepper.

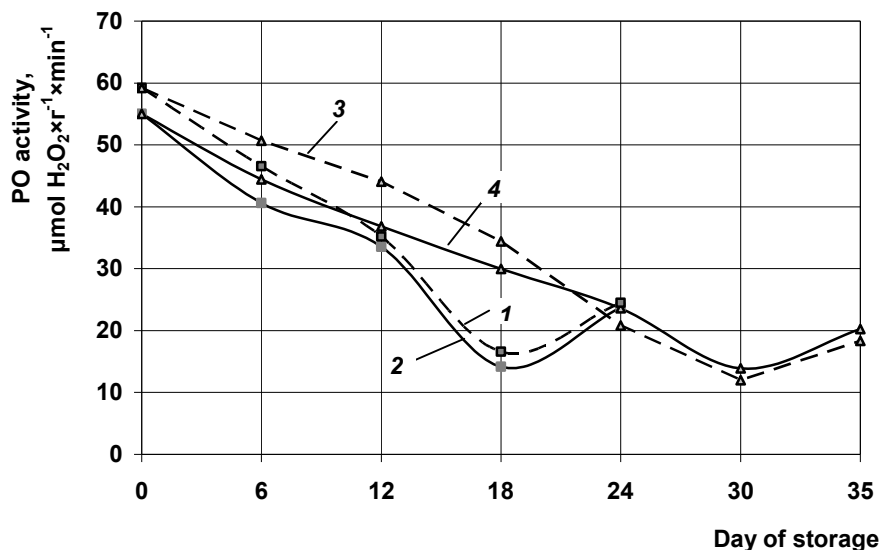


Fig. 6. PO activity in bell peppers during storage:

- 1 - fruits of Nikita hybrid without treatment; 2 - fruits of Hercules hybrid without treatment; 3 - temperature preconditioning with antioxidants of Nikita hybrid fruits; 4 - temperature preconditioning with antioxidants of Hercules hybrid fruits.

In pepper fruits peroxidase activity is directly correlated to the SOD and CAT activity and reverse correlated to the content of MDA (table 3).

Such correlation dependences testify to the direct influence of peroxidase on the degree of antioxidant defense of pepper tissues. In the conditioned samples strength of correlation between enzymes is higher than in control group, which shows increasing of antioxidant defense system balance for successful neutralization of ROS.

Table 3

Correlation between peroxidase, catalase, superoxide dismutase activity and content of MDA in sweet bell pepper fruits during shelf life

Year	Hercules						Nikita					
	Without treatment			Temperature preconditioning with antioxidants			Without treatment			Temperature preconditioning with antioxidants		
	SOD	CAT	MDA	SOD	CAT	MDA	SOD	CAT	MDA	SOD	CAT	MDA
2009	0,73	0,87	-0,89	0,90	0,92	-0,83	0,83	0,86	-0,92	0,88	0,93	-0,81
2010	0,87	0,82	-0,90	0,93	0,96	-0,90	0,84	0,93	-0,94	0,86	0,93	-0,92
2011	0,80	0,87	-0,92	0,94	0,93	-0,92	0,87	0,94	-0,92	0,91	0,97	-0,91

Conclusions

Using of heat preconditioning with antioxidants before storage of sweet bell pepper fruits leads to decreasing of oxidative damage induced by cooling. Heat preconditioning with antioxidants has a noticeable influence on the content of lipid peroxidation products. In studied groups of pepper level of malondialdehyde stays stable in both hybrids till 12th day of storage. If storage was continued, growth of lipid peroxidation products in conditioned samples has been maintained minimal. Level of MDA in studied samples is lower in 1,7 times in average for Hercules hybrid and twice for Nikita hybrid. This is a confirmation of lipid peroxidation inhibition by exogenous antioxidants.

Heat preconditioning with antioxidants decreases speed of SOD deactivation by 25% and CAT by 30...50%. Heat treatment with antioxidants decreases also speed of peroxidase deactivation in sweet bell pepper fruits and postpone a moment of increasing of its activity. Activity of this enzyme in studied fruits of both hybrids on 18th day is twice higher compared to the control. This lets longer maintenance of hydrogen peroxide concentration on the stationary level. Strong reverse correlations between the content of malondialdehyde and activities of superoxide dismutase, catalase and peroxidase were found and testify to antioxidant functions of these enzymes in control and studied groups of fruits.

Combination of heat and antioxidant treatment for preparation to the storage of sweet bell pepper fruits lets prolong maintenance of functioning of the system responsible for reactive oxygen species utilization.

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Laser light scattering by milk particles

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Abstract

Keywords:

Milk
Fat
Casein
Serum
Protein
Laser
Scattering

Article history:

Received 11.01.2016
Received in revised
form 29.01.2016
Accepted 24.03.2016

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Introduction. The main objective of this article is the theoretical and experimental study of laser light scattering by milk particles and the analysis of the character of size distribution of milk particles depending on the concentration and the heating of the samples during the technological processes.

Materials and methods. Theory of light scattering and the dependence of the coefficient of scattering on the wavelength of light and parameters of milk particles are discussed. The experimental investigation of light scattering by milk particles was done with the photon correlation spectrometer *System 4700c*.

Results and discussion. Different types of scattering such as Rayleigh Scattering, Rayleigh-Gans Scattering, Anomalous Diffraction, and corresponding coefficients of scattering are discussed. Histograms of size distribution of the diluted ($C \leq 10^{-3}$ %) milk (temperature 25°C) and samples of milk with different fat concentration (4.2%; 5.2%; 7.4% at temperature 20°C and 7.4% at temperature 50°C), just as the dependence of the intensity of scattered light on the angle of observation were studied.

The method of light scattering can be used for analysis of the effect of milk content and technological processes which are related to heating (pasteurization, sterilization, homogenization) on the size distribution of milk particles.

Conclusions. Laser light scattering is an advanced technique that can be used to determine the size distribution profile of milk particles. It is characterised with high accuracy, non-destructive action and does not require calibration standards.

Introduction

Analytical methodologies. Milk is a complex mixture of fat globules (0.1-10 μm), micelles of casein (0.1-0.2 μm), and particles of serum proteins (0.01-0.02 μm), and the turbidity of milk depends on the dimensions and concentration of these components. Such properties of milk as its texture, colloidal stability, flavor, and sensory feel (mouth feel) depend on the particle size of the milk particles.

There are various analytical methodologies that give an information about distribution of particle sizes in milk: nanoparticle tracking analysis, scanning electron microscopy, size exclusion chromatography, cell electrophoresis, analytical ultracentrifugation, near infrared spectroscopy etc. [Alexander and Dalgleish, 2006 ; Anema et al., 2005; Mootse, 2014; Thu Tran Le et al., 2008; Dejan, 2010; Vasco et al., 2010 ; Posudin et al., 2015 ; Posudin and Kostenko, 2015].

Laser light scattering is an advanced technique that can be used to determine the size distribution profile of milk particles in solution. It is characterised with high accuracy, non-destructive action and does not require calibration standards.

The main objective of this article is the study of laser light scattering by milk particles and the analysis of the character of size distribution of milk particles depending on the concentration and the heating of the samples during the technological processes.

Theory of Light Scattering. Theoretically the process of light scattering is characterized by the so-called *coefficient of scattering* Q [Hulst, 1982; Walstra, 1964]. This coefficient depends on the wavelength of light λ , refractive indices of particles n_1 and medium n_2 , and the size d of particles. The effect of these factors is determined with such parameters as relative refraction index $m = n_1/n_2$, and phase shift $\rho = 2x(m - 1)$, where $x = nd / \lambda$. There are different types of scattering depending on the values of all these parameters. The complete theory of light scattering is given in [Hulst, 1982]; several examples of the relation of coefficient of scattering with above-mentioned parameters are listed below.

1. *Rayleigh Scattering.* This type of scattering is demonstrated by the spherical particles which have relative refractive index equal to about 1 and dimensions less than the wavelength:

$$m \rightarrow 1 \quad (1)$$

$$Q_R = \frac{32}{27} x^4 (m - 1) \quad (2)$$

The following expression was proposed for very small ($mx \ll 1$) particles [Walstra, 1965]:

$$Q_R = \frac{8}{3} x^4 \left(\frac{m^2 - 1}{m^2 + 2} \right)^2 \quad (3)$$

2. *Rayleigh-Gans Scattering.* There are two possible situations for the particles which have relative refractive index equal to about 1 and very small phase shift $2x(m - 1) \ll 1$:

a. Rayleigh Scattering

$$x \ll 1 \quad Q_R = \frac{32}{27} x^4 (m - 1) \quad (4)$$

b. Rayleigh-Gans Scattering

$$x \gg 1; \quad Q_{RG} = 2(m - 1)^2 \quad (5)$$

3. *Anomalous Diffraction.* This type of scattering is demonstrated by the spherical particles which have relative refractive index equal to about 1 and dimensions which exceed the wavelength:

$$m \rightarrow 1; x \gg 1; \rho \gg \lambda \quad (6)$$

$$Q_N = 2 - \frac{16m^2 \sin \rho}{(m+1)^2 \rho} + 4 \frac{1 - m \cos \rho}{\rho^2} + 7.54 \frac{z - m}{z + m} x^{-0.772} \quad (7)$$

where $z = [(m^2 - 1)(6x/\pi)^{2/3} + 1]^{1/2}$.

If the dimensions of particles are small or moderate, the coefficient of scattering can be written as:

$$Q_S = (1.26m - 0.44)/\rho - 2.558(m - 1)^{1.273} - 0.843 \quad (8)$$

Application of Theory of Light Scattering to Milk Particles

The method of light scattering seems to be one of the perspective approaches. The dependence of coefficient of scattering on the spectral region and parameters such as x , ρ , and d is given in Table 1.

Table 1
Dependence of coefficient of scattering on the spectral region and parameters x , ρ , and d

Milk Particles	Ultraviolet Region ($\lambda = 0.2 \mu\text{m}$)			Ultraviolet Region ($\lambda = 0.6 \mu\text{m}$)			Infrared Region ($\lambda = 1.2 \mu\text{m}$)		
Fat globules $d \gg \lambda$ $d_{min} = 1.2 \mu\text{m}$	15.71	3.14	Q_N	5.0	1.0	Q_N	2.62	0.52 $1.6 < \rho < 2.5$	Q_S
$d_{max} = 10 \mu\text{m}$	157.1	31.4	Q_N	49.9	10.0	Q_N	26.12	5.2 $\rho \gg 2.5$	Q_N
Casein micelles $d < \lambda$ $d \sim 0.15 \mu\text{m}$	2.36	0.47	Q_{RG}	0.75	0.15	Q_R	0.39	0.08	Q_R
Casein micelles $d < \lambda$ $d \sim 0.015 \mu\text{m}$	0.24	0.05	Q_R	0.075	0.015	Q_R	0.04	0.01	Q_R

Dependence of the coefficient of scattering on the wavelength of light is presented in Figs. 1 and 2 for milk globules, and in Figs. 3 and 4 for casein micelles and serum protein particles. Thus, the analysis of the dependence of light scattering on the wavelength and angle of observation can make it possible to estimate quantitatively the dimension of milk particles.

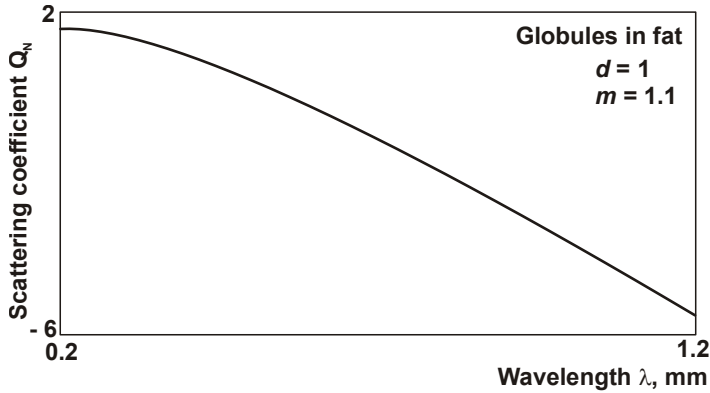


Figure 1. Dependence of the coefficient of scattering of the wavelength of light for milk globules (diameter 1 μm)

It is clear that the transfer from visible to infrared part of the spectrum induces the decreasing of coefficient Q_N 1.35 times for fat globules of diameter 10 μm and 4.41 times for fat globules of diameter 1 μm . That is why it is expedient to pass to the infrared region in order to determine the fat content in milk and to avoid the effect of small particles.

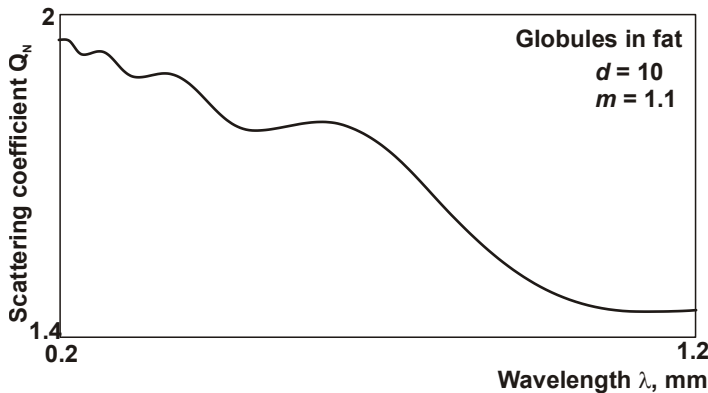


Figure 2. Dependence of the coefficient of scattering of the wavelength of light for milk globules (diameter 10 μm)

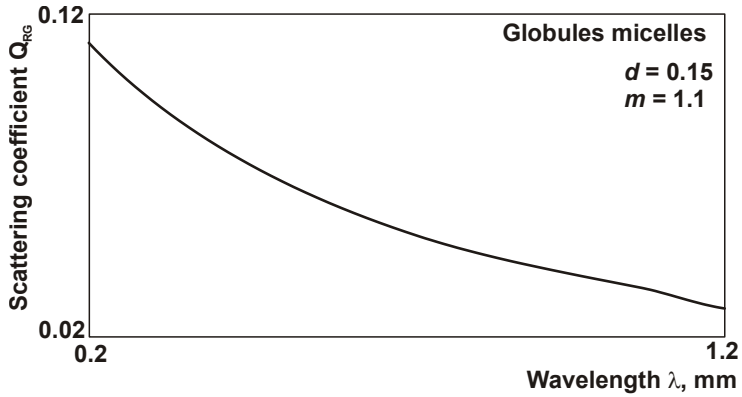


Figure 3. Dependence of the coefficient of scattering of the wavelength of light for casein micelles

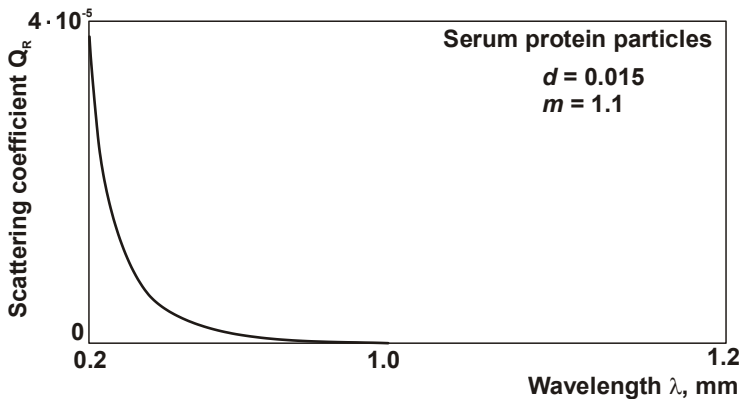


Figure 4. Dependence of the coefficient of scattering on the wavelength of light for serum protein particles

Materials and methods

The experimental investigation of light scattering by milk particles was done with the photon correlation spectrometer *System 4700c*. The schematic representation of this spectrometer is presented in Fig. 5. The focused radiation of laser 1 is directed to the sample 2 of milk that is placed on the goniometer table 3. The scattered radiation is directed to the readout system which consists of photomultiplier 4, correlator 5, computer 6, and printer 7. A sample holder is supplied with a system of temperature control 8, peristaltic pump 9, and electric motor 10. A signal from the output of photomultiplier gives the information about the dependence of light scattering on the angle of observation. Milk, aqueous solution of lactose, dry milk, and milk mixtures were used as the samples [Posudin, 1988, 1993, 2007; Posudin and Kostenko, 1994].

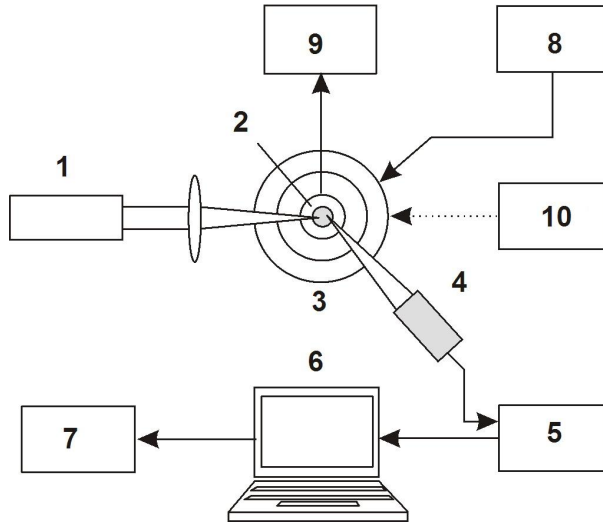


Figure 5. Schematic representation of photon correlation spectrometer System 4700c:
1 - laser; 2 - sample; 3 - goniometer table; 4 - photomultiplier; 5 - correlator; 6 - computer; 7 - printer;
8 - system of temperature control; 9 - peristaltic pump; 10 - electric motor

Results of light scattering by milk particles

The typical histograms of size distribution of these samples are given in Figs. 6-10.

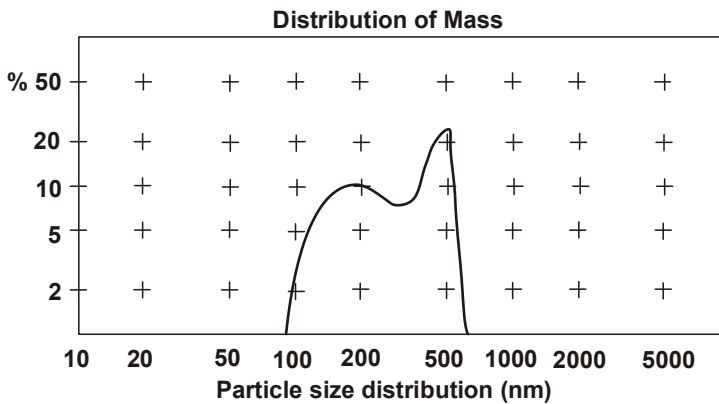


Figure 6. Typical histogram of size distribution of the diluted ($C \leq 10^{-3}$ %) milk. Temperature 25°C

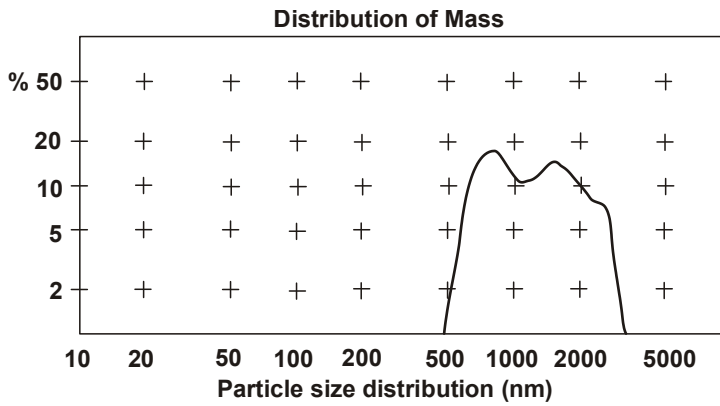


Figure 7. Histogram of size distribution of milk. Concentration of fat 4.2%; temperature 20°C

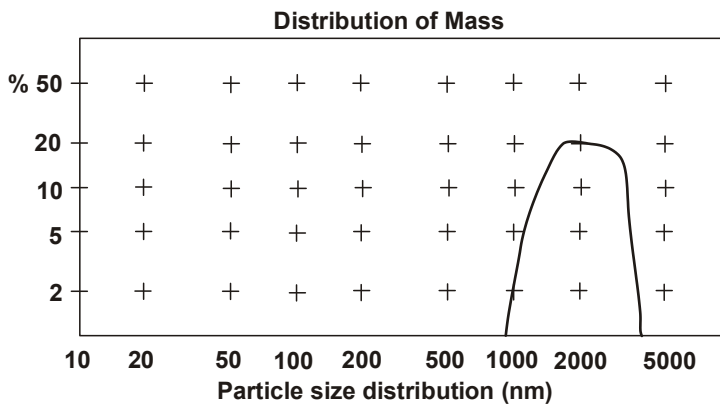


Figure 8. Histogram of size distribution of milk. Concentration of fat 5.2%; temperature 20°C

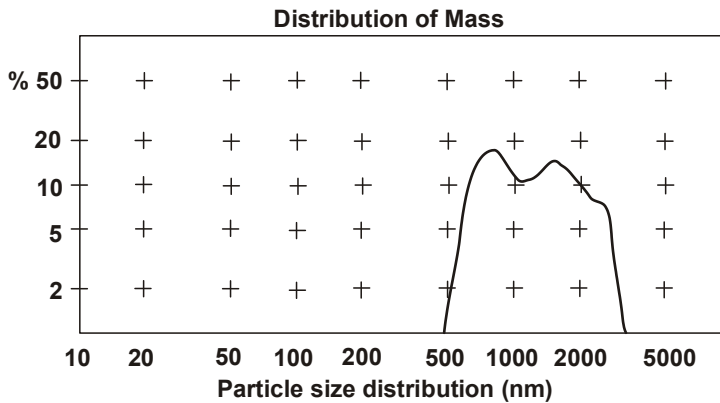


Figure 9. Histogram of size distribution of milk. Concentration of fat 7.4%; temperature 20°C

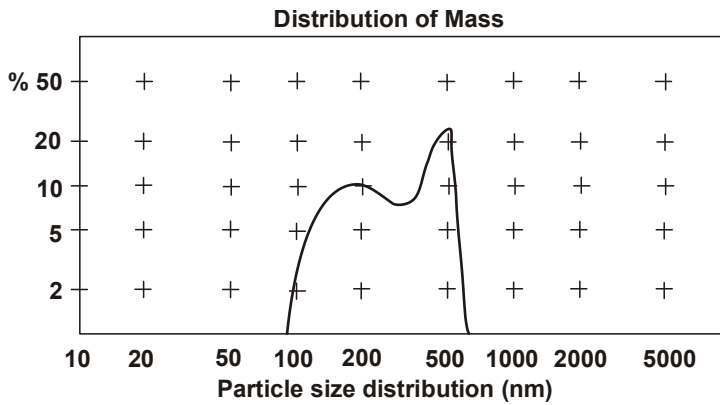


Figure 10. Histogram of size distribution of milk. Concentration of fat 7.4%; temperature 50°C

It is clear that the maximum of size distribution of milk particles is shifted with increasing concentration of particles from 100- 500 nm ($C \leq 10^{-3} \%$) to 500-2,000 nm ($C = 7.4 \%$). The character of size distribution of milk particles depends strongly on the concentration (Figs. 6-9) and the heating of the sample (Figs. 9-10).

Fig. 11 demonstrates the dependence of the intensity of scattered light on the angle of observation.

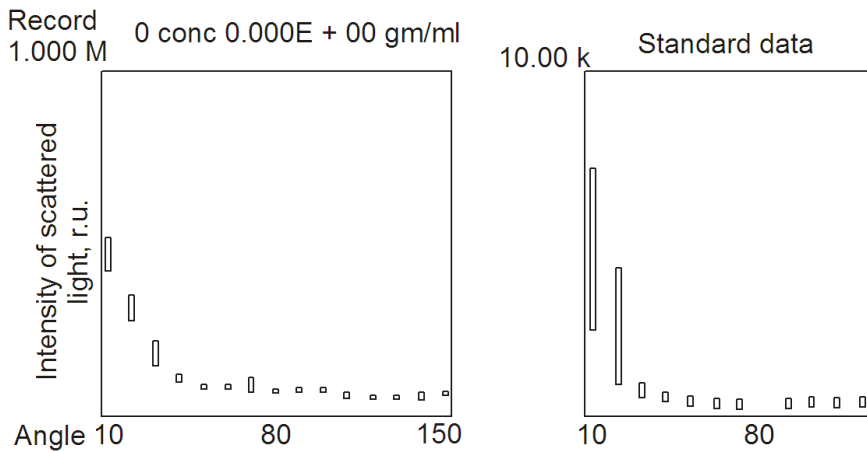


Figure 11. Dependence of the intensity of scattered light on the angle of observation

Conclusion

In such a way, the method of light scattering can be used for analysis of the effect of milk content and technological processes which are related to heating (pasteurization, sterilization, homogenization) on the size distribution of milk particles.

Acknowledgments. *The author is grateful to his colleagues A.D. Alyokhin, G.I. Gaydidey, M.G. Skrishevskaya, E.M. Galushko, N.G. Golubeva, M.M. Krolivets and V.A. Myasnikov.*

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Production and quality evaluation of tapioca substituted with fermented bambara flour

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Abstract

Keywords:

Tapioca
Bambara
Pasting
Fermentation
Cassava

Article history:

Received 07.12.2015

Received in revised
form 16.01.2016

Accepted 24.03.2016

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Introduction. Tapioca grit is a partly gelatinized dried starch obtained from cassava. Fortifying tapioca with fermented bambara flour may be important to improve its nutritional value.

Materials and methods. The pasting, functional, nutritional and sensory properties of tapioca meal fortified with fermented bambara flour at varying levels of 10, 20, 30, 40 and 50% were investigated using standard methods.

Results and discussion. Carbohydrate was the major nutrient in the control tapioca (84%) and the fortified tapioca samples (64-80%). However, with increasing levels of fermented bambara flour, the protein contents of the tapioca meal progressively increased. The higher protein content of the fortified samples is expected since bambara is a good source of protein. Fortified tapioca meals showed a slight decrease in water absorption capacity but significant increase in swelling power. Increased swelling of the tapioca meal could be due to the effect of heat which may have enhanced starch disruption. Further, the increase in protein content of the fortified samples may also have contributed to their higher swelling power. Peak viscosity of tapioca samples decreased with increasing levels of fermented bambara flour up to 30%, but thereafter increased. This behaviour was attributed to increase in starch content. Cyanide contents of the control tapioca and fortified samples (5.82-6.34 mg/kg) are within the acceptable limits in foods. Tapioca cake fortified with 10% fermented bambara flour compared favourably with the control sample in colour, taste, aroma, consistency and overall acceptability.

Conclusions. Acceptable tapioca meal with improved swelling power and nutritional value can be produced from cassava starch fortified with 10% fermented bambara flour.

Introduction

Cassava (*Manihot esculenta crantz*) is a major food crop in many parts of the world including Nigeria [1]. It is highly perishable, and must be processed immediately after harvest into highly stable products such as tapioca. Tapioca cake is a partly gelatinized dried starch obtained from cassava tubers which appears as flakes of irregular shaped granules [2]. It is consumed in various forms such as soaking and subsequently cooking in water to form meals. Sugar and milk may be added depending on individual preferences. In Africa, tapioca meal is consumed as a convenience food [2, 3]. Cooked tapioca is similar to oatmeal in appearance and taste [4]. However, tapioca meal is nutritionally inferior compared to oatmeal. Further, unlike oatmeal that is widely consumed all over the world, the consumption of tapioca meal seems to be limited to production areas of cassava. Nutritionally, tapioca contains substantially high amount of carbohydrate (78-96%) [4-6] in the form of starch. The protein content of tapioca is very low and may vary between 0.31 and 1.20% depending on the cassava variety [4-6]. Attempts by previous studies to improve the nutritional value of tapioca meal using defatted soybean flour [5] full fat soybean flour [4, 6], spices [7] coconut and banana pulp [8] have proved successful. The protein content of tapioca fortified with 50% full soybean flour was reportedly higher (approx. 37 times) compared to the unfortified control tapioca [6]. In a similar study, the addition of defatted soybean flour at 20% was found to substantially increase the protein content of tapioca meal. [5].

The addition of legume flour to tapioca meal may influence its physicochemical properties including pasting, functional and sensory properties. For instance, with increasing amount of full fat soybean flour, the swelling power of tapioca was reported to decrease significantly, while the water absorption increased [9]. The peak, breakdown, set back and final viscosities of the fortified tapioca meal also decreased with increasing amount of full fat soybean flour [9]. Further, the inclusion of full fat or defatted soybean flour to tapioca meal has been found to increase its overall acceptability [5, 6].

The use of many underutilized legumes including bambara in enriching staple foods is currently being encouraged. Many of these crops have the potential to reduce protein malnutrition especially among communities where their staple is majorly carbohydrate foods. Bambara groundnut (*Vigna subterranea*) is a protein-rich legume (19-25%) [10-13] similar to those of cowpea [14]. It is highly drought tolerant and well adapted to the changing climate. However, bambara remains a neglected and underutilised legume grown mainly for subsistence [11]. In Nigeria, bambara is traditionally used in making puddings and has been fermented for improved nutritional value [15-17]. The use of fermented bambara to fortify tapioca meal may be one of several ways to unlock the potential of this crop and enhance value addition. Therefore, the objective of this work was to enhance the nutritional quality especially protein content of tapioca meal using fermented bambara nut flour.

Materials and methods

Cassava tubers (sweet variety) were obtained from the research farm of Ladoke Akintola University of Technology, Ogbomoso. Bambara grains were purchased from a local market in Ogbomoso, while the innoculum (*R. Oligosporus*) was from the Indonesian Embassy, Lagos Nigeria. All other chemical used in this study were of food grade.

Fermentation of bambara groundnut. Bambara was fermented as described in the literature [18]. The fermented bambara grains were blanched for 20 min, drained and dried at 55°C for 24 h in hot air oven. The dried samples was milled, sieved and used immediately for fortification.

Preparation of the Tapioca cake. Tapioca cake was prepared as previously described [9]. Fermented bambara flour was added at 10%, 20%, 30%, 40% and 50% into the moist cassava starch prior to roasting. Tapioca without bambara flour served as the control.

Pasting and functional properties. The pasting properties of the tapioca mixes were determined using a RVA (3D New port Scientific, Australia). The functional properties including water absorption capacity, swelling capacity and solubility index were determined using previous methods [9]

Chemical composition. The pH of the tapioca cakes were determined using a pH meter, cyanide contents by Bradbury, Egan [19], while moisture, fat and ash contents were determined using standard methods [20]. Protein content was determined by Kjeldahl method ($6.25 \times N$) and total carbohydrate was calculated by difference.

Sensory evaluation. Consumers acceptability of cooked tapioca meal was conducted using semi-trained panelist (N=50) consisting of students in the Department of Food Science and Engineering, Ladoke Akintola University of Technology, Nigeria. Most panelists were between the ages: 18–30 and these were regular consumers of tapioca meal. A 9 point hedonic scale (1- extremely dislike; 9- extremely like) was used.

Statistical analysis. All experiments were conducted in duplicate. Data were analysed using analysis of variance (ANOVA) and means were compared using Fischer's Least Significant Difference Test ($p < 0.05$).

Results and discussion

Chemical composition. The major nutrient in unfortified tapioca control and the fortified samples was carbohydrate (Table 1). As expected, the protein content of the tapioca meal progressively increased with increase in the level of the fermented bambara flour (Table 1). Similarly, the fat, ash, crude fibre and moisture content of the tapioca meal increased significantly ($p < 0.05$) following the addition of fermented bambara nut flour. The protein contents of tapioca meal containing 50% fermented bambara nut flour was substantially higher (approx. 10 times) compared to the tapioca control. Bambara groundnut is a good source of protein and its inclusion into tapioca meal expectedly increased the protein content of the tapioca mixes. Previous studies similarly reported improvement in the nutritional value of tapioca meal fortified with full fat or defatted soybean flour [5, 6]. However, the protein content (12.07%) obtained in this study for tapioca meal fortified with 50% fermented bambara flour was slightly lower (approx. 1.6 times) compared to those previously reported for tapioca meal fortified with 50% full fat soybean flour [6]. Further, the fat content reported by these authors for the same sample was almost double the fat content in this study. Bambara groundnut is a pulse while soybean is an oil seed with higher protein and fat contents. Thus, the difference in protein and fat contents may be attributed to the higher protein and fat content in soybean compared to those in bambara flour.

Table 1

Proximate composition of tapioca-bambara flour mixes (%)

Tapioca	Bambara	Moisture	Protein	Fat	Ash	Crude fiber	CHO
100	0	10.47±0.00 ^b	1.24±0.01 ^a	0.65±0.01 ^a	1.85±0.01 ^a	1.77±0.00 ^d	84.00±0.01 ^f
90	10	10.33±0.01 ^a	3.95±0.01 ^b	1.35±0.01 ^b	2.43±0.01 ^b	1.81±0.01 ^b	80.10±0.02 ^e
80	20	10.47±0.01 ^b	4.75±0.15 ^c	3.76±0.01 ^c	2.62±0.01 ^c	2.12±0.01 ^c	76.26±0.12 ^d
70	30	10.55±0.01 ^c	7.63±0.01 ^d	5.85±0.01 ^d	2.75±0.01 ^d	2.25±0.01 ^d	70.95±0.04 ^c
60	40	10.62±0.01 ^d	10.55±0.00 ^e	6.54±0.01 ^e	2.95±0.01 ^e	2.36±0.01 ^e	66.96±0.02 ^b
50	50	10.54±0.01 ^c	12.07±0.01 ^f	7.46±0.02 ^f	3.11±0.02 ^f	2.41±0.01 ^f	64.39±0.03 ^a

Mean ± SD. Mean with the same superscript within a column are not significantly different (p<0.05).

pH and cyanide contents. The tapioca control had pH value slightly lower compared to the tapioca samples containing fermented bambara flour (Fig. 1). However, the difference was not significant. The cyanide contents of tapioca samples varied between 5.82 and 6.34 mg/kg for tapioca meal with 50% fermented bambara flour and the control sample respectively (Fig. 1). These values are within the range previously reported [6]. The cyanide content of the tapioca cake decreased slightly with increase in fermented bambara flour. This is expected since the tapioca which is the source of cyanide in the formulation reduced with increasing concentration of the fermented bambara flour. Tapioca sample containing 50% fermented bambara flour showed approximately 8.2% reduction in cyanide content.

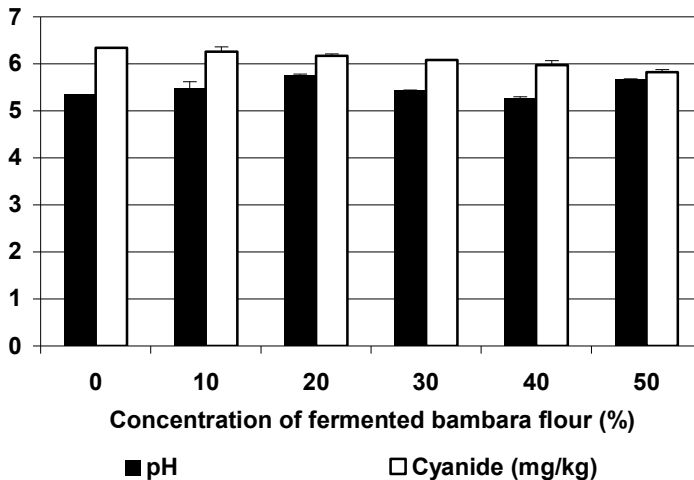


Fig 1. pH and Cyanide content of fortified tapioca

Error bars indicate n=2

Functional properties. Water absorption capacity of tapioca meal showed a minimal decrease with increasing concentration of fermented bambara flour (Fig. 2).

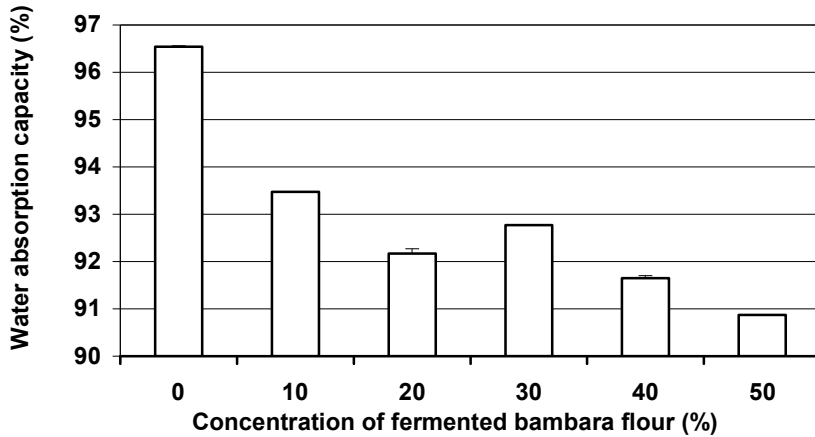


Fig 2. Water absorption capacity of fortified tapioca
Error bars indicate n=2

However, when the tapioca mixes were heated, they showed tangible swelling (Fig. 3). The applied heat may have enhanced starch disruption and subsequent leaching of amylose into the paste. Further, the high protein content of the fortified tapioca (Table 1) could also have contributed to the increased swelling power. Otegbayo, [9] working with tapioca meal reported a reduction in swelling power following the addition of full fat soybean. These authors attributed the reduction in swelling power to possible formation of amylose-lipid complex, which has been found to inhibit swelling in starch [21, 22]. However, in this study, fermented bambara which was used to fortify the tapioca cake are not rich sources of fats when compared to soybean. The lower fats contents of the bambara may have accounted for the relatively higher swelling power of the tapioca mixes when compared to those reported by Otegbayo, [9]. The solubility profile of the tapioca mix fortified with fermented bambara flour followed a similar trend as observed for swelling power (Fig. 3).

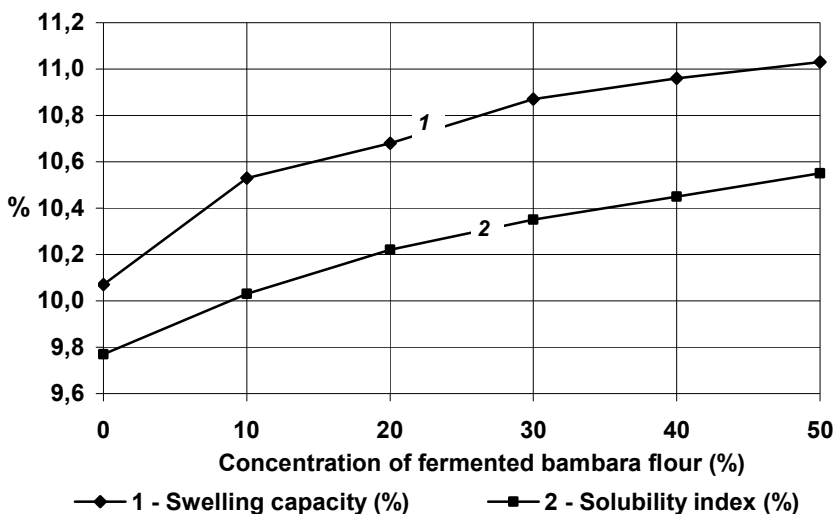


Fig 3. Swelling capacity and solubility index

Pasting. With the exception of pasting temperature (approx. 81°C) which appear similar, other pasting properties of tapioca mixes were significantly ($p < 0.05$) influenced by the added fermented bambara flour (Table 2). In general pasting time increased with increasing levels of fermented bambara flour (Data not shown). The peak viscosity also referred to as swelling peak of the control tapioca (163.16 RVU) decreased consistently with increasing level of fermented bambara flour up to 10%, 20% and 30%. However, the peak viscosity tends to increase when the concentration of fermented bambara flour in the tapioca meal reached between 40 and 50%. The increase in peak viscosity may be attributed to high starch content [23, 24]. This seems plausible since the bulk of carbohydrate in bambara grain is starch [11]. The high peak viscosity displayed by 50% substitution implies that the tapioca may be suitable for products requiring high gel strength and elasticity [25]. Previous research on tapioca meal fortified with 50% full fat soybean flour similarly reported reduction in peak viscosity of tapioca [9]. These authors reported approximately 53% reduction in peak viscosity for tapioca fortified with 25% full fat soya bean. In this study, with similar concentration (20%) of added fermented bambara flour, the reduction in peak viscosity was approximately 22%. This value is much lower compared to values reported by the authors working with tapioca meal fortified with full fat soybean [9]. The difference in peak viscosity reduction may be attributed to the higher fat content of soya bean compared to that of bambara flour. This seems plausible since lipids may interact with starch restricting granule hydration and swelling [26]. In addition, the fermentation process may have altered the composition of bambara groundnut flour.

The breakdown viscosity of the tapioca meal varied from 28.16 to 84.25 RVU for the control tapioca and tapioca with 50% fermented bambara flour respectively. High breakdown viscosity is generally associated with poor resistance to thermal disruption. Thus, the higher breakdown viscosities of the fortified tapioca mix suggest that these samples will show higher disintegration during heating.

The control tapioca meal showed higher tendency for starch retrogradation as indicated by its higher setback value of 108.59 RVU compared to the fortified tapioca (40.08-102.96 RVU). Many other authors have reported the reduction in setback value of tapioca fortified with legumes [9].

Final viscosity ranged from 124.31-243.28 RVU for the control tapioca and tapioca with 50% fermented bambara flour respectively. The variation in the final viscosity might be due to the re-association of starch molecules in the samples during cooling.

Table 2

Pasting properties of tapioca-bambara flour mixes

Tapioca	Bambara	PV (RVU)	BDV (RVU)	FV (RVU)	SBV (RVU)	PT (°C)
100	0	163.16±0.01 ^c	28.16±0.01 ^b	243.28±0.42 ^t	108.59±0.02 ^c	80.42±0.01 ^b
90	10	100.48±0.13 ^a	40.91±0.01 ^c	170.07±0.01 ^b	102.42±0.01 ^d	82.26±0.01 ^d
80	20	128.11±0.56 ^a	41.07±0.01 ^d	186.90±0.67 ^c	98.95±0.08 ^c	81.45±0.01 ^c
70	30	128.51±0.02 ^b	22.08±0.00 ^a	229.34±0.01 ^d	102.96±0.06 ^t	80.63±0.04 ^b
60	40	221.59±0.67 ^a	81.66±0.02 ^t	233.01±0.02 ^a	92.56±0.01 ^a	80.75±0.57 ^a
50	50	239.35±0.67 ^e	84.25±0.00 ^b	124.31±0.02 ^a	40.08±0.01 ^a	79.60±0.57 ^a

Mean ± SD. Mean with the same superscript within a column are not significantly different ($p < 0.05$).

PV: Peak viscosity, BDV: Breakdown viscosity, FV: Final viscosity, SBV: Setback viscosity, PT: Pasting temperature

Sensory evaluation. In general, tapioca meals fortified with fermented bambara flour had lower ratings compared to the control sample (Table 3). Tapioca cake fortified with 10% fermented bambara flour compared favourably with the control sample in colour, taste, aroma, consistency and overall acceptability. At higher concentrations (>10%) of fermented bambara flour the ratings for the tapioca meal was low when compared to the control. However, since the objective of this study was to improve the nutritional value of the tapioca meal, there may be a need to add some food additives especially to improve the taste. The colour of tapioca is generally accepted to be white in colour so the low rating recorded for the colour of the fortified samples is expected. Previous studies similarly reported lower ratings for aroma when tapioca was fortified with full fat soybean flour [6]. These authors, however, reported higher ratings for taste and overall acceptability, which may be attributed to the higher oil content of the soybean flour used for fortification.

Table 3
Mean sensory scores of tapioca mixes

Tapioca	Bambara	Colour	Taste	Aroma	Consistency	Overall acceptability
100	0	8.46 ^{cd}	8.40 ^{cd}	8.20 ^{cd}	8.60 ^c	8.88 ^d
90	10	7.60 ^c	7.46 ^c	7.53 ^c	8.13 ^c	8.00 ^d
80	20	5.80 ^a	6.40 ^b	5.66 ^a	6.86 ^b	6.00 ^b
70	30	7.13 ^c	5.80 ^a	6.26 ^b	5.73 ^a	6.60 ^c
60	40	5.26 ^a	4.66 ^a	5.20 ^a	5.66 ^a	4.60 ^a
50	50	6.33 ^b	5.40 ^a	6.06 ^b	5.26 ^a	5.46 ^b

Mean with the same superscript within a column are not significantly different ($p < 0.05$).

Conclusion

The inclusion of fermented bambara flour improve the swelling power and nutritional value of tapioca cake. Pasting properties of tapioca were significantly affected by fermented bambara flour. Acceptable tapioca meal can be produced using 10% fermented bambara flour. However, higher amounts of fermented bambara flour up to 50% can be used provided that additives to enhance taste and aroma will be added. Fortified tapioca meal could serve as valuable intervention to combat Protein energy malnutrition syndrome in Africa.

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High pressure in the technology of milk and soft cheese

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Abstract

Keywords:

High-pressure
Milk
Cheese

Article history:

Received 18.01.2016
Received in revised
form 03.03.2016
Accepted 24.03.2016

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Introduction. This work is devoted to the use of technology of high pressure in the production of milk and soft cheese, the substantiation of rational options of treating by high pressure of domestic raw milk in the production of drinking milk and soft cheese.

Materials and methods. The objects of research are: milk, processed with high pressure, soft cheese, produced with the use of high pressure. The mineral composition of milk and sour-milk cheese were determined by atomic and absorption spectrophotometry on the atomic and absorption spectrophotometer «C - 115 PC», the rheological properties of soft cheese were tested on electromechanical universal testing machine SANS CMT2503.

Results and discussion. With the help of research the mechanism of pressure and duration of treatment on the micro flora of milk and soft cheese has been found. Processing options have been selected at which inactivating effect of micro flora of milk and soft cheese is achieved.

In the process of experimental studies the rational processing options have been established and proved: for milk – the pressure of 300-330 MPa, the temperature is 40-45°C, the duration of exposure 30*60¹s; for cheese – the pressure 450-580 MPa, the temperature is 18°C and the duration of exposure – 20-30*60¹c.

In assessing physical-chemical characteristics of milk and cheese in comparison with control samples it was established that the total content of protein, fat, lactose and mass fraction of solid substances varies slightly.

The content of essential vitamins in milk and soft cheese is a sign of the maximum preservation. In milk treated by high pressure fat soluble vitamins are stored in 4-6 times more and water soluble in 2-3 times more than in pasteurized milk. In the soft cheese vitamins content is stored in 1,5-2 times more than in those produced by traditional technology.

According to the evaluating marks of sensor characteristics the products produced by the technology of high pressure received the highest scores: milk has got 98,6 points, soft cheese has got 96,4. Decline in consumer properties of control samples during storage was significantly faster than in the samples treated with high pressure, which are the result of enzymatic activity and the development of surviving micro organisms.

Conclusions. Technology of milk and soft cheese by using high pressure allows for microbial sterility of products to increase their shelf life, to preserve enzyme-vitamin complex feedstock.

Introduction

For the last decades high pressure technology has been widely applied in food industry that makes it possible to manufacture preservatives-free products of new quality, thus eliminating loss of vitamins and precious nutrients with improved taste and aromatic properties. High pressure processing of food products has been successfully applied in Europe, North America, Japan and New Zealand.

Fundamental scientific basis created by our predecessors, constant scientific research and numerous experimental work in research institutes and laboratories, extensive testing and clinical trials expanded significantly the idea of importance of milk and dairy products in human diet. Therefore, the main trends in the development of dairy industry aimed primarily on constant search, development and introduction of new technologies which allow obtaining high quality products with high biological and nutritional value.

The importance and the need in the use of high pressure in food industry are predetermined by numerous available scientific developments and the existing international experience.

Taking into consideration the needs of the industry and market requirements, the scientific laboratory of high pressure has made a complex of scientific research work in high pressure effects on safety, nutritive and biological value of milk and milk products, the increasing of their shelf life, the expansion of assortment by creating new dairy products with improved consumer properties [1].

Recent research in the sphere of the use of high-pressure technology with the aim to create high-quality dairy products are widely reflected in scientific works of many foreign researchers. The greatest interest are the studies, highlighted over the last five years in the works of scientists: Bruno Ricardo de Castro Leite Júnior and others [2], Bibiana Juan, Anna Zamora and others [3], Luciana M. Costabel, Carina Bergamini and others [4], G.G. Amador-Espejo and others [5], Evelyn, Filipa V.M. Silva [6], Francisca I. Bravo, Xavier Felipe, Rosina López-Fandiño, Elena Molina [7, 12], Sergio I. Martínez-Monteaquedo, Michael G. Gänzle, Marleny D.A. Saldaña [8], Genaro Gustavo Amador Espejo, M.M. Hernández-Herrero, B. Juan, A.J. Trujillo [9], G.G. Amador-Espejo and others [10], Has Mukh A. Patel, Thom Huppertz [11], Bravo FI, Molina E, López-Fandiño R. [13].

In Ukraine, the problem of the use of high pressure technology does not lose its relevance, the solution of which will help to receive products with high sanitary-bacteriological indexes without losing the natural food properties.

This work is devoted to scientific developments in the use of technology of high-pressure in the production of milk and soft cheese, basing the expediency and prospects of introduction of this technology in the domestic industry.

Materials and methods

The objects of research are: milk, processed by high pressure, soft cheese, produced with the use of high pressure. Control samples are raw cow's milk, heat-treated (pasteurized), mild cheese, produced by traditional technology, described in works [14, 15].

For the processing of raw milk high pressure, the sealed, in a special container, samples were placed in an optical camera installation of high pressure (up to 1000 MPa). The camera device allows to measure temperature changes in the range of 5 to 95 ° C.

High pressure processing parameters on the stage of research are the following: the pressure from 300 to 600 MPa, the processing time: from 10x60s to 30x60s, milk cures temperature 40-45°C; cheese process temperature of the product at the moment of

completing its self pressing: $18 \pm 2^\circ\text{C}$. Experimental samples of cheese were made from unpasteurized standardized milk with fat content 2.4%.

The mineral composition of milk and soft cheese were determined by atomic and absorption spectrophotometry on the atomic and absorption spectrophotometer «C - 115 PC», the rheological properties of mild cheese was tested for electromechanical universal testing machine SANS CMT2503 production «Shenzhen SANS Testing Co. Ltd.».

The content of Vitamin A was examined by colorimetric method; Vitamin C was determined by indophenols method; vitamins B₁ (thiamine) and B₂ (riboflavin) - by fluorimetric method.

The number of mesophilic aerobic and facultative anaerobic microorganisms in milk and soft cheese were determined by the method of quantitative crop of diluted samples of tested material on nutrient media. To do this, there were prepared Tenfold dilution of prototypes, followed by entering it in the amount of 1 ml sterile Petri cup and poured it melted and cooled to $40\text{--}45^\circ\text{C}$ with nutrient agar. After incubation determined number of colony forming units per unit volume of research material.

Organoleptic evaluation of the test samples of milk and cheese was made by the developed 5-point scale based on weight coefficients for each indicator. The change of flavoring properties was assessed by the profile method [16].

Results and discussion

In order to improve parameters of high pressure processing we have performed complex researches of microbiologic, physical and chemical properties of products.

According to data of the performed tests there has been adopted biophysical model which enables grounding of inactivation mechanism of milk microbial flora depending on technology parameters of treatment process and appropriateness of the chosen modes.

Concentration of microbial population in milk depends on temperature and pressure as shown in the Fig. 1.

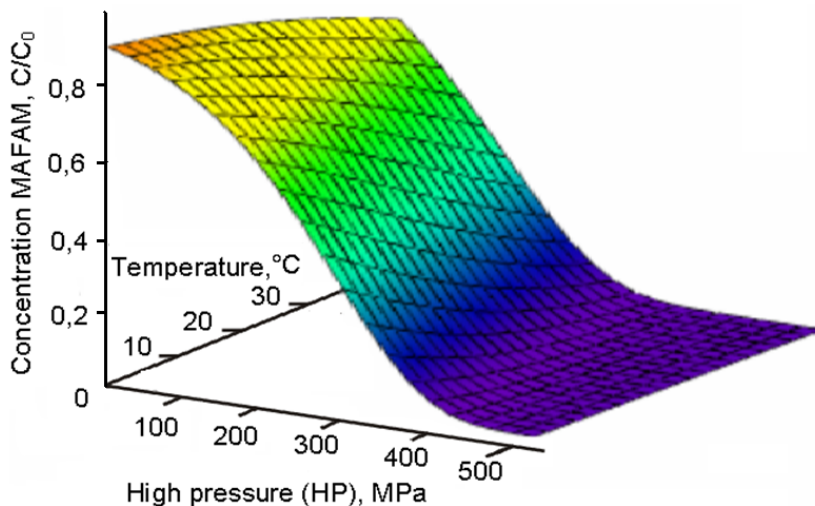


Fig. 1. Response surface and isolines of sections of concentration dependence of aerobic mesophilic count, colony forming unit/ sm^3 in milk from influencing factors (pressure, temperature)

In case of high pressure processing at lower temperature (up to 40°C) high inactivating effect may be achieved by applying pressure of 400-600 MPa. Increase of temperature shifts inactivation threshold to lower pressure, namely at temperature of 40-45°C it is enough 300-330 MPa to achieve the desirable effect. Influence of processing by pressure of 400, 500, 600 MPa corresponds to sterilization effect, but in that milk there occur denaturation processes. The most inactivating effect in result of milk processing by high pressure has been achieved at temperature 40-45°C and duration of processing 30*60¹C.

Thus, in soft cheese processed by high pressure of 300 MPa during 10, 20, and 30 minutes there have been found coliform bacteria. Application of 450 MPa pressure and higher during more than 10 minutes effectively inactivates opportunistic and pathogenic microflora.

According to optimization of high pressure processing there have been established the following parameters of high pressure technology (pressure, temperature, duration of processing): for milk – 300-330 MPa, 40-45°C, 30*60¹ c; for cheese – 450-580 MPa, 18°C, 20-30*60¹ c. These parameters make it possible to the best advantage to preserve biochemical properties of milk and are strong inhibiting factors as to microflora of the product.

In assessing physical-chemical characteristics of milk and cheese in comparison with control samples it was established that the total content of protein, fat, lactose and mass fraction of solid substances varies slightly.

Results of determination of basic vitamins content have proved maximal preservation thereof. There have been investigated change of quantity of basic vitamins A, B₁ and B₂, PP, C, which are important components of milk and soft cheese, and partially form biologic value thereof (tab.1).

Table 1

Content of basic vitamins in the tested milk and soft cheese, mg %

Vitamins, mg %	Content in milk		Content in milk, processed by high pressure		Content in soft cheese	
	Raw milk	Pasteurized	300MPa -40°C- 30·60 ¹ c	330MPa - 40°C- 30·60 ¹ c	Control samples (Cheese, produced from the pasteurized milk)	Produced with the use of HIGH PRESSURE
Vitamin A	0,031	0,018	0,027	0,025	0,211	0,254
Vitamin B ₁ (thiamin)	0,042	0,037	0,036	0,037	0,032	0,051
Vitamin B ₂ (riboflavin)	0,160	0,124	0,160	0,160	0,354	0,405
Vitamin PP (niacin)	0,09	0,09	was not determined	was not determined	0,309	0,407
Vitamin C	1,43	0,92	1,23	1,21	0,62	1,12

The comparative characteristic of the obtained results proves more favorable influence of high pressure on milk and soft cheese if compared to traditional production technology. Thus, in high pressure processed milk there have been preserved 4-6 times as much of fat-soluble vitamins and 2-3 times as much of water-soluble vitamins; in soft cheese produced with applying high pressure processing there are basic vitamins 1,5-2 times as much.

We have tested changes of several basic mineral components of milk and soft cheese playing an important role for stability of milk protein system and its transformation in the process of soft cheese production. Results of determination mineral elements in milk and cheese are given in the tables 2 and 3.

According to the data shown in the table it is obvious that high pressure processes and milk pasteurizing lead to change of salt composition. Thus, upon pasteurizing soluble calcium and phosphor levels decrease by 13% and 29.8% that may occur due to calcinations of casein complex at heating up to higher temperatures. High pressure processing involves increase of calcium content by 7% at 300 MPa, and to 12% at 330 MPa. Phosphor content in the result of high pressure milk processing is increased to 36.2%. Increase of calcium and phosphor levels probably takes place due to casein micelle fragmentation into smaller sub-micelles connected by mineral potassium phosphate (citrate), and as a result salts transfer from colloid state into solution.

Table 2

Content of basic mineral elements in milk processed by various means

Processing options	Content mineral elements (mg/ml)				
	Ca	P	Na	K	Mg
Raw milk	1,0	0,47	0,68	1,14	0,11
Pasteurized milk	0,87	0,33	0,73	1,12	0,12
300MPa -40°C-30-60 ¹ c	1,07	0,64	0,71	1,1	0,12
330MPa -40°C-30-60 ¹ c	1,12	0,64	0,71	1,12	0,13

High pressure of milk processing results in increase of Na concentration at an average of up to 7% and 4% if compared to check sample.

Content of potassium and magnesium salts upon various processing remained almost the same.

For determination of salts in cheese soft cheese has been taken as a check sample. The cheese has been produced without pasteurizing of milk mixture, as in the result of milk pasteurizing balance between various forms of potassium salts become destabilized, and as a consequence its ability to change into enzyme rennet declines.

Test data prove slight decrease of Ca and Na in soft cheese (by 0.85% and 0.83% consequently), produced with application of high pressure processing technology; content of Mg, K, Fe, Zn, Cu salts remained almost the same, cadmium is not found.

Table 3

Content of basic mineral elements in soft cheese

Mineral elements	Content mineral elements in soft cheese, mg/100 g	
	Control samples (Cheese, produced without pasteurization of milk), mg/100 g	Produced with the use of HIGH PRESSURE, mg/100 g
Ca	549,59	544,96
Mg	27,41	27,39
K	85,68	85,66
Na	1231,13	1221,02
Fe	0,61	0,7
Zn	0,84	0,82
Cu	0,01	0,02
Cadmium	Not found	Not found

We have tested rheological indices of soft cheese “penetration coefficient”, “limit cut tension” and “cutting operation” performed by electromechanical universal test machine SANS CMT2503 of Shenzhen SANS Testing Co. Ltd.

Analysis of change dynamics of the above mentioned indices revealed, that penetration coefficient and cutting operation significantly differentiate from these parameters in check sample and significantly depend on processing pressure. Limit cut tension has been to lower degree influenced by processing parameters and if compared to the check sample changes insignificantly.

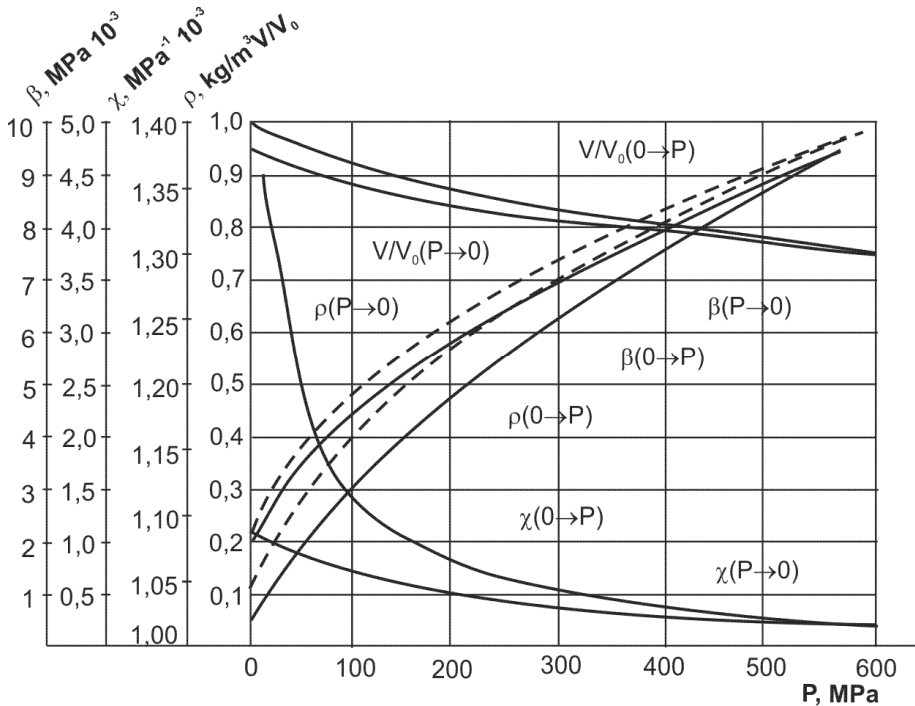


Fig. 2. Change of compression indices high pressure processed of soft cheese

In the result of experimental researches of compression properties of soft cheese (Fig. 2) it has been determined that during processing by 0 – 600 MPa pressure there take place changes in its rheological indices as follows:

- Relative product volume decreases up to 0.76, but upon pressure decrease it increases to 0.95 of the initial value;
- Product density increases by 29% (from 1,047 kg/m³ up to 1,351 kg/m³); upon pressure decrease density falls to 1,085 kg/m³, that equals to 3.6% of the initial value;
- Volume compression modulus in case of pressure rise increases almost 17 times as much (from 0,55×10⁻³ MPa up to 9,4×10⁻³ MPa) and then returns to the value of 2,0×10⁻³ MPa;
- Isothermal compression coefficient in case of pressure rise decreases by 77.3% (from 1,1×10⁻³ up to 0,25×10⁻³ MPa⁻¹) and then upon pressure decrease its value exceeds 4,5×10⁻³ MPa⁻¹.

In order to use the obtained experimental values of the parameters for predicting product status with various process parameters, calculations and projecting of technology equipment for production of soft cheese by applying high pressure technology experimental curves have been described by mathematical functions.

Functional $\chi = f(P)$ и $\beta = f(P)$ connections are described by general function (1):

$$y = a + b \cdot e^{-\frac{x}{c}} + d \cdot e^{-\frac{x}{g}}, \quad (1)$$

Functional $\rho = f(P)$ и $V/V_0 = f(P)$ connections are described by general function (2):

$$y = a + b \cdot x + c \cdot e^{-\frac{x}{d}}. \quad (2)$$

Results of processing and statistic analysis of the obtained connections are given in the table 4.

Thus, in the result of experimental researches there have been obtained values of rheological indices for various parameters of production process and there has been performed comparative analysis for samples of soft cheese produced by traditional technology and high pressure technology.

Table 4

Results of mathematical processing of the functional connections

Parameters		R ²	F-statistics	Values of the coefficients				
				a	b	c	d	g
$\chi = f(P)$	0 → P	0,997	1835,22	0,393	149,86	326,59	-149,25	329,28
	P → 0	0,998	4980,93	0,061	6,98	15,5	2,42	157,42
$\beta = f(P)$	0 → P	0,999	17668	19,4	-0,84	64,46	-16,45	1152,56
	P → 0	0,999	36032	16,94	-0,41	44,49	-15,92	770,89
$\rho = f(P)$	0 → P	0,999	29087	820,5	0,96	-586,16	221,16	-
	P → 0	0,999	17527,4	2544,52	-0,41	-	2140,68	710,94
$V/V_0 = f(P)$	0 → P	0,998	4425,07	0,84	-	0,0002	0,159	192,07
	P → 0	0,997	3180,8	0,63	3·10 ⁻⁵	0,312	428,8	-

Complex evaluation of organoleptic indices has made it possible to determine that products produced by high pressure technology show the highest induces: milk – 98.6 points, soft cheese – 96,4 points.

Profile analysis of taste properties of high pressure processed milk has revealed high level thereof. Raw milk and pasteurized milk are included into your rating with 89.5 points and 81.2 points consequently, and rank as good quality products. First and foremost, it is connected with appearance of fatty film on the surface and peculiar pasteurizing taste, as the signs of denaturation of whey protein. Change of taste properties of high pressure processed milk if compared to raw and pasteurized milk are represented by profile charts (fig 3, 4).

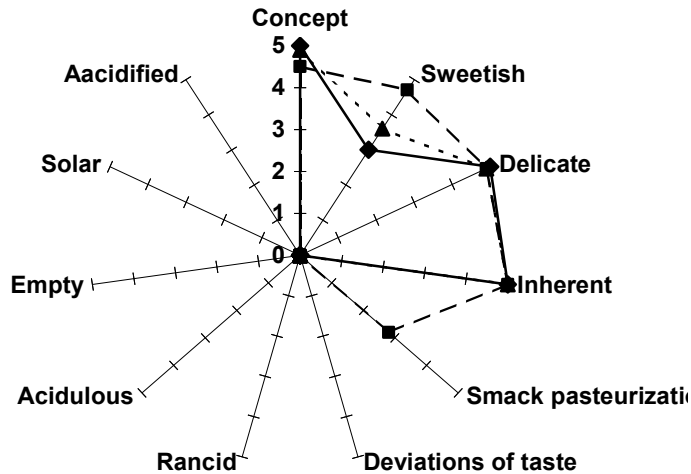


Fig 3. Taste profile chart of the tested samples at the beginning of storage period:

- raw milk;
- pasteurized milk;
- high pressure processed milk.

Taste profiles of high pressure processed milk are similar to raw milk taste, except of more pronounced sweet flavor in case of high pressure processing. These differences are more characteristic of pasteurized milk.

According to results of organoleptic evaluation both samples of cheese are of high quality and possess highest quality indices. Soft cheese produced with application of high pressure technology ranks higher if compared to the check sample, thus, it may be concluded that new high pressure technology positively influenced organoleptic properties of soft cheese (fig 5, 6).

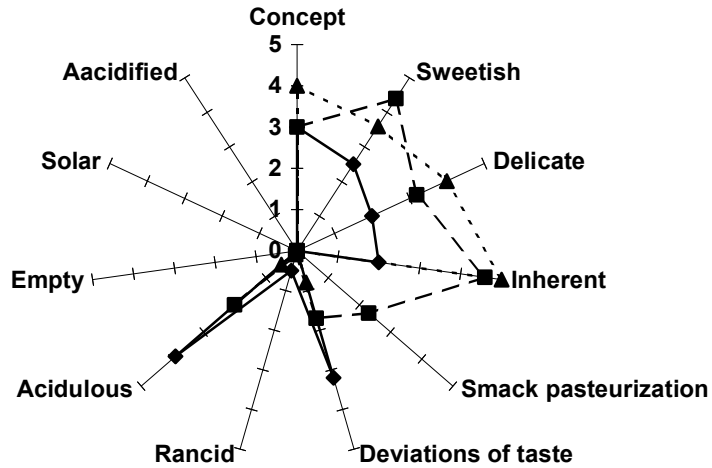


Fig 4. Taste profile chart of the tested samples during the storage period:

- raw milk;
- pasteurized milk;
- high pressure processed milk.

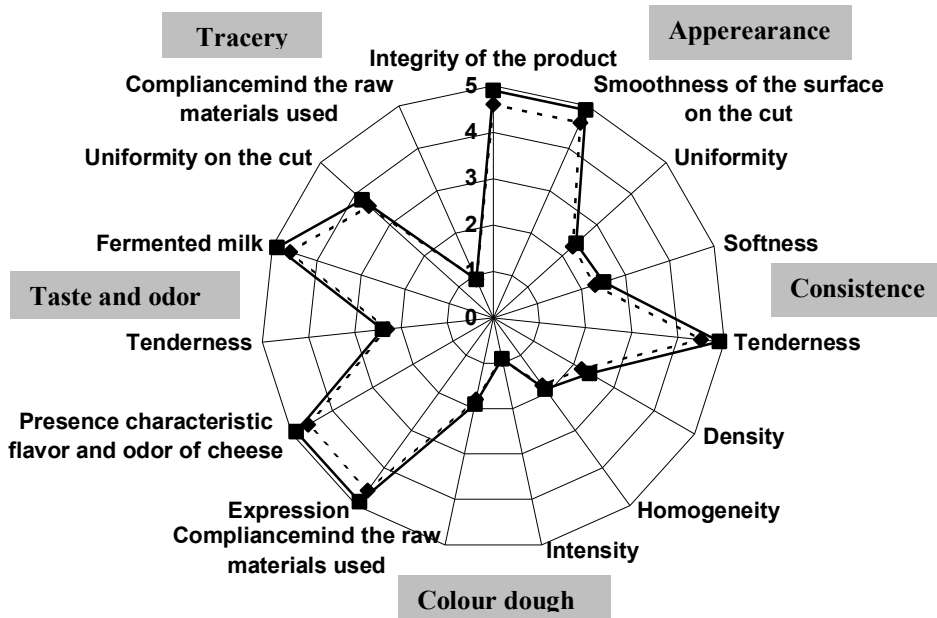


Fig 5. Profile chart of organoleptic indices soft cheese

- Control sample
- Soft cheese produced with the use of high pressure

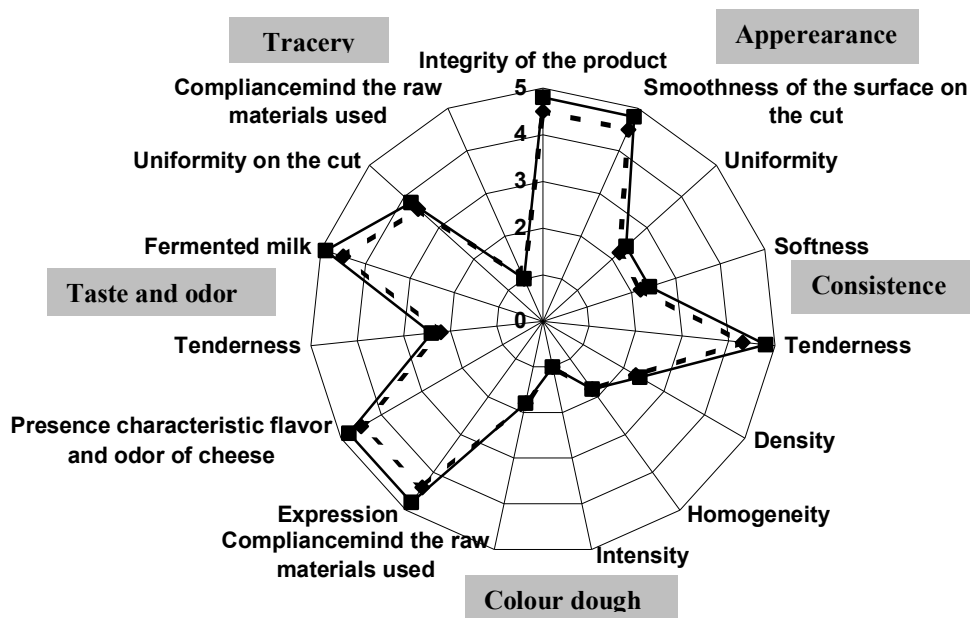


Fig 6. Profile chart of organoleptic indices soft cheese during the storage period

----- Control sample

———— Soft cheese produced with the use of high pressure

If compared to the check sample soft cheese looks more attractive with more pronounced flavor if compared to the check sample. Its body is more homogeneous, soft and elastic, while the check sample is harder and a little crumbly. To our opinion such changes in soft cheese have become possible due to increase of attached moisture volume under high pressure.

Decline in consumer properties of control samples during storage was significantly faster than in the samples treated with high pressure, which is the result of enzymatic activity and the development of surviving micro organisms.

Conclusions

The research and study of the mechanism of high pressure combined with a thermal treatment on inactivation of microflora and change the components of milk and soft cheese gave grounds to determine rational modes of the use of high pressure technology.

During the storage of milk processed by high pressure in the first three days, a decrease of the concentration of microorganisms is happened, at the expense of processing efficiency increases to 98.6%. Further storage of such milk accompanied by increased microbial numbers, but with a lower level of intensity than in raw milk and processed high temperatures.

The changes in physic-chemical and biochemical parameters together determine the pattern of changes milk quality and soft cheese, processed by high pressure, in particular vitamin, mineral composition, rheological and organoleptic properties.

The presented analytical summary of high pressure effects on the quality of milk and soft cheese proves the prospects in implementation of this technology in the dairy industry with the aim of producing products with high biological value of durability.

It should be noted that the research in this area continues, in particular, the most important is the question of technological properties of milk processed by high pressure and prospects for its further use in the manufacture of dairy products.

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Sensor analysis of functional biscuits

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Abstract

Keywords:

Sensor
Analysis
Functional
Biscuits
Inulin
Fibregum

Article history:

Received 24.02.2016
Received in revised
form 20.03.2016
Accepted 24.03.2016

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Introduction. The biscuits are important bakery products which are favorable due to lower production costs, convenience and long shelf life. Usually are consumed as a dessert or as a light snack between meals. The classic biscuits have functional properties, so it is necessary to change the composition recipe by adding different functional components. In that context by adding inulin and acacia gum the obtained biscuits are with functional features and then they are sensory assessed.

Materials and Methods. This paper is made as sensory evaluation of three types of functional biscuits "Fructi"; "Fructi + Inulin" and "Fructi + Fibregum". The biscuits were assessed by 46 evaluators from R. Macedonia and Bulgaria. Biscuits evaluators assessed according to the following sensory attributes: appearance, structure and breaking, smell, taste and chewable.

Results and discussion. It is necessary to know the sensory characteristics of an appropriate product because they determine its quality. From the conducted sensory evaluation of three types of functional biscuits is determined that the with the highest scores in terms of appearance are assessed biscuits "Fructi + Fibregum" (3.45). The same biscuits with highest proportions of points are assessed in terms of the structure and breaking, as well as in terms of smell and taste. In terms of chewing biscuits "Fructi" are rated with a higher number of points (19.3) compared to biscuits "Fructi + Inulin" and "Fructi + Fibregum". With highest total average sensory evaluation are assessed biscuits "Fructi + Fibregum" (16.42).

Conclusion. From the conducted sensory analysis can be concluded that the biscuits "Fructi + Fibregum" are featured with the best sensory characteristics.

Introduction

On the road to optimal nutrition which presents ambitious long-term goal, "functional food" look new, interesting concept. This concept should be built on solid scientific foundations, while being accepted by consumers [1].

In accordance with FUFOSSE functional food is characterized by the following features: conventional or daily food or supplements; natural components present in food; a proven beneficial effect on certain functions outside the nutritional value of the product; possess conclusive scientific studies proving the enhanced well-being and health and / or reducing the risk of disease and / or improve the quality of life including physiological and psychological improvement [2].

In a broad concept of functional food Mishan [1] lists the: natural nutrition rich food, food that is enriched with functional ingredients, food which excludes certain ingredients, food in which are changed the properties of certain components, food in which the bioavailability of one or more components has been modified and all combinations of these possibilities.

Biscuits are type of cookies with a grain base and containing a large quantity of sugars and fat levels [3]. The composition of biscuits includes a number of raw materials, different enhancers and other accessories, so they differ in appearance, composition, mass, consistency, structure and production technology [4]. There are possibilities for the production of dietetic biscuits with sugar replacement, using fats with different characteristics, as well as enrichment of biscuits with different functional components [5].

Dietary fiber have many characteristics which include them as an important ingredient in the recipes for production of functional foods [6,7].

Inulin is used in food industry to increase the proportion of dietary fiber in the final product. Advantage over "traditional" dietary fiber is that inulin does not possess a distinctive raw taste and does not contribute to increased viscosity of the final product, so its usage results in products enriched with dietary fibers that retain the organoleptic properties of the standard recipe composition [8].

Acacia gum is used in food industry for decades as an additive to diet. When used as a food, the acacia gum has a role as an improver, thickener, stabilizer, emulsifier, and coating agent. Such different functions of a product makes it unique [9].

The sensor quality (shape, color, smell, taste, texture) is of great importance for all food products. It is a feature that every customer everyday evaluates and based on that assessment makes the decision whether that product will be bought [10].

Since biscuits belong to the group of confectionery which are characterized by attractive appearance, pleasant taste and aroma it is very important to analyze their sensory characteristics.

In this paper we examined the sensor characteristics of three types of functional and fortified biscuits and is determined their sensory quality.

Materials and methods

In this paper was made a sensor analysis of three kinds of functional biscuits ("Fructi", "Fructi + Inulin" and "Fructi + Fibregum"). Biscuits "Fructi" are the basic type of functional biscuits, while biscuits "Fructi + Inulin" are with added inulin to the basic recipe composition, and biscuits "Fructi + Fibregum" are with added Fibregum (Acacia gum), whereby the biscuits "Fructi" further are enriched with fiber.

The biscuits are made in a confectioner's shop "Sweet Pleasures" in Veles, Republic Macedonia in accordance with HACCP standards.

The produced biscuits were evaluated using the scoring method [10] by 46 assessors. All sensor properties are clearly defined and described in the form of sensor evaluation of biscuit cookie (Table 1).

Table 1

Application form for sensor evaluation of biscuit cookie [4]

Quality Factor	O	FZ	Evaluation	Requirement of quality
Appearance (size and shape)	5	0,8		Adequate characteristic (circular) shape, without damage to upper and lower surface.
	4			Very slight deviation from the circular shape.
	3			Deviation of the shape, size.
	2			Great deviation from the shape and size.
	1			Deformed, a large deviation of the biscuit cake.
Structure and snapping	5	0,8		Soft, uniform fine structure.
	4			Very small deviation of porosity.
	3			The biscuit cake is dry, slightly crumbling.
	2			The biscuit cake is expressed dry.
	1			The biscuit cake is very dry.
Chewing	5	0,8		The biscuit cake is soft, evenly softens in the mouth.
	4			Very little deviation from the optimal.
	3			The biscuit cake is dry, hard with a sharp taste.
	2			There is rather atypical chewing.
	1			Expressed rather atypical chewing.
Scent	5	0,6		Characteristic, slightly sensitive, aromatic. Constant for specific time period.
	4			Characteristic, less pronounced aromatic.
	3			Slight odor, low aromatic.
	2			Slight odor.
	1			Atypical strange smell.
Flavor	5	1,0		Characteristic, slightly sensitive, aromatic. Constant for specific time period.
	4			Pleasant permanent taste.
	3			Moderate pleasant taste.
	2			Slight pronounced taste.
	1			Atypical strange taste.

The individual parameters are corrected with FZ (correction factor), by which have been obtained points (PB), whose assembly is obtained to total number of points for sensor quality of the product. Depending on the sum of points is determined in which category the product belongs to by sensor quality (Table 2).

Table 2

Category of sensor quality depending on the sum of gained points (PB)

Quality category	Sum (PB)
Excellent	19.1-20.0
Very good	16.1-19.0
Good	13.1-16.0
Frail	11.1-13
Not match	<11.1

Results and discussion

Sensor or organoleptic properties of food, as an aspect of quality, are associated with a sense of comfort that food can provide when consuming, and include those attributes that can be perceived with the senses of sight, smell, taste, touch, and even with hearing. Sensor properties are the first and often the only parameters on which the majority of consumers assess the quality of food [11]. The results of the sensor analysis are given in (Table 3).

Table 3
Results of sensor analysis of functional biscuits

Quality Factor Type of biscuits	Appearance	Structure and snapping	Scent	Chewing	Flavor	Total PB
“Fructi”	3.43	2.98	2.44	3.19	3.59	15.63
“Fructi+Inulin”	3.41	2.98	2.34	3.09	3.71	15.53
“Fructi+Fibregum”	3.45	3.28	2.62	3.01	4.06	16.42

* The values in this table are calculated according to the average values from the results of the evaluators.

Appearance. The appearance or optical property is based on the sense of sight, and it includes the capacity that can be visually examined. Every merchant knows that the appearance is often the sole purpose of which is based the decision whether something is to be bought and eaten or not. About the size and shape are important: the length, thickness, width, size of particles, the geometric shape (square, circular, etc.) [12].

The color affects the appearance, as well. Color is important because it is used as a control parameter during baking of biscuits [13]. Many factors influence the formation of the final texture of products, including temperature, air speed, humidity and heat transfer in the sample [14].

From Table 3 can be concluded that in terms of appearance with the highest score were assessed biscuits "Fructi + Fibregum" (3.45), and the least biscuits "Fructi + Inulin" (3.41). Biscuits "Fructio" are evaluated with 3.43 points.

The external appearance of biscuits with a different recipe composition is evaluated by Yadav et al. [15] who examined the effect of partly skimmed peanut flour on nutritional, organoleptic and physico-chemical characteristics of biscuits. Best assessed in terms of external appearance were biscuits which recipe composition contained 0% partly skimmed peanut flour.

Structure and snapping. Based on the results obtained from sensor analysis of biscuits (Table 3) is determined that the biscuits "Fructi" and "Fructi + Inulin" are characterized by the same number of points, regarding the structure and snapping (2.98 points). With a higher number of points (3.28) compared to the same parameter, are featured biscuits "Fructi + Fibregum".

Yenkar et al. [13], who made sensor analysis of biscuits made of Rajgira and Sabudana (local types of flours that are rich in vitamins and minerals) in a different ratio, found that in terms of structure best are biscuits made of flour Rajgira and Sabudana in the ratio 3: 2.

Best rated biscuits are from skim peanut flour in terms of structure which in their biscuit recipe have composition of 5% of partly skimmed peanut flour [15].

Jothi et al. [16] did tests on crackers (a type of biscuits) made with various flours (potato flour, rice flour without gluten, a type of Italian mix of flours ...) in a different ratio, using as a control sample, in which they took biscuits from wheat flour. Regarding the structure, the best rated were the control types of cookies (biscuits made of white wheat flour).

In contrast of this, during sensor analysis of biscuits made in a different proportion of white wheat flour and skim sesame flour, the best structure according to the evaluators had biscuits made from 80% white wheat flour and 20% skimmed flour sesame, while the worst biscuits were made only of white wheat flour (control) [17].

Govindaraj et al. [18] made surveys of biscuits fortified with FeSO_4 and NaFeEDTA and varying amounts of tartaric and citric acid. Between biscuits fortified with FeSO_4 and different amounts of tartaric acid (60, 80 and 10 mg) and between biscuits fortified with FeSO_4 and different amounts of citric acid (60, 80 and 10 mg), structure is best evaluated in the control sample in which nothing is added. Also, during fortification of biscuits with NaFeEDTA and adding a predetermined quantity of tartaric acid (60, 80 and 10 mg) and fortification of biscuits with NaFeEDTA and different amounts of citric acid, the control sample of biscuits is best assessed in terms of structure.

Scent. The scent and taste act pleasant, define and consist of basic scent and taste derived from the aromatic compounds of the essential ingredients, and aromatic additives in raw materials composition.

The scent of the product will meet the required standards, if is characteristic scent of the raw material from which it is derived, without foreign uncharacteristic odors [19].

According to the established average values for the scent of biscuits, with the highest score 2.62 are assessed biscuits "Fructi + Fibregum". The least number of points of 2.34 are assessed biscuits "Fructi + Inulin", and biscuits "Fructi" are evaluated with 2.44 points.

Between biscuits prepared by types of flour Rajgira and Sabudana, which were added at different ratio, in terms of scent, best were rated biscuits made of flour Rajgira and Sabudana in the ratio 2: 3 [13].

From biscuits in which in the recipe composition is added a certain amount of skimmed peanut flour, Yadav et al. [15] noticed highest grades of scent at biscuits containing 5% partly skimmed peanut flour.

The biscuits made of 70% flour and 30% skimmed sesame flour in terms of scent were the worst evaluated during sensor analysis, made by Gernah et al. [17].

Chewing. When chewing the biscuits, the senses of touch, scent and taste register all impressions and evaluate internal quality properties [19].

In terms of chewing biscuits "Fructi" are assessed with a higher number of points (19.3) as compared to biscuits "Fructi + Inulin" and "Fructi + Fibregum", which compared to the same parameters are assessed with 3.09 and 3.01 point (Table 2).

Taste. From the data given in Table 3 is show that with highest average value for taste (4.06 points out of a possible 5) are assessed biscuits "Fructi + Fibregum" and least (3.59 points) the biscuits "Fructi". Biscuits "Fructi + Inulin" are evaluated with 3.71 points.

During sensor analysis of biscuits made of Rajgira and Sabudana, Yenkar et al. [13] is concluded that the biscuits prepared from flour Rajgira and Sabudana in a ratio of 3: 2 are the best in terms of taste.

Based on the results obtained by Yadav et al. [15] during examination of the effect of partly skimmed peanut flour on the characteristics of biscuits, it is shown that in terms of taste are best assessed biscuits which in the its recipe consist of 0% partly skimmed peanut flour.

Jothi et al. [16] based on the results of the conducted sensor analysis of different types of biscuits concluded that white flour biscuits compared to ones which in its composition contain different types of flour show the best quality features in terms of taste.

On the contrary, Gernah et al. [17] concluded that biscuits prepared from 90% white wheat flour and 10% skimmed sesame flour are best compared to the same parameter.

Total sensor evaluation. The sum obtained from individual points of appearance, structure and breaking, scent, chew and taste gives the total assessment (total points) of sensor analysis of biscuits, which in our case is a maximum of 20 points.

According to the results from the performed calculations for total points shown in Table 2, with highest average total mark are assessed biscuits "Fructi + Fibregum" (16.42), less biscuits "Fructi" (15.63), and least biscuits "Fructi + Inulin" (15.53).

Based on sensor evaluation (Table 3) is defined the category of sensor quality in which belong the obtained biscuits (Table 1). Biscuits "Fructi" and "Fructi + Inulin" belong to the group of products with good quality and biscuits "Fructi + Fibregum" in the group of products with very good quality.

From the performed sensor analysis of the biscuits fortified with *Cyperus esculentus* L. as partial substitute for wheat flour (10, 20 and 30%) by Ahmed et al. [20], it is provided the best overall score for biscuits which were with 20% *Cyperus esculentus* L.

Other authors presented their results of sensor analyzes of different types of cookies. For example, Yenkar et al. [13] concluded highest total sensor evaluation of biscuits made from flour Rajgira and Sabudana in the ratio 3:2. Yadav et al. [15] in sensor evaluation of biscuits in which recipe composition is added a certain amount of peanut flour skimmed best, overall final assessment was found the biscuits recipe which composition does not contain skimmed peanut flour.

Between biscuits analyzed by Johti et al. [16] as the best biscuits in the acceptability are assessed control biscuits ie white flour, while others biscuits which in its composition contain different types of flour (potato flour, rice without gluten, a type of Italian mix of flour) are assessed lower. In terms of total acceptance of cookies with different ratio of white wheat flour and sesame skimmed flour, Gernah et al. [17] best results found in biscuits made from 90% white wheat flour and 10% skimmed flour sesame.

Conclusion

From the conducted sensor analysis can be concluded that the biscuits "Fructi + Fibregum" are featured with the best sensor characteristics. Biscuits "Fructi + Fibregum" are most highly rated in terms of layout, structure and crashing, scent and taste, and in terms of chewing are best assessed biscuits "Fructi". Based on the results, can be concluded that the biscuits "Fructi" and "Fructi + Inulin" belong to the group of best products, and biscuits "Fructi + Fibregum" to the group of very good products.

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Influence of packaging on the quality of soft brine cheese fortified with seaweed additive

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Abstract

Keywords:

Cheeses
Packing
Storage
Quality
Brined

Article history:

Received 23.01.2016
Received in revised
form 20.03.2016
Accepted 24.03.2016

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Introduction. The aim of the article is to study the influence of different ways of packaging and storage on consumer properties of soft brine cheeses, as well as changes in quality during storage. Soft brine cheeses fortified with dietary supplement have organically bound iodine that can prevent a lack of iodine in human nutrition [1-3].

Materials and methods. The objects of research were new soft brined cheeses: "Sample 1" - with the addition of supplement from brown seaweed in an amount of 0.5% to weight of cheese, "Sample 2" - with supplement of brown seaweed in an amount of 0.5% and with serum albumin in an amount of 0.3% to weight of cheese. "Sample 3" - produced by thermoacid method with the addition of supplement from brown seaweed in an amount of 0.5% to weight of cheese. "Control 1" - was soft brine cheese produced by the traditional method. "Control II" - soft brine cheese produced by thermoacid method according to traditional recipe. Soft brine cheeses were investigated immediately after production and on 2, 3, 5, 10, 30, 45, 60 days. Sensory evaluation of quality the new soft brine cheeses was carried out according to the developed by us 5-point scale. The amino acid composition - by ion-exchange liquid-column chromatography (the amino acid automatic analyzer T 339).

Results and discussion. According to research results brine cheeses maturation for 14 days in brine and subsequent packaging and storage (at 2 ... 5 °C for 60 days) in plastic bags "Saran" has a positive effect on its quality. In the soft brined cheese "Control 1" quantity of free amino acids have increased by 19.8%, whereas at the soft brined cheese "Sample 1" quantity of free amino acids have increased by 21.2%, at "Sample II" - quantity of free amino acids have increased by 20.8%. "Control II" was characterized by an increase in the quantity of free amino acids by 7.7% in case of packaging in plastic film. At the same time, "Sample III" was characterized by increasing of quantity of free amino acids to 22.2%. This method of storage pickled cheeses slows down the process of moisture loss, promotes intensification of proteolysis and increased levels of free amino acids, which positively affects the organoleptic characteristics.

Conclusions. The obtained results allow us to improve the method of ripening and packaging of soft brine cheeses, their consumer properties.

Introduction

One can store and form the application properties of brined cheese at the proper level under condition of using an appropriate package, packing, creating the optimal regimes and periods of storage, transportation [4].

Consumer demand and requirements of trade cause various types of packaging, the range of which is also affected by changes in the environment. Forming and packing equipment offers new opportunities. In a market conditions an appearance, label design and quality of packing materials are important. The main trends in packaging of brined cheese are diversification, improvement of quality indicators, attractive packaging and label, convenience when consumed [5].

In Ukraine, along with the classic type of packaging such as glass, paper, and polymer packaging plays an important role in the food industry that is widely used for packaging of cheese [6].

Such requirements are imposed on polymer films packing [7]:

- general - non-toxicity, mechanical strength, neutral to taste and smell of packed product;
- special: low oxygen permeability (less than $400 \text{ sm}^3/\text{m}^2$ per day) to prevent mold; limited moisture permeability (less than $0.02 \text{ kg}/\text{m}^2$ per day) to prevent drying of the product; permeability to CO_2 , which is formed during cheese ripening ($500 - 2500 \text{ sm}^3/\text{m}^2$ per day).

In general the packaging, especially the consumer packaging, is important for all dairy products and specifically for cheese. Firstly, because these products are perishable, unless they are stored in the appropriate conditions. Secondly, the storage conditions are too individual for different kinds of cheese. Using different methods and conditions of use of brined cheese, the manufacturers of these products are constantly improving packaging, matching it considering the shelf life, packaging weight, ease of use. The following effects on choice of packaging for cheese: consumer demand, which depends on its income, demographic changes, local habits and traditions of food consumption [8].

“Active” and “smart” packaging which regulates microbiological processes occurring inside the package, and which reports on the status of the product, guaranteed health and safety of consumers can save quality of products.

“Active” and “smart” packaging emerged in economically developed countries (USA, Australia, New Zealand, Japan and Western Europe) according to the project of the European Union ASTIRAK (1999-2001), which provides an assessment of safety, impact of "active" and "smart" packaging on environment, creation in the field of legal rules [9].

The main function of "active" packaging is the impact on a product extension of its shelf life and storage characteristics: taste, color and so on. The material from which the packing is made is biologically active: a polymer matrix tightly holds immobilized supplements (e.g., potassium carbonate compounds, enzymes, gas and moisture absorbers, fragrances, antimicrobials).

The abovementioned packing allows you to regulate microbial balance inside the packaging, shelf life of the product. Modified and adjustable air is mostly used in the "active" packaging. This quite expensive technology, however, is compensated through its use.

The advantage of "active" packaging is that the migration of chemicals in foods is close to the minimum. "Smart" packaging is intended to respond to the impact of the environment on the condition of the product and to inform consumers of this state. The cost of such packing is high, as it requires the use of modern dyes (thermochromatic dye 767

VK) and even microprocessors. However, a significant part of materials for "smart" packaging is still under development and experimentation.

Packaging for cheese is characterized by general trends of development - this is the minimization of material resources through the use of polymer films, including multilayer, which prolong the shelf life of the cheese due to the high barrier properties. It is important that manufacturers of cheese take into account the customer requirements regarding usability of packaging when products are consumed.

For packing of brined cheese the consumer and transport packaging are used. A bag of polyethylene film, polypropylene containers (plastic containers with a capacity of 350, 500g), glass jars or other containers made of polystyrene serve as consumer packaging. The metal containers are rarely used (up to 5 kg). Brined cheese is packed in shipping containers of a net up to 5.0 kg. Packing of cheese in brine in buckets of polystyrene is allowed under the current legal documents.

During packaging the hygienic indicators, water and gas impermeability, and level of protection against light are important to preserve the quality of a food product. During packaging under vacuum the film should fit tightly to the surface of the cheese. During vacuum packaging the oxygen level inside the packaging is reduced to less than 1%. Barrier properties of polymer films prevent the penetration of oxygen into the vacuum packaging. Due to the vacuum plastic packaging the shelf life is increased depending on the type of cheese from 60 to 90 days [10, 12].

Polymer film of polyvinyl chloride (PVC) is often used in the production of brined cheese. This is explained by ease of its use, thermal stability in the temperature range (from -10 °C to 75 °C), the possibility of overprint, low permeability of air, steam, gas and odors; resistance to fats and oils. Ripening of brined cheese in a film helps to reduce the cost of labor associated with caring for cheese during ripening, the decrement of product loss. The use of film in vacuum at cheese packaging has a positive effect on the quality of cheese and accelerates their ripening.

The prolonged ripening and storage of cheese in brine environment is the result of the traditional conditions of the brine cheese production in Ukraine. Although brine well preserves the product from damage, but this method of ripening and storage degrades the quality of cheese during prolonged staying in brine. Therefore, we conducted research on the impact of various types of packaging on the quality of soft brine cheese.

Materials and methods

The objects of research were new soft brined cheeses: "Sample 1" - with the addition of supplement from brown seaweed in an amount of 0.5% to weight of cheese, "Sample 2" - with supplement of brown seaweed in an amount of 0.5% and with serum albumin in an amount of 0.3% to weight of cheese. "Sample 3" produced by thermoacid method with the addition of supplement from brown seaweed in an amount of 0.5% to weight of cheese. "Control 1" - was soft brine cheese produced by the traditional method. "Control 2" – soft brine cheese produced by thermoacid method according to traditional recipe. Soft brine cheeses were investigated immediately after production and on 2, 3, 5, 10, 30, 45, 60 days. Sensory evaluation of quality the new soft pickled cheeses was carried out according to the developed by us 5-point scale. The amino acid composition - by ion-exchange liquid-column chromatography (the amino acid automatic analyzer T 339).

Results and discussion

According to Table 1 brined cheese ripening research during 14 days in brine and ripening during 45 days in the film has a positive effect on its quality.

Table 1
Organoleptic assessment of quality of soft brine cheese depending on packaging

Packaging	Taste and smell		Texture		Total
	Characteristics	Point	Characteristics	Point	
Control 1					
The ripening in brine during 60 days	Pure, satisfactory	4,2	Satisfactory, slightly dense	4,0	4,1
The ripening in brine during 14 days and after the ripening in the film during 45 days	Pure, good	4,5	Good	4,5	4,5
Sample 1					
The ripening in brine during 60 days	Pure, good	4,5	Good	4,8	4,7
The ripening in brine during 14 days and after the ripening in the film during 45 days	Pure, excellent	5,0	Excellent	5,0	5,0
Sample 2					
The ripening in brine during 60 days	Pure, good	4,7	Good	4,7	4,7
The ripening in brine during 14 days and after the ripening in the film during 45 days	Pure, excellent	4,9	Excellent	5,0	5,0
Control 2					
The ripening in brine during 60 days	Pure, satisfactory	4,4	Satisfactory, slightly dense	4,2	4,3
The ripening in brine during 14 days and after the ripening in the film during 45 days	Pure, good	4,9	Excellent	5,0	5,0
Sample 3					
The ripening in brine during 60 days	Pure, good	4,6	Good	4,8	4,7
The ripening in brine during 14 days and after the ripening in the film during 45 days	Pure, excellent	5,0	Excellent	5,0	5,0

Organoleptic indicators of cheeses “Sample 1”, “Sample 2”, “Sample 3” under study have more scoring than those related control brined cheeses.

In the above-mentioned method of brined cheese packing the improvement of their organoleptic characteristics as taste, smell and texture occurs. If the control cheeses are characterized by good organoleptic characteristics after the ripening in brine during 14 days and after the ripening in the film during 45 days, the experimental cheeses are characterized by excellent quality.

According to Table 2 cheese, ripening in the film slightly speeds up proteolytic processes.

Table 2
Content of free amino acids in soft brine cheese depending on packaging mg/100 g of cheese

Content of free amino acids	Brine cheese	
	The ripening in brine during 60 days	The ripening in brine during 14 days and after the ripening in the film during 45 days
1	2	3
Control 1		
Total	65	85
Sample 1		
Total	240	300
Sample 2		
Total	245	300
Control 1		
Total	57	72
Sample 3		
Total	235	290

Low water vapor permeability of polymer package prevents desiccation of brined cheese, promotes uniform distribution of moisture and table salt all over its surface. Due to this the properties of cheese remain constant over the entire surface.

Conclusions

Ripening of brined cheese in the film contributes to lower moisture loss during storage, which positively effects on the proteolytic processes in brined cheese [11]. The use of plastic film for packaging of brined cheese increases the amount of lactic acid microorganisms at all stages of its ripening in comparison with the cheese with the traditional method of packing.

Using a combined method of packaging has contributed to increase of the content of free amino acids. If the number of free amino acids in a raw cheese "Control 1" increased by 19.8%, then in the brined cheese “Sample 1” under research - by 21.2%, “Sample 2” - by 20.8%. Brined cheese “Control 2” is characterized by an increase of the content of free amino acids by 7.7% when packing in the polymer film. However, in the raw “Sample 3” their number increased by 22.2%. The use of a polymeric film for ripening of soft brine

cheeses under study contributed to the increase of free amino acids by an average of 20.6%, which had a positive effect on their organoleptic properties. Figure 1 graphically presents the results of the research of the content of free amino acids in soft brine cheese depending on the method of packaging.

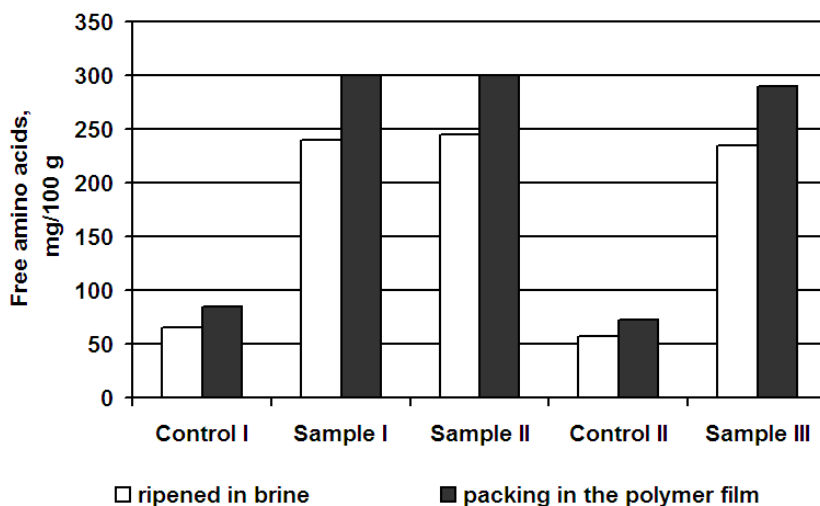


Fig. 1. Effect of packaging on the content of free amino acids of soft brine cheeses ($\tau = 60$ days, $t = 2 \dots 5^\circ \text{C}$)

Thus, the production of soft brined cheese is expedient using the next method of ripening - 14 days in brine, followed packaging in a polymer film under vacuum and storage of 45 days at a temperature of $2 \dots 5^\circ \text{C}$.

Further research will be aimed at a complex estimation of quality of soft brine cheeses.

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Research of high oleic sunflower oil properties under the hydrothermal effect

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Abstract

Keywords:

Sunflower
Oil
High oleic
Hydrothermal
Pastry

Article history:

Received 15.01.2016
Received in revised
form 28.02.2016
Accepted 24.03.2016

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Introduction. The modern life trend causes necessity of formation of the profile of choux pastry products consumer properties according to the principles: available, tasty, useful, comfortable. Studying the properties of the fat recipe component of the choux pastry in the conditions of technological process modeling will allow to control and provide the obtaining of competitive products.

Materials and methods. To evaluate the transformations that occur in the high oleic sunflower oil-water model systems during hydrothermal effect physicochemical research methods, namely the determination of acid (AV), peroxide (PV) and saponification values, were used.

Results. Obtained data confirm the growth of rate of hydrolysis and the accumulation of free fatty acids in the oil-water model systems due to medium's temperature (from 20 to 100° C) and pH increase. AV magnitude is in the range 0,22...0,41 mg KOH/g at pH 4,5, 0,19...0,34 mg KOH/g at pH 6,0, 0,32...0,38 mg KOH/g at pH 8,0. The rapid growth of peroxide values is observed due to the temperature increase from 80...100° C and is 3,90...4,70 mmol 1/2O/kg. The saponification values of model samples depends on the medium reaction and does not change in the temperatures' range from 20 to 100° C. Those oil samples that were subjected to hydrothermal treatment at the alkaline medium (pH = 8,0) are characterized by almost unchanged acylglycerols' composition compared to the untreated oil and SN value of 191,0±06 mg KOH/g. The SN value of treated samples is some less and amounts at pH 4,5 – 186,0±0,5; pH 6 – 184,0±0,4 mg KOH/g. Thus obtained dependences of AV, PV, SV of high oleic sunflower oil on medium reaction of oil-water (1,0:2,5) model system and hydrothermal effect temperature evidence of its sufficient stability. The values of these indicators are within 0,19...0,41 mg KOH/g, 0,95...4,70 mmol 1/2O/kg, 184,0...191,0 mg KOH/g, respectively.

In the oil-water model system at pH=6,0 and hydromodule 1,0:2,5 insignificant accumulation of free fatty acids and primary oxidation products is observed, and AV and PV do not exceed 0,17 mg KOH/g and 1,55 mmol 1/2O/kg respectively at 20° C.

The largest triacylglycerol oxidation rate during the first 20-60 s of hydrothermal treatment is observed for model system with pH 8,0, where PV increases in 2,3 times. PV increases in 1,4 and 1,2 times in systems with pH 4,5 and 6,0.

Conclusions. High oleic sunflower oil using as a source of fat in culinary products technology, particularly in technology of products based on choux pastry was experimentally proved extremely perspective.

Introduction

In the structure of human nutrition an important place is occupied by culinary products, among which the choux pastry products are in high demand [1-2]. Market research has shown that the range of culinary and pastry products with choux pastry at the domestic market is limited, the quality does not meet the requirements of today and their production technology needs an improvement. The modern trend of life dictates necessity of creation of a clearly formed profile of choux pastry products consumer properties, which will meet modern requirements: available, tasty, useful, comfortable. Performing of the aforementioned requirements will provide obtaining of the competitive products with high quality characteristics. It is known, that the realization of technological process of choux pastry products manufacturing is determined mainly by technical and technological properties of the fat component. The undisputed fact is that the final product's quality depends on the behavior of recipe components in the technological stream. The aim of this research was to study high oleic sunflower oil properties under the hydrothermal effect.

Analysis of scientific studies

Theoretical and practical aspects of the high oleic sunflower oil properties were studied by such scientists as K.M. Schaich, Xiaoqing Yang, Robin A. Boyle, S.M. Ghazani, A.G. Marangoni, Olesea Roman, G. Talbot, Bertrand Heyd, Bertrand Broyart, Roberto Castillo, Maria Teresa Rodriguez-Estrada, Claudia Belingheria, Barbara Giussani, Antonio Ferrillo, Elena Vittadinia, . Analyze of existing technologies of choux pastry products determined that the fat component performs the following functions: recipe component, softener, complexing agent, baking powder, and a source of food and nutritional value [3, 8, 12-16].

Fat component of choux pastry is an important complexing agent. It interacts with other recipe components of the dough. By adsorbing at the surface of starch grains and wheat protein micelles fat screens part of hydrophilic groups, preventing their interaction with water and the formation of strong flour paste. Fat increases the plasticity of dough by weakening the connection between protein micelles and also starch polysaccharides of wheat flour. Found that in the process of making dough and baking it the intensive binding of lipids occurs – more than 75% of free lipids, including 90% of glycolipids and phospholipids and 66% of glycerides [4].

The activity of fat in the process of complexation greatly depends on its chemical composition, so different choux pastry fat components take part in forming of the structure of choux pastry and baked semi-finished products in different ways. Fats, that contain the mixture of triglycerides with the following composition: saturated fatty acids – 10...20% and unsaturated – 80...90%, show the greatest activity in interaction with flour proteins. The vegetable oil is characterized by this ratio of fatty acids.

Fat products, which are used in technology of dough products, perform the role of softeners of dough structure and improvers of product quality. Fats, which are included to recipe composition of choux pastry semi-finished products, perform the similar function. Monoglyceroles show plastic characteristic more than diglyceroles. According to the researches of Mikhailov V.S., Chekmariova I.B. and others the melting temperature and physical state of fat during the dough making process largely determine the degree of dough plasticizing.

One of the traditional fat components in the technology of choux pastry semi-finished product cooking is butter. Fatty acid composition of butter mainly comprises saturated fatty acids (63...65%) - palmitic and stearic, less monounsaturated (33...34%) – oleic acid and very small amount of polyunsaturated (1,1...3,6%) – linoleic acid. Deficiency of essential fatty acids and high price for butter urge consumers to search for alternative replacement to other raw material, despite butter's high nutritional value.

In world practice, there is some production experience of floury confectionery products with the oil addition. The use of oil allows to enrich products with unsaturated fatty acids, primarily the essential, and to reduce its cost by excluding butter and margarine products and attracting domestic raw materials [5-7].

S.M. Ghazani, A.G. Marangoni (Healthy Fats and Oils) discussed the main healthy minor components present in fats and oils, as well as their fatty acids composition. The major oils produced in the world were discussed, focusing on beneficial compounds naturally present in these oils. The effects of refining on the removal of desirable and undesirable minor components are also reviewed [8].

Gruner B.C. and Scherbova E.A. investigated the possibility of vegetable oil use instead of butter in the manufacturing of choux pastry semi-finished products. The researchers used sunflower and corn oil for this purpose. The oil pressing off didn't occur during baking the dough pieces. The taste and smell of cooked choux pastry semi-finished products did not change significantly. The specific volume of choux pastry semi-finished products increased by 5-10% compared to the control samples, made using butter (replacement was equivalent 1:1, i.e. excluding the content of lipids in fat products) [9].

Mikhailov V.S. and Borodina T.P. showed the influence of vegetable oil (50 to 100%) amount on the quality of choux pastry semi-finished products [10]. In mentioned work it was revealed that the volume of semi-finished products increases with the increasing of vegetable oil content. However, the authors baked semi-finished products in non-traditional way, i.e. by dough pieces portioning at the pastry sheet, but in the forms, so these data can not be the basis for the development of choux pastry semi-finished products recipe, that uses vegetable oil as a fat component. Seeing during the baking at the pastry sheet, i.e. with the bigger surface of water yielding, the increase of vegetable oil amount in the dough would lead to a sharp decrease in dough's viscosity which would not let to obtain the semi-finished products of satisfactory quality.

Andrews S.L., Harte J. B. (Ingredient Functionality and Dough Characteristics) considered that fat contributes tenderness, or shortness, to pastry. Depending on the type of pastry, the fat content can range from 25% to almost 75% of the dough. Fat tenderizes pastry by waterproofing flour particles. The polar groups in water have an affinity for the polar groups in both the protein and starch. Polar carbonyl groups and the double bonds in unsaturated fatty acid moieties make it possible for fat to unite with polar groups on the surface flour particles. The remaining portions of the fat molecule have no affinity for the flour or water and act as a mechanical barrier, preventing contact of the water and protein in the flour. A fat's ability to interfere with gluten formation is known as its shortening power. Pure fats have more shortening power than do butter or margarine which contain 16% water. Even pure fats, such as lard, hydrogenated shortenings, and oils, exhibit different characteristics in a pastry product. Oil is more dense than lard, which is more dense than shortening. In addition, liquid fats have more spreading power and are able to coat flour more evenly and completely. The higher the ratio of liquid to crystals, the greater the covering power of the fat. [17].

Analytical data showed that the studies are preliminary, it is impossible to give scientifically grounded choux pastry semi-finished products recipe, which provides full or

partial butter replacement with vegetable oil, on their base. However, giving preference to vegetable oil including choux pastry semi-finished products technology will let to get a number of advantages. Therefore, the question of butter replacement with vegetable oil in choux pastry semi-finished products recipe is actual.

All this created the preconditions for searching for oil with the required properties. Researchers of the Plant Production Institute nd. a. V. Ya. Yuryev of NAAS managed to create sunflower hybrid whose oil is characterized by significantly increased oleic acid content and has similar to olive oil properties. The high oleic sunflower oil (HOSO) use will provide high nutritional value and oxidative stability of finished products. It should be noted HOSO's high ability to form complexes because of the peculiarities of the fatty acid composition, which affects the reactivity and the ability to form complexes with starch and protein substances contained in the dough.

Materials and methods

The dynamics of chemical reactions of high oleic sunflower oil under the conditions of hydrothermal process was studied in the oil-water model systems with volume ratio of 1,0:0,5; 1,0:2,5; 1,0:3,0. The composition and the ratio of fat and water (hydromodule) in a model system are chosen on the base of traditional choux pastry recipe, according to which it is 1,0:2,1, and with less and larger part of water in the system.

To estimate changes in oil-water model systems that occur during hydrothermal effects physicochemical research methods were used. Data of acid value (AV) showed the rate of hydrolysis of triacylglycerol in oil. AV was determined according to standard procedure as the number of potassium hydroxide milligrams required to neutralize free fatty acids contained in 1 gram of oil. For what oil sample was dissolved in a neutral mixture of ethanol with diethyl ether to further titration with alcoholic solution of potassium hydroxide in the presence of phenolphthalein.

Determination of primary triacylglycerol oxidation products in oil was carried out with standard iodometric method by peroxide value (PV). PV was determined by the number of active oxygen ($1/2O$) millimoles equivalent to I_2 separated from potassium iodide in glacial acetic acid by peroxides and hydroperoxides contained in 1 kg of oil. The peculiarity of the method was that sample after adding of the aqueous solution of potassium iodide to oil dissolved in a mixture of glacial acetic acid and chloroform was kept for 40-60 s in the darkness. Further formed iodine was titrated with sodium thiosulfate in the presence of starch solution.

In order to detect the possible deepening of hydrolysis with change of acylglycerol composition of oil due to hydrothermal influence an important for identification of fat indicator – saponification value (SV) was studied. SV as the number of potassium hydroxide milligrams required for saponification of acylglycerols and free fatty acids contained in 1 gram of oil was determined according to standard procedure. Samples of oil were treated with a solution of potassium hydroxide in ethanol and heated for 60² s in a boiling water bath, further the sample was titrated with hydrochloric acid solution in the presence of phenolphthalein.

The transformation of triacylglycerols of high oleic sunflower oil during hydrothermal influence was studied at different medium reaction pH (4,5; 6,0; 8,0), temperature (20...100° C) and the duration of the process (5, 10, 20, 40-60 s).

The required medium reaction 4,5; 6,0; 8,0 of model system was provided by the addition to the water of citric acid, salt, baking soda accordingly in a certain quantity to achieve pH.

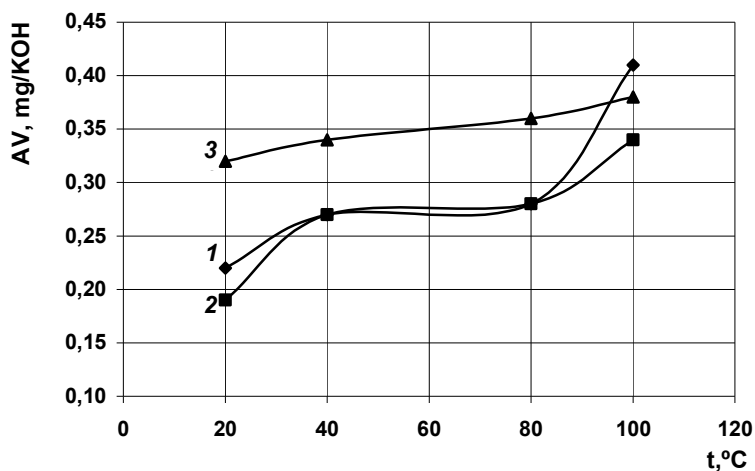
Maximum temperature and duration interval of hydrothermal exposure were chosen according to the traditional technology of choux pastry cooking, based on the dough boiling operation parameters (95...100° C within no more than 10·60 s).

For an objective judgment about the degree of probability of the data obtained the mathematical treatment of the obtained results was made. The reliability of the results obtained was determined with the help of Student's coefficients for the taken statistical significance level of $P = 0.05$ and corresponding $(n-1)$ degrees of freedom.

Results and discussion

Hydrothermal processes belong to multifactorial difficult thermal processes that significantly affect the quality of the finished products. The dynamics of changes in the properties of fats in the technological system under the hydrothermal treatment depends on the temperature, time of treatment, the ratio of fat-containing products and water, i.e. hydromodule, presence of electrolytes, alkalis, acids solutions among the other food components [11-17].

Research results of high oleic sunflower oil physicochemical characteristics dependence on the pH and temperature in oil-water model systems with ratio 1,0:2,5 are presented accordingly in Fig. 1 and 2.

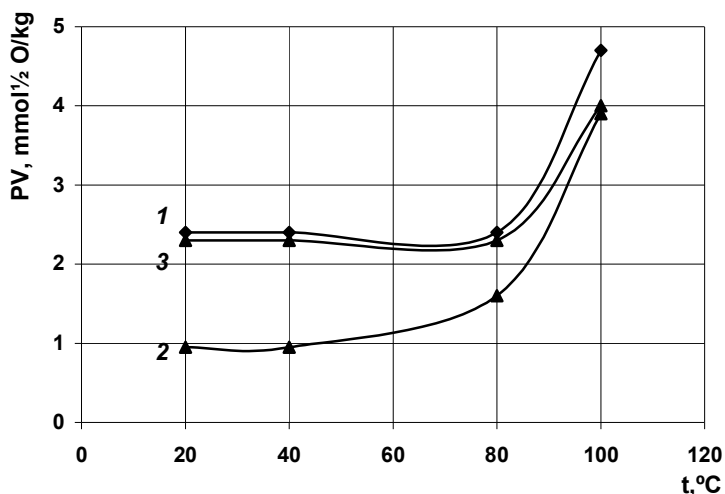


**Fig. 1. Dependence of fat's acid value of oil-water (1,0:2,5) model systems on the temperature of hydrothermal influence and pH:
1 – 4,5; 2 – 6,0; 3 – 8,0**

The experimental data (Fig. 1) show that the acid value of samples increases with the increase of hydrothermal influence temperature from 20 to 100° C in model systems with acidic, slightly acidic and alkaline medium. AV magnitude is in the range 0,22...0,41 mg KOH/g at pH 4,5, 0,19...0,34 mg KOH/g at pH 6,0, 0,32...0,38 mg KOH/g at pH 8,0. Obtained data confirm the growth of rate of hydrolysis and the accumulation of free fatty

acids in model systems due to medium's temperature and pH increase. The greatest growth occurs when hydrothermal process flows in acidic medium (1) at the temperature of 100° C. However, in alkaline medium (3) transformation is accompanied by the accumulation of more free fatty acids for the entire temperature range (20...100 ° C) of hydrothermal influence.

The process of hydroperoxides' accumulation is confirmed by the results of the study of fat peroxide value change dynamics of oil-water model systems with ratio 1,0:2,5 respectively (Fig. 2).



**Fig. 2. Dependence of fat's peroxide value of oil-water (1,0:2,5) model systems on the temperature of hydrothermal influence and pH:
1 – 4,5; 2 – 6,0; 3 – 8,0**

It is clear from Fig. 2 that peroxide value increases for all samples of model systems at the investigated pH range with the temperature increase. The rapid growth of peroxide values is observed due to the temperature increase from 80...100° C and is 3,90...4,70 mmol 1/2O/kg.

The results of dependence determination of fat's saponification number of model systems with oil-water ratio equal 1,0:2,5 respectively on high oleic sunflower oil base on medium reaction and hydrothermal treatment temperature are given in Table 1.

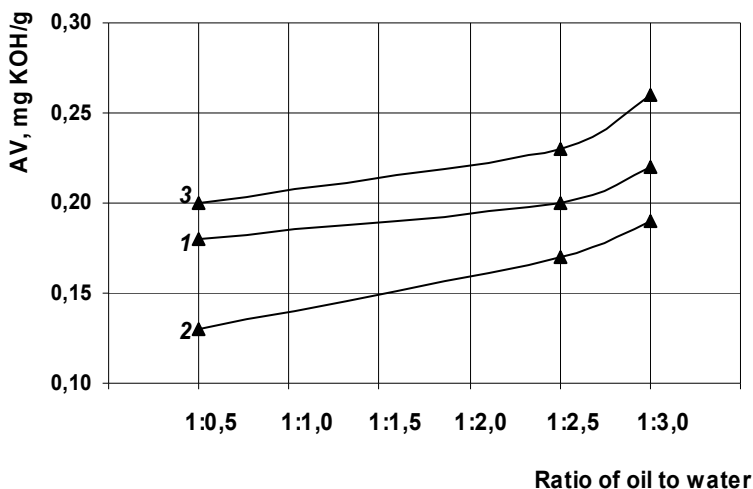
**Table 1
Dynamics of the changes of fat's saponification value of oil-water (1,0:2,5) model systems due to pH and the temperature of hydrothermal influence**

Temperature t, ° C	Saponification value, mg KOH / g		
	pH=4,5	pH=6,0	pH=8,0
20° C	186,0±0,5	184,0±0,5	191,0±0,6
40° C	186,0±0,4	184,0±0,5	191,0±0,5
80° C	186,0±0,5	184,0±0,4	191,0±0,5
100° C	186,0±0,5	184,0±0,4	191,0±0,6

Data from Table 1 show that the saponification value of model samples depends on the medium reaction and does not change in the temperatures' range from 20 to 100° C. Those oil samples that were subjected to hydrothermal treatment at the alkaline medium (pH = 8,0) are characterized by almost unchanged acylglycerols' composition compared to the untreated oil and SV value of 191,0±06 mg KOH/g. The SV value of treated samples is some less and amounts at pH 4,5 – 186,0±0,5; pH 6 – 184,0±0,4 mg KOH/g.

Thus obtained dependences of AV, PV, SV of high oleic sunflower oil on medium reaction of oil-water (1,0:2,5) model system and hydrothermal effect temperature evidence of its sufficient stability. The values of these indicators are within 0,19...0,41 mg KOH/g, 0,95...4,70 mmol 1/2O/kg, 184,0...191,0 mg KOH/g, respectively.

Dependence of model systems fat's AV and PV on hydromodule and pH at the temperature of 20° C is shown in Fig. 3 and 4.



**Fig. 3. Dependence of fat's acid value of oil-water model systems at the temperature of 20° C on hydromodule and pH:
1 – 4,5; 2 – 6,0; 3 – 8,0**

Acid value (Fig. 3) increases in all oil-water model systems with increasing of water share that is the defining factor of oil hydrolysis deepening. Experimentally determined that the biggest AV and accumulation of free fatty acids characterize model system with hydromodule 1,0:3,0 at pH=8, which amounts 0,26 mg KOH/g, and the least – a model system with ratio 1,0:0,5 at pH=6, which amounts 0,13 mg KOH/g.

Growth dynamics of peroxide value in all model systems (Fig. 4) has a similar character. PV for systems with acidic (pH=4,5) and alkaline (pH=8,0) medium reaction almost coincide within the limits of experimental error. Larger PV characterizes oil-water model systems with ratio 1,0:3,0 respectively, and is: 2,15 mmol 1/2O/kg at pH 4,5; 2,31 mmol 1/2O/kg at pH 8,0; 1,91 mmol 1/2O/kg at pH 6,0. The oil-water system with hydromodule 1,0:0,5 respectively has the lowest PV: at pH 4,5 – 1,36 mmol 1/2O/kg; at pH 8,0 – 1,39 mmol 1/2O/kg; at pH 6,0 – 1,24 mmol 1/2O/kg. Peroxide value increase due to increasing the share of water in the system indicates an increase of oxidation rate of oil acylglycerols and accumulation of primary oxidation products, mainly hydroperoxides.

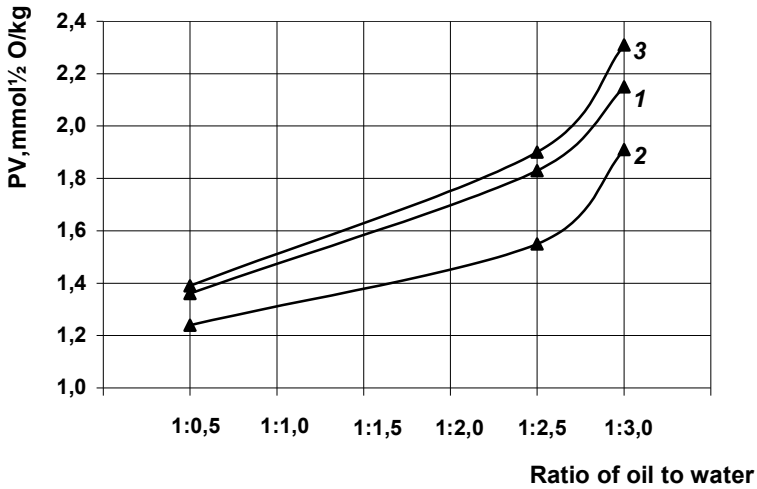


Fig. 4. Dependence of fat's peroxide value of oil-water model systems at the temperature of 20° C on hydromodule and pH:
1 – 4,5; 2 – 6,0; 3 – 8,0

Therefore in oil-water model system at pH=6,0 and hydromodule 1,0:2,5 insignificant accumulation of free fatty acids and primary oxidation products is observed, and AV and PV do not exceed 0,17 mg KOH/g and 1,55 mmol 1/2O/kg respectively at 20° C.

Effect of hydrothermal influence duration at the temperature of 100° C on fat's AV and PV of oil-water model systems with hydromodule 1,0:2,5 at the different medium reaction is illustrated by dependencies, shown in Fig. 5 and 6.

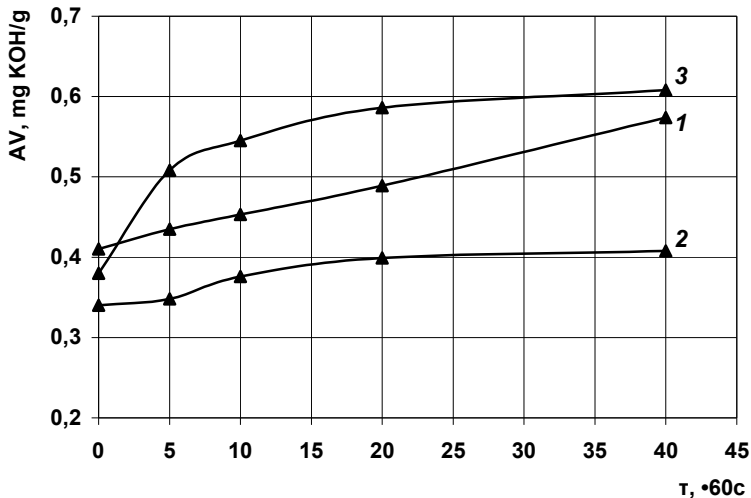
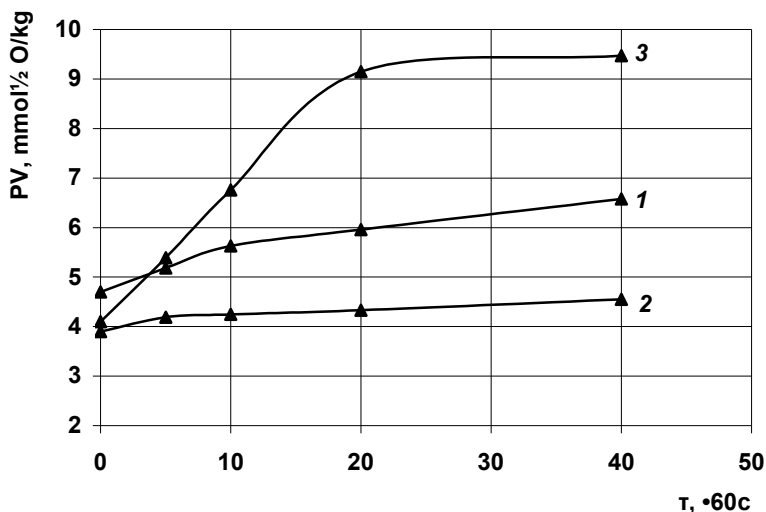


Fig. 5. Dependence of fat's acid value of oil-water (1,0:2,5) model systems on temperature treatment duration (100° C) and pH: 1 – 4,5; 2 – 6,0; 3 – 8,0

Hydrothermal treatment duration increase leads to fastening of oil hydrolysis, what is demonstrated by the AV increase in model systems. Acid value increases during 40·60 s of hydrothermal influence at pH 4,5 in 1,4 times; pH 6,0 in 1,2 times; pH 8,0 in 1,6 times.



**Fig. 6. Dependence of fat's peroxide value of oil-water (1,0:2,5) model systems on temperature treatment duration (100° C) and pH:
1 – 4,5; 2 – 6,0; 3 – 8,0**

The largest triacylglycerol oxidation rate during the first 20·60 s of hydrothermal treatment is observed for model system with pH 8,0, where PV increases in 2,3 times. PV increases in 1,4 and 1,2 times in systems with pH 4,5 and 6,0.

Conclusions

It was determined that chemical transformations of high oleic sunflower oil triacylglycerols, such as hydrolysis and oxidation, are not accelerated so significant during hydrothermal processes in oil-water model systems with different medium reaction (pH 4,5; 6,0; 8,0). Oil reveals sufficient thermal stability and resistance to peroxidation, and maximum AV and PV values do not exceed 0,61 mg KOH/g and 9,50 mmol 1/2O/kg respectively under the conditions of increasing of water proportion in the system (1,0:0,5; 1,0:2,5; 1,0:3,0), raising of the temperature to 100° C and thermal treatment duration to 40·60 s. Rational conditions for hydrothermal process for model systems oil-water were determined, under which the AV and PV do not exceed 0,34 mg KOH/g and 4,10 mmol 1/2O/kg respectively. According to these conditions temperature is 95...100° C, duration is 5·60 s, oil-water hydromodule– 1,0: 2,5. High oleic sunflower oil using as a source of fat in culinary products technology, particularly in technology of products based on choux pastry was experimentally proved extremely perspective.

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Influence of chocolate frosts on their qualities and usage in food industry

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Abstract

Keywords:

Chocolate
Frosts
Viscosity

Article history:

Received 19.01.2016

Received in revised
form 09.03.2016

Accepted 24.03.2016

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Introduction. The aim of this work was to get surface active substances on the basis of fat and vegetable origin for reducing their viscosity in chocolate frosts

Materials and methods. Chocolate frosts composed of cocoa products, whey powder and fruit concentrate. Surface-active substances obtained from waste palm oil and fat, having got by the method of glycerolysis.

Results and discussion. Addition 0.4% of lecithin and mixture MG, DG, TG of fat allow to decrease the viscosity of chocolate frosts to needed characteristics (2500-2900mPa•s), but at the addition of mixture MG, DG, TG of palm oil this result is achieved already at 0.2%.

Addition 0.4% mixture MG, DG, TG of fat allows to reduce the viscosity of chocolate frosts with whey powder till 2690 mPa•s, but adding lecithin or mixture MG, DG, TG of waste palm oil this result is achieved already at 0.3% SAS.

When adding 1% SAS the most reducing of viscosity of chocolate frosts with fruit concentrate is achieved with the usage of mixture MG, DG, TG of fat (3400mPa•s). With the usage of lecithin the result was 3900mPa•s, but with the mixture MG, DG, TG of waste palm oil was 3600mPa•s.

Surface-active substances reduce viscosity but don't influence the taste and feeling of melting in the mouth researched items of frosts and whey powder and fruit concentrate enriched taste and aroma of chocolate frosts.

Addition of mixture MG, DG, TG of waste palm oil and fat made it worse the stability of chocolate frosts as to the turning gray, especially frost with whey powder.

Lecithin and surface-active substances obtained on the bases of waste palm oil and fat have thinning ability more than 0.8%.

Conclusions. Surface active substances from waste palm oil and fat, received by this method are preferably to be used in the recipes of chocolate frosts to reduce the viscosity qualities of the prepared product.

Introduction

Nowadays there is a great assortment of confectionery, which is under the process of chocolate frosting. But there is a problem of working out new recipes in order to enrich and to improve the taste of finished articles.

By using a chocolate frosts the terms of storage of confection can be improved also as a physical configuration with a hiding of some defects. The committing of that way provide to cost saving in production.

To characterize the chocolate frosts they make definitions which show the rheological properties, to be exact, its viscosity. If viscosity is rather high (more than 12 Pa•s), the lay of frosts is rather thick, but if it is low (less than 2 Pa•s), then frosts become watery and the layer becomes too thin. One of the main organoleptic evaluations of the chocolate frosts is the feeling of fusibility just in the mouth, as due to it one can feel their real taste. As for taste and aroma of the frosts it must be like original chocolate without any strange scent or taste. Its color may be from light brown to dark brown but in harden state it may be turn grey inside and outside.

Consistence must be at temperature 16°C – hard, but at temperature above +40°C – fluid.

Viscosity reducing for all recipes of chocolate frosts is necessary, because it leads to increasing the currents of frosts and in the long run, they will cover equally the confectionery.

This process will make frosting better. These investigations are thought to develop the recipes of chocolate frosts aiming to reduce their basic prices, but to keep their biological value, the figures of quality and characteristics, which respond to modern standards, having been used in food industry.

Hence of it, with a purpose of partial substitution of cocoa butter, the fruit juice is added together with whey and surface active substances (SAS). The necessary deterioration of viscosity is reached with (SAS in mixture) 0.4 -0.6%.

Materials and methods

To get SAS they used waste palm oil (AN=1.9mgKOH/g, IN=50.1gI₂/100g, melting point=25°C, congelation point=35°C) and fat (AN=2.0mgKOH/g, IN=33.0gI₂/100g, melting point=23°C, congelation point=28°C).

To analyze the mixtures mono-, de-, threeglicerides of fat acids were used having got by the method of glycerolize [1], the indices of quality were given in table 1.

Table 1
Physicochemical properties SAS

Index	Mixture MG, DG, TG:	
	Waste palm oil	Fat
acidic number, mgKOH/g	7.19	8.87
peroxide number, mmol ½O/kg	50.3	49.46
iodine number, gI ₂ /100g	53.71	43.15
melting point, °C	29	33
congelation point, °C	20	26

The investigation was made for influence of all the mixtures (MG, DG, TG) exhausted palm oil and fat as to the viscosity of chocolate frosts without any whey powder (2%) and fruit concentrate (3.25%). For these investigation all the frosts were chosen in table 2.

Table 2

Recipes of chocolate frosts

Chocolate frosts	Cocoa butter, %	Coca powder, %	Sugar powder, %	Whey powder, %	Fruit concentrate, %
Without addition	18.42	42.48	39.1	–	–
With whey powder	18.42	42.48	37.1	2	–
With Fruit concentrate	18.5	42.5	35.75	–	3.25

As a standard SAS was choosed a lecithin which is used in the food industry.

Results and discussion

The main criteria of chocolate frosts are their rheological properties which are under the influence of SAS. The dependence of viscosity of chocolate frosts from content of SAS is on the figure 1.

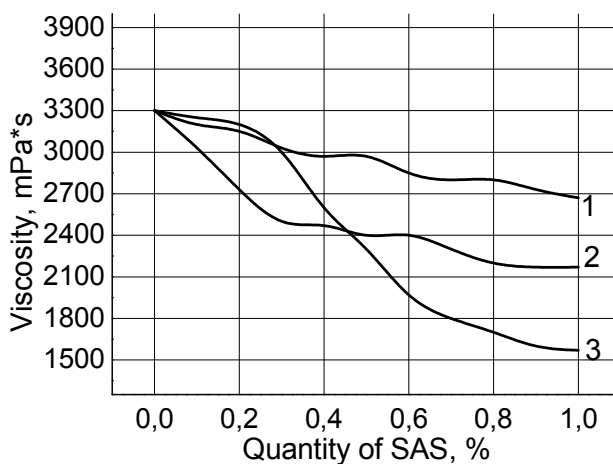


Figure 1. Dependence of viscosity of chocolate frosts on the content of SAS:
 1 - frost with lecithin, 2 - frost with MG, DG, TG of waste palm oil,
 3 - frost with MG, DG, TG of fat

It can be observable that addition 0.4% of lecithin and mixture MG, DG, TG of fat allow to decrease the viscosity of chocolate frosts to needed characteristics (2500-

2900mPa*s), but at the addition of mixture MG, DG, TG of palm oil this result is achieved already at 0.2%.

The results of influence of SAS at viscosity of chocolate frosts with whey powder are at figure 2.

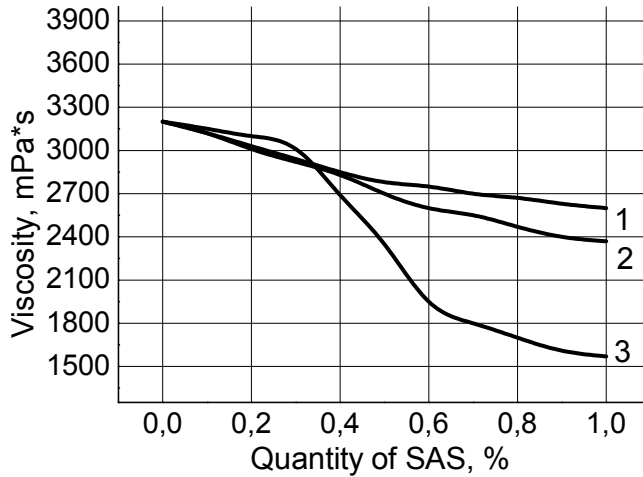


Figure 2. Dependence of viscosity of chocolate frosts with whey powder on the content of SAS:
1 -frost with lecithin, 2 - frost with MG, DG, TG of waste palm oil,
3 - frost with MG, DG, TG of fat

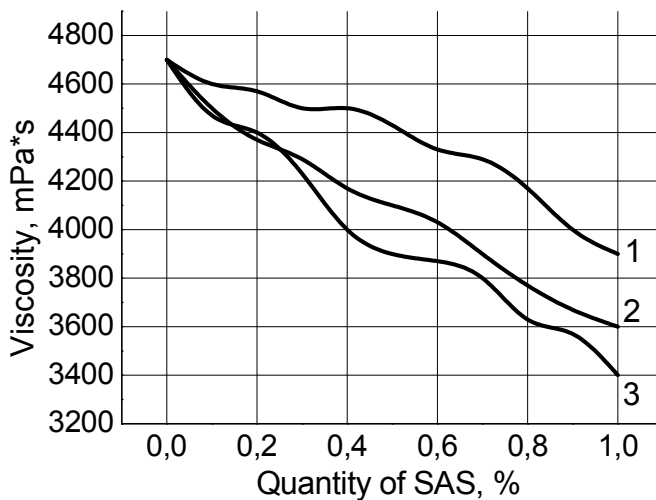


Figure 3. Dependence of viscosity of chocolate frosts with fruit concentrate on the content of SAS:
1 -frost with lecithin, 2 - frost with MG, DG, TG of waste palm oil, 3 - frost with MG, DG, TG of fat

It is seen that with the addition of whey powder, viscosity reduces to 3200 mPa•s in the comparison with viscosity of frosts without any additions which 3300 mPa•s. Addition 0.4% mixture MG, DG, TG of fat allows to reduce the viscosity of chocolate frosts till 2690 mPa•s, but adding lecithin or mixture MG, DG, TG of waste palm oil this result is achieved already at 0.3% SAS. Thus, the most effective SAS for chocolate frosts with whey powder is mixture MG, DG, TG of waste palm oil.

The results of the investigations of dependence viscosity chocolate frosts with fruit concentrate and various kinds of SAS are given at figure 3.

It is seen that viscosity of the frosts with fruit concentrate has increased till 4700mPa•s, in comparison with viscosity of frosts without any additions. When adding 1% SAS the most reducing of viscosity is achieved with the usage of mixture MG, DG, TG of fat (3400mPa•s). With the usage of lecithin the result was 3900mPa•s, but with the mixture MG, DG, TG of waste palm oil was 3600mPa•s. Thus, the most effective SAS for chocolate frosts with fruit concentrate is mixture MG, DG, TG of fat.

The organoleptic characterists are shown in table 3.

Table 3

The research of organoleptic data of chocolate frosts

Chocolate frosts	Fusibility	Testle	Scent
Without addition: - without SAS; - with lecithin; - with the mixture MG, DG, TG of waste palm oil; - with the mixture MG, DG, TG of fat.	average good average good	Bitterly-sweet	Typical chocolate frosts without any foreign scent
With whey powder: - without SAS; - with lecithin; - with the mixture MG, DG, TG of waste palm oil; - with the mixture MG, DG, TG of fat.	average good average good	Sweet with milk	Typical chocolate frosts without any foreign scent
With fruit concentrate: - without SAS; - with lecithin; - with the mixture MG, DG, TG of waste palm oil; - with the mixture MG, DG, TG of fat.	average good good good	Sour-sweet	Typical chocolate frosts without any foreign scent

From given results at table 3 it is evident, that SAS reduce viscosity but don't influence the taste and feeling of melting in the mouth researched items of frosts and whey powder and fruit concentrate enriched taste and aroma of chocolate frosts.

The appearance of frosty confectionery is also important. But turning grey is available, but not desirable. That's why it is necessary to investigate the stability to turn grey the made chocolate frosts. The results of these investigations are at table 4.

Table 4

Stability investigation of the chocolate frosts to turn grey

Chocolate frosts	Stability investigation of the chocolate frosts to turn grey
without addition: - without SAS; - with lecithin; - with the mixture MG, DG, TG of waste palm oil; - with the mixture MG, DG, TG of fat.	process are not done process are not done minor change minor change
with whey powder: - without SAS; - with lecithin; - with the mixture MG, DG, TG of waste palm oil; - with the mixture MG, DG, TG of fat.	process are not done minor change minor change minor change
with fruit concentrate: - without SAS; - with lecithin; - with the mixture MG, DG, TG of waste palm oil; - with the mixture MG, DG, TG of fat.	process are not done process are not done minor change process are not done

From given results at table 5, is clear that the addition of mixture MG, DG, TG of waste palm oil and fat made it worse the stability of chocolate frosts as to the turning gray, especially frost with whey powder. It's also established that taste, scent and melting point of chocolate frosts in the mouth did not change after their saving.

In table 5 there are data as to the appearance and quantity SAS, effective to each example, and to the main results of the received products.

The effect of SAS under the production of chocolate frosts is characterized also by its thinning ability. It's taken by the quantity of cocoa butter, necessary to reduce viscosity at 0.4% SAS [2].

The compared characteristics thinning ability of used SAS are shown in table 6.

Table 5

The components and quality results of chocolate frosts

Index	Types of SAS	Content of SAS, %	Viscosity, mPa·s	Temperature of freezing, °C	Temperature of fusibility, °C
Model of frosts					
without addition	mixture MG, DG, TG of waste palm oil	0.2	2530	23	29
with whey powder	lecithin	0.3	2940	22	33
with fruit concentrate	mixture MG, DG, TG of fat	1	3400	21	35

Table 6

Thinning ability of received SAS

Types of SAS	Thinning ability, %
lecithin	0.80
mixture MG, DG, TG of waste palm oil	0.85
mixture MG, DG, TG of fat	0.85

It's clear from these figures that lecithin and SAS obtained on the bases of waste palm oil and fat have thinning ability more than 0.8%.

The results can be related to the peculiarity of the molecular structure of palm oil and fat. Also special is the impact of the ratio of MG, DG and TG in the SAS.

It's evident that they are effective SAS for reducing viscosity of chocolate frosts and give the possibility to economize nearly 1% of cocoa butter in the ready to use product.

Conclusions

The results of those investigations are shown that with addition of whey powder and fruit concentrate the gustatory sense of made frosts is increasing. It is enriched with the vitamins, micro-elements necessary for a man. Besides, the physical data is also improving. For example, frosts with whey powder the melting point is more than 33°C, and viscosity of frosts reduce to 2940mPa·s. With the addition of fruit concentrate congelation point are reduce to 21°C. At the sometime viscosity of the frost significantly increase, that why it's necessary to add SAS to reduce these characteristics until 3400mPa·s. But the addition of the fruit concentrate provide to eliminate the turn gray and makes tasty qualities better and to use the lecithin, mixture MG, DG, TG of waste palm oil and fat is also good for ready to use product.

As a SAS are need to use lecithin, mixture MG, DG, TG of waste palm oil or fat in depending of components of final product.

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Antibacterial biodegradable films for foods

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Abstract

Keywords:

Biodegradable
Films
Food
Nanocomposite
Titanium dioxide

Article history:

Received 25.01.2016
Received in revised
form 16.03.2016
Accepted 24.03.2016

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Introduction. Titanium dioxide (TiO₂) has antibacterial properties, but It has not been used for antibacterial films. It becomes necessary to study the direction of its antibacterial properties in packing as well as the influence of its packaging on the quality of food.

Materials and methods. Films based on polyvinyl alcohol (PVA) with various concentration of TiO₂ were studied. Elongation, tensile strength, glass transition and melting temperature of the films from polymer materials were defined. Microbiological studies to establish inhibitory action of nanodispersed TiO₂ powder on some pathogens were carried out. Antibacterial properties of TiO₂ films were studied by agar diffusion test.

Results and discussion. Addition of TiO₂ nanodispersed powder to the film decreases its flexibility by 20-45%, depending on the added amount, however improves its tensile strength. Moreover, adding of TiO₂ powder by more than 1% increases the tensile strength of plastic film (46,7 MPa). The melting point (transition temperature to the viscous flow state) (175 °C) and a glass transition temperature (78 °C) were the best for the sample of 1% filler. The least deformation was observed for the same sample, indicating that most interacted system is associated with the maximum number of hydrogen bonds. The best approach was to handle TiO₂ with a UV radiation, since this way the minimum concentration (2,5 %) of TiO₂ suspension was used. While not conducting UV treatment, it was necessary to use 10 - 20% of TiO₂ mixture. TiO₂ solutions do not suppress the action of fungi and yeast. TiO₂ deposited on the film inhibits the growth of bacteria (*E. coli* IEM-1, *B. subtilis* BT-2), growth retardation was observed.

Conclusions. Introduction of biodegradable packaging with antibacterial properties is necessary and requires designing or revising the existing regulatory documents. Low prices of proposed nanoparticle additives do not significantly affect the cost of packaging, which is extremely important in the difficult economic conditions.

Introduction

Active nanocomposites are polymer composites containing nanoparticles with antimicrobial and antioxidant properties [1]. The priority types of nanomaterials to improve the barrier properties are nanoscale titanium dioxide (titanium oxide IV), zinc oxide, colloidal silver, nanoclay, wax and paraffin [2-4].

Titanium dioxide (TiO₂) – the white dye is widely used as an additive in foods, personal care products, cosmetics, and pharmaceutical shells. Other uses of TiO₂ include antimicrobial coatings, photocatalysts for air purification and water-resistant coating of welding electrodes and molds, paints and others. Office for Global Operations as part of the Food and Drug Administration (FDA) has approved TiO₂ as a food additive (dye), provided that the consumption does not exceed 1% of the weight of the product. Also, approved by the FDA shell component food products E 171 – refer to the EU Food Additives (TiO₂) as white color [5].

As of today, Ukraine has an official permission to use some nanostructured food additives in food production, synthetic amorphous silica (silicon dioxide) and titanium dioxide [6, 7].

TiO₂ is used as a material for not only photocatalytic sterilization in pharmaceutical and food industries, but addressing environmental problems through photocatalytic decomposition of harmful organic contaminants in water and air as well as destroying a wide range of harmful bacteria and viruses [8]. The combination of titanium oxide treatment with ultraviolet (UV) radiation was proposed as one of the best technology disinfectants because, unlike the others, it does not form dangerous compounds [9].

Films with TiO₂ are used as a self-sterilization surface due to their property to form reactive oxygen species (ROS) when irradiated with ultraviolet light. These ROS attack bacteria and kill them. It is a new way to enhance the bactericidal activity of TiO₂-films [10]. Bionanocomposites were developed by casting/evaporation of wheat gluten, cellulose nanocrystals and TiO₂ nanoparticles. The antimicrobial activity of the coated papers, against *Saccharomyces cerevisiae*, Gram-negative bacteria *Escherichia coli* and Gram-positive bacteria *Staphylococcus aureus*, was investigated and expressed in terms of reduction % of surviving number (CFU) of the tested organisms. More than 98,5% reduction in CFU was observed against the organisms compared to TiO₂-free coated paper [11]. A total of six different coating suspensions were prepared by mixing TiO₂ nanoparticles with three different types of binders (shellac, polyurethane, and polyacrylic) at a 1:4 to 1:16 to binder weight ratio. Bactericidal activity of these TiO₂ coatings against *Escherichia coli* was determined at three different UV-A light intensities (0,25, 0,5 and 0,75 mW/cm²) for 3 h. The results of this study showed promise in developing durable TiO₂ coatings with strong photocatalytic bactericidal property on food contact surfaces using appropriate binding agents to help ensure safe food processing environment [12].

Analytical analysis of the literature showed that TiO₂ has antibacterial properties, however studies using food and packing have not been conducted. Promising direction is to study the antimicrobial properties of TiO₂ as a food packaging component and its impact on their quality.

Materials and methods

Packaging was made from polyvinyl alcohol (PVA) with molar mass of 10350 g / mol, apple pectin produced by «Cargill» with a degree of etherification of 66,9 % glycerol,

and nanodispersed powder of TiO₂ (20-50 nm particle size, surface area – 50 ± 5 m²/g, patent PCT/US2007/025504, 06.09.2011). PVA was dissolved in hot distilled water. While stirring on a magnetic stirrer pectin sample was added, followed by glycerol and the full volume of TiO₂ suspension. To prevent the formation of air bubbles in the ready-mades samples solutions were degassed using a vacuum. Films were then poured onto Teflon surface and dried at 25 °C.

The physical, mechanical and thermo-mechanical properties, namely elongation (ε, %), tensile strength (σ, MPa), glass transition temperature (T_g, °C) and transition temperature to the viscous flow state (T_f, °C) were studied. Determination of elongation and strength of the film on the break were carried on the tensile machine F-1000 without previous conditioning of the samples. Thermo-mechanical studies were performed by penetration in the uniaxial constant stress mode (σ = 0,5 MPa) on the UYP-70 M machine. Gradual heating of the samples was carried out at a speed of 2,5 °C / min. in the temperature range of 0 to 350 °C. Thermo-physical characteristics established by differential scanning calorimetry (DSC) on the TA Instruments DSC Q 2000 instrument with a heating rate of 20 °C / min. in the temperature range of 20 to 250 °C. DSC data determined the glass transition temperature (T_g) and the glass transition interval (ΔT), the heat capacity jump (ΔC), as well as the melting temperature (T_f) and the melting enthalpy of fusion (ΔH). To obtain leveling background designs and achieve equilibrium state, samples were subjects to heating twice. Glass transition temperature (T_g) was defined as a midrange temperature of glass transition interval.

Inhibitory effect of nanodispersed TiO₂ powder on some bacteria (*E. coli* IEM-1, *B. subtilis* BT-2, *C. albicans* D-6, *A. niger* P-3) was defined at the surface. In a sterile conditions, 1 ml suspension of the specific species of microorganisms, selected and mixed by pipette 0,1 ml and made the center of the frozen environment (for determining bacteria – MPA, fungi and yeast – Saburo) was placed in a test tube with a solution of a certain concentration of TiO₂. Sterile spatula was rubbed across the surface of the medium in a cup, placed in the thermostat at 30 °C (bacteria – 72 h., fungi – 5-7 days). Number of microorganisms in 1 ml was determined by multiplying the number of colonies on the sample dilution and dividing by the number of introduced seeds. In the absence of the cell growth inhibition concluded on the growth of certain microorganisms [13, 14]. Definition of antagonistic properties of TiO₂ film was performed by agar diffusion test. In sterile conditions discs cut from films were imposed in the culture medium (for determining bacteria – MPA, fungi and yeast – Saburo) and put in the thermostat at 30 °C for 24 hours to measure the diameter of the delayed the growth of the microorganisms [14].

Results and discussion

The composition of the films, their physical, mechanical and thermal characteristics are shown in Fig. 1-4 and Table 1.

Results in Table 1 indicate that the introduction of additional particulate filler has led to a deterioration of elasticity, but for samples 1 and 2 % of TiO₂ values of elongation and tensile strength were quite acceptable. Thus for these samples the tensile strength exceed the strength of plastic film (46,7 MPa), Fig. 1.

Table 1

The composition and quality of nanoparticle TiO₂ films

№	Ingredients, %				ε, %	σ, MPa	T _g , °C	T _f , °C
	PVA	Pectin	Glycerol	TiO ₂				
1	60,0	10	30	0,0	492	68,8	54	171
2	59,5	10	30	0,5	268	31,4	62	182
3	59,0	10	30	1,0	384	48,3	78	175
4	58,0	10	30	2,0	380	52,7	57	179
5	56,0	10	30	4,0	292	38,8	54	175

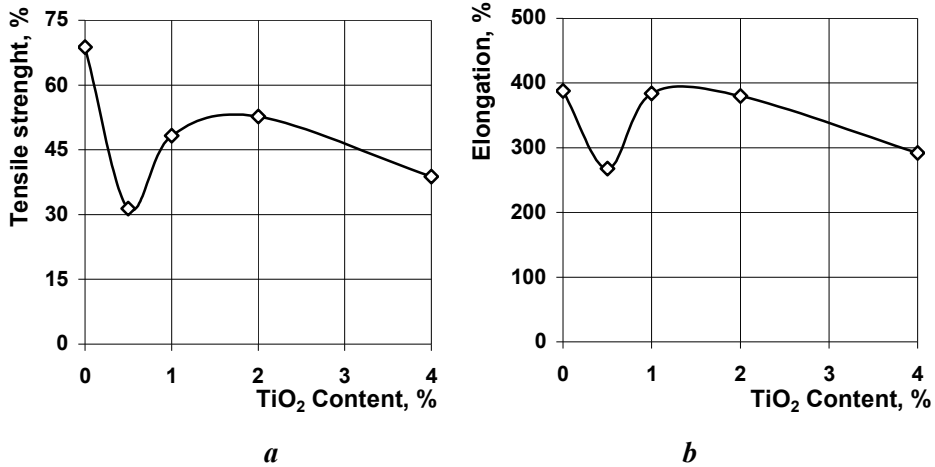


Fig. 1.

a – tensile strength (σ, MPa)
 b – elongation (ε, %) of films with different content of TiO₂

Defining characteristics of polymeric materials such as T_g and T_f is important considering the conditions of exploitation and processing. The variation of T_g of polymer systems with the addition of fillers describes the interaction between the components. The increase in T_g is usually associated with the decreased mobility of the polymer chains and the flexibility of the system.

Fig. 2 shows thermo-mechanical curves of samples with different contents of TiO₂. For the samples with 0,5 and 1 % TiO₂ (curves 2, 3) observed a decrease in strain and T_g increase, indicating the increase in the number of hydrogen bonds between the PVA, pectin and TiO₂, as well as the even distribution of filler without the formation of agglomerates. Instead, with the content of the filler 2 and 4 % increase in distortion and T_g reduction was likely caused by aggregation of the inorganic component.

The discrepancy between the values of T_g established by the methods of thermo-mechanical analysis and DSC (Table 2), can be explained by the peculiarities of obtaining samples and experimental procedure. Films formed from aqueous solutions, physically hold a certain amount of solvent, which has a plasticizing effect. DSC method involves cyclic tests, where the same sample is heated twice in the cell with its thermal characteristics being recorded. Accordingly, the sample loses moisture during the first cycle of heating.

Therefore, thermal characteristics recorded from the second cycles tend to be shifted towards the higher temperatures. T_g temperature in the first heating cycle was identified for outgoing compounds only – PVA and pectin, and the results support each other: T_g is 48 and 47 °C for pectin and 43 and 42 °C for the plant, according to the thermo-mechanical analysis and DSC, respectively. The value of the melting temperature (T_m) according to DSC and the transition temperature (T_f) to the viscous state according to thermo-mechanical analysis are also close.

Results of the study of the physical and chemical properties of the obtained films of various compositions are shown in Fig. 3 (the first heating run), 4 (the second heating run) and in Table 2. An analysis of the Table 2 shows that the enthalpy of fusion (ΔH) of all composites is lower than the original PVA, indicating a decrease in the degree of crystallinity of the system.

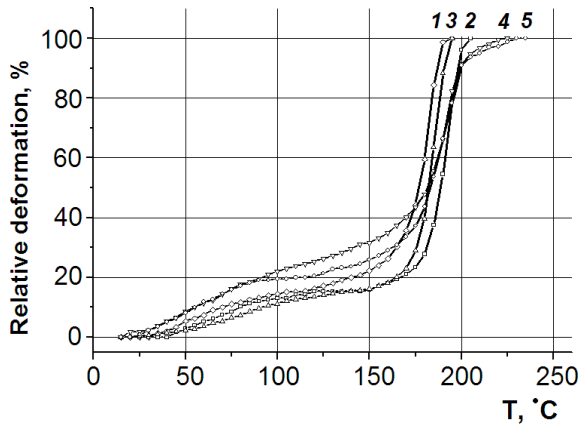


Fig. 2. Thermo-mechanical curves of films:

1 – PVA - Pectin - Glycerol –60:10:30 (control); (2 - 5) – with the adding of TiO_2 : 2 – 0,5 %; 3 – 1 %; 4 – 2 %; 5 – 4 %

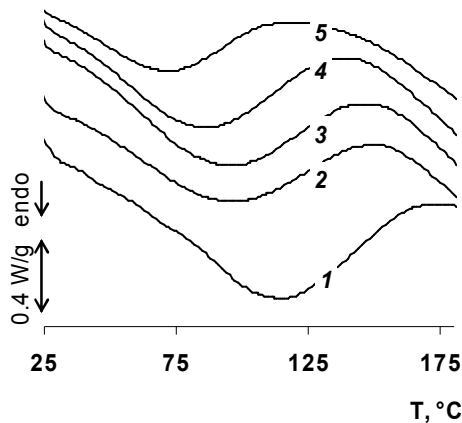


Fig. 3. DSC thermogram of the first heating run of films:

1 – PVA; 2 – PVA- Glycerol 70:30; 3 – PVA - Pectin - Glycerol – 60:10:30 (control); 4, 5 – with the addition of TiO_2 : 4 – 1 %; 5 – 2 %

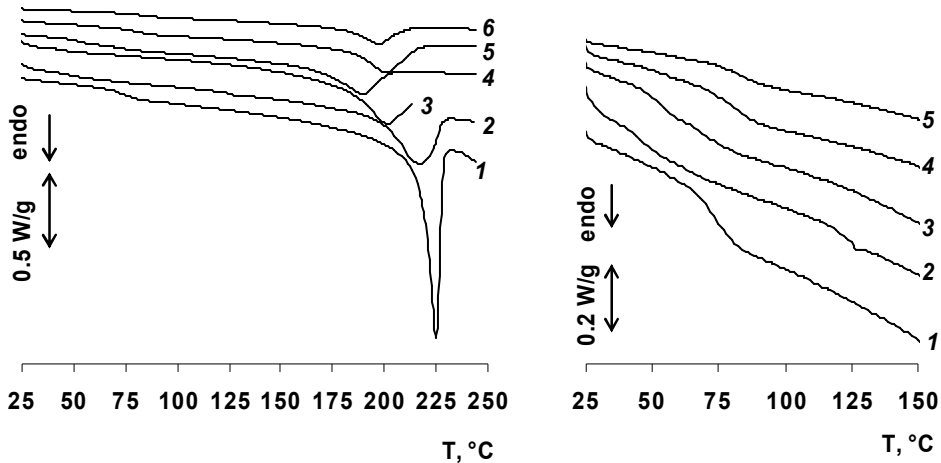


Fig. 4. DSC thermogram of the second heating run of films:
 1 – PVA; 2 – PVA- Glycerol 70:30; 3 – PVA- Pectin - Glycerol – 60:10:30 (control); 4, 5 – with the adding of TiO₂: 4 – 1 %; 5 – 2 %

Results of the research of influence of films on glass transition temperature (T_g) and melting temperature (T_m) are shown in Table 2.

Table 2

The value of T_g and T_m of films with various compositions

Composite	T _g			T _m			ΔH, J/g
	start	mid.	end	start	max.	end	
PVA	69,1	74,8	80,4	221,3	225,5	237,5	49,5
PVA - Glycerol 70:30	41,9	46,5	51,0	199,6	216,8	236,3	30,0
PVA - Pectin - Glycerol 60:10:30							
without additives	66,0	72,0	78,0	168,5	188,9	219,3	18,1
with 1 % TiO ₂	72,5	80,7	88,9	182,7	201,0	225,7	16,4
with 2 % TiO ₂	75,0	83,0	91,1	180,3	197,0	226,0	10,5

Determination of the inhibitory effects of nanodispersed TiO₂ powder to test bacterial cultures was determined at the surface. As objects of study used: *E. coli* IEM-1, *B. subtilis* BT-2, *C. albicans* D-6, *A. niger* P-3. In sterile conditions, 1 cm³ suspension of the specific species of microorganisms, thoroughly mixed and selected with a 0,1 cm³ (drop) pipette and made the center of frozen environment was placed in a test tube with a solution of an appropriate concentration of TiO₂ (2,5 %; 5 %; 10 %; 20 %). Sterile spatula was gently rubbed across the surface of the medium in a cup. Cups were then placed in the thermostat at 37° C for bacteria and 28 °C for the fungi and incubated for 72 hours for bacteria and 5-7 days for fungi [13, 14]. Research of inhibitory effect of TiO₂ solution to culture bacteria and fungi are also conducted in daylight and in the thermostat with UV radiation.

The research results appear on Table 3-5.

Table 3

Inhibitory effect of different concentrations of TiO₂ solution in daylight

Concentration of TiO ₂ , %	Total bacterial count, CFU/g			
	<i>E. coli</i> IEM-1	<i>B. subtilis</i> БТ-2	<i>C. albicans</i> Д-6	<i>A. niger</i> P-3
2,5	8,9×10 ⁴	9,8×10 ⁴	7,4×10 ⁶	7,2×10 ⁶
5	6,7×10 ⁴	6,9×10 ⁴	8,5×10 ⁵	3,8×10 ⁴
10	+	+	4,2×10 ⁶	2,2×10 ⁴
20	+	+	5,3×10 ⁶	3,4×10 ⁴

Table 4

Inhibitory effect of different concentrations of TiO₂ solution in UV radiation

Concentration of TiO ₂ , %	Total bacterial count, CFU/g			
	<i>E. coli</i> IEM-1	<i>B. subtilis</i> БТ-2	<i>C. albicans</i> Д-6	<i>A. niger</i> P-3
2,5	+	+	4,4×10 ⁶	1,2×10 ⁵
5	+	+	5×10 ⁶	1×10 ⁵
10	+	+	4,2×10 ⁶	5,2×10 ⁴
20	+	+	5,3×10 ⁶	1,2×10 ⁵

Table 5

Inhibitory effect of different concentrations of TiO₂ solution in the thermostat

Concentration of TiO ₂ , %	Total bacterial count, CFU/g				C
	<i>E. coli</i> IEM-1	<i>B. subtilis</i> БТ-2	<i>C. albicans</i> Д-6	<i>A. niger</i> P-3	
2,5	8,7×10 ⁴	9,7×10 ⁴	7,4×10 ⁶	2×10 ⁵	1,6×10 ⁶
5	4,9×10 ⁴	5,5×10 ⁴	3,2×10 ⁶	1,6×10 ⁵	5,7×10 ⁶
10	+	+	2,5×10 ⁶	1,6×10 ⁵	1,8×10 ⁷
20	+	+	2,7×10 ⁶	1,1×10 ⁴	2,3×10 ⁵

Note. «+» – completely overwhelming; «C» – the initial concentration of microorganisms without TiO₂.

Solutions of different concentrations of TiO₂ in different conditions (daylight, thermostat 30°C, UV radiation) did not exhibit inhibitory effect on *C. albicans* D-6 and *A. niger* P-3, since as compared to their initial concentrations (1,8×10⁷, 2,3×10⁵) significant differences in values were not observed (Table 3-5). A small difference in values may be due to possible sampling of TiO₂, where the content differed.

Best option is handling of TiO₂ with UV radiation, because the minimum concentration (2,5%) of TiO₂ suspension can be used this way. When UV treatment is not possible, it is necessary to use either 10 or 20 % of TiO₂ mixture.

In addition, a study to determine the antimicrobial properties of TiO₂ films by agar diffusion test was conducted [14]. The research results are presented in Table 6.

Table 6

Antimicrobial properties of films with TiO₂

Test culture	Zone delays, mm			
	Control sample ₁	Control sample ₂	Sample ₁	Sample ₂
<i>E. coli</i> IEM-1	0,00	0,00	9	15
<i>B. subtilis</i> БТ-2	0,00	4	6	7
<i>C. albicans</i> Д-6	0,00	0,00	0,00	0,00
<i>A. niger</i> P-3	0,00	0,00	0,00	0,00

Note. Control sample₁, Control sample₂ – PVA-Glycerol - Pectin – 60:10:30
Sample₁, Sample₂ – with the addition to a control sample of 1 and 2 % TiO₂

Evaluating the results (Table 6), it can be concluded that the TiO₂ caused in the film, actually inhibits the growth of bacteria (*E. coli* IEM-1, *B. subtilis* BT-2), because there are areas of stunted growth. The livelihood of fungi and yeast TiO₂ was not affected.

Conclusions

It is appropriate to introduce packaging with antibacterial properties that would require designing or revising the existing regulatory documents. Proposed nanoparticle additives are inexpensive and do not significantly affect the cost of production, which is extremely important in difficult economic conditions.

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Persistence and survival of some food borne pathogens in neutralized unripe grape products

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Abstract

Keywords:

Grape
Neutralization
Antimicrobial
Survival
Foodborne
Pathogen

Article history:

Received 30.01.2016
Received in revised
form 12.03.2016
Accepted 24.03.2016

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Introduction. In this study, the persistence and survival of some food borne pathogens (*E. coli*, *L. monocytogenes*, *S. Typhimurium* and *S. aureus*) in neutralized unripe grape products (verjuice and sour grape sauce) which are particularly rich in antioxidants and organic acids were evaluated.

Materials and methods. The survival patterns of these pathogens in un-neutralized unripe grape products were determined previously. The test pathogens were inoculated in neutralized unripe grape products at two different inoculum doses (2 and 6 log CFU/mL) and all the samples were kept at room temperature (approximately 25°C) for 0, 5, 15 and 30 minutes after inoculation with pathogens, separately.

Results and discussion. The presence of initial microflora is important for food quality and safety. It was mentioned that the unripe grape products had no competitive microflora that could be affect the survival patterns of inoculated pathogens. The initial cell number of *E. coli*, *L. monocytogenes*, *S. Typhimurium* and *S. aureus* were counted as to 2.50, 2.38, 2.52, and 2.21 log CFU/mL for low inoculation dose and 6.00, 6.49, 6.45, and 6.57 log CFU/mL for high inoculation dose, respectively. No viable cells were detected in negative controls. The decreasing numbers of tested pathogens were significant at low inoculation doses after 30 minutes ($p < 0.05$), while there was no significant difference at high inoculum doses in the same treatment time ($p > 0.05$). The unripe grape products have self-protection systems and they could be assumed as ‘microbiologically safe products’ when they were contaminated with pathogens at low levels, and it was associated with the phenolic content they have. However, food borne pathogens, at high contamination levels could survive in unripe grape products in case where the acidic environment was neutralized.

Conclusions. The inhibitory activity of unripe grape products generally based on phenolic compounds and organic acid contents, and the organic acids and phenolic compounds inhibit the pathogens in a synergistic way.

Introduction

Natural products are chemical compounds or substance obtained from a living organism or presented in nature which has pharmacological or biological activity [1]. Living organisms produce secondary metabolites that can be used as antimicrobial agent against to food borne pathogens [2, 3]. These secondary metabolites can be extracted from different origins as microorganisms, animals and plants [4]. These natural antimicrobials could yield better results than synthetic/chemical preservatives that especially have adverse effects on human health [5, 6]. The synthetic preservatives could be the reason of hives, itching, asthma, allergies, lung irritation, tumors, antibiotic resistance in human as well as mutagenic and carcinogenic effects on metabolism [7, 8, 9]. Therefore, natural antimicrobials, especially plants are been given more attention in the consumers due to their properties of ensuring the food safety by preventing the survival of pathogenic microorganisms [5, 6, 10].

In recent years there are lots of studies have been indicating the antimicrobial activity of plant based products. These studies have been mentioned that there are over 1340 plants with antimicrobials activities which are defined, and over 30.000 compounds have been isolated from plants that shown antimicrobial properties [10, 11]. The plants themselves (leaves, stems, buds, flowers, fruits, juices, seeds, bulbs and rhizomes) or the compounds held from plants (extracts, essential oils) have been used as plant based antimicrobials to ensure the food safety in these studies [10, 12, 13, 14]. The plant based products such as fruit and vegetables or their juices, herbs and spices, essential oils, extracts, and fermented products such as vinegar have been used to extend shelf life of foods with ensuring the food safety and quality [15, 16, 17].

The antimicrobial effect of fruit and vegetable juices as grape, pomegranate, noni, garlic, lemon, unripe papaya, raspberry, black currant, gooseberry, jostaberry, radish, leek, and onion were stated against *Bacillus* spp., *Bacteroides* spp., *Citrobacter* spp., *Clostridium* spp., *Micrococcus* spp., *Mycoplasma* spp., *Neisseria* spp., *Salmonella* spp., *Serratia* spp., *Shigella* spp., *Staplococcus* spp., *Aspergillus niger*, *Candida albicans*, *Corynebacterium xerosis*, *Cronobacter sakazakii*, *Enterococcus faecalis*, *Escherichia coli*, *Escherichia coli* O157:H7, *Klebsiella pneumoniae*, *Kluyveromyces marxianus*, *Listeria monocytogenes*, *Mucor indicus*, *Pseudomonas aeruginosa*, *Penicillium citrinum*, *Rhizopus oryzae*, *Rhodotorula rubra*, and *Trichoderma reesei* in various studies [18 - 31]. In addition, extensive researches have investigated the antimicrobial mechanism of these products against food borne pathogens. The mechanism is mainly attributed to organic acids, as well as, phenolic compounds [21, 31 - 33]. The several organic acids like benzoic, capric, fumaric, lactic, malic, tartaric, and acetic are found in foods [17]. The organic acids inhibit the microorganism by targeting their cell wall, membrane, metabolic enzymes, protein synthesis, and genetic material [34]. The phenolic compounds such cinnamic acid, caffeic acid, gallic acid play an important role in antimicrobial activity of fruit and vegetable juices [35, 36]. The antimicrobial mechanisms of phenolic compounds associate with damaging the cytoplasmic membrane, collapsing the PMF (proton motive force), disruption of electron flow and depletion of active transport. In a result of these factors, the cell components become coagulated [5, 37].

Unripe grape products such verjuice and sour grape sauce are acidic juices with sour flavor [38]. Nikfardjam (2008) has studied with 7 verjuice samples obtained from different origins. The values of titratable acidity, sugar content, and total phenolic matter ranged between 19.6-39.6 g/L, 0.1-95.1 g/L, and 200-1330 mg/L, respectively [39]. In another research, the mean of pH, titratable acidity, and total phenolic matter were determined as 2.94, 2.74%, and 6900 mg/L [40]. The physicochemical and phytochemical properties, as well as antioxidant capacity of unripe grape products which are also used as material in this study have investigated previously. In that previous study, the mean values of pH, titratable acidity (%) and total phenol content

were 2.42, 3.84%, 473.97 mg/L, 1.036 μmol Trolox/mL (FRAP), and 0.421 μmol Trolox/mL (TEAC) [41]. The impact of unripe grape products on some food borne pathogens were also investigated and the minimum inhibitory concentrations (MICs) of five verjuice and five sour grape sauce samples on *Bacillus cereus*, *Escherichia coli*, *Listeria monocytogenes*, *Salmonella* Typhimurium, and *Staphylococcus aureus* were determined [42]. Analyze was performed for both un-neutralized and neutralized products to detect whether the inhibitory effect depends on the organic acid content of the samples or not. According to the results, the antimicrobial effect of these products is mostly related to their organic acid content. Nonetheless, the inhibitory effect is also dependent on their phenolic compounds.

As mentioned before, there are many studies on fruit or vegetable juice about their antimicrobial properties. The researches are mostly carried out on their original pH values to mention their inhibition mechanisms. However, there are limited studies on the antimicrobial effect of the neutralized juices on pathogenic microorganisms. Thus, this study was aimed to detect inhibition effect of neutralized unripe grape products on food borne pathogen due to their rich phenolic properties.

Materials and methods

Unripe grape products. Two kind of unripe grape products such as verjuice and unripe grape sauce were used in this study. Five verjuices and five unripe grape sauces were tested and the product details were represented in Table 1. The products were obtained by different production methods as traditional, laboratory scale, and industrial. The laboratory method was based on traditional one. The verjuice is produced by squeezing the berries, holding fresh juice by discarding the pomace. The unripe grape sauce has a heat treatment step after extraction of the mash. Some ingredients such as salt and/or olive oil could be added optionally before bottling in the production of the both products. The flow diagram of the production was shown in the Figure 1. The all of the samples were kept at -80°C until analyses and they were held at $+4^{\circ}\text{C}$ during a night for thawing before analyzing. The samples were aseptically neutralized to pH 7.00 (± 0.20) with sterile NaOH solutions (106462, Merck, U.S.A.).

Test cultures. Four different microorganisms were used for this research work as target pathogens and they were obtained from Gaziosmanpaşa University, Faculty of Engineering and Natural Science, Department of Food Engineering, Food Microbiology Laboratory. The target pathogens were *Escherichia coli* (ATCC 25922), *Listeria monocytogenes* (ATCC 19115), *Salmonella* Typhimurium (ATCC 14028) and *Staphylococcus aureus* (ATCC 25923). Stock cultures were kept at -80°C in Brain Heart Infusion Broth (BHIB, Lab M, LAB049, UK) with 20% glycerol (1.04092.2500, Merck, Germany). The stock cultures were regenerated twice in BHIB at $37\pm 2^{\circ}\text{C}$ for 18-24 hours.

Preparation test cultures for inoculation. High and low inoculum doses were performed in this research. The final inoculum dose in unripe grape products was approximately 6 log CFU for high dose, and 2 log CFU/mL for low dose for each pathogen. In the pre-treatment, the growth curves of target pathogens were detected for inoculating the pathogens while they were in the exponential (log) phases of the growth. According to these results, the regenerated cultures were incubated in BHI Broth at $37\pm 2^{\circ}\text{C}$ for 1-8 hours. The bacterial cultures were diluted with 0.1% sterile peptone water (PW, Merck, 1.07224, Germany) accordingly to achieve 7 log CFU/mL for high dose and 3 log CFU/mL for low dose. The initial inoculum doses of pathogens were counted onto Brain Hearth Infusion Agar (BHIA, Lab M, LAB 048, UK) by spread plate method for detecting inoculation dose exactly. BHIA plates were incubated at 37°C for 24-48 hours.

Table 1

Details about the unripe grape product samples tested

Samples	Region	Varieties	Ingredients	Production Method	
VERJUICE	1	İzmir city	Yediveren	Verjuice, Salt (0.4%)	Laboratory production
	2	Antalya city	Margaz	Verjuice, Salt (0.4%), Olive oil (3%)	Traditional production
	3	Antalya city	Müşküle	Verjuice, Salt (0.4%), Olive oil (3%)	Traditional production
	4	Ankara city	Kalecik Karası	Verjuice	Laboratory production
	5	Tokat city	Narince	Verjuice, Olive oil (3%)	Laboratory production
SOUR GRAPE SAUCE	6	Aydın city	Yediveren	Verjuice, Salt (0.4%), Olive oil (3%)	Traditional production
	7	Aydın city	Yediveren	Verjuice, Salt (0.4%), Olive oil (3%)	Traditional production
	8	Tokat city	American Rootstock	Verjuice	Laboratory production
	9	Industrial product 1	Cabernet Sauvignon, Shiraz, Merlot	Verjuice	Industrial manufacturing
	10	Industrial product 2	Cabernet Sauvignon, Shiraz, Merlot	Verjuice, Salt (0.5%)	Industrial manufacturing

Inoculation of neutralized unripe grape samples. Firstly, nine milliliter of neutralized unripe grape samples were poured into sterile test tubes and then one milliliter of the test culture (at 7 log CFU/mL for high dose and at 3 log CFU/mL for low dose) was placed into the same tube, aseptically. The test tubes were homogenized (Velp, F202A0173, Europe) at 3000 rpm for 5 seconds. So, high (6.0 log CFU/mL) and low (2.0 log CFU/mL) inoculum doses have been achieved finally. For detecting the survival pattern of the test microorganisms, all treated samples were kept at room temperature (approximately 25°C) during 0, 5, 15 and 30 minutes. The viable cell numbers were established by surface plating method on BHIA right after serial dilutions were prepared with 0.1% PW. Then, the BHIA plates were incubated at 37°C for 24-48 hours. The pathogen cultures were assessed as positive control and the products without pathogens as negative control.

Statistical analyses. All experiments were carried out with two replicates and two parallels. The significant difference between the means was established by ANOVA variance analysis and Duncan tests. The results were analyzed with the SPSS statistical package program (SPSS 17.0 for Windows Evaluation Version, 17.0.3); SPSS Inc., Chicago, USA). Independent-Samples T-Test was used for comparing the means of verjuice and unripe grape sauce.

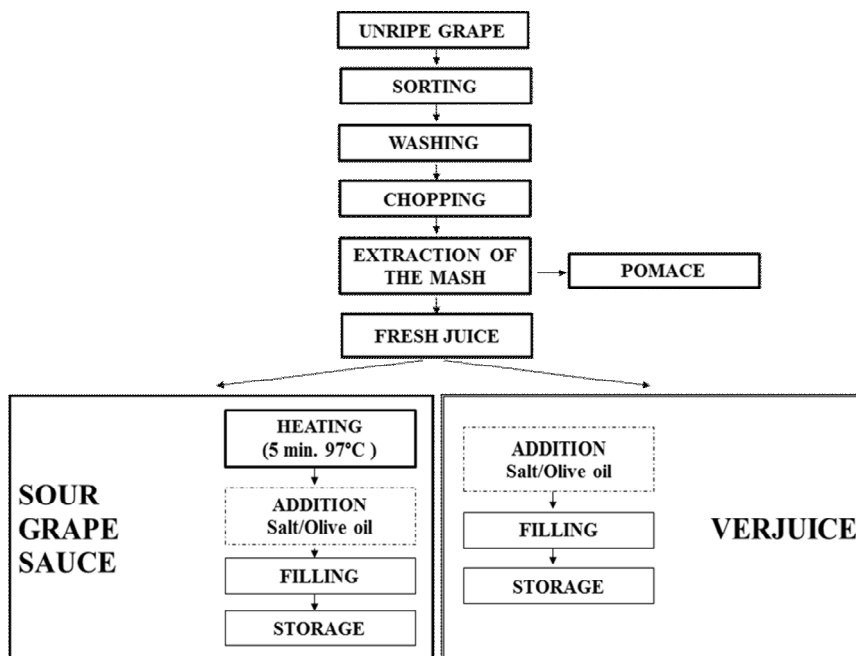


Fig. 1. Flow diagram for laboratory production of verjuice and sour grape sauce based on the traditional methods

Results and discussions

The microbiological properties of unripe grape products which were used in this research had been examined in the previous study. The unripe grape products were tested for enumeration analyses of total mesophilic aerobic bacteria, total psychophilic aerobic bacteria, yeasts and molds, lactic acid bacteria, *Bacillus cereus*, *Clostridium perfringens*, *Staphylococcus aureus*, total coliform bacteria and total fecal coliform bacteria. In the same time, the products were also tested for the presence of *Escherichia coli*, *Escherichia coli* O157:H7, *Salmonella* spp. and *Listeria monocytogenes*. The presence of initial microflora is important for food quality and safety. It was mentioned that the unripe grape products had no competitive microflora that could be affect the survival patterns of inoculated pathogens [42].

The initial cell number of *E. coli*, *L. monocytogenes*, *S. Typhimurium* and *S. aureus* were counted as to 2.50, 2.38, 2.52, and 2.21 log CFU/mL for low inoculation dose and 6.00, 6.49, 6.45, and 6.57 log CFU/mL for high inoculation dose, respectively. No viable cells were detected in negative controls. There was significant differences between the positive control and treatment times in low inoculum doses ($p < 0.05$) while there was no significant difference in high doses ($p > 0.05$). These results possibly depend on increasing pathogens numbers while the antimicrobial ingredient amount is stable. Because of the effective component is constant, the increasing numbers of inoculated cells could not be inhibited effectively (tab. 2-5).

The neutralized products were produced a slight reduction on the number of *E. coli* in low dose ($p < 0.05$), and this reduction was continued throughout the treatment period (tab. 2). The effect of samples 1, 2, and 4 were increased by treatment time. The differences between the

mean values of verjuices and sour grape sauces were not significant ($p > 0.05$). However, in high inoculum doses the neutralized grape products had no inhibitory activity against the *E. coli* during the application times.

Results in Table 3 showed that, *S. Typhimurium* were inhibited significantly ($p < 0.05$) at the beginning of treatment (0. min) by all the samples except samples 1 and 10 at low doses. The samples 1 and 10 also had inhibitory activity by increasing application time ($p > 0.05$). On the other side, the neutralized products had no inhibitory activity against *S. Typhimurium* even after the 30 minutes of treatment time at high doses ($p > 0.05$). The mean values of the verjuice and sour grape sauce were not significantly different ($p > 0.05$).

The count results of *S. aureus* which were inoculated to neutralized products at low and high doses were shown in Table 4. The inhibitory activity of neutralized products on *S. aureus* at low doses were significant ($p < 0.05$) when compared with the high doses ($p > 0.05$).

The inhibitory activity of neutralized products against *L. monocytogenes* was indicated in Table 5. Some of the samples (1, 3, 5, 7, and 8) had inhibitory activity on *L. monocytogenes* at the beginning of the treatment in low doses. However, the sample 5 only had significant differences from the positive control during the application time ($p < 0.05$). The inhibitory effects of verjuices and sour grape sauces were significant only at initial application time ($p < 0.05$). At high doses, samples 1, 2, 8, and 9 were shown inhibitory activity at the beginning, and after 5 minutes of treatment ($p < 0.05$), but the inhibition disappeared by increasing treatment time. The viable cell numbers were not significant compared to positive control after 30 minutes ($p > 0.05$). Also, the inhibitory effects of verjuices and sour grape sauces were not significant ($p > 0.05$).

The survival and growth patterns of *E. coli* O157:H7, *S. Typhimurium* and *L. monocytogenes* in neutralized black carrot juice (pH 7.00) were investigated during incubation period at 4°C and 37°C for 7 days. The initial counts of *E. coli* O157:H7, *S. Typhimurium* and *L. monocytogenes* were 6.25, 6.37, and 6.21 log CFU/mL at 37°C and 6.20, 6.24, and 6.16 log CFU/mL at 4°C, respectively. All the pathogens tested were counted as less than 1 log CFU/mL in neutralized black carrot juice samples stored at 37°C for 7 days. However, all the pathogens could survive in the samples stored at 4°C up to 7 days and the viable cell numbers were counted as 5.30, 4.13, and 3.12 log CFU/mL for *E. coli* O157:H7, *S. Typhimurium* and *L. monocytogenes* at the end of 7th day, respectively [43]. The survival and growth patterns of *S. Typhimurium* and *L. monocytogenes* were observed in neutralized sour orange juice during 4°C and 37°C for 7 days. *S. Typhimurium* and *L. monocytogenes* were separately inoculated in neutralized sour orange juice and the initial test cultures were counted as 6.26 and 6.11 log CFU/mL for both storage temperatures. The survivor numbers of *S. Typhimurium* and *L. monocytogenes* was not significant after 1 and 3 hours of application and was found as 5.29 and 5.76 log CFU/mL after 7 days at 4°C. However, it was detected that *S. Typhimurium* and *L. monocytogenes* could survive - even grown - in neutralized juice sample during 1 and 2 days incubation at 37°C. Conclusively, the numbers of pathogens was decreased to undetectable level after 7 days [44]. Survival of *S. Typhimurium* and *E. coli* O157:H7 in neutralized black mulberry juice was studied by Karabiyikli et al. (2012) [4]. The juice samples were inoculated with test pathogens (6 log CFU/mL) separately and were incubated at 4°C and 37°C for 7 days. The viable population of pathogens was increased up to day 2, and was not detected in the end of the treatment at 37°C. However, population of both pathogens in neutralized black mulberry juice samples was decreased slowly over 7 days. The researchers were investigated survival pattern of *L. monocytogenes* in neutralized black mulberry juice in another study under the same conditions [46]. The juices inhibited approximately 1.5 log unit cells at 37°C after 1 day incubation, and only approximately 1 log reduction was observed at 4°C after 7 days.

Table 2
Inhibitory effect of neutralized unripe grape products on *E. coli* (log CFU/mL)

SAMPLES ¹		Low Inoculation Dose				High Inoculation Dose			
		0. min	5. min	15. min	30. min	0. min	5. min	15 min	30 min
VERJUICE	1	2.18 (±0.02) Ba ²	1.98 (±0.10) Ca	1.90 (±0.00) Cc	1.90 (±0.03) Cbcd	5.86 (±0.10) Aa	5.81 (±0.06) Ab	5.96 (±0.08) Aabc	6.12 (±0.34) Aa
	2	2.13 (±0.04) Ba	2.11 (±0.10) Ba	1.78 (±0.07) Cd	1.84 (±0.04) Ccd	5.82 (±0.11) Aa	5.89 (±0.11) Aab	5.88 (±0.00) Ac	5.95 (±0.50) Aa
	3	2.09 (±0.19) Ba	2.15 (±0.09) Ba	2.20 (±0.02) Ba	2.15 (±0.09) Ba	5.90 (±0.04) Aa	5.86 (±0.16) Aab	5.99 (±0.03) Aabc	6.19 (±0.34) Aa
	4	2.10 (±0.12) Ba	1.99 (±0.02) Ba	1.75 (±0.01) Cd	1.80 (±0.00) Cd	5.91 (±0.25) Aa	5.95 (±0.03) Aab	5.88 (±0.12) Ac	6.21 (±0.07) Aa
	5	1.97 (±0.12) Ba	2.19 (±0.12) Ba	2.10 (±0.01) Bb	2.05 (±0.10) Babc	5.94 (±0.00) Aa	5.88 (±0.04) Aab	5.93 (±0.04) Abc	5.95 (±0.64) Aa
	Mean	2.09 (±0.11) x ³	2.03 (±0.18) x	1.97 (±0.17) x	1.94 (±0.14) x	5.88 (±0.10) x	5.85 (±0.11) x	5.92 (±0.07) x	6.35 (±0.32) x
UNRIPE GRAPE SAUCE	6	2.18 (±0.05) Ba	2.03 (±0.21) Ba	2.10 (±0.05) Bb	2.04 (±0.08) Babc	5.88 (±0.08) Aa	5.91 (±0.08) Aab	5.98 (±0.02) Aabc	6.15 (±0.50) Aa
	7	2.07 (±0.16) Ba	1.94 (±0.09) Ba	1.98 (±0.01) Bc	2.03 (±0.03) Babc	5.90 (±0.19) Aa	5.93 (±0.22) Aab	6.15 (±0.04) Aa	6.29 (±0.50) Aa
	8	2.16 (±0.08) Ba	2.08 (±0.14) Ba	2.10 (±0.00) Bb	2.07 (±0.05) Bab	5.94 (±0.37) Aa	5.89 (±0.22) Aab	6.06 (±0.02) Aabc	6.11 (±0.45) Aa
	9	2.02 (±0.04) Ba	1.97 (±0.18) Ba	1.91 (±0.04) Bc	2.02 (±0.18) Babc	5.98 (±0.07) Aa	6.00 (±0.04) Aab	5.98 (±0.11) Aabc	6.08 (±0.85) Aa
	10	2.15 (±0.12) Ba	2.11 (±0.00) Ba	2.14 (±0.01) Bab	2.15 (±0.07) Ba	5.99 (±0.02) Aa	6.07 (±0.09) Aa	6.09 (±0.14) Aab	6.22 (±1.24) Aa
	Mean	2.11 (±0.10) x	2.02 (±0.12) x	1.98 (±0.20) x	2.06 (±0.08) x	5.93 (±0.15) x	5.96 (±0.13) x	6.65 (±0.09) y	6.42 (±0.60) x
Positive Control		2.50 (±0.07) Aa				6.00 (±0.21) Aa			

¹All the samples were tested as negative control before inoculation, and initial microflora could not be detected (<1.00 log CFU/mL)

²n=4, (± standard deviation), different lowercase letters indicate differences between rows and different capital letters indicate differences between columns (p<0.05).

³n=20, mean values of the groups, x and y letters indicate differences between rows (p<0.05).

Table 3
Inhibitory effect of neutralized unripe grape products on *S. Typhimurium* (log CFU/mL)

SAMPLES ¹		Low Inoculum Dose				High Inoculum Dose			
		0 min	5 min	15 min	30 min	0 min	5 min	15 min	30 min
VERJUICE	1	2.13 (±0.10) Aba ²	2.11 (±0.15) ABabcd	2.05 (±0.15) Babc	2.03 (±0.10) Bbc	5.38 (±0.80) Abc	5.82 (±0.10) Aabc	6.36 (±0.45) Aa	6.15 (±0.44) Aa
	2	2.09 (±0.14) Ba	1.81 (±0.07) Ccd	1.86 (±0.06) BCbcd	1.92 (±0.12) BCcde	5.91 (±0.47) Aabc	5.95 (±0.35) Ac	6.19 (±0.33) Aa	6.37 (±0.28) Aa
	3	2.08 (±0.18) Ba	2.17 (±0.05) ABab	2.15 (±0.04) Aba	2.16 (±0.06) ABb	5.75 (±0.80) Aabc	5.54 (±0.76) Aabc	6.60 (±0.48) Aa	6.60 (±0.77) Aa
	4	2.07 (±0.13) Ba	1.80 (±0.03) BCd	1.71 (±0.18) Cd	1.83 (±0.10) BCde	6.16 (±0.30) Aab	5.75 (±0.54) Aabc	6.21 (±0.57) Aa	6.51 (±0.70) Aa
	5	2.12 (±0.09) Aa	2.33 (±0.31)Aabc	2.10 (±0.10) Aab	2.07 (±0.04) Abc	6.42 (±0.06) Aa	6.55 (±0.08) Aab	6.48 (±0.74)A	6.56 (±0.78) Aa
	Mean	2.10 (±0.10) x ³	2.00 (±0.20) x	1.97 (±0.19) x	2.00 (±0.13) x	5.92 [†] (±0.56) x	5.72 (±0.63) x	6.62 (±0.52) x	6.85 (±0.55) x
UNRIPE GRAPE SAUCE	6	2.05 (±0.03) Ba	1.99 (±0.18) Bbcd	2.02 (±0.15) Babc	1.99 (±0.08) Bbcd	6.50 (±0.19) Aa	6.48 (±0.23) Aab	6.35 (±0.94) Aa	6.39 (±0.88) Aa
	7	2.18 (±0.08) Ba	2.09 (±0.06)Babc	2.12 (±0.06) Ba	2.11 (±0.00) Bbc	6.18 (±0.10) Aab	6.35 (±0.19) Aab	5.71 (±0.62) Aa	6.10 (±0.31) Aa
	8	2.16 (±0.06) Ba	2.00 (±0.06) Bbcd	1.80 (±0.00) Ccd	1.74 (±0.13) Ce	5.13 (±0.32) Cc	5.48 (±0.04) BCbc	6.36 (±0.50) Aba	6.51 (±0.72) Aa
	9	2.06 (±0.16) Ba	2.34 (±0.06) Aa	2.26 (±0.04) Aba	2.37 (±0.05) Aa	6.45 (±0.10) Aa	6.13 (±0.85) Aa	6.33 (±0.7) Aa	6.43 (±0.9) Aa
	10	2.28 (±0.06) ABa	2.14 (±0.06) Cabc	2.09 (±0.08) Cab	2.16 (±0.01) BCb	6.52 (±0.11) Aa	6.27 (±0.74) Aab	6.24 (±0.04) Aa	6.38 (±0.86) Aa
	Mean	2.14 (±0.10) x	2.10 (±0.15) x	2.05 (±0.17) x	2.07 (±0.22) x	6.18 (±0.59) x	6.26 (±0.59) x	6.43 (±0.68) x	6.84 (±0.69) x
Positive Control	2.38 (±0.03) Aa				6.49 (±0.25)A Ba				

¹All the samples were tested as negative control before inoculation, and initial microflora could not be detected (<1.00 log CFU/mL)

²n=4, (± standard deviation), different lowercase letters indicate differences between rows and different capital letters indicate differences between columns (p<0.05).

³n=20, mean values of the groups, x and y letters indicate differences between rows (p<0.05).

Table 4
Inhibitory effect of neutralized unripe grape products on *St. aureus* (log CFU/mL)

SAMPLES ¹		Low Inoculum Dose				High Inoculum Dose			
		0 min	5 min	15 min	30 min	0 min	5 min	15 min	30 min
VERJUICE	1	1.92 (±0.04) Bc	1.92 (±0.04) Bbc	1.81 (±0.04) Cabc	1.77 (±0.00) Cb	5.87 (±0.69) Aa	5.81 (±0.90) Aa	5.79 (±0.48) Aa	5.65 (±0.33) Aa
	2	1.47 (±0.00) Bf	1.47 (±0.00) Bd	1.45 (±0.21) Bc	1.38 (±0.12) Bc	5.20 (±0.14) Ba	6.03 (±0.52) Aba	5.85 (±0.66) Aba	5.71 (±0.27) Aba
	3	1.65 (±0.07) Be	1.69 (±0.12) Bcd	1.73 (±0.05) Bbc	1.65 (±0.07) Bb	6.03 (±0.57) Aa	5.99 (±0.42) Aa	6.15 (±0.69) Aa	5.75 (±0.42) Aa
	4	1.77 (±0.10) Bd	1.53 (±0.08) BCd	1.45 (±0.21) BCc	1.38 (±0.12) Cc	5.78 (±0.85) Aa	5.70 (±0.77) Aa	5.78 (±0.54) Aa	5.69 (±0.60) Aa
	5	2.33 (±0.04) Aa	2.36 (±0.07) Aa	1.73 (±0.05) Bbc	1.80 (±0.14) Bb	6.20 (±0.28) Aa	6.30 (±0.32) Aa	6.01 (±0.61) Aa	5.87 (±0.59) Aa
	Mean	1.83 (±0.30) x ³	1.78 (±0.32) x	1.63 (±0.19) x	1.60 (±0.20) x	5.81 (±0.55) x	5.96 (±0.51) x	5.91 (±0.47) x	5.73 (±0.35) x
UNRIPE GRAPE SAUCE	6	2.37 (±0.01) Ba	2.21 (±0.01) Cab	2.19 (±0.01) Ca	2.17 (±0.00) Ca	5.70 (±1.13) Aa	6.01 (±0.76) Aa	5.95 (±0.50) Aa	6.07 (±0.55) Aa
	7	2.38 (±0.04) ABa	2.17 (±0.14) Bab	2.17 (±0.08) Ba	2.14 (±0.15) Ba	6.43 (±0.12) Aa	6.14 (±0.52) Aa	6.02 (±0.35) Aa	5.81 (±0.41) Aa
	8	1.92 (±0.03) ABc	1.71 (±0.34) Bcd	1.66 (±0.42) Bc	1.38 (±0.12) Bc	5.40 (±1.05) Aa	5.70 (±0.59) Aa	5.60 (±0.50) Aa	5.67 (±0.68) Aa
	9	2.17 (±0.04) Bb	2.20 (±0.07) Bab	2.13 (±0.02) Bab	2.14 (±0.04) Ba	6.24 (±0.51) Aa	6.24 (±0.56) Aa	6.26 (±0.65) Aa	6.10 (±0.78) Aa
	10	2.30 (±0.03) Ba	2.22 (±0.10) Bab	2.21 (±0.01) Ba	2.17 (±0.14) Ba	6.45 (±0.00) Aa	6.26 (±0.35) Aa	6.32 (±0.50) Aa	6.05 (±0.42) Aa
	Mean	2.21 (±0.16) y	2.10 (±0.24) y	2.06 (±0.28) y	2.00 (±0.33) y	6.04 (±0.70) x	6.06 (±0.47) x	6.02 (±0.46) x	5.94 (±0.47) x
Positive Control	2.52 (±0.05) Aa				6.45 (±0.07) Aa				

¹All the samples were tested as negative control before inoculation, and initial microflora could not be detected (<1.00 log CFU/mL)

²n=4, (± standard deviation), different lowercase letters indicate differences between rows and different capital letters indicate differences between columns (p<0.05).

³n=20, mean values of the groups, x and y letters indicate differences between rows (p<0.05).

Table 5

Inhibitory effect of neutralized unripe grape products on *L. monocytogenes* (log CFU/mL)

SAMPLES ¹		Low Inoculum Dose				High Inoculum Dose			
		0 min	5 min	15 min	30 min	0 min	5 min	15 min	30 min
VERJUICE	1	1.66 (±0.37) BCab	1.22 (±0.20) Cb	1.91 (±0.06) Aba	1.92 (±0.10) Aba	5.22 (±0.20) Cb	5.68 (±0.08) Dab	6.42 (±0.08) Aa	6.65 (±0.27) Aa
	2	1.61 (±0.29) ABab	1.72 (±0.12) ABab	2.05 (±0.01) Aa	1.26 (±0.49) Bb	5.80 (±0.54) Aab	6.27 (±0.33) Aab	6.02 (±0.99) Aa	6.60 (±1.17) Aa
	3	1.67 (±0.05) Bab	2.07 (±0.08) Aa	2.07 (±0.06) Aa	2.00 (±0.15) Aa	5.64 (±0.23) Cab	6.67 (±0.07) Aa	6.51 (±0.33) Aa	6.62 (±0.15) Aa
	4	1.72 (±0.14) ABab	1.73 (±0.39) ABab	1.67 (±0.41) Aba	1.24 (±0.39) Bb	6.11 (±0.14) Aab	6.33 (±0.24) Aab	6.32 (±0.41) Aa	6.61 (±0.50) Aa
	5	1.45 (±0.07) Bb	1.72 (±0.29) Bab	2.08 (±0.03) Aa	1.71 (±0.06) Bab	6.24 (±0.47) Aab	6.56 (±0.23) Aa	6.68 (±0.41) Aa	6.75 (±0.40) Aa
	Mean	1.61 (±0.19) x ³	1.69 (±0.34) x	1.95 (±0.21) x	1.62 (±0.40) x	5.80 (±0.46) x	6.48 (±0.24) x	6.34 (±0.63) x	6.72 (±0.29) x
UNRIPE GRAPE SAUCE	6	1.87 (±0.00) Aab	1.97 (±0.21) Aa	1.79 (±0.29) Aa	1.99 (±0.18) Aa	6.11 (±0.50) Aab	5.93 (±1.06) Aab	4.86 (±2.69) Aa	5.34 (±2.35) Aa
	7	1.77 (±0.13) Bab	1.83 (±0.19) Aba	1.85 (±0.20) Aba	1.80 (±0.14) ABab	5.97 (±0.58) Aab	5.42 (±0.17) Ab	5.29 (±0.78) Aa	5.40 (±0.60) Aa
	8	1.69 (±0.07) Bab	2.16 (±0.16) Aa	1.97 (±0.06) Aa	2.08 (±0.08) Aa	5.52 (±0.73) Cab	6.28 (±0.30) ACab	6.65 (±0.33) Aa	6.77 (±0.20) Aa
	9	1.90 (±0.28) Aab	1.87 (±0.35) Aa	1.83 (±0.18) Aa	1.84 (±0.23) Aa	6.41 (±0.05) Ba	6.39 (±0.08) Bab	6.50 (±0.08) Aa	6.76 (±0.53) Aba
	10	2.00 (±0.08) Aa	1.72 (±0.03) Bab	2.00 (±0.08) Aa	2.04 (±0.18) Aa	6.53 (±0.24) Aa	6.47 (±0.56) Aa	6.77 (±0.01) Aa	6.92 (±0.29) Aa
	Mean	1.84 (±0.15) y	1.91 (±0.22) x	1.88 (±0.16) x	1.95 (±0.17) x	6.10 (±0.52) x	6.17 (±0.65) x	6.15 (±1.34) x	6.30 (±1.17) x
Positive Control	2.21 (±0.01) Aa				6.57 (0.03) ABa				

¹All the samples were tested as negative control before inoculation, and initial microflora could not be detected (<1.00 log CFU/mL)

²n=4, (± standard deviation), different lowercase letters indicate differences between rows and different capital letters indicate differences between columns (p<0.05).

³n=20, mean values of the groups, x and y letters indicate differences between rows (p<0.05).

Conclusions

As a conclusion, unripe grape products have intrinsic characteristics as low pH values, high titratable acidities and rich phenolic content that create a hostile environment for bacterial growth and survival. Hereby, the present study is focused on evaluating the surviving of *E. coli*, *L. monocytogenes*, *S. Typhimurium* and *Staphylococcus aureus* in these neutralized products at room temperature for low and high inoculum doses. Although, statistically significant reductions were observed, the survived population is remarkable at high doses. The inhibition effect on the tested pathogens seems to be very limited or completely disappeared when the inoculation dose is increased. Even though, the phenolic composition of products may be varied due to their species, regions, harvesting time and ripening period, generally the inhibitive activity on target bacteria among the products was not significantly different ($p > 0.05$). Therefore, the phenolic content of the samples indicate that these products could have antimicrobial effects on food borne pathogens – besides organic acid compositions.

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Microbiological quality during storage of pork *balangu* inoculated with *nisin* producing *Lactococcus lactis* subsp. *lactis*

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Abstract

Keywords:

Balangu
Lactococcus
Biopreservative
Activity
Spoilage
Safety

Article history:

Received 20.11.2015
Received in revised form
24.12.2015
Accepted 24.03.2016

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Introduction. The aim of this study was to characterize *Lactococcus* spp. isolates from pork *balangu* and use suitable strain in the biopreservation of the product.

Materials and methods. Isolation of *Lactococcus* isolates was carried out using traditional methods while their presumptive identities were obtained using phenotypic method involving biochemical reactions. Their full identification was obtained by the use of 16S rDNA sequencing. Production of antimicrobial agents was done by the use of reported methods. The abilities of suitable isolate(s) to control spoilage and pathogenic organism of food origin were examined.

Results and discussion. Thirty four *Lactococcus* spp. were isolated from pork *balangu*; their phenotypic characterization revealed they were composed of seven groups, members of which shared similar biochemical reactions. The phenotypic identities of seven isolates of the *Lactococcus* isolates, including one from each group, were confirmed by their 16S rDNA sequencing as *L. raffinolactis*, *L. garviae*, *L. lactis* subsp. *cremoris*, *L. lactis* subsp. *hordniae*, *L. lactis* subsp. *lactis*, *L. plantarum* and *L. piscium*. Among them *L. lactis* subsp. *lactis* produced higher concentration of lactic acid (21.45 g/10⁷ CFU) than others, and also produced *nisin* (~608 bp), active against the spoilage organism of meat *Brochothrix thermosphacta*. Inoculation of *nisin* producing *L. lactis* subsp. *lactis* in pork *balangu* showed 4 log reductions in the counts of *Listeria monocytogenes* and *B. thermosphacta* from their initial 6 log in the meat product *in situ*.

Conclusion. The *nisin* producing *L. lactis* subsp. *lactis* could be used in the shelf life extension of pork *balangu* beyond the day of production. The *Lactococcus* strain could therefore be applied as biopreservative culture to promote safety of meat products in West Africa and other parts of the world.

Introduction

In Nigeria, meat is normally cut into various thin slabs, spiced and grilled over glowing charcoals, resulting in products which are called by various names, including *suya*, *tsire*, *balangu* etc, depending on the specific method adopted during preparation. One of such popular grilled meat product is *balangu*, and may be produced from the muscle of cow, mutton, lamb, chicken or other animals, although preparation from beef is common. *Balangu* is a traditional Nigerian meat product, commonly prepared from raw meat, especially beef with the addition of various spices and cooked by grilling. It is usually eaten as delicacies and has associated sensory characteristics that play important role in its acceptance by consumers [1]. The meat product is consumed by many people as delicacies, mostly during leisure. Generally, producers of grilled meat products in Nigeria do not normally exhaust their sales on the day of production, thereby leaving remains until the second day or beyond. Unfortunately due to poor storage facilities, the remains are kept at ambient temperature of approximately 30°C. This temperature may encourage spoilage by opportunistic microorganisms and have adverse effect on the physicochemical properties of the product due to deterioration by spoilage organisms. This has resulted in product's rejection by customers, although such has not been reported in the literature due to limited research studies [1].

Food security, the availability of food and its accessibility to people in safe forms, has been an important concern in most developing countries where food preservation techniques have been very inadequate [2]. In Nigeria, this has adverse effects on grilled meat products, especially *balangu*, where losses have been experienced due to poor storage facilities [1]. Biopreservation refers to the extension of the shelf-life and improvement of the safety of foods using microorganisms and/ or their metabolites [3]. One of the common biological agents used in the biopreservation of food is lactic acid bacteria (LAB). LAB have a GRAS (generally regarded as safe) status and have been widely used as starters in the industrial preservation of meats [4]. LAB can secrete many antimicrobial substances with presumptive antimicrobial effect in foods that could be exploited in preventing many foodborne pathogens and spoilage organisms [5]. LAB are used to ensure safety, preserve food quality, develop characteristic new flavours, and to improve the nutritional qualities of food [6]. Many LAB cultures like *Leuconostoc* spp., *Lactococcus* spp., *Pediococcus* spp., and *Lactobacillus* spp. are being used in meat processing and few of these cultures could produce bacteriocins, a useful factor which has been noted to enhance their choice as starters [7]. The ability of LAB to inhibit the growth of undesirable bacteria has been reported and inhibition may be due to the production of organic acids, hydrogen peroxide, carbon dioxide, acetaldehyde, diacetyl or bacteriocins [3,8].

Some strains of LAB have been identified and used as starters in the preservation of some meat products [5,8,9]. There is limited research studies on the Nigerian traditional grilled meat product *balangu* and more efforts are required to protect consumers and ensure safe availability of the product in promoting food security in Nigeria. The only known study was reported by Olaoye [1] who investigated some physicochemical factors of *balangu* as possible indicators of spoilage during storage. The present study therefore reports on characterization of *Lactococcus* isolates from pork *balangu* and the use of a *nisin* producing *L. lactis* subsp. *lactis* in the biopreservation of the product.

Materials and methods

Source of materials. The pork meat used in this study was obtained from a Nigerian retail shop in the city of Nottingham, UK. The meat samples were conveyed over ice crystals to the laboratory in clean polyvinyl chloride (PVC) bags and stored briefly at -5°C (Haier Thermocool, BD-66A, Westgate Ealing, London, UK) before use. Other materials used which included ground red pepper (*Capsicum* sp.), ginger (*Zingiber officinalis*), groundnut (*Arachis hypogaea*), salt and groundnut oil were all purchased from the same source.

Bacterial strains and culture conditions. The bacteria cultures, used in challenge experiments against the starter culture (nisin producing *Lactococcus lactis* subsp. *lactis*) in the pork meat product *in situ*, included *Brochothrix thermosphacta* NCIMB 10018 (STAA, oxid; 30°C), *Listeria monocytogenes* NCTC 11994 (OX, oxid; 30°C) and *Salmonella typhimurium* (XLD, oxid; 30°C) which were obtained from culture collection unit of Food Microbiology, Division of Food Sciences, University of Nottingham, UK. Frozen cultures were maintained in BHI broth media (Oxoid, UK) containing 20% glycerol at -80°C [10].

Isolation of Lactococcus from pork balangu. Isolation of *Lactococcus* isolates was done according to a modification of the method of Onilude *et al.* [8]. A weighed quantity (10 g) of fresh pork meat was immersed in a sterile 10% (w/v) sucrose solution for about 7 min to stimulate LAB growth [11]. This was then homogenized in 90 ml sterile distilled water (SDW) in standard stomacher bags (BA 6141, Seward, UK), using a Seward stomacher (model 400 circulator, P/4/518, 50-60Hz, UK). The resultant homogenate was made into 10 fold dilutions, 1 ml of suitable dilution was measured into sterile petri dishes after which 10-15 ml of molten deMann Rogosa and Sharpe, MRS (-45°C ; Oxoid UK) agar was carefully poured while swirling gently. The plates were allowed to cool and then incubated in micro-anaerobic jars (18 - 24 h, 37°C). Upon observation for growth, Gram positive, catalase and oxidase negative colonies were picked for sub-culturing on sterile MRS agar. Streaking of single colonies was done repeatedly to obtain pure cultures which were examined microscopically for cell appearing as cocci in pairs or short chains, which are characteristic *Lactococcus* [12]. Pure cultures of presumptive *Lactococcus* isolates were kept for characterization and identification.

Phenotypic characterization and identification of the Lactococcus isolates. Seven presumptive isolates of *Lactococcus* were selected based on their biochemical reactions were characterized using API 20 Strep kit (API Systems, Biomerieux Sa, France). Presumptive identification of the isolates was done using the results obtained in the API website (www.apiweb.biomerieux.com).

Full identification of the presumptive Lactococcus isolates. Confirmation of full identities of the presumptive *Lactococcus* isolates was carried out using PCR-16S rDNA . DNA was extracted by a modification of the boiling method described by Suwanjinda *et al.* [13]. PCR amplification was carried out with specific primers targeting approx. 390 bp of the 16S rDNA (V4 regions) in the *Lactococcus* isolates. This was in a 50 μl reaction volume containing 1.25 units of *Taq* DNA polymerase (ABgene, Thermo Fischer, UK), 2.5mM magnesium chloride (Promega, Southampton, Southamptonshire, UK), 0.2mM dNTPs (Promega), 0.1 μl of each reverse 5'-CCGTC AATTCCTTTGAGTTT-3' and forward primer 5'- CAGCAGCCGCGGTAATAC-3' [14], 5 μl PCR buffer and 5 μl of DNA template. Volume was made up with SDW.

Electrophoresis of the PCR products was performed on the Bio-Rad Contour-Clamped Homogenous Electric Field (CHEF) DRII electrophoresis cell (Hemel Hempstead, UK). Agarose gel (Biogene Kimbolton Cambs, UK), 1.5% (w/v), stained with 0.5 µg/ml ethidium bromide, was used in 1 X TAE (Tris-Acetate EDTA) buffer at 84 volts for 1.5 - 2 h. A 100 bp molecular size marker was as ladder.

Sequencing of 16S rDNA genes was done by resolving 40 µl of the PCR products in 1% Agarose gel. Amplicons were excised from gel, purified using the Wizard PCR Preps DNA Purification System (Promega) and sent to Germany (MGW-Biotech, Germany) for sequencing. The nucleotide sequences were used in the GenBank database using BLAST at the website (<http://www.ncbi.nlm.nih.gov/blast/>) to determine the closest known relatives of the 16S rDNA gene sequences.

Selection of starter culture. The *Lactococcus* isolates were tested for abilities to produce organic acids (lactic acid and acetic acid), which is characteristic of the fermentative ability of LAB in lowering pH of food samples and thereby creating uncondusive environment for the growth of many spoilage/pathogenic organisms. Possible production of bacteriocin by the isolates was also evaluated in *in vitro* assay against sensitive indicator organism.

Production of organic acids was tested using the method of high performance liquid chromatography (HPLC) described by Olaoye and Onilude [15].

The modified method described by Suwanjinda *et al.* [13], involving overlaying of M17 plates, containing live colonies of *Lactococcus*, with indicator organism (*Brochothrix thermosphacta* NCIMB 10018) was used to detect antagonism of the *Lactococcus* isolates. Serial dilutions were made and used to obtain plates containing 10-50 colonies of *Lactococcus* on M17 agar; the plates were carefully overlaid with test indicator strain (50 µl of an overnight culture in 10 ml of BHI broth containing 0.7% agar). Plates were allowed to solidify and then incubated at 30°C for 24 h. The plates were examined for presence of zones of inhibition around the *Lactococcus* colonies.

The paper disc assay method [16] was also used detect antimicrobial activity of the *Lactococcus* isolates against *Brochothrix thermosphacta* NCIMB 10018. The *Lactococcus* isolates were grown in M17 broth for 24 h at 30°C. The cultures were then centrifuged at 5000 × *g* for 15 min (Centrifuge Falcon 6/300 series, CFC Free, UK) and the cell free supernatants (CFS) were collected for use in antimicrobial assay.

A sterile filter paper disc (Whatman AA, 6mm, Fisher Scientific, UK) was soaked in CFS for 30 min, and then applied on plates previously seeded with BHI broth (with 0.7% agar) containing 50 µl of indicator organisms. The plates were incubated overnight at 30°C for 24 h and then observed for zones of inhibition. Clear zones extending for 1 mm or more were considered as positive for inhibition [17].

To test for possible production of bacteriocin, the CFS was neutralized to pH 6.5 using NaOH to eliminate activity of organic acids. To also get rid of influence of hydrogen peroxide, the neutralized CFS was treated with 300 units/ml of horseradish peroxidase (Sigma-Aldrich); this gives a crude bacteriocin used in bacteriocin assay [18] against the sensitive indicator organism (*Brochothrix thermosphacta* NCIMB 10018) using the paper disc method as previously described.

The *Lactococcus* isolates, that their CFS displayed antagonism against the sensitive indicator organism *B. thermosphacta* NCIMB 10018, were tested for presence of bacteriocin (*nisin*) encoding genes by PCR. DNA templates were obtained using the extraction method of Suwanjinda *et al.* [13]. PCR reactions were same as for 16S rDNA. The primers used include 5'-CTATGAAGTTGCGACGCATCA-3' (Forward) and 5'-

CATGCCACTGATACCCAAGT-3' (Reverse), targeting the 608 bp of the *nisin* operon in *Lactococcus* (Suwanjinda *et al.*, 2007). The same methods as used for PCR amplification conditions, resolving and visualization of PCR products, sequencing and identification of 16S rDNA genes were used for the *nisin* gene(s).

Preparation of pork balangu. The traditional technique was mimicked for preparation of pork *balangu* with little modification [1]. Fresh pork meat was rinsed in SDW and then cut into chunks (25 x 16 x 1.2 cm), each weighing about 75±2.3g. They were allowed to drain for 15 min in clean perforated plastic containers. The entire surface of each of the pork chunks was covered with mixed ground spices, consisting of red pepper (23%), groundnut powder (52%), ginger (20%) and salt (5%). The spiced pork chunks were then spread on wire gauze and grilled over glowing charcoals at 100-120°C for 30-45 min. They were intermittently turned to ensure even cooking while groundnut oil was sprinkled on them at regular intervals during the grilling process to simulate the traditional technique of avoiding burning or charring [8].

Inoculation of pork balangu with bacteriocin producing *Lactococcus lactis* subsp. *lactis* as starter culture. The *nisin* producing *Lactococcus lactis* subsp. *lactis* culture was subcultured three times in M17 broth at 30°C for 24 h. Filter sterilised glucose solution (~5% w/v final concentration) was applied to the pork *balangu* pieces [19] and then placed separately in aluminium foils. Two sets (Ing-lc and N-Ing-lc) of the pork meat were inoculated with the *Lactococcus* culture to about 10⁶ cfu/g of meat, wrapped in sterile transparent bags and incubated at 30°C for 5 days. Un-inoculated samples (Ing-cont and N-Ing-cont) served as control. The ability of the *Lactococcus lactis* subsp. *lactis* to control *LS. monocytogenes*, *Salmonella typhimurium* and *Brochothrix thermosphacta* was tested in challenge experiments by inoculating them separately on different sterile pork *balangu* samples (Ing-lc and N-Ing-lc) at 10⁶ cfu/g prior to inoculation with *nisin* producing *Lactococcus* isolate. The pathogenic/spoilage organism were previously grown for 24 h in BHI broth (Oxoid) before their application on the meat product.

Microbial enumeration of pork balangu during storage. Pork *balangu* samples (10 g) were homogenized in standard stomacher bag (BA 6141, Seward, West Sussex, UK) containing 90 ml maximum recovery diluent (MRD) for 3 min at 230 rpm, using a Seward stomacher (model 400 circulator, P/4/518, 50-60Hz, UK). One millilitre (1 ml) of homogenate was serially diluted in 9 ml of MRD to obtain 10 fold dilutions; 1 ml of appropriate dilutions were spread or pour plated in replicates on selected agar media. The media used included deMan Rogosa Sharpe (MRS, Oxoid) for LAB, incubated at 30°C for 48 h; Rose Bengal Chloramphenicol Agar (RBCA, Oxoid) for yeast and moulds at 25°C for 72 h; Mannitol salt phenol red agar, MSPRA (Sigma-Aldrich, St. Louis, Missouri, USA) for *Staphylococcus* at 37°C for 24 h; MacConkey Agar (Oxoid) for coliforms at 37°C for 24 h; Violet red bile glucose agar (VRBGA, Oxoid) for *Enterobacteriaceae*, at 30°C for 48 h; Xylose lysine deoxycholate (XLD, Oxoid) for *S. typhimurium* at 37°C for 24 h; Oxford formulation (Oxoid) for *LS. monocytogenes* at 30°C for 48 h; and STAA agar (Oxoid) for *B. thermosphacta* at 30°C for 24-48 h. Emerging colonies were counted and the results expressed in colony forming units per gram (cfu/g) of meat.

pH measurement. pH of the pork *balangu* samples was monitored during storage by taking 10 g of sample and homogenized in standard stomacher bags (BA 6141, Seward, UK) containing 100 ml SDW, using a Seward stomacher (model 400 circulator, P/4/518,

50-60Hz, Leighton Buzzard, UK). The pH was then measured by a pH meter (pH 212 Microprocessor, Hanna Instruments, USA) using the method of Marugg *et al.* [20].

Statistical analysis. The effect of storage time on the respective pork *balangu* samples was evaluated by subjecting the data obtained on each day of storage to analysis of variance using the statistic software, Design expert (version 6.0.6; Stat-Ease Inc., East Hennepin Ave, Minneapolis, MN). Significant differences were determined at $p < 0.05$.

Results and discussion

Thirty four presumptive isolates of *Lactococcus* were isolated from pork *balangu*, with the objective of selecting suitable isolate for use as starter culture in biopreservation of the meat product. The biochemical characteristics of the *Lactococcus* isolates (Table 1) indicate the presence of seven groups, with each member having similar biochemical reactions. The members of the groups were Gram positive cocci, appearing in pairs or short chains which are characteristic of the genus *Lactococcus* [12]. The phenotypic (biochemical) characteristics of the isolates were used in their phenotypic identification with particular reference to the Bergey's manual of determinative bacteriology [12]. The isolates of each group were therefore phenotypically identified as *Lactococcus piscium*, *L. garviae*, *L. lactis* subsp. *cremoris*, *L. lactis* subsp. *hordniae*, *L. lactis* subsp. *lactis*, *L. plantarum* and *L. raffinolactis* respectively. Their full identifications were obtained by sequencing of their 16S rDNA genes (V4 region) that were amplified by PCR (Figure 1); PCR products of approximately 400 bp in size were obtained. The 16S rDNA nucleotide sequences were used in the genBank database to obtain their closest relatives. The *Lactococcus* isolates showed between 99 and 100% homologies to the respective closest relatives, thus giving acceptable full identifications [21].

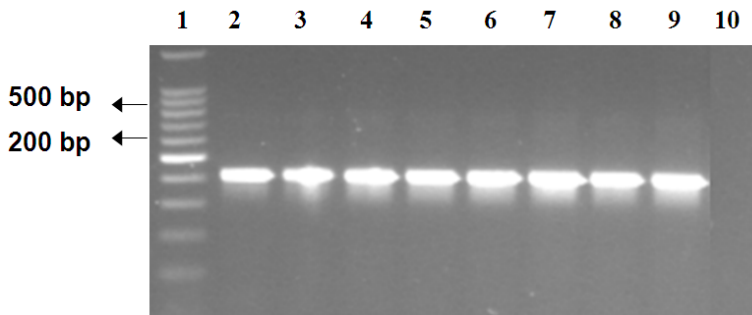


Figure 1. PCR amplification of 16S rDNA -V4 region of the phenotypically identified *Lactococcus* isolates from pork *balangu*

1 - 100 bp DNA marker; 2, -presumptive *Lactococcus piscium*; 3 3 presumptive *L. garviae*; 4 - presumptive *L. lactis* subsp. *cremoris*; 5 - presumptive *L. lactis* subsp. *hordniae*; 6 - presumptive *L. lactis* subsp. *lactis*; 7 - presumptive *L. plantarum*; 8 - presumptive *L. raffinolactis*; 9 - positive control - *L. lactis* NCIMB 4918; 10 - Negative control - sterile deionized water.

Table 1. Phenotypic/biochemical characteristics and presumptive identities of *Lactococcus* isolates from PFGE clusters

Cell shape	cocci in pairs or short chains	cocci in pairs or short chains	cocci in pairs or short chains	cocci in pairs or short chains	cocci in pairs or short chains	cocci in pairs or short chains	cocci in pairs or short chains
Grams reaction	+	+	+	+	+	+	+
Acid production from:							
<i>Amygdalin</i>	+	+	-	-	-	-	-
<i>Galactose</i>	+	+	+	+	+	+	+
<i>Lactose</i>	+	-	-	-	-	-	-
<i>Maltose</i>	+	+	+	+	+	+	+
<i>Melibiose</i>	+	-	-	-	-	-	-
<i>Melezitose</i>	+	-	-	-	-	-	-
<i>α-Methyl-D-glucoside</i>	+	-	-	-	-	-	-
<i>α-Methyl-D-mannoside</i>	+	-	-	-	-	-	-
<i>D-Raffinose</i>	+	-	-	-	-	-	-
<i>Sucrose</i>	+	+	+	+	+	+	+
<i>Trehalose</i>	+	+	+	+	+	+	+
<i>D-Turanose</i>	+	-	-	-	-	-	-
<i>D-Xylose</i>	+	-	-	-	-	-	-
Growth at 0°C	+	-	-	-	-	-	-
Growth at 40°C	-	+	+	+	+	+	+
Growth 4% NaCl	-	+	+	+	+	+	+
Production of:							
APP ^a	+	+	+	+	+	+	+
<i>Arginine Dehydrolyase</i>	-	+	+	+	+	+	+
<i>α-Galactosidase</i>	+	+	-	-	-	-	-
<i>β-Galactosidase</i>	+	+	+	+	+	+	+
<i>β-Mannosidase</i>	+	-	-	-	-	-	-
PYR ^{A^b}	-	+	-	-	-	-	-
<i>Hippurate hydrolysis</i>	-	-	+	+	+	+	+
Probable identity	<i>L. piscium</i> (5)	<i>L. garmae</i> (8)	<i>L. lactis</i> subsp. <i>cremoris</i> (5)	<i>L. lactis</i> subsp. <i>borderiae</i> (6)	<i>L. lactis</i> subsp. <i>lactis</i> (3)	<i>L. plantarum</i> (3)	<i>L. reffinolactis</i> (4)

+, positive; -, negative; APPA, Alanyl-phenylalanyl-proline arylamidase; PYRA, Pyrrolidonyl arylamidase

Among the *Lactococcus* isolates, only the neutralized supernatant of *L. lactis* subsp. *lactis* was shown to display antagonism against a *nisin* sensitive indicator organism *B. thermosphacta* in an *in vitro* assay. The broth medium was neutralized in order to eliminate the presence of organic acids which may interfere with the antimicrobial assay [13]. Additionally, the broth supernatant was treated with peroxidase enzyme to get rid of hydrogen peroxide that may also interfere in the assay [16]. The antimicrobial activity recorded against the indicator organism by the neutralized broth supernatant of *L. lactis* subsp. *lactis* suggested the presence of bacteriocin encoding gene in the isolate. The positive PCR amplification of the bacteriocin (*nisin*) gene of about 608 bp in the *Lactococcus* isolate with specific primer was used to confirm this finding, this was similar to those reported in earlier investigations [22,23]. Beside production of *nisin* by *L. lactis* subsp. *lactis*, its ability to produce considerable quantities of organic acids, especially lactic acid, was considered in its choice as starter culture for pork *balangu*. During preliminary experiments, *L. lactis* subsp. *lactis* produced higher quantity of lactic acid ($21.45 \text{ g}/10^7 \text{ CFU}$) than others, giving it an advantage as starter culture for the pork meat product. The ability of LAB to produce lactic acid that contributes to reduction in the pH of food products is one of the very important factor in their antagonistic activities against spoilage and pathogenic organism [5,23,24]. The lowering of pH due to production of organic acids can also cause characteristic changes in flavour and texture of meat products; however, this does not normally have any adverse effect on the sensory appeal of the product [11,25].

The specific microbial organisms that were evaluated in the meat samples during storage exhibited their defined colony characteristics on respective media; LAB as whitish or milky; coliforms as pinkish or reddish; Enterobacteriaceae as pink; *Staphylococcus* had red or yellow colourations around them; *Ls. monocytogenes* as black; *Salmonella typhimurium* appeared as red with black centre colourations; and *B. thermosphacta* appeared as straw coloured colonies. The specific organisms emerging from respective growth media were also confirmed by PCR-16S rDNA, and their nucleotide sequences used in the genBank database to know the closest relatives.

There was no detection of coliforms in the pork *balangu* samples until after 72 h of storage (Figure 2). Lower counts of coliforms were recorded in the starter culture inoculated samples (SCIS) compared to the uninoculated control samples (UCS), suggesting possible inhibitory action by the starter culture. Kalalou *et al.* [11 reported the reduction of coliform counts during storage of a Moroccan fermented meat product inoculated with LAB. In similar studies, Olaoye *et al.* [5] and Olaoye and Onilude [25] reported reduction of coliforms in fresh meat inoculated with LAB starter cultures. The researchers concluded that the use of LAB as starter culture was moderately effective for the control of coliforms. Reduction of coliforms by the LAB cultures could be attributed to production of antimicrobial agents by the latter [15,26]. The non-detection of coliforms in the early period of storage in the *balangu* samples could be due to the lethal action of heat on them during the grilling process.

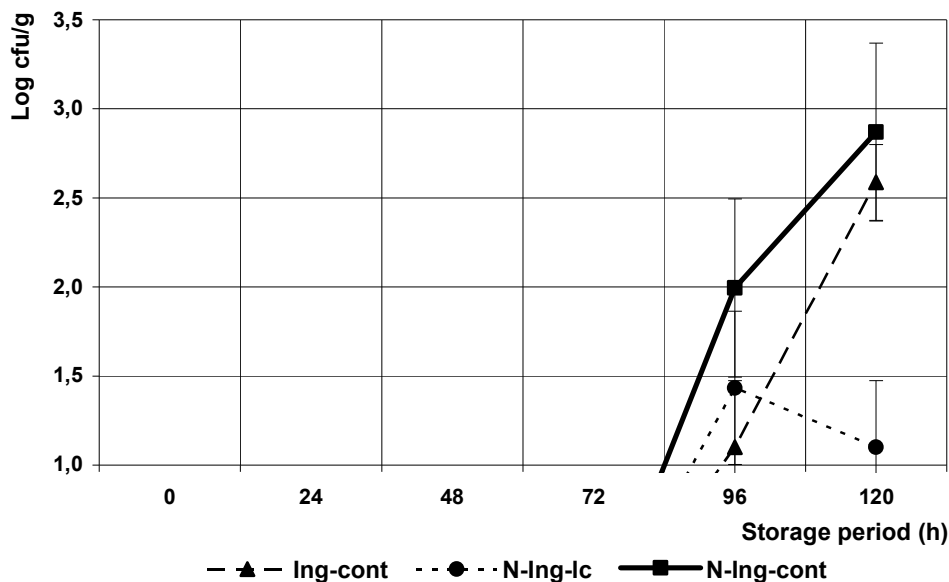


Figure 2. Counts of Coliforms in pork *balangu* during storage

Ing-lc, pork *balangu* containing spices and inoculated with *L. lactis* subsp. *lactis*; Ing-cont, pork *balangu* containing spices and uninoculated with starter culture; N-Ing-lc, pork *balangu* containing no spices and inoculated with *L. lactis* subsp. *lactis*; N-Ing-cont, pork *balangu* containing no spices and uninoculated with starter culture.

From the results of counts of total bacteria (CTB), a decrease was recorded in the SCIS compared UCS (Figure 3). While CTB decreased from the initial count of about 3.81 at 0 h to 2.84 at 48 h in the Ing-lc samples, an increase from 3.79 to 7.88 was recorded for the Ing-cont samples over same period. In all pork *balangu* samples, CTB was comparatively lower in the SCIS than UCS, indicating the effect starter culture on the microflora associated with the meat product. The effect could possibly be due to production of antimicrobial agents by the *Lactococcus* starter culture. Similar findings have been reported on the reduction of CTB in meat products treated with cultures of LAB [9,19,27]. Yeast and moulds were not detected in the Ing-lc samples during storage; however, counts (log count/g) of 3.45 and 4.82 and above were recorded from 72 h in the N-Ing-lc and N-Ing-cont respectively (Figure 4). Counts were generally lower in the SCIS compared to UCS ($p < 0.05$); increment of 3-5 log was observed in the latter samples compared to 0-2 log recorded for the former. Casaburi *et al.* [28] observed a reduction in yeast and mould counts of Italian style sausages on storage after inoculation with LAB starter culture. In another study, Erkmén [27] made similar observation in a Turkish sausage treated with LAB. Similarly, Olaoye [9] reported reduction in the counts of yeast and moulds in a Nigerian stick meat *tsire* inoculated with cultures of LAB during storage. The researchers concluded that the LAB cultures reduced the growth of yeast and moulds in the meat products.

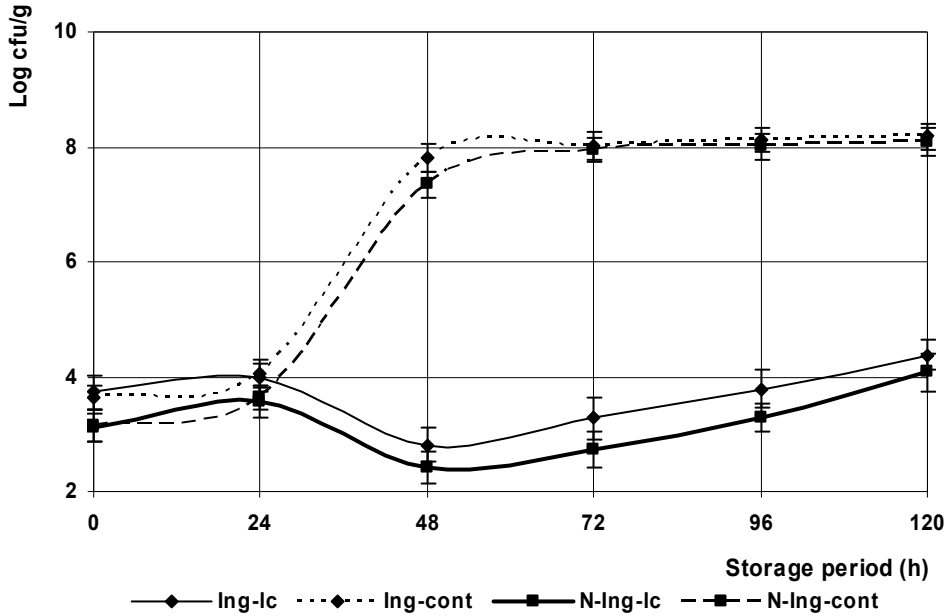


Figure 3. Counts of total bacteria in pork *balangu* during storage

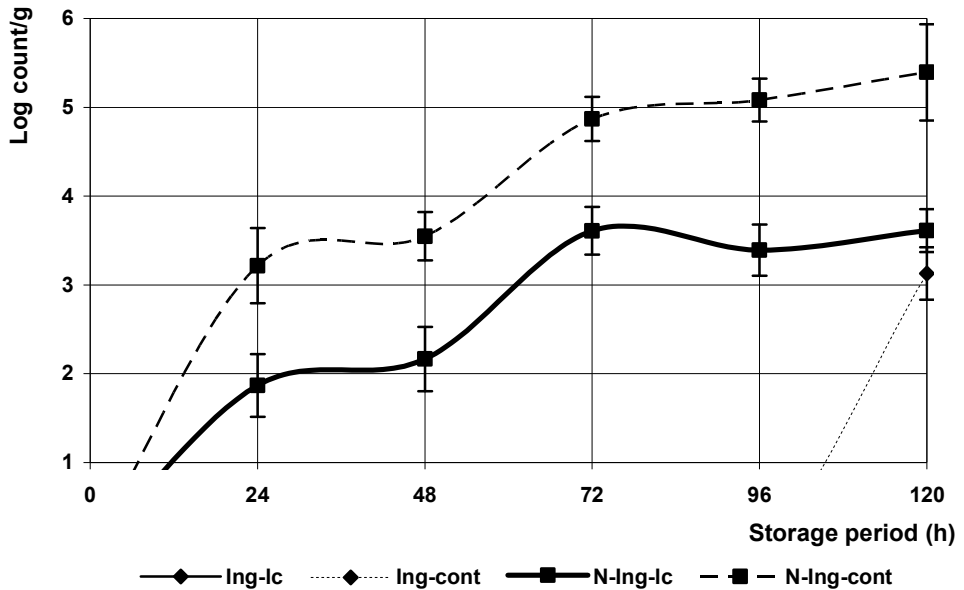


Figure 4. Counts of yeast and moulds in pork *balangu* during storage

Ing-lc, pork *balangu* containing spices and inoculated with *L. lactis* subsp. *lactis*; Ing-cont, pork *balangu* containing spices and uninoculated with starter culture; N-Ing-lc, pork *balangu* containing no spices and inoculated with *L. lactis* subsp. *lactis*; N-Ing-cont, pork *balangu* containing no spices and uninoculated with starter culture.

Figure 5 presents the *Ls. monocytogenes* counts in the pork *balangu* samples during storage. Prior to storage, the meat product was inoculated with approximately 6 log cfu/g of the pathogenic organism to challenge the starter culture. Counts of *Ls. monocytogenes* decreased by about 1.6 and 4 log at 24 h and 48 h respectively in the Ing-lc samples, and at 72 h were below 2 log. On the contrary, an increase of up to 5 log was recorded in the UCS, i.e. Ing-cont and N-Ing-cont samples. Counts increased from 7.07 at 0 h to 11.78 at 120 h in Ing-cont, while similar increase from 6.79 to 11.18 was recorded in N-Ing-cont during same period. Sensitivity of *Ls. monocytogenes*, an important foodborne pathogen, to *nisin* produced by *Lactococcus lactis* subsp. *lactis* was reported by Cintas *et al.* [29]. The risk of *Ls. monocytogenes* in causing serious threat to food safety and health of consumers has been noted [30]. The pathogen has also been known to contaminate meat and meat products during slaughter, processing and production while it can also persist and grow at low pH values, at low water activity and at refrigeration temperatures [15]. Hence the need for its control, especially by the use of biopreservative agents is of ultimate importance in order to safeguard public health [31]. The control of *Ls. monocytogenes* recorded in the *tsire* samples by the *Lactococcus* culture is therefore very important, as no previous report is available on its control in the Nigerian traditional meat product *balangu*.

The pork *balangu* was also inoculated with approximately 10^6 cfu/g of *Salmonella typhimurium* to challenge the starter culture in the meat product. The graph (Figure 6) shows that there was about 3 log reduction in the counts of *S. typhimurium* in the SCIS samples during storage. An increase of approximately 4 log was however recorded in UCS. The antagonism recorded against *S. typhimurium*, a foodborne pathogen, may be attributed to production of antimicrobial agents, such as organic acids and hydrogen peroxide, by the *Lactococcus* starter culture. The *nisin* produced by the starter culture may not contribute to the antagonism of the pathogen because bacteriocins of LAB have been reported to be ineffective against gram negative organisms [18,29]. Furthermore, *Brochothrix thermosphacta*, a meat spoilage organism, was inoculated at 10^6 cfu/g of pork *balangu* to challenge the *nisin* producing *L. lactis* subsp. *lactis* used as tarter culture during storage. Result indicates that there was reduction of about 4 log in the Ing-lc sample at 24 h, after which period the spoilage organism was no more detected throughout storage (Figure 7). A similar observation was noted in the N-Ing-lc sample, where 3 and 4 log reductions were recorded at 24 and 48 h of storage respectively. There was however an increase of 4 log of the spoilage organism in the UCS. The *nisin* produced by the *L. lactis* subsp. *lactis* may have contributed mainly to the inhibition of the spoilage organism, as the indicator organism is known to be sensitive to the bacteriocin. In a related study, Ercolini *et al.* [32] demonstrated effective antimicrobial activity of purified *nisin* against *B. thermosphacta* in meat during storage. Hence, the antagonistic activity of *Lactococcus* starter culture against this spoilage organism in the present study could be very significant towards curtailing spoilage and extending shelf life of pork *balangu* in Nigeria.

The result of pH monitored in the pork *balangu* samples showed that values in SCIS were lower than 5 from day 2 of storage, unlike in the UCS where values of above 6 were recorded (Figure 8). The decrease in pH values of SCIS may be due to production of organic acids by the starter culture. Lowering of pH in food products inoculated with lactic acid bacteria has been recorded by other researchers [1,5,33] and this has been noted as an important factor in the control of undesirable microorganisms in food [34]. The reduction in pH in starter treated samples may thus contribute to the lower counts recorded in those samples than in their uninoculated control counterparts.

Statistically, results generally indicate that there were significant differences ($p < 0.05$) between pork *balangu* samples inoculated with *nisin* producing *Lactococcus lactis* subsp. *lactis* and the uninoculated control samples.

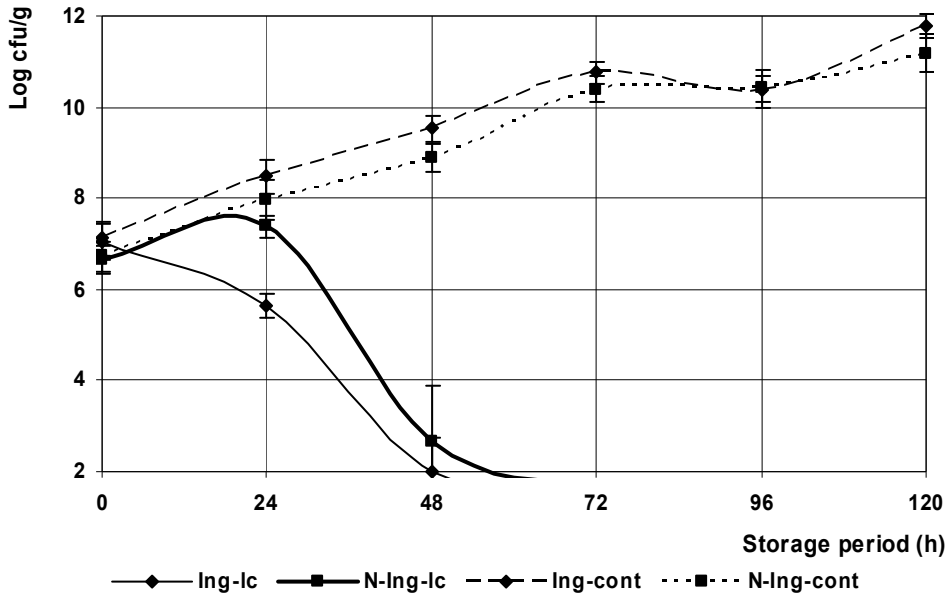


Figure 5. Counts of *Listeria monocytogenes* in pork balangu during storage

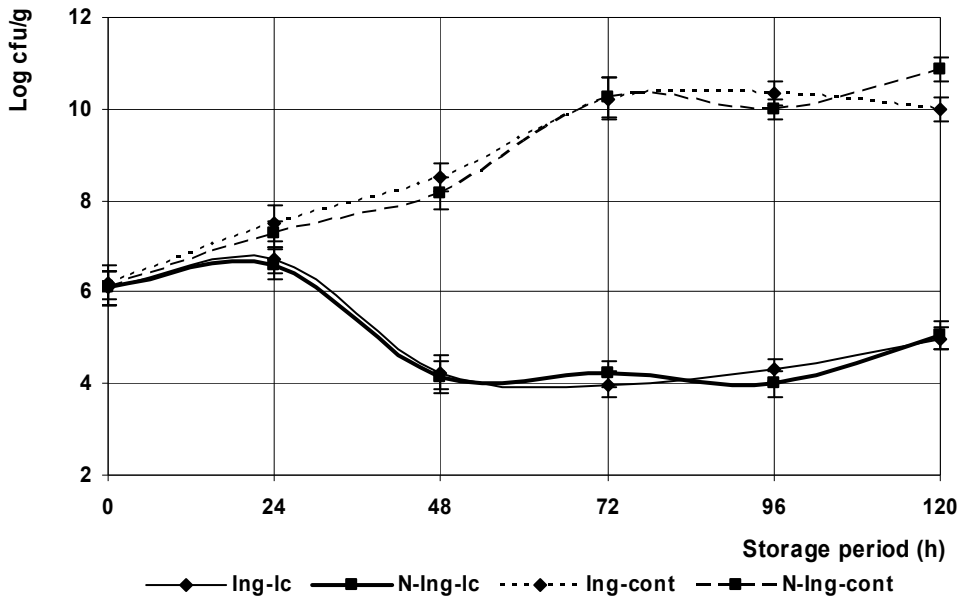


Figure 6. Counts of *Salmonella typhimurium* in pork balangu during storage

Ing-lc, pork balangu containing spices and inoculated with *L. lactis* subsp. *lactis*; Ing-cont, pork balangu containing spices and uninoculated with starter culture; N-Ing-lc, pork balangu containing no spices and inoculated with *L. lactis* subsp. *lactis*; N-Ing-cont, pork balangu containing no spices and uninoculated with starter culture.

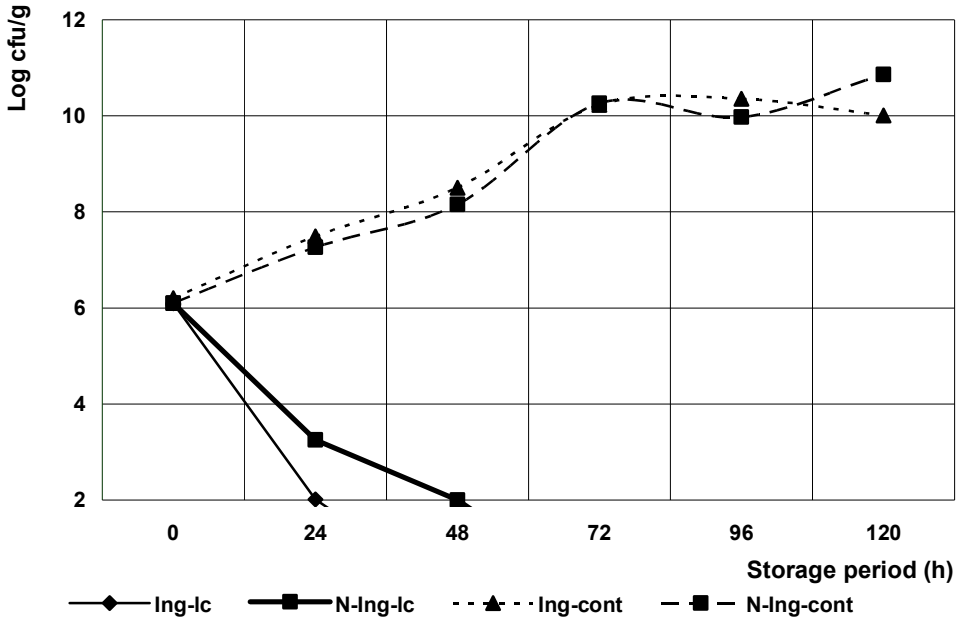


Figure 7. Counts of *Brochothrix thermosphacta* in pork *balangu* during storage

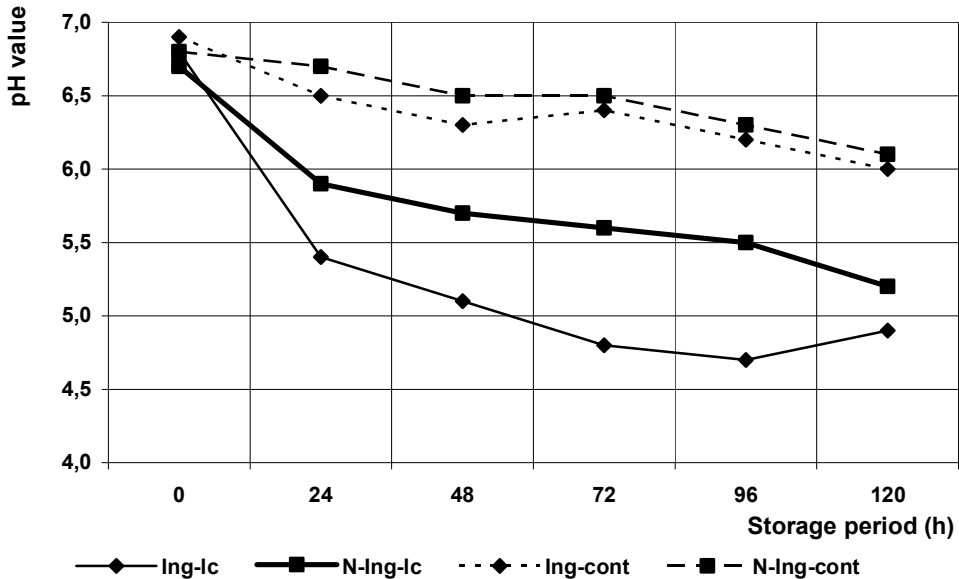


Figure 8. pH patterns of pork *balangu* during storage

Ing-lc, pork *balangu* containing spices and inoculated with *L. lactis* subsp. *lactis*; Ing-cont, pork *balangu* containing spices and uninoculated with starter culture; N-Ing-lc, pork *balangu* containing no spices and inoculated with *L. lactis* subsp. *lactis*; N-Ing-cont, pork *balangu* containing no spices and uninoculated with starter culture.

Conclusion

The *nisin* producing *Lactococcus lactis* subsp. *lactis* used as starter culture in pork *balangu* demonstrated effective control of spoilage and pathogenic organisms in the meat product. Of most important is the control recorded against the pathogens *Ls. monocytogenes* and *S. typhimurium*, and spoilage organism *B. thermosphacta* which are commonly associated with meat products. The findings of this study may therefore be every useful in the preservation of the product, as possible transformation into practical applications could constitute an important approach for improving safety and availability.

Acknowledgement. Authors thank the Division of Food Sciences, University of Nottingham, UK, where certain aspects of this study was carried out.

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Oleoresins effect on cooked poultry sausages microbiological stability

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Abstract

Keywords:

Sausage
Poultry
Oleoresin
Storage

Article history:

Received 13.01.2016
Received in revised
form 14.03.2016
Accepted 24.03.2016

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Introduction. The effect of coriander, mace and black pepper oleoresins on microbiological stability of a cooked poultry sausages during refrigeration storage was investigated.

Materials and methods. Cooked poultry sausages with different part non-meat raw material were examined. Microbiological attributes, such as Quantity of Mesophilic Aerobic and Facultative Anaerobic Microorganisms (QMAFAnM), coliforms, *Salmonella*, Sulfite-reducing clostridia, *Proteus*, *Listeria monocytogenes*, *Staphylococcus aureus*, yeasts and molds were determined methods accepted in general lines.

Results and discussion. There was no significant difference between initial QMAFAnM for all samples after the thermal treatment (day 0). QMAFAnM for CO samples increase during entire research. At the end of storage, the count was increased to $1,1-8,5 \times 10^5$ cfu/g and was significantly higher than the other samples, except CO40. The QMAFAnM in MO samples was initially $1,0 \times 10^1 - 1,5 \times 10^2$ cfu/g and was maintained at this level until the seventh day of storage. However, after 13 days the increase of QMAFAnM was significant. Sausages with BPO showed stable meaning of QMAFAnM during storage time. After 13 days of storage, the BPO samples contributed to significantly lower QMAFAnM count than the CO and MO samples. The initial population of moulds was <10 cfu/g while on day 4 of storage a count of $2,5-7,0 \times 10^1$ cfu/g was recorded for treatments with CO. MO and BO samples demonstrated stable meaning of moulds during entire research, only on 13th day of storage BPO100 and MO60 samples showed $2,0 \times 10^1$ and $2,5 \times 10^1$ cfu/g respectively. Yeasts from MO and BPO samples did not differ after 7 d of storage, but were significantly lower than counts from CO samples. Yeasts from CO samples increased during entire storage. Sausages with MO showed stable meaning of yeasts during storage time. Initial meaning of BPO80 yeasts was $3,0 \times 10^1$ cfu/g, although on 7th day of storage inhibition of yeasts was observed. The samples with BPO and MO had lower yeasts counts than CO samples during the entire storage period.

Conclusions. Mace oleoresin and black pepper oleoresin have more antimicrobial activity than coriander oleoresin. Only black pepper oleoresin has shown antimicrobial effect during refrigeration storage more than 10 days. In processing meat containing products with oleoresin it's necessary to make accent on black pepper oleoresin addition.

Introduction

Qualitative characteristics of meat products preservation during storage is one of important task and is vital for meat industry.

The object of the present research was to study the coriander (CO), black pepper (BPO) and mace (MO) oleoresins effect on the microbiological stability and the shelf-life of a cooked chicken meat (more than 60% of meat in formulation) and meat containing (less than 60 % of meat in formulation) products.

Spice oleoresins application for food, in particular meat products analysis

In the last decade, chicken-based meat products have become increasingly popular worldwide due to their high nutritional quality and low cost and are available as either fresh or precooked (i.e. fried) chicken products, which after subsequent packaging are usually stored under refrigeration [1]. Additionally, frozen chicken-based meat products also available on the market include specialties such as: nuggets, meatballs, hamburgers, frankfurters, etc. Susceptibility of chicken meat and chicken-based meat products to microbial spoilage presents a potential health hazard, since poultry meat may harbor pathogenic microorganisms [2].

Poultry and poultry products are a highly perishable food and their shelf-life varies between 3 and 10 days under refrigeration. Deterioration depends mainly on the microbiological quality of the poultry carcasses, as poultry meat offers the perfect environment, pH, nutrients and humidity conditions for microorganism development.

The use of natural preservatives in foods has been widely accepted by consumers, who increasingly seek for natural and healthier products, free of synthetic additives [3, 4]. In addition, consumers are used to the presence of herbs and spices commonly added to provide flavor and aroma in meats.

Black pepper and coriander are the most widely spread in meat products processing, unlike mace.

The quality of black pepper depends on the contents of piperine and essential oil. Both pepper and piperine exert liver protective action. Kaul and Kapil found that piperine reduces *in vitro* and *in vivo* lipid peroxidation [5]. This is a very significant property, as lipid peroxidation causes free radical production that causes tissue damage. Pepper has antioxidant activity which is attributed to the tocopherol and polyphenol contents in pepper. Supercritical carbon dioxide extracts of ground black pepper have been found superior in reducing lipid oxidation of cooked ground pork [6]. The antioxidative activity of black pepper can, at least partially, be ascribed to the presence of glycosides of the flavonoids kaempferol, rhamnetin and quercetin [7], as well as to the phenolic amides. Nakatani *et al.* established that all the five phenolic amides present in pepper possess very good antioxidant property, which is even superior to that of the synthetic antioxidants like butylated hydroxy toluene and butylated hydroxy anisole [8]. Addition of pepper to foods increases their keeping quality and prevents their spoilage, due to the antimicrobial properties of pepper. The essential oil of pepper is found to be inhibitory to *Vibrio cholerae*, *Staphylococcus albus*, *Clostridium diphtheriae*, *Shigella dysenteriae*, *Streptomyces faecalis*, *Bacillus* spp., *Pseudomonas* spp., etc. Pepper oil stopped the growth and aflatoxin production by *Aspergillus parasitics* at a concentration of 0,2–1%. Pepper leaf oil also exhibits antifungal activity.

The ethanol extract of *Coriandrum sativum* leaves is an excellent antioxidant, which is stable at high temperature and can serve as a substitute for synthetic antioxidants [9]. Further studies carried out by Melo *et al.* indicated that the four coriander extract fractions obtained from the crude extract using chromatography in silica gel possessed similar antioxidant activities, which can be measured by the β -carotene/linoleic acid system. The antioxidant activity was due to several phenolic acids and caffeic acid, which were present in all four fractions [10].

The greater antioxidant effect of a crude extract of coriander compared to its component fractions suggested a synergistic action between the carotenoids. Assessment of the total antioxidant activity of methanol and water extracts coriander leaves and stems using an iron-induced linoleic acid oxidation model system showed that the methanol-derived leaf extracts exhibited significantly greater radical-scavenging activity towards both lipid- and water-soluble radicals, which was attributed to the total phenolic content [11].

Coriander has strong antibacterial activity against *Staphylococcus aureus*, *Salmonella typhi* and *Escherichia coli* [12]. In 2002, a study carried out by Delaquis *et al.* reported that coriander oil strongly inhibited gram-positive bacteria (*Listeria monocytogenes* and *S. aureus*), but had little effect against gram-negative bacteria (*Pseudomonas fragi*, *E. coli*, *S. typhi*) [13].

Mace possess antioxidant properties. Checker *et al.* reported that the lignans present in the aqueous extract of fresh mace also possess antioxidant properties [14]. Acetone extract of mace-containing lignans inhibited lipid oxidation and prevented oxidative damage to cells [15]. Mace oil is inhibitory to the growth of *A. parasiticus* and *F. moniliforme* [16] and prevented the formation of aflatoxins.

Herbs and spices, and the oleoresins and essential oils extracted from them, are widely recognized as powerful agents for the preservation of food quality. Spices and herbs, in addition to contributing taste and aroma to foods, also contain a variety of bioactive substances which are of considerable use from the standpoint of food science and technology. These may be used singly or in combination, and some act synergistically to control spoilage of foods [17]. Their use has been well documented in terms of their ability to increase safety and shelf-life of pork, beef and poultry products through their antimicrobial [18, 19, 20] and antioxidant [21, 22, 23] capacities.

Therefore, oleoresins can be considered a good choice of natural preservatives for meat and meat products.

Oleoresin is a concentrated form of the spice containing the volatile essential oils as well as non-volatiles such as fixed oils, antioxidants, and pigments materials.

Oleoresins contain all of the volatile and nonvolatile flavor components and the natural antioxidants of the spices. In comparison to the ground spices, they are hygienic and can be standardized for acceptable flavour levels by blending. Unlike the essential oils, oleoresins contain natural antioxidants of the corresponding spices, which make them more stable. Oleoresin contains essential oils that make up the aroma, oleoresin also contains resins and compounds that did not volatile determine the characteristic flavor of spices. Moreover, the resin part in the oleoresins acts as natural fixatives to more volatile components. Oleoresins are quite concentrated and have good replacement value.

However, despite these advantages over ground spices, spice oleoresins exhibit sensitivity to light, heat and oxygen, and have short storage lives if not stored properly.

The process used for extraction depends on the nature of vegetable matter, and depending on its thermal instability, the operating temperature ranges from ambient to the boiling point. Oleoresin extraction is generally done with organic solvents, such as acetone, ethanol, methanol, hexane, ether and isopropyl alcohol. The choice of solvent affects the

quality and quantity of oleoresin obtained. Oleoresins were used in food processing safer extracted using ethanol solvent [24]. Extraction with polar solvents such as ethanol will be produced oleoresin with a low fat content.

In recent years, many researchers have evaluated the antioxidant properties of oleoresins from different spice and herbs [25, 26]. Oleoresin has been studied for its antimicrobial activity [27, 28]. Rosemary and onion oleoresins showed antioxidant effects in both raw and cooked irradiated pork loins. When these oleoresins were used in combination with α -tocopherol, the antioxidant activity was more distinct in irradiated cooked pork. Rosemary oleoresin–tocopherol had stronger antioxidant effect than onion–tocopherol [29]. The incorporation of nanoparticle paprika oleoresin in to meat using carrier system, demonstrated that the marinating performance and sensory acceptability of marinated meat products can be improved and optimized by the utilization of nanoparticle ingredients in marinating operations [30].

It appears a meat containing products with large part non-meat raw material problem shelf-life and ensuring microbiological deterioration.

Materials and methods

The present study focused on the monitoring of the following species of micro-organisms: Quantity of Mesophilic Aerobic and Facultative Anaerobic Microorganisms (QMAFAnM), coliforms, *Salmonella*, Sulfite-reducing clostridia, *Proteus*, *Listeria monocytogenes*, *Staphylococcus aureus*, yeasts and molds.

After the preparation of each chicken batter, oleoresin was added according to the following formulations (Table 1).

Table 1
Formulation of poultry sausages with coriander, mace and black pepper oleoresins

	CO 100	BPO 100	MO 100	CO 80	BPO 80	MO 80	CO 60	BPO 60	MO 60	CO 40	BPO 40	MO 40
Poultry	100	100	100	80	80	80	60	60	60	40	40	40
Soyabean meal	–	–	–	10 (2%)	10 (2%)	10 (2%)	20 (4%)	20 (4%)	20 (4%)	30 (6%)	30 (6%)	30 (6%)
Protein	–	–	–	10	10	10	20	20	20	30	30	30
Water	30	30	30	30	30	30	30	30	30	30	30	30
Salt	2,2	2,2	2,2	2,2	2,2	2,2	2,2	2,2	2,2	2,2	2,2	2,2
Oleoresin	0,1	0,1	0,1	0,1	0,1	0,1	0,15	0,15	0,15	0,2	0,2	0,2
Smoke	–	–	–	0,2	0,2	0,2	0,2	0,2	0,2	0,2	0,2	0,2

Minced poultry from each treatment was formed into sausages using a meat former. The sausages were boiled in a vessel at a water temperature of 75±1°C until a core temperature of 71±1°C was reached. After cooling in iced water, the sausages were stored at 5±1°C for 13 days.

Microbiological analysis of chicken sausages was carried out on days 0, 4, 7 and 13 of storage. At each sampling day, three independent samples from three different manufactured date for each treatment were analyzed.

10 g of each sample were aseptically placed into a stomacher bag. Afterward, 90 ml of Peptone Saline Solution (PSS) was added and homogenized using a stomacher for 60 s at

room temperature. Serial 10-fold dilutions were prepared by diluting 1 ml of homogenate in 9 ml of PSS.

Serial decimal dilutions were inoculated (1 ml) onto nutrient agar for QMAFAnM and onto Sabouraud agar for yeasts and molds. Plates were incubated at 30 ± 1 °C for 72 h for QMAFAnM and 24 ± 1 °C for 120 h for yeasts and molds.

Coliforms were determined on nutrient medium Kessler after incubation at 37 ± 1 °C for 48 hours. After that, one loop of nutrient medium was streaked onto the surface of Endo agar and incubated at 37 ± 1 °C for 48 h.

Staphylococcus aureus was determined on Saline Solution after incubation at 37 ± 1 °C for 48 hours. After that, was streaked on Baird Parker agar and incubated at 37 ± 1 °C for 48 h.

Sulfite-reducing clostridia were determined on Iron sulfite agar (Wilson-Blair) after incubation at 37 ± 1 °C for 3 days.

Proteus was determined on nutrient broth. After incubation at 37°C for 48 h the one loop was streaked on nutrient agar and incubated at 37°C for 48 h.

Presence of *Listeria monocytogenes* was determined by suspending 25 g of sausage into 225 ml enrichment medium with reduced concentration of selective agents (half-Fraser broth) with incubation at 30 ± 1 °C for 24 h. Then one loop was re-seeding in a selective liquid enrichment medium with full concentration of selective agents (Fraser broth) and streaked on PALCAM agar and incubated at 37 ± 1 °C for 48 h. After incubation Fraser broth one loop was streaked on PALCAM agar and incubated at 37 ± 1 °C for 48 h.

Presence of *Salmonella* was determined by suspending 25 g of sausage into 225 ml buffered peptone water followed by incubation at 37 ± 1 °C for 20 h. Then the culture was re-seeding on Rappaport-Vassiliadis medium and incubated at 37 ± 1 °C for 24 h. After that, one loop was streaked onto the surface of two selective solid media: Brilliant green agar and bismuth sulphite agar, both incubated at 37 ± 1 °C for 48 h.

After incubation, two plates with nutrient agar and Sabouraud agar for each sampling point were counted. Results were expressed as a number of colony forming units per gram (cfu/g). The article contains average meaning of three independent samples from three different manufactured date for each treatment. All plates were examined visually for typical colony types and morphological characteristics that were associated with each growth medium.

The lowest detection limit for QMAFAnM, yeasts and molds analysis was <10 cfu/g, for coliforms, *Staphylococcus aureus*; *Proteus*; Sulfite-reducing clostridia; *Salmonella* and *Listeria monocytogenes* the detection limit of which were absence in 1,0; 0,1; 0,01; 25 g, respectively.

Results and discussion

The changes in the QMAFAnM during storage are shown in Figs. 1, 2, 3 and 4.

There was no significant difference between initial QMAFAnM for all samples after the thermal treatment (day 0). It characterizes homogeneity of the initial condition.

QMAFAnM for CO samples increase during entire research. At the end of storage, the count was increased to $1,1-8,5 \times 10^5$ cfu/g and was significantly higher than the other samples, except CO40. It is attributable to low functional effect of this oleoresin component.

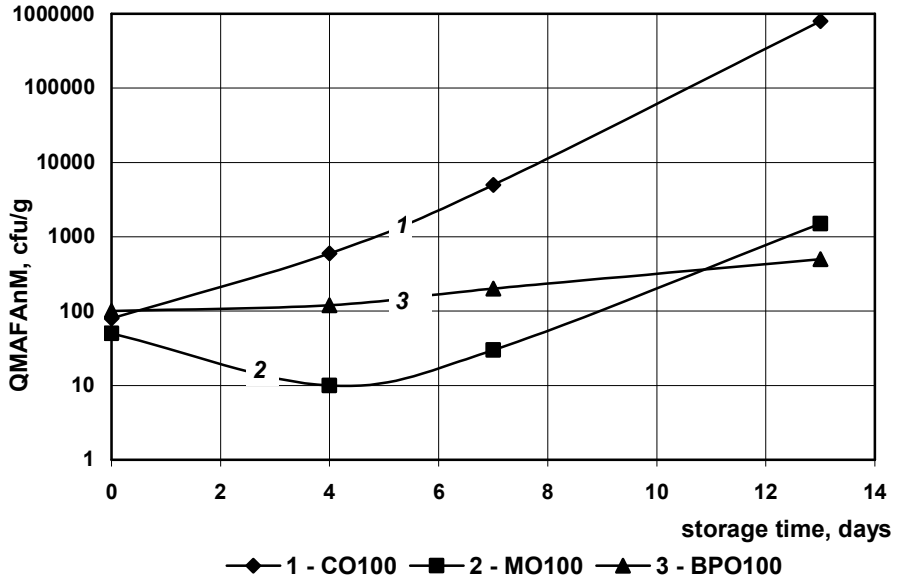


Fig. 1 Changes in QMAFAnM in cooked poultry sausage with 100% of meat stored under refrigeration ($4\pm 1^\circ\text{C}$) during 13 days

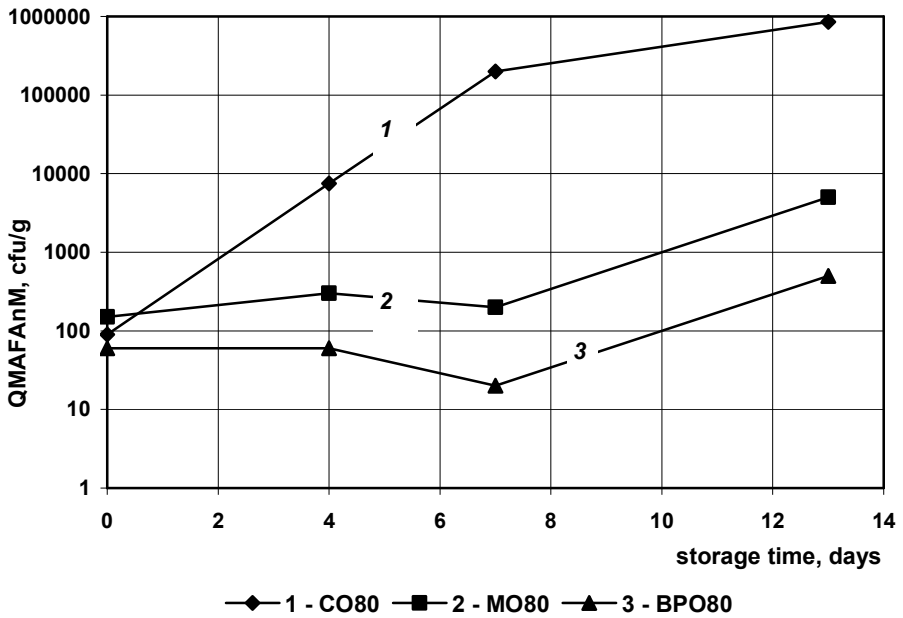


Fig. 2 Changes in QMAFAnM in cooked poultry sausage with 80% of meat stored under refrigeration ($4\pm 1^\circ\text{C}$) during 13 days

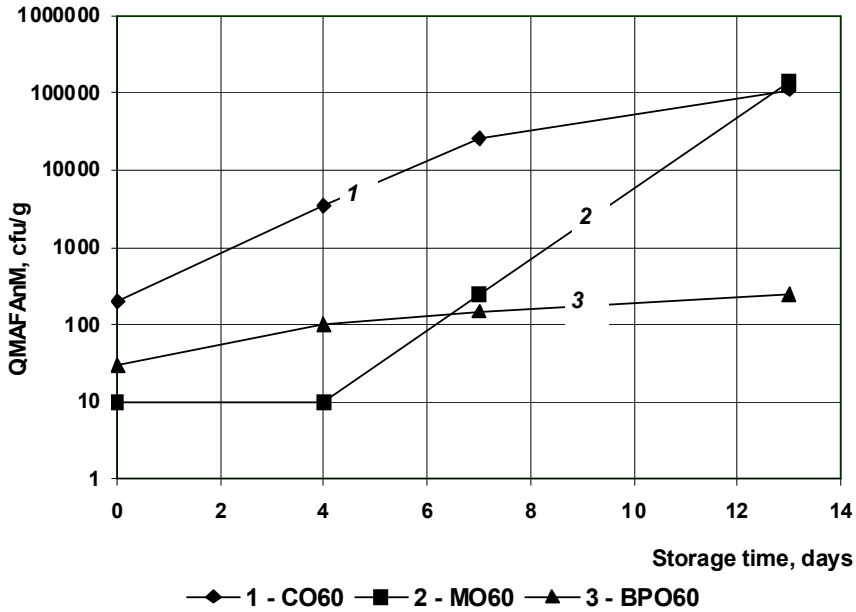


Fig. 3 Changes in QMAFAnM in cooked poultry sausage with 60% of meat stored under refrigeration ($4\pm 1^\circ\text{C}$) during 13 days

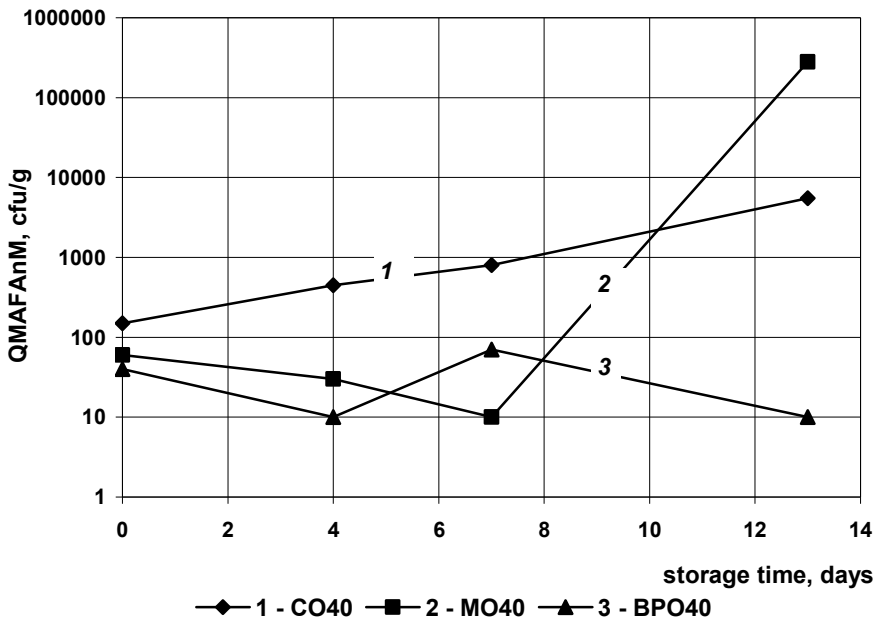


Fig. 4 Changes in QMAFAnM in cooked poultry sausage with 40% of meat stored under refrigeration ($4\pm 1^\circ\text{C}$) during 13 days

The QMAFAnM in MO samples was initially $1,0 \times 10^1 - 1,5 \times 10^2$ cfu/g and was maintained at this level until the seventh day of storage. However, after 13 days the increase of QMAFAnM was significant and was more expressive in meat-containing samples than in the meat products. QMAFAnM shift character with MO addition shows this oleoresin as possible bacteriostatic agent.

Numbers QMAFAnM recovered from meat and meat-containing samples treated with MO and BPO were not significantly different from the numbers recovered from sample with 100% meat raw material.

Mace oleoresin and black pepper oleoresin were more effective than coriander oleoresin on reducing QMAFAnM.

After 13 days of storage, the BPO samples contributed to significantly lower QMAFAnM count than the CO and MO samples.

Sausages with BPO showed stable meaning of QMAFAnM during storage time, that pointed to BPO bactericidal effect and afford its recommendation as basic component for oleoresins mix, including meat containing products processing. It's approved by the fact that QMAFAnM was significantly lower for BPO40 samples as compared to all other treatments during storage.

Of the treatments examined in the present study, black pepper oleoresin was the most effective for the inhibition of QMAFAnM during storage.

The initial population of moulds (Table 2, 3) was <10 cfu/g while on day 4 of storage a count of $2,5 - 7,0 \times 10^1$ cfu/g was recorded for treatments with CO.

Table 2

Changes of moulds in meat products during refrigeration storage

Days of storage	Moulds, cfu/g					
	CO100	BPO100	MO100	CO80	BPO80	MO80
0	<10	<10	<10	<10	<10	<10
4	$2,5 \times 10^1$	<10	<10	$5,5 \times 10^1$	<10	<10
7	<10	<10	<10	$3,2 \times 10^2$	10	<10
13	<10	$2,0 \times 10^1$	<10	$1,0 \times 10^3$	<10	<10

Table 3

Changes of moulds in meat-containing products during refrigeration storage

Days of storage	Moulds, cfu/g					
	CO60	BPO60	MO60	CO40	BPO40	MO40
0	<10	<10	<10	<10	<10	<10
4	$5,0 \times 10^1$	<10	<10	$7,0 \times 10^1$	<10	<10
7	$3,0 \times 10^1$	<10	<10	<10	<10	<10
13	$3,0 \times 10^1$	<10	$2,5 \times 10^1$	$4,0 \times 10^1$	<10	<10

CO samples demonstrated moulds increase during storage, so coriander oleoresin did not inhibit moulds.

MO and BO samples demonstrated stable meaning of moulds during entire research, only on 13th day of storage BPO100 and MO60 samples showed $2,0 \times 10^1$ and $2,5 \times 10^1$ cfu/g respectively.

The development of the yeasts for the samples is shown in Table 4, 5.

Table 4

Changes of yeasts in meat products during refrigeration storage

Days of storage	Yeasts, cfu/g					
	CO100	BPO100	MO100	CO80	BPO80	MO80
0	<10	<10	<10	<10	$3,0 \times 10^1$	<10
4	$3,0 \times 10^1$	10	<10	$2,3 \times 10^2$	$2,5 \times 10^1$	10
7	$1,0 \times 10^2$	<10	<10	$1,5 \times 10^2$	<10	<10
13	$1,5 \times 10^3$	10	<10	$4,5 \times 10^3$	<10	10

Table 5

Changes of yeasts in meat-containing products during refrigeration storage

Days of storage	Yeasts, cfu/g					
	CO60	BPO60	MO60	CO40	BPO40	MO40
0	$2,0 \times 10^1$	<10	<10	<10	<10	<10
4	$9,0 \times 10^1$	<10	<10	$8,0 \times 10^1$	<10	<10
7	$5,5 \times 10^2$	<10	<10	<10	10	10
13	$5,5 \times 10^2$	$2,0 \times 10^1$	10	$3,5 \times 10^1$	$2,5 \times 10^1$	<10

Yeasts from MO and BPO samples did not differ after 7 d of storage, but were significantly lower than counts from CO samples.

Yeasts from CO samples increased during entire storage.

Sausages with MO showed stable meaning of yeasts during storage time.

Initial meaning of BPO80 yeasts was $3,0 \times 10^1$ cfu/g, although on 7th day of storage inhibition of yeasts was observed.

The samples with BPO and MO had lower yeasts counts than CO samples during the entire storage period.

Concerning yeasts and moulds, no significant differences between all treatments with BPO and MO were observed.

Mace oleoresin and black pepper oleoresin was a lot more effective in reducing yeast and moulds populations than coriander oleoresin.

The sample with CO showed a higher QMAFAnM, yeasts and molds count than the samples with MO and BPO. The mentioned microbial groups are considered as spoilage microorganism, and their presence in high amounts could affect the organoleptic properties of the samples. The relatively high population of yeasts and molds also may cause the formation of slime and greening on the sample surface. Therefore, it confirmed more powerful inhibitory action of oleoresins on QMAFAnM as well as yeasts and molds development.

In this work, the presence of coliforms, *Salmonella*, Sulfite-reducing clostridia, *Listeria monocytogenes*, *Proteus*, *Staphylococcus aureus* were not detected in any sausage samples, regardless of storage time (data not shown).

Conclusions

Meat and meat containing systems with coriander oleoresin demonstrated rapid increase of QMAFAnM, moulds and yeasts during entire research, so coriander oleoresin application in meat-containing products processing as microbial stabilizing agent is not recommended.

Meat systems with mace oleoresin kept microbiological stability during 10 days, meat containing products kept microbiological stability during 8 days. That's why for meat and meat containing systems with mace oleoresin could be recommended shelf life 7 and 5 days respectively.

Samples with black pepper oleoresin kept microbiological stability during all storage time. For meat and meat containing systems with black pepper oleoresin could be recommended shelf life 10 days.

Mace oleoresin and black pepper oleoresin have more antimicrobial activity than coriander oleoresin.

Only BPO has shown antimicrobial effect during refrigeration storage more than 10 days.

Offered shelf life for samples containing mace and black pepper oleoresins is in accord with trivial recommendation for cooked sausages.

In processing meat containing products with oleoresins mix inhibiting microorganisms growth it's necessary to make emphasis on black pepper oleoresin addition.

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Mathematical modelling of the separation of suspension process on the filter with self-purifier filter element

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Abstract

Keywords:

Filtering
Suspension
Pores
Clogging
Capillaries
Regeneration

Article history:

Received 20.12.2015
Received in revised form
26.02.2016
Accepted 24.03.2016

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Introduction. The aim of the work was to build a mathematical model of the process of suspension filtering on the filter with self-purifier filter element that is designed as a cylindrical compression spring.

Materials and methods. Research of the filtering process was performed on the filter with self-purifier filter element. As a studied suspension the milk whey was used, obtained in the production of cottage cheese. The concentration of the dispersed phase in the milk whey was defined by centrifugation of samples followed by further drying sludge in a drying oven.

Results and discussion. The gained mathematical model is based on the model of the filtering process with clogging each pore with the individual particle.

The model takes into account that not all of the dispersed phase particles that are larger than the width of the filter holes, will clog them, but only their particle that is directly proportional to the ratio of living area to the total area of the filter surface.

The mathematical model allows to determine the length of the filtering process based on the volume of suspension, and to set the rational period between regenerations of self-purifier filter element.

Comparing the parameters obtained by mathematical modelling with the real process of filtering milk whey indicates that the mathematical model adequately reflects the separation process of suspension on the filter with self-purifier filter element with the volume of filtrate from 0 to 5 m³ per 1 m² filter surface - the average relative deviation of the results obtained with the help of the mathematical model of the experiment is 11%.

Conclusions. The mathematical model can be applied in calculating parameters of the process of suspension filtering on the filter with self-purifier filter element.

Introduction

Regimes of suspensions filtering process are due to the following main factors: properties of the suspension, properties of the filtration surface and the design properties of the filter. Regarding filtration properties of the sludge, the impact of both properties of dispersed environment [1-4] and dispersion [5, 6] should be noted.

Filtering through a layer of sludge is described by Darcy [7]:

$$W = \frac{1}{\mu r_0} \cdot \frac{\partial P}{\partial h}, \quad (1)$$

where W – speed of filtering, m/sec;
 r_0 – filtering resistivity, m^{-2} ;
 μ – dynamic viscosity of suspension, Pa·sec;
 P – pressure, Pa;
 h – height of sludge layer, m.

In the mathematical description of the filtering process the compressibility of the sludge and continuity equation of the solid and liquid phase are also taken into account [8]:

$$\frac{\partial W}{\partial h} = -\frac{\partial \varepsilon}{\partial \tau}, \quad (2)$$

where ε – porosity of the sludge layer;
 τ – duration of filtering, sec.

For consideration of sludge's compressibility the law of compression can be used under which the infinitely small change in sludge's porosity is directly proportional to the infinitely small change of pressure:

$$G = \frac{\partial e}{\partial P}, \quad (3)$$

where G – compressibility module of sludge, Pa;
 e – coefficient of porosity.

Solving this equation considering filtration and compression properties of sludge is given in the work [8]

Also [8] the work highlights the need for representation filtering equation as follows:

$$W - e\sigma = \frac{1}{\mu r_0} \cdot \frac{\partial P}{\partial h}, \quad (4)$$

where σ – speed of the solid phase movement, m/sec.

In the work [1] it is proposed to consider the dependence of the resistivity to filtering of the pressure filtering.

In the work [9] non-Newton properties of the dispersion phase, considering Rut's equation are proposed:

$$\frac{dq}{d\tau} = \left[\frac{\Delta R}{kr_0 x (V - V_e)} \right]^{\frac{1}{n}}, \quad (5)$$

where q – specific volume of the filtrate obtained of the area unit of filter surface, m^3/m^2 ;
 ΔR – hydraulic resistance to the layer of sludge and filter surface, m^{-1} ;
 k – consistency index;
 n – stream index of filtrate;
 x – ratio of volumes of the obtained sludge and filtrate;
 V – volume of filtrate, m^3 ;
 V_e – equivalent volume of filtrate, during the passage of which a sludge layer with resistance to filtering that equals resistance to filter surface may be formed, m^3 .

Secure features of the filtering process are considered in the monograph [1], in particular filtration at a constant speed, constant pressure of the process, etc.

Common to all these works is filtration with a sludge formation on the filter surface and change of the layer of sludge's height. However, structures of filtering machines the height of the sludge layer of which is minimal and unchanged throughout the whole process are known. This includes filtering centrifuges with continuous diversion of sludge and filters with self-purifier filter elements [7]. In this case, resistance to filtering, which creates a layer of sludge, can be considered as resistance of the filter surface, and filtration process as such that proceeds with clogging pores.

Filtering theory [7] in this case considers the following cases: filtering with clogging every pore with a separate solid particle (complete clogging of pores) filtering with a gradual clogging of every pore with many solid particles.

Mathematical model of filtering every pore process with a separate solid particle is presented in the publication [7]. The author proposes to consider that on the filtration surface with area of 1 m^2 is N identical cylindrical capillaries of radius r and height, corresponding to the height of the sludge's layer. The initial speed of filtration is proposed to be determined of the expression:

$$W_0 = AN, \quad (6)$$

where W_0 – initial speed of filtering, m/sec ;
 N – number of capillaries;
 A – volume of filtrate that passes through the capillaries per second, m^3/sec , is found of Hagen–Poiseuille equation:

$$A = \frac{\pi r^4 \Delta P}{8 \mu h}. \quad (7)$$

After passing the filtrate in the amount V the number of the clogged capillaries is the following:

$$N_c = nV, \quad (8)$$

where N_c – number of the clogged capillaries;
 n – amount of the particles of a dispersed phase in one m^3 of suspension that clog capillaries, $1/\text{m}^3$.

In these conditions, the dependence of filtering speed of the volume of filtrate will be described by the equation:

$$W=A(N - nV) \quad (9)$$

However, the formulation of the problem in this form does not allow taking into account design features of filter and suspension.

The aim of the work was to build a mathematical model of the process of suspension filtering with every pore clogging of the self-purifier filter element with one solid particle in the absence of the sludge layer.

Materials and methods

The object of the research was the process of suspension filtering and theoretical description of this process. Theoretical analysis was performed for the installation with self-purifier filter element.

This refers to the self-purifier filter element that is designed as a cylindrical spring of compression, the size of the gap between the turns of which corresponds to the size of the smallest particle of the dispersed phase that should be impeded [10].

Experimental instalment with a cylindrical filter element consisted of a cylindrical body 1 (Fig. 1), nozzles 2 and 3, supply and removal of whey respectively, guide glass 4 with a screw 5, tube to remove sludge 6, filter element 7, ring 8, electromagnet 9, shaft 10, sludge tank 11 and measuring tank 12. Material of the filter element is stainless steel; the gap between the turns of the filter element - 0.9 mm, the proportion of living cut - 38%.

As the pilot suspension the milk whey was used, obtained in the production of cottage cheese in a periodic way (fat content in the finished product - 9%) using cheese-producing baths of brand VS-5000.

Firstly a concentration of dispersed particles of protein in whey was defined. For this case sampling of whey with volume of 500 ml was made every 3 minutes during the whole time of pouring the whey out of the cheese-producing bath. Then all samples were poured into one container, mixed and 12 portions of 5 ml each were selected of the total volume of whey. Then four portions of whey were poured into four tubes, closed with rubber stoppers and placed into a centrifuge of mark OPN-12 with plugs to center. The whey was centrifuged for 5 min at speed of 6000 rev/min (centrifugation duration was measured from the moment of gaining rotational speed - 6000 rev/min). After centrifugation the tubes were removed from the centrifuge, opened and the liquid phase was poured so that only the sludge remained in the tube. The obtained sludge was dried in the oven for 60 minutes at the temperature of 105° C. The dried sludge was weighed. The concentration of dispersed particles of protein in the whey was defined as the ratio of dry sludge to the volume of whey of which it was obtained (2·10⁻⁵ m³). The experiment was repeated three times. The concentration of dispersed particles of protein in the whey was 3.0 kg/m².

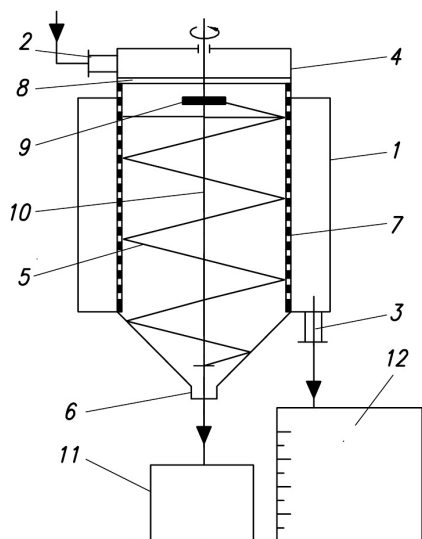


Figure 1. Experimental installation with self-purifier filter element
 1-body; 2-3, respectively tubes of supply and removal of whey; 4-guide glass;
 5-screw; 6-nozzle to remove sludge; 7 self-purifier filter element; 8-ring; 9;
 9- electromagnet; 10-shaft; 11-sludge tank; 12-measuring filtrate tank.

Further purification of the whey was performed. Milk whey was given to the experimental installation directly of cheese-producing bath (without pump) under the pressure of 3.0 kPa. The whey through the pipe 2 was tangentially given to the glass 4. From the glass it passed through the filter element 7. The ring 8 with periodic switch of electromagnet 9 was moved down along the axis of the shaft 10 and thus compressed filter element 7 that ensured its regeneration. The duration of the regeneration was 1 sec. The sludge that remained on the filter element was transported with screw 5 into the bottom of the body 1, where it was removed periodically through the pipe 6 into the tank 11. The filtrate was removed of the filter through the pipe 3 into the measuring tank 12.

Screw rotation rate was 9 rev/min. During the experiment every 10 sec the amount of the sludge was fixed.

After filtering whey samples were collected out of the measuring tank 12, 5 ml each, and according to the methodology described above, the concentration of the dispersed phase in the filtrate was defined. It was 1.6 kg/m³.

Results and discussion

In the mathematical model developing the following assumptions were made: during the accumulation of sludge in the gaps between the turns of the spring the capillaries were formed, diameter of which is equal to the distance between the coils of the spring; the height of the capillary is equal to the wall thickness of the filter element (diameter of the coil spring); dispersed phase particles are uniformly distributed over the entire area of the filter surface.

It was believed that the number of capillaries N is proportional to the living cut area of filter surface:

$$N = \frac{S_l}{\pi r_c^2}, \quad (10)$$

where S_l – living cut area of filter surface, m^2 ;
 r_c – capillary radius, m .

Then we assume that not all of the dispersed phase particles that are larger than the diameter of capillaries will clog them, but only their particle that is directly proportional to the ratio of living cut area to the total area of the filter surface. The remaining particles will be laid on coils of the spring and will be transported by screw. Then n will be:

$$n = n_0 \frac{S_l}{S}, \quad (11)$$

where n – number of the dispersed phase particles in $1 m^3$ of suspension that are larger than pore diameter, $1/m^3$;

S – filtering surface area, m^2 .

We change in equation (9) W to $dV/d\tau$:

$$\frac{dV}{d\tau} = A(N - nV). \quad (12)$$

We divide variables:

$$\frac{dV}{A(N - nV)} = d\tau, \quad (13)$$

$AN = W_0$, mark $A \cdot n = m$.

After integration of equation (13) from 0 to V and from 0 to τ , with boundary conditions $V = 0$ and $\tau = \tau_0$, we obtain an equation for the duration of filtering:

$$\tau = \tau_0 - \frac{1}{A \cdot n} \ln \left(\frac{N - nV}{N} \right), \quad (14)$$

where τ_0 – duration of the filtering with a clean filter surface (none of capillaries is clogged), sec .

Verification of the mathematical model for adequacy was performed on the example of the process of milk whey filtering. Sequence of checking was as follows: experimental study of the milk whey filtering process in production conditions was made; mathematical modelling of the milk whey filtering process was performed; data obtained in the experimental way with the appropriate calculations were compared; relative deviation of the mathematical model from the real process of filtering was defined according to the formula:

$$\Omega = \frac{\sum_{i=1}^j (|X_i - Y_i|)}{\sum_{i=1}^j X_i} \cdot 100\%, \quad (15)$$

where Ω – relative deviation, %;
 X_i – experimental value of filtering duration to filtrate volume V_i , sec;
 Y_i – calculated value of filtering duration (according to the formula (14)) for the filtrate volume V_i , sec;
 j – number of measurements, $j = 10$.

Mathematical modelling was carried out in the following sequence.

1. Number of capillaries was defined according to the formula (10);
2. Average radius of particles larger than the diameter of the pores of the expression was defined:

$$r_{\text{i\ddot{a}d}} = \frac{\sum_{i=1}^3 (r_i g_i)}{\sum_{i=1}^n g_i}, \quad (16)$$

where r_i – average radius of i -fraction of protein particles larger than the width of the filter surface gap;
 g_i – share of i -fraction protein particles, %.

3. average weight of one protein particle was found:

$$m_{p.av.} = \frac{3}{4} \pi (r_{c.av.})^3 \rho_p, \quad (17)$$

where $m_{p.av.}$ – average weight of protein particles larger than the diameter of the pore, kg;
 $r_{c.av.}$ – average radius of capillaries, m;
 ρ_p – density protein, kg/m³.

4. Number of particles in 1 m³ of suspension larger than the size of the pore was defined with the help of the formula:

$$n_0 = \frac{m_{\Sigma}}{m_{p.av.}}, \quad (18)$$

where m_{Σ} – total mass of particles larger than the diameter of the pore, kg.

5. Number of the dispersed phase particles in 1 m³ of suspension that clog the pores according to the formula was defined (11);

6. Volume of the filtrate that passes through the capillary in one second according to the equation was found (7);

7. Length of the filter with a clean filtration surface was defined:

$$\tau_0 = \frac{V}{FW_0}, \quad (19)$$

where F – area of filter surface, m².

8. Calculation of the duration of the filtering process at $V = 0, 1, 2, 3 \dots 10 \text{ m}^3$ according to the formula (14) was performed.

Comparison of the data obtained by mathematical modelling with the real filtering process of milk whey (figure 2) shows that the mathematical model adequately reflects the process of suspension separation on the filter with self-purifier filter element with the specific volume of filtrate up to $5 \text{ m}^3/\text{m}^2$ - the average relative deviation of the results obtained with the help of the mathematical model of the experiment is 11%. With further increase of the specific volume of the filtrate the relative deviation of the mathematical model increases.

As the concentration of dispersed particles in suspension remains stable and the number of unclogged filter openings is inversely proportional to the volume of filtrate, at a constant speed of filtering the process of clogging is eventually accelerated. So, the lines showing the filter length dependence on the volume of filtrate are of non-linear character (figure 2).

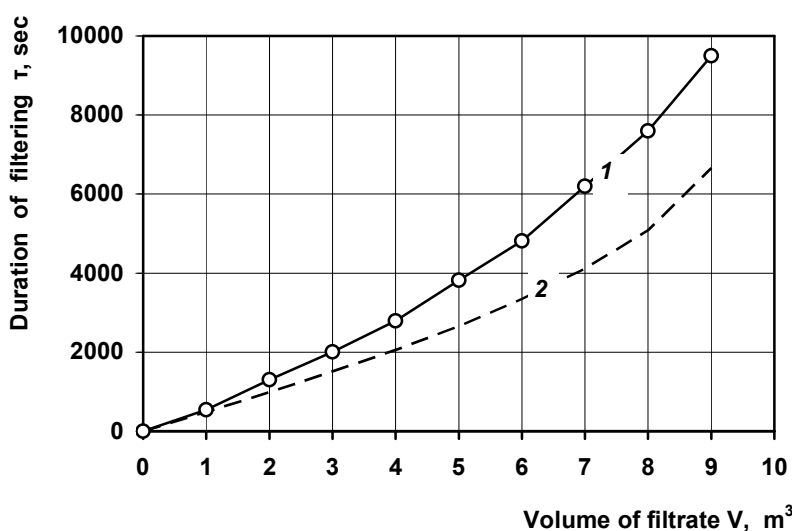


Figure 2. Dependence of filtering milk whey duration filtrate on the volume of filtrate for 1 m^2 of filter surface:

1 – experimental data; 2 – calculation according to the formula (14).

Conclusions

The proposed mathematical model of suspension filtering process with every pore clogging of self-purifier filter element with a separate particle at absence of sludge layer allows to predict the duration of filtering depending on the volume of the obtained filtrate and to set a rational value of regeneration period of the filter element.

The mathematical model adequately reflects the separation process of suspension on the filter with self-purifier filter element with at the volume of filtrate from 0 to 5 m^3 per 1

m² of filter surface - average relative deviation of results obtained with the help of the mathematical model of the experiment is 11%.

It can be used in calculation of the regeneration process of self-purifier filter element and when designing new filter installations.

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Kinetic laws of the process of obtaining complex humic-organic-mineral fertilizers in the fluidized bed granulator

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Abstract

Keywords:

Sunflower
Fertilizer
Fluidization
Granulation
Humate
Ash

Article history:

Received 20.12.2015
Received in revised
form 27.02.2016
Accepted 24.03.2016

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Introduction. The main part of wastes from the sunflower oil production is sunflower ash that contains useful substances. The aim of the work to determine kinetic laws of the process of obtaining complex organic-mineral granulated fertilizers using the sunflower ash.

Materials and methods. Dehydration and granulation of liquid heterogeneous systems that contained mineral, humic substances and sunflower ash were held in the fluidized bed apparatus equipped with a special gas distribution device for creating a jet-pulsating mode of fluidization by supplying gas heat carrier.

Results and discussion. A stable kinetics of granulation process of the humic-organic-mineral fertilizers which contain $K:N:Ca:P:Mg:S:Hum.=23:9:5:2:6:15:2$ with a coefficient of granule formation $\psi \geq 90\%$ was achieved with an average meaning of the heat carrier temperature difference at the entrance to the granulator and in fluidized bed $\Delta T=117^\circ C$. The obtained (resulting) product has a spherical shape, a uniform distribution of components at the micro level throughout the volume of granules, strength $\sigma \geq 35$ Newtons per granule that is more than 3 times higher than standard indicators. An increasing of the average specific load of bed's surface by moisture divided by the efficient temperature difference $A_f = 0,006 \div 0,0066 \text{ kg}_{\text{moisture}} / (\text{m}^2 \cdot \text{h} \cdot \text{deg})$ was achieved when applying a jet-pulsating hydrodynamic mode of fluidization.

The research results can be applied when creating an industrial equipment for production of humic-organic-mineral fertilizers with the use of mineral and organic nutrients. The use of sunflower ash in creating of new humic-organic-mineral fertilizers will provide rational usage of natural resources with the preservation of natural food chain and will improve the environmental safety as a result of recycling of wastes from fat and oil production.

Conclusions. The developed method allows to utilize wastes of sunflower oil production by their use in the producing of new complex humic-organic-mineral fertilizers.

Introduction

A dynamic development of food industry is accompanied by increasing of food products realization volumes, among which fat and oil industry occupies a special place [1] and is focused on the production of sunflower oil and allied products. Sunflower occupies more than 90% of the total oilseeds production in Ukraine and at least 10% of the sowing areas structure [2].

By-product of sunflower oil production is an oil meal and an oil cake that constitute 17-20% from the initial seed weight. However, in order to reduce an energy costs by the past 10 years almost all large oil and fat combines and oil-extraction plants of Ukraine have implemented technology of husk burning and pellets or briquettes from it, which is 80% or 312 tons per year.

Nevertheless, the quantity of residues (ash) after husk burning reaches up to 10% of the total volume - 31.2 thousand tons per year [3], which contains useful substances.

Thus, from an environmental point of view, the need for rational utilization of sunflower ash arises. The main components that belong to its composition, constitute 95.67% of the total mass, table 1, the rest (4,33%) are Zn, C, Co, Mn, Fe, Mo [4].

Table 1

The chemical composition of sunflower ash

Chemical compounds	K ₂ O	CaO	MgO	SO ₃	P ₂ O ₅
wt. %	31,40	19,07	18,58	13,68	10,94

Presence of potassium and phosphate components deficiency in soils, which increases significantly while growing sunflowers and ether-oil crops, stipulates an advisability of ash returning to an agricultural cycle in form of fertilizers. Moreover, a large removal of nutrients from the soils causes the necessity in restoration of their fertility. Therefore, one of the ways of using wastes from oil and fat industry after burning is the creation of organic-mineral fertilizers, into which structure nitrogen containing components and humic substances are additionally included.

There are known methods of a sunflower ash water solution granulation in rotating drum granulators [4-7]. However, the lack of nitrogen-containing components and humic substances reduces the effectiveness of their use.

The firm "Ecoplant" [8] has organized a production of complex granulated fertilizers by pressing mixture, which is composed of sunflower ash, ammonium sulfate and humic-containing substance (brown coal). However, in this case the components distribution occurs at the macro level and the final product has a low strength and great ability to clumping. Furthermore, the addition of water causes the formation of calcium hydroxide, which reacts with ammonium sulfate or with carbamide and causes an intensive allocation of ammonia.

As world practice shows, the compensation of the soil fertility losses by means of using the mineral fertilizers with an increasing number of active substance up to 500 kg per hectare does not give desired results [9-12]. Therefore, the use of organic-mineral and especially humic-organic-mineral fertilizers, which contain nutrients of an organic origin refers to an effective ways of soil fertility preservation [13].

Materials and methods

To create a composite humic-mineral fertilizers containing NPK, macro and micro impurities of mineral and humic substances a method of obtaining solid humic and mineral fertilizers by dehydration of composite liquid systems in a fluidized-bed apparatus is developed [Patent of Ukraine 4465 IPC C05 G 1/01. A method for production of granulated organic-mineral fertilizers].

Thanks to a specially developed method and construction of the granulator realization of the granulation mechanism with a layer structure ensures a uniform distribution of mineral and organic components throughout the volume of granules [14].

The aim of experimental researches is determination of conditions of sunflower ash utilization and kinetic regularities of the process of obtaining granulated comprehensive organic-mineral fertilizers.

For studying the kinetics of a granule formation process was created the sample of an experimental-industrial equipment with a chamber of granulator size $A \times B \times H = 0,1 \times 0,3 \times 1,5$ m (Fig. 1), that was equipped with a special gas distribution device with sizes $A \times B = 0,1 \times 0,3$ m for creating a jet-pulsating regime of fluidization. [Patent of Ukraine 84680 IPC B01 J 8/44. Section of the fluidized bed apparatus].

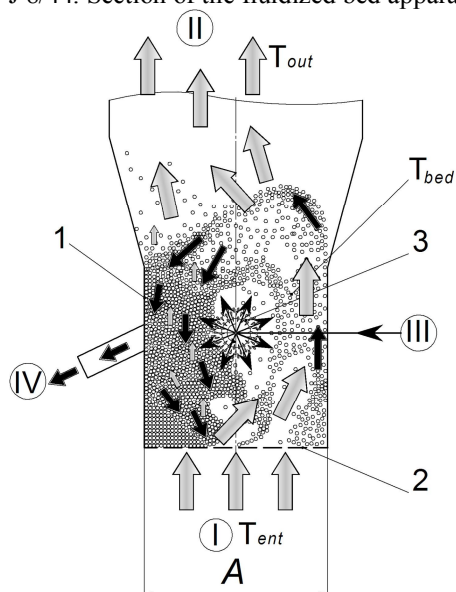


Fig.1. Scheme of the process in chamber of the granulator:

- I – inlet heat carrier; II – outlet heat carrier;
- III – initial solution; IV – granulated product;
- 1 – chamber of granulator; 2 – gas distribution device (GDD); 3 – dispergator;

As the heterogeneous liquid phase a water solution of ammonium sulfate (AS) with impurities of humates (H), sunflower ash (SA) and bentonite (B) was used.

All of the experiments were performed by the condition of keeping a constant bed mass in the chamber of granulator and was expressed by hydraulic resistance of bed $\Delta P_{bed} = 1962$ Pa (± 50) which was fixed by indications of a differential manometer. Every excess of a granular material was unloaded from the bed of granulator.

An interaction of AS with a $\text{Ca}(\text{OH})_2$ compound (which is formed when adding water to SA) in the initial solution causes the allocation of ammonia. To prevent this a certain amount of an acid must be added to it for the formation of useful calcium compounds. Beside this, in order to reduce deposition rate of the suspended particles of SA to the initial solution is added 15% of bentonite. Ratio of mass percent of dry components in the solution is $[\text{SA}]:[\text{AS}]:[\text{B}]:[\text{H}]=25,6:21,4:1,5:1,0$. The component composition of inlet liquid systems for dehydration in apparatus with a fluidized-bed are given in Fig. 2.

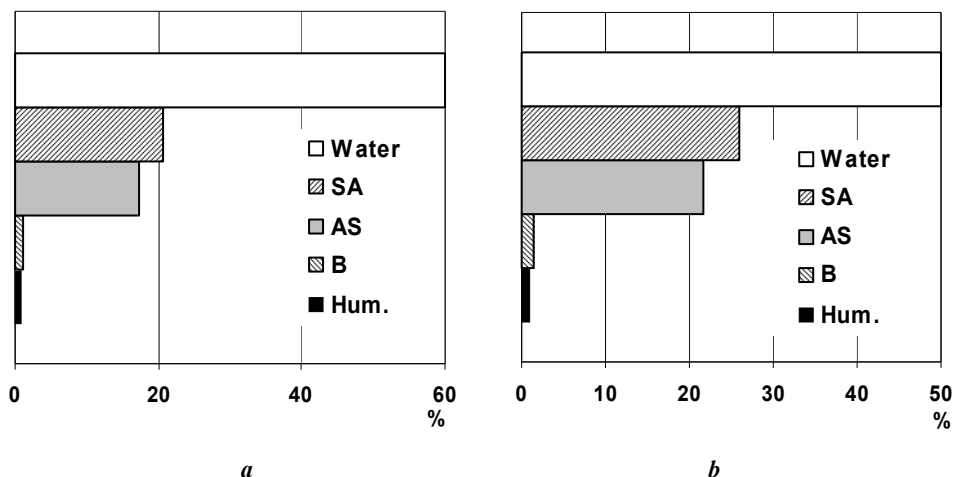


Fig. 2. Diagrams of the component composition of composite liquid systems: *a* - research №1; *b* - research №2

Solids content in a liquid phase is determined by rheological properties of the initial solution and ensuring of the layering mechanism of granule formation with "onion" structure [15, 16].

From the point of view on dehydration efficiency of the process a water content must be reduced to its minimum ($\leq 40\%$), but in this case there are difficulties with realization of the layering mechanism of granulation. Therefore, the influence of this parameter will be more thoroughly investigated in subsequent experiments, associated with development of a special unit of entering the initial solution.

As an initial granulation centers were used the granules with equivalent diameter $D_e = 2,3$ mm consisting of ammonium sulfate with impurities of humic substances [17].

Initial working solution III was injected in the fluidized bed by mechanical dispergator 3. Heat carrier temperature at the entrance to granulator ($T_{ent.}$) was maintained at the range $T_{ent.}=200\pm 10^\circ\text{C}$. The temperature of a layer of granular material (T_{bed}) was fixed by an electronic potentiometer and was holding in the set range $T_{bed} = 96 \pm 4^\circ\text{C}$ by injecting of a liquid phase III.

The coefficient of granule formation (ψ) was calculated by the equation, %:

$$\psi = G_{dry} / G_{g.p.} \cdot 100\%$$

where $G_{g.p.}$, G_{dry} – productivity by a granulated product and by dry substances which are enjected to the apparatus with a liquid phase III respectively [18], kg/h.

Estimation of efficiency of the granule formation process when applying the hydrodynamic regime of fluidization is a value of specific load of the surface of bed by moisture [19] divided by the useful temperature difference (ΔT), $\text{kg}_{\text{moist.}}/(\text{m}^2 \cdot \text{h} \cdot \text{deg})$:

$$A_f = G_{\text{moist.}} / (f_{\text{bed}} \cdot \Delta T),$$

where $G_{\text{moist.}}$ – moisture consumption enjected to the apparatus with an initial working solution III, $\text{kg}_{\text{moist.}}/\text{h}$; f_{bed} – the total surface of a bed of granular material, m^2 ; $\Delta T = T_{\text{ent.}} - T_{\text{bed}}$ – the useful temperature difference, $^{\circ}\text{C}$.

A generalization of results of an experimental researches was carried out as a dependence of the coefficient of granule formation (ψ) from the complex $\Delta P_{\text{bed}}/(g \cdot D_e)$ and from the number of fluidization (K_w) – $\psi = f(\Delta P_{\text{bed}}/(g \cdot D_e); K_w)$, which allows to identify areas of rational realization of the process.

Results and discussion

Dynamics of changes of the equivalent diameter of granules D_e is shown in Fig. 3.

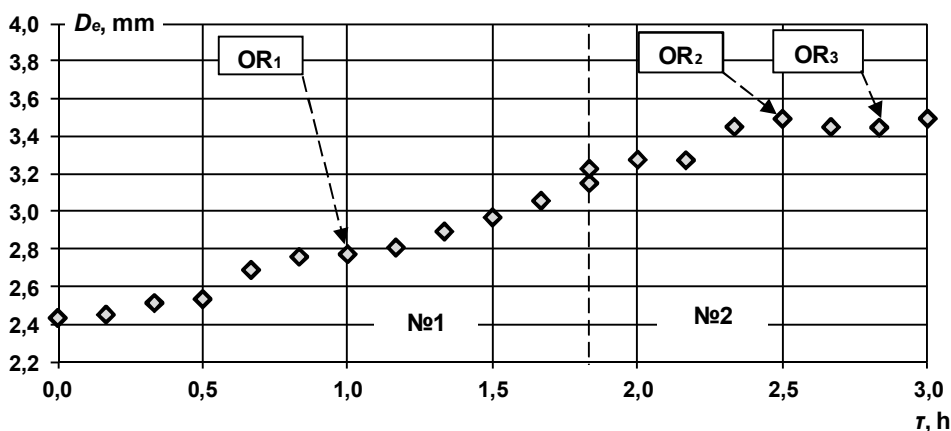


Fig. 3. Dynamics of changes of the equivalent diameter of granules $D_e = f(\tau)$:
OR₁, OR₂, OR₃ – marks of the recycle injection

When injecting a 40% initial water solution (research №1) there was a gradual increase of the granules diameter from $D_e = 2,43$ mm to $D_e = 3,15$ mm with a growth rate $+\frac{dD}{d\tau} = 0,35$ mm/h, time $0,00 \leq \tau \leq 1,83$ h, which indicates a stable kinetics of granule formation, Fig. 3.

The layering mechanism of granule formation confirmed by the dynamics of changing of mass percent of certain fractions Fig. 4. Namely, in the considered time range a decrease of fraction +2,0 mm is accompanied by a corresponding increase of the next fraction by size – +3,0 mm.

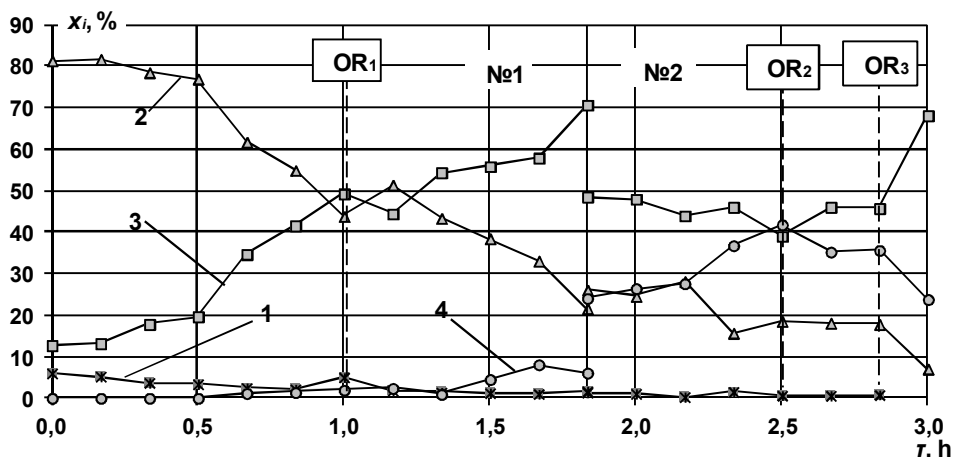


Fig. 4. Dynamics of changing in mass content percent of certain fractions $x_i=f(\tau)$ with the size of granules: 1 – +1,0 mm; 2 – +2,0 mm; 3 – +3,0 mm; 4 – +4,0 mm; OR₁, OR₂, OR₃ – marks of the recycle injection

Injection of the outer recycle (OR) for stabilization of the dispersed composition at $\tau = 1,0$ h (Fig. 3, 4) resulted the temporary increase of mass percent of the fraction +2,0 mm at $\tau = 1,17$ h (OR₁). However subsequently, after $\tau = 1,17$ h and to completion of the research №1 there was an increase of mass percent of fraction +3,0 mm to 70%, and the content of the fraction +2,0 decreased to 20%, Fig. 4. This gradual transition of granules from the smaller fractions to bigger ones demonstrates the layering mechanism of granule formation and the absence of crushing granules.

After $\tau=1,83$ h D_e was instantly changed to 3,23 mm by injection of the outer recycle. A 50% initial water solution was injected for dehydration (research №2). However, further there was a gradual increase of the granules diameter from $D_e = 3,23$ mm to $D_e = 3,49$ mm with a growth rate $+\frac{dD}{d\tau} = 0,389$ mm/h, time $1,83 \leq \tau \leq 2,50$ h, that increased proportionally with increasing concentration of the initial solution. After injection of OR₂ at time $\tau = 2,50$ h was observed insignificant decrease of D_e to 3,45 mm was observed by increasing of a mass percent of fraction + 2,0 mm. A disperse composition of granules stabilized after $\tau = 2,66$ h to $D_e = 3,45$ mm.

The feature of a granule formation process is that it was achieved a coefficient of granule formation $\psi \geq 90\%$ (Fig. 5) while was applying the jet-pulsating regime of fluidization with the number of fluidization $K_w \leq 1,43$ (Fig. 6).

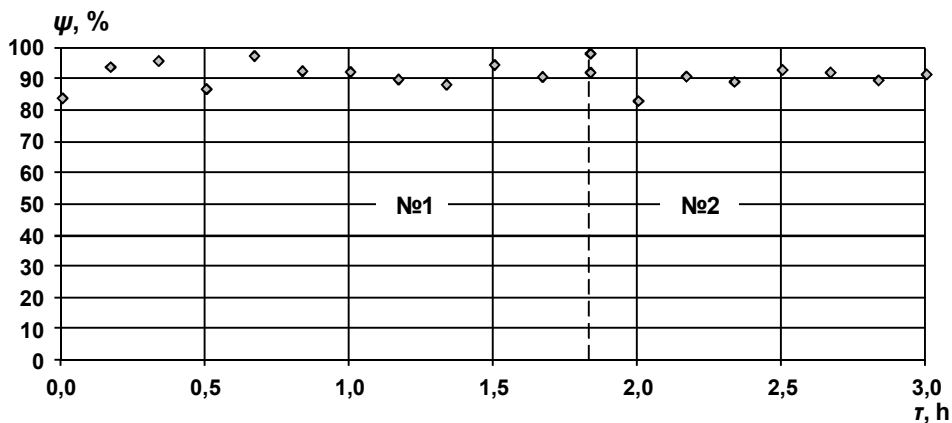


Fig. 5. Dynamics of changes of granule formation coefficient $\psi=f(\tau)$

Another advantage of application of the fluidization technology is possibility of injecting a heat carrier into the working area with a high temperature, which significantly exceeds the melting point of ammonium sulfate.

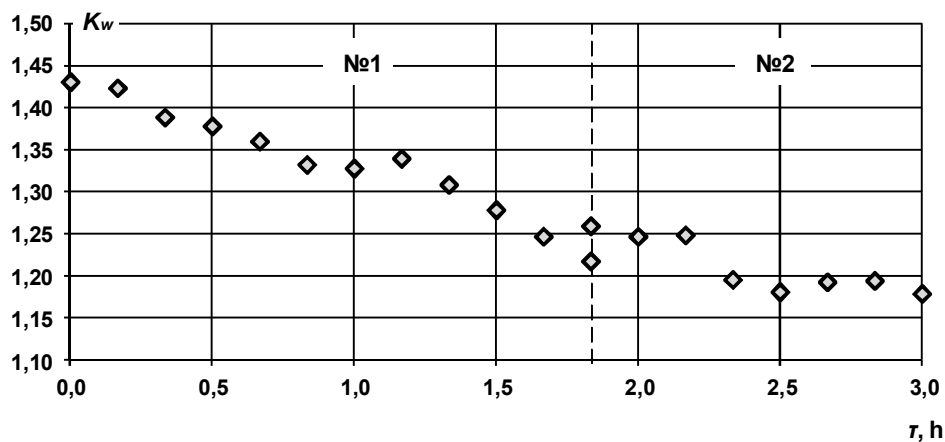


Рис. 6. Dynamics of changes of the number of fluidization $K_w=f(\tau)$

Dynamics of changes of a heat carrier temperature at the entrance to the apparatus, in a fluidized bed of granular material and the efficient temperature difference are shown in Fig. 7.

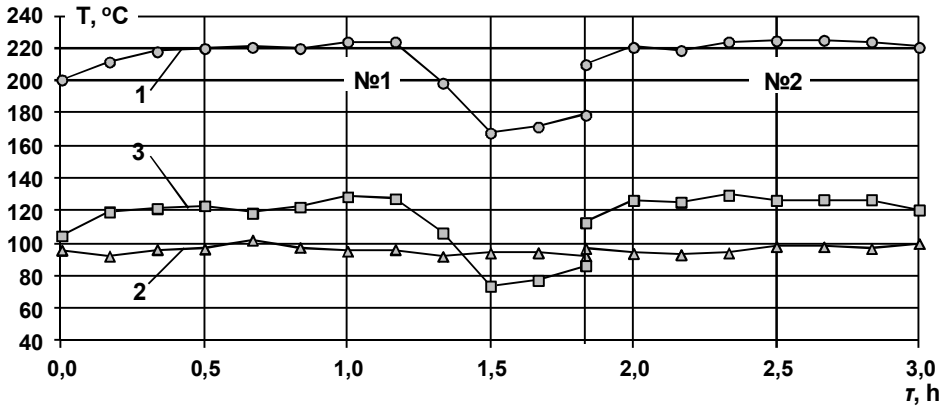


Fig. 7. Dynamics of temperature changes in the granulator:

1 – $T_{ent.}$ (temperature of heat carrier at the entrance to granulator); 2 – T_{bed} (temperature in a fluidized bed); 3 – ΔT (an efficient temperature difference ($\Delta T = T_{ent.} - T_{bed}$)).

The obtained values of the average specific load of the surface of bed by moisture divided by the efficient temperature difference $A_f = 0,006 \div 0,0066 \text{ kg}_{\text{moist.}}/(\text{m}^2 \cdot \text{h} \cdot \text{deg})$ confirm the effectiveness of the applying of a jet-pulsating regime of fluidization (Fig. 8). However, it is necessary to repeat researches in future to confirm the results obtained in the research №2 in which the maximum value A_f is almost 2 times higher than a value that were obtained for the ordinary bubbling fluidization regime [20].

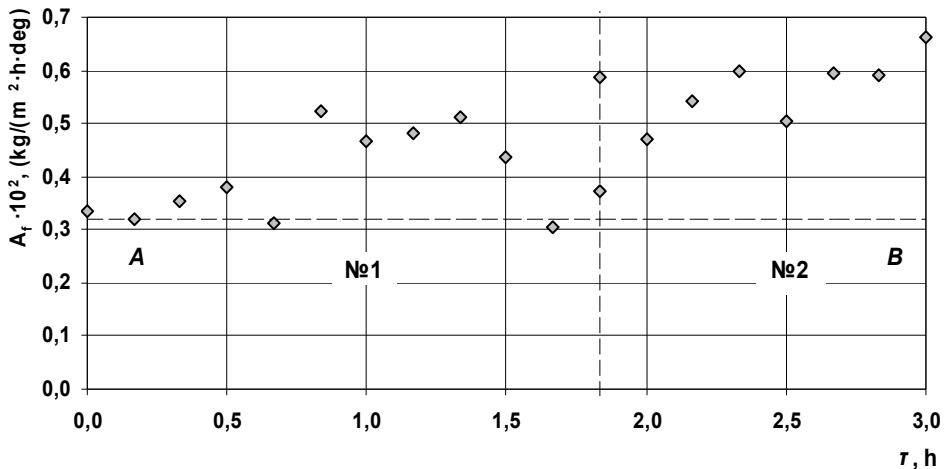


Fig. 8. Dynamics of changes of the average specific load of the surface of bed by moisture divided by the efficient temperature difference $A_f = f(\tau)$:

line AB – the maximum value of this parameter obtained for the bubbling regime of fluidization

A generalization of results of experimental researches was carried out as a dependence $\psi = f(\Delta P_{bed}/(g \cdot D_c); K_w)$, which allows to identify areas of rational realization of the process (Fig.9).

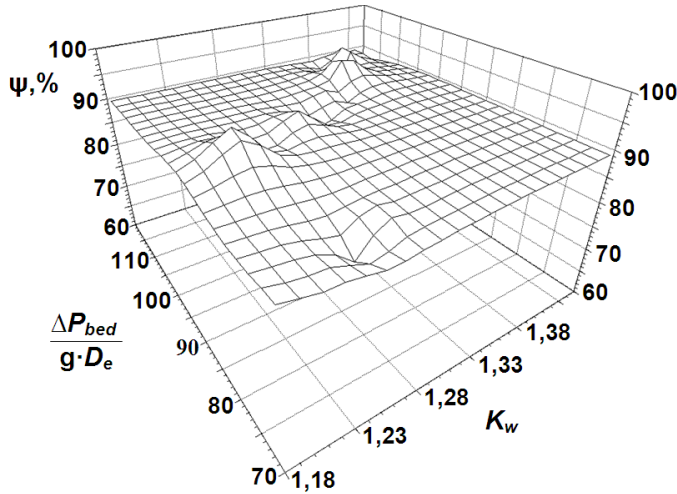


Fig. 9. Experimental dependence $\psi = f(\Delta P_{bed}/(g \cdot D_e); K_w)$

Thereby, values of the coefficient $\psi \geq 90\%$ achieves at $90 \leq \Delta P_{bed}/(g \cdot D_e) \leq 115$ with $2,43 \leq D_e \leq 3,15$ mm (research №1) and $68 \leq \Delta P_{bed}/(g \cdot D_e) \leq 75$ with $3,2 \leq D_e \leq 3,5$ mm (research №2) with value of the number of fluidization $1,2 \leq K_w \leq 1,4$.

A general view of the humic-organic-mineral fertilizers which contain K:N:Ca:P:Mg:S:Hum.=23:9:5:2:6:15:2 is shown in Fig. 10. A granular product has a spherical shape 2÷4% and strength $\sigma \geq 35$ Newtons per granule.

The granule cut is given in Fig. 11, which confirms the layering mechanism of granule formation.

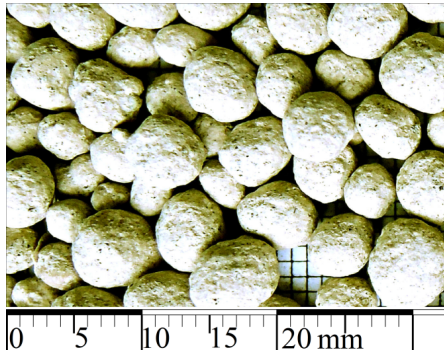


Fig. 10. A general view of the humic-phosphorus-calcium-nitrogen-potassium fertilizers with a stimulating action which contain:
K:N:Ca:P:Mg:S:Hum.=23:9:5:2:6:15:2

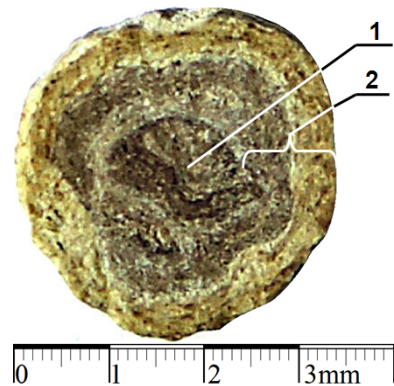


Fig. 11. The granule cut

In Fig. 11 is distinctly pronounced the center of granulation 1 (a nitrogenous-humic composite) with "onion" structure, around which is formed a multilayer structure from the new material 2 in the coaxial form.

Conclusions

The technological parameters of a stable kinetics of granulation process of the complex humic-organic-mineral fertilizers with a coefficient of granule formation $\psi \geq 90\%$ are established.

The obtained (resulting) product has a spherical shape with an equivalent diameter $D_e = 1,2 \div 4,5$ mm, a uniform distribution of components at the micro level throughout the volume of granules and strength $\sigma \geq 35$ Newtons per granule that is more than 3 times higher than standard indicators.

Applying of the jet-pulsating hydrodynamic mode of fluidization allowed to increase an average specific load of the surface of bed by moisture divided by the efficient temperature difference to $A_f = 0,006 \div 0,0066$ $\text{kg}_{\text{moist.}}/(\text{m}^2 \cdot \text{h} \cdot \text{deg})$, that allows to determine the area of an efficient mass transfer process.

For the first time completed studies for the first time allowed to determine the conditions of a sustainable process kinetics of the wastes recycling from the enterprises of fat and oil industry of Ukraine by continuous dehydration and granulation of fertilizers with set properties, obtained by dehydration of highly concentrated water solutions of sunflower ash and ammonium sulphate with impurities of humic substances.

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Effects of osmotic pressure environments lethal effects on the level of microorganisms in the conditions of evacuation

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Abstract

Keywords:

Medium
Vacuuming
Cell
Boiling
Microorganisms

Article history:

Received 14.01.2016
Received in revised
form 18.03.2016
Accepted 24.03.2016

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Introduction. The article deals with the possibility of osmotic pressure environment to act as a factor that, in certain quantities causes bacteriostatic effects and provides information about the strength of cell membranes and mechanisms of interaction with the environment. Experimental studies to determine the action of osmotic pressure environment at the level of lethal effects on different physical interactions.

Materials and methods. Research performed at chamber vacuum packing machine Easy PACK (Germany). The temperature environments in all cases were kept with the restriction on the maximum value $t \leq 38$ °C, which excluded lethal effects on this indicator. Vacuum pump composed laboratory facility provided residual pressure in the vacuum chamber at 0,002-0,004 MPa, corresponding to boiling temperature range environments $\sim 20 \dots 30$ °C. Therefore, the initial ambient temperature $t_{(in)} \leq 15$ °C boiling point not taken place, and the transition to the initial ambient temperature of $20 < t_{(in)} \leq 38$ °C provided the boiling adiabatic mode.

Results. It is known that exposure of yeast in the water accompanied by increased physical pressure in the cells at the osmotic pressure of the cell sap. Transferring in osmotically active yeast solution leads to a reduction of physical pressure, which should be appropriately reflected by vacuuming. Increasing the osmotic pressure of the solution reduces the level of lethal effects of vacuuming, but nevertheless a significant impact on the achievement of the organization lethal effects adiabatic boiling environments. The influence of the level of lethal effects on microorganisms indicators such as exposure time environment and the dynamics of change of pressure in the vacuum chamber, the amount of solids in the environment and the importance of temperature conditions, and the presence or absence of boiling adiabatic environment under vacuuming.

Conclusions. Vacuuming biological environment by reducing the constant saturation gas liquid phase creates limitations in the mass transfer between microbiological cells and environment, and level of limits depends on the osmotic pressure of the solution. Achieved possibility of lethal effects on microorganisms accompanying the food environment, due to the aggregate impact of osmotic pressure and vacuuming.

Introduction

A large number of food and drink to some extent be attributed to the solutions. Last importance of the existence of biological systems, since power transfer biological objects made food components in solution.

The main properties of solutions based on experimental studies and focuses on the phenomenon of osmosis - the penetration of the solvent into the solution through a semipermeable barrier (membrane), impermeable to solute, the solution when it is separated from the pure solvent [1-4].

Osmosis phenomena play an important role in the life of flora and microorganisms, because cell membranes are easily permeable to water and almost impermeable to substances that are part of the cell sap. Penetrating into the cell water creates increased pressure in their shell and stretches, keeping them in a state of stress.

The typical protoplasmic cells derived from bags, that are filled with aqueous solutions of various substances (cell sap). The magnitude of the osmotic pressure determined by the equation Van't - Hofa

$$P_{ism} = \frac{n}{V}RT, \quad (1)$$

where n - number of moles of solute in a volume V of the solution; R - universal gas constant; T - absolute temperature.

Osmotic pressure of the cell sap on the border of the water is between 0,4 - 2 MPa. In solution with a high osmotic pressure of the cell loses water protoplasmic sac. This phenomenon is called plasmolysis and in this state is achieved bacteriostatic effects.

As the osmotic pressure acts as a factor which, in certain quantities causes bacteriostatic effects, it is advisable to stay on the strength of information on the mechanisms of cell membranes and interactions with the environment.

Among the microorganisms that accompany microbiological and food production include yeast, mold, actinomycetes and bacteria. The dimensions of the cells typically 0.5 - 10 microns. In general, the construction of cells of animals, plants and microorganisms are the same and the environment protected cell membrane.

The cell wall of yeast, for example, is about 15% of the mass of the cell and its thickness of 400 nm. The structure of the cell membrane are protein-polysaccharide complexes and lipids. Approximately 70% of the dry weight of the cell membrane of yeast polysaccharides mannan up and glucan. It polysaccharides play an important role in maintaining its mechanical strength.

The effects of osmotic pressure on microorganisms recorded in different technologies and therefore made some adjustments in manufacturing processes. Thus, the increase in osmotic pressure of the solution leads to increased levels of molecular diffusion of water from the microbial cells or vice versa, which eventually leads to equilibrium at a new level.

Therefore, it is logical for research to determine the action of osmotic pressure on the media level for lethal effects of different physical effects.

Materials and methods

Among the planned vacuuming was attributed liquid media (water, beer, wort, sugar solutions) with microorganisms. This was provided for the possibility of vacuuming without boiling environment and adiabatic mode boil. The temperature environments in all

cases were kept with the restriction on the maximum value $t \leq 38 \text{ }^\circ\text{C}$, which excluded lethal effects on this indicator.

Organization experiments without boiling and boiling media carried out by different initial temperature environments. Vacuum pump composed laboratory facility provided residual pressure in the vacuum chamber at 0,002-0,004 MPa, corresponding to boiling temperature range environments $\sim 20 \dots 30 \text{ }^\circ\text{C}$. Therefore, the initial ambient temperature $t_{(in)} \leq 15 \text{ }^\circ\text{C}$ boiling point not taken place, and the transition to the initial ambient temperature of $20 < t_{(in)} \leq 38 \text{ }^\circ\text{C}$ provided the boiling adiabatic mode.

Results and discussion

It is known that exposure of yeast in the water accompanied by increased physical pressure in the cells at the cellular osmotic sap. Transferring in osmotically active yeast solution leads to a reduction of physical pressure, which should be appropriately reflected by vacuuming.

Generalized experimental results are presented in table. 1.

The table shows that increasing the osmotic pressure of the solution reduces the level of lethal effects of the evacuation, which in this group of experiments continued for 10 minutes. However, a significant impact on the achievement of the organization lethal effects adiabatic boiling environments.

Studies have shown that the level of lethal effects influenced by such factors as exposure time environment in a vacuum chamber and dynamics of change of pressure in the vacuum chamber of the maximum to minimum values. Summary data of this series of studies given in the table. 2.

Table 1

The results of studies to assess the lethal effects of vacuum

Environment	The initial temperature environment, $^\circ\text{C}$	The vacuum in the vacuum chamber, MPa	The level of the lethal effects, %	The initial temperature environment, $^\circ\text{C}$	The vacuum in the vacuum chamber, MPa	The level of the lethal effects, %
	Without boiling adiabatic			With adiabatic boiling		
Water +yeast	15	0,002	75	38	0,002	90
15 % sugar solution in water + yeast	15	0,002	50	38	0,002	70
20 % sugar solution in water + yeast	15	0,002	30	38	0,002	60

Table 2
Impact assessment and evacuation time dynamics pressure changes in the level of lethal effects

Environment	The initial temperature environment, °C	Time pressure reduction, c	Dwell time in a vacuum chamber, c	The level of the lethal effects, %
Water +yeast	8	90	60	20
Water +yeast	8	90	120	30
Water +yeast	8	90	1200	100
Water +yeast	8	15	10	95
Water +yeast	8	15	30	100
Water +yeast	8	15	60	100

Stabilized at 8 ° C reference temperature environments provided no adiabatic boiling. However, from the table 2 shows a significant effect of reducing the rate of pressure from the start of vacuuming until its completion at the level of lethal effects.

The following table 1 and 2 summarizes the results of studies make it possible to assess the impact of the choice of parameters. This does not exclude the presence of mutual influences of factors and the transition to a comprehensive assessment advisable to look towards multivariate experiment.

The list of important factors and the lower and upper levels shown in the table. 3. Matrix plan that complies presented in table. 4.

Table 3

Parameter	Unit of measurement	Code	levels			The interval of variation
			lower	null	upper	
The initial temperature environment, t	°C	z_1	8	23	38	15
Concentration of solids	%	z_2	0	7,5	15	7,5
Dwell time in a vacuum chamber, τ	C	z_3	10	605	1200	595
The rate of change of pressure in the vacuum chamber, $\frac{dP}{dt}$	MPa/s	z_4	0,0001	0,0003	0,0005	0,0002

Research performed at chamber vacuum packaging machine Easy PACK firm WEBOMATIC (Germany), and the results determine the levels of lethal effects are presented in table 4.

Mathematical processing of experimental results presented in the form of equation (2). Data processing research based on methods of mathematical statistics, the use of which is possible under the assumption that the results have a normal distribution.

$$\begin{aligned}
 \bar{y} = & 64,812 + 7,937z_1 - 7,687z_2 + 17,312z_3 - 0,187z_4 + \\
 & + 2,9375z_1z_2 + 2,3125z_1z_3 + 4,1875z_1z_4 - 7,6875z_2z_3 + \\
 & + 3,5625z_2z_4 + 2,3125z_3z_4 + 2,9375z_1z_2z_3 + 1,6875z_1z_3z_4 - \\
 & - 1,4375z_2z_3z_4 + 2,9375z_1z_2z_4 + 2,937z_1z_2z_3z_4
 \end{aligned}
 \tag{2}$$

Table 4

№	z_1	z_2	z_3	z_4	z_0	The level of the lethal effects, %
1	-	-	-	-	+	50
2	+	-	-	-	+	60
3	-	+	-	-	+	40
4	+	+	-	-	+	50
5	-	-	+	-	+	95
6	+	-	+	-	+	100
7	-	+	+	-	+	60
8	+	+	+	-	+	65
9	-	-	-	+	+	30
10	+	-	-	+	+	50
11	-	+	-	+	+	40
12	+	+	-	+	+	60
13	-	-	+	+	+	95
14	+	-	+	+	+	100
15	-	+	+	+	+	45
16	+	+	+	+	+	97

Equation (2) shows that the most effective towards achieving the lethal effects of a factor z_3 - exposure time biological system in a vacuum chamber after reaching the final pressure. At that time exposure in the vacuum chamber is the most effective factor indicates a correlation coefficient of z_3 (equal to +17.312). The physical nature of the impact of this factor explains the complex causes, among which include the change in the rates of mass transfer processes, especially CO_2 .

In connection with the above it must be concluded that the decrease in osmotic and physical pressure of cell sap should lead to a decrease in lethal effects, *ceteris paribus*. To achieve these pressure reducing cell sap possible by increasing the osmotic pressure of the solution. In table 4 can be seen that the double effect of osmotic pressure of the sugar solution (factor z_2) largely eliminates the effect given by the violation rate mass transfer processes.

This is underlined by the fact that in all the experiments in which the concentration of sugar is highest, lethal effects are significantly reduced, which also corresponds to a correlation coefficient of z_2 , even -7.687.

Only in those experiments where a combination of the maximum and minimum factor $z_3 z_2$ lethal effects close to 100%.

Assessment on the impact factor z_3 leads to the conclusion that increasing the osmotic pressure of the environment in which microorganisms are, creates a barrier to achieving lethal effects.

Another factor influencing the intensity towards achieving lethal effects in experiments determined the initial temperature environment (factor z_1). The correlation coefficient of this factor amounts to +7.937. Turn to steam experiments (table 4), which differ only in the upper and lower levels of the factor z_1 (experiments 1-2; 3-4; 5-6 ...). At a time when there is adiabatic boiling, increment of lethal effects is about 10%.

The results for the aggregate impact of these factors enable prediction and delineation of areas obtained using scientific information.

Vacuum packaging of food products is carried out by the relevant standards parameters and generally such factors as the osmotic pressure of the solution is stabilized. Among the parameters controlled with the related temperature, level of residual pressure vacuuming and the exposure time vacuumed environment.

Information obtained in experiments gives reason to explain instability results in the lethal effects for vacuum processing, such as drinking water. The vacuum and the delay time so stabilized system should be well-defined. However, one should not neglect the opportunity to organize adiabatic boiling the treated environment.

In experiments conducted by the time determined initial boiling adiabatic energy potential of the medium, which was largely temperature. Similar conditions occur and when the vacuum packaging products.

Definitely increase the level of state aseptic products meet increasing initial temperature. However, this figure for each product in some way regulated.

As for the vacuum aseptic water treatment exists conditional restrictions related to energy consumption for a given initial temperature.

The share impact of adiabatic boiling point as compared to the vacuuming and the exposure time can be increased by external supply of heat to the treated environment and continued so time point.

Using vacuum processing to achieve aseptic condition in some way limited production capacity given packaging for products, but in terms of technological processing of these limitations are removed.

Adiabatic boiling in conjunction with vacuuming relate to purely physical factors that rightfully belong to environmentally friendly technologies.

It is on this basis must find a way to microbiological purity of drinking water as a general and special software. Other points of application of vacuum technology with elements of adiabatic boiling is the production of beverages, juices, compotes.

The results of experimental studies clearly indicate a reduction of lethal effects with increased osmotic pressure environments. This is a strengthening effect the transition to a medium boil adiabatic regime that derives its vacuuming. The explanation of these factors needs to develop relevant hypotheses.

Painting intracellular structures in a solution of methylene blue indicates that there is a destruction of cytoplasmic and cell membranes. It could happen for two groups of factors, which include internal and external influences. Among the outer include pressure, temperature, cavitation modes adiabatic boil and even geometry volume, which are located environment. The latter is due to the fact that the residual pressure in the vacuum chamber and the height of the column with the environment can be commensurate and the resulting destabilized adiabatic boiling in full.

Adiabatic boiling is active displays on the various components of the solution are, however, cells of microorganisms thermodynamic parameters of the medium is not achieved. This is due to the fact that physical pressure cell sap equal to its difference in osmotic pressure and the osmotic pressure of the medium can far exceed the physical pressure in the vacuum chamber. The latter leads to the conclusion that in such circumstances the establishment of the solvent vapor phase in the middle of the cell is impossible.

But with the approach of osmotic pressure and cell sap environment is expected to reduce the physical pressure in the cells. Thus we can say that with the decrease of osmotic pressure environment (down to zero for distilled water), the possibility of the formation of vapor phase reduced, and in some limiting value π_2 vapor phase formation becomes impossible.

But are among the factors operating in the cell sap dissolved gases. Liquid environment saturated with dissolved gases, among which include nitrogen and carbon dioxide. The level of saturation with the law meets Henry.

Active reduce the pressure in the vacuum chamber reduces physical pressure in microbiological cell so disturbed equilibrium. This should lead to rapid selection of the gas phase, enhance mass transfer for CO₂ and additional internal pressure in the cells.

These internal factors, such as creating a vapor phase and the allocation of dissolved gases may occur in parallel or the first may be missing. Therefore, a relatively small osmotic pressure environments involving increased physical pressure in microbial cells predominant influence of dissolved gases.

Thus, by dissolving gases (most convenient CO₂) significantly expands the range of variation of pressure and physical phenomena that accompany them.

Conclusions

1. Vacuuming biological environment by reducing the constant saturation gas liquid phase creates limitations in the mass transfer between microbiological cells and environment.
2. Level restrictions mass transfer processes and state of microbial cells depends on the osmotic pressure of the solution.
3. Limitation of metabolic processes in cells and in the "cell-environment" due to osmotic pressure in the last, leads to an increase protective function and bacteriostatic effects. Due to the increase in osmotic pressure of the solution and the migration of water from the cell sap physical pressure in the cells decreases and decreases the solubility of gases in the sap. The result is a restriction of lethal effects.

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Modeling of heat transfer in free down flowing laminar liquid films with development wavy structure at the regime of evaporation from the interface

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Abstract

Keywords:

Films
Waves
Heat transfer
Temperature
Evaporation

Article history:

Received 20.01.2016
Received in revised
form 16.03.2016
Accepted 24.03.2016

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Introduction. Within long vertical boiling tubes thermohydrodynamic processes in liquid film take place at a regime with cycling mixing of the film by the powerful waves. The existing Heat Transfer models of such phenomena do not take into account such processes.

Materials and methods. The heat transfer and hydrodynamic processes that take place in down flowing film of water and sugar solutions at the regimes of evaporation from the interface have been studied. There were developed mathematical models, which then were compared with the result of direct experimentation of heat transfer in tubes at the regimes of solutions concentrations.

Results and discussion. A mathematical model of heat transfer in laminar, heated to the saturation temperatures liquid films with the developed wavy structures on the free interface have been developed. The model takes into consideration cyclic relaxation of transient temperature field which happens right after the passage of a powerful big wave. The developed mathematical model describes the time history of the two dimensional temperature fields as a function of the Peclet number and the core characteristic of the wavy motion (the length of big waves). Based upon the proposed model a set of correlations have been obtained. These are proposed as a means for the generalization of heat transfer experimental data, obtained within the experimental studies of liquid films, heated to the saturation temperatures and evaporating from the interface. A generalized equation has been derived, which can be used for the calculations of Heat Transfer Coefficients (HTC) to the saturated sugar solutions liquid films. This equation contains wavy characteristics of down flowing films and valid within the range of parameters characteristic for the sugar industry evaporators, namely: concentrations – 0...70 % dry matter; liquid mass flow rate density – $0.01 \times 10^{-3} \dots 0.6 \times 10^{-3} \text{ m}^2/\text{sec}$, the Peclet number range – 400...25000. The mathematical model of the temperature field cyclic relaxation turned out efficient for generalization of heat transfer experimental data not only laminar, but turbulent liquid films either, despite of the fact that the transport equations do not contain turbulent characteristics.

Conclusions. A correlation between the liquid film wavy structure with the heat transfer has been established. The correlation is based upon the model of temperature field cyclic relaxation after passage of big waves. The respective correlations have been presented.

Introduction

A wavy structure is being formed on the interface of the liquid film down flowing over the vertical surface even at a lowest density of liquid flow rate. At a distance to 2...2.5 from the liquid film inlet a so called regime of the wavy motion saturations takes place [1]. This regime is characterized by a completely structure of big waves which move on the interface at a so called “bulldozer” regime. On top of this, big waves even in case of viscose concentrated solutions contain a swirl [2]. Therefore, their movement on the surface entails to the intensive mixture of liquid and, respectively, if accompanies by a noticeable deformation of velocity and temperature profiles, and thus by equalization of the concentrations over the film thickness. Studies into the wavy structures of liquid films – as an adequate form of liquid film movement is important and becomes a point of interest for many modern foreign publications [3...12]. It is clear that within the time period between the passage of two consecutive big waves a transient process of temperature and velocity fields relaxation takes place. It is quite natural that exactly this process has a decisive influence on the heat transfer. This statement corroborated by the deviation of the heat transfer coefficient curve from the Nusselt line [13]. The fact that the said deviation happens at laminar regime of film flow substantiates a decisive effect of the interface wavy structures on the film heat transfer.

A major number of theoretical studies dedicated to the film wavy motion [14...16] directed at the aspect of the development and structures of regular waves. The portion of these waves on the length of heat transfer tubes of industrial evaporator is small when compared to the regions at which big waves take place. A big number of scientific publications aimed at the studies of heat transfer in down flowing films, the effect of wavy structures upon the heat transfer have being taken into account indirectly by the turbulence parameters and liquid film interface shear stress. A majority of experimental results obtained with evaporating films heated to the saturation temperatures at the conditions which model industrial evaporators [17...19]. The obtained data therefore contain information of the heat transfer at the simultaneous action of a number of influencing factors. It is impossible to separate the effect of some individual factor in these conditions. That is why the existing correlations for the calculations of heat transfer coefficients to the saturated liquid films in industrial evaporators are sufficiently limited within the range of regime parameters in which the process had been modeled. Similarly, the said correlations are limited in terms of the geometry of the experimental unit. The proposed project is aimed at the development of the theoretical description of temperature field relaxation which takes place in the film after passing of a big wave.

As a result of this, an interrelation between the liquid film wavy structure with the heat transfer at a regime of liquid film evaporation from the free interface is established.

Materials and methods

A direct experimentation of heat transfer in down flowing liquid films heated to the saturation temperatures with sugar solutions as model liquids has been carried out at the experimental unit with the independent formation of phases' mass flow rates and heat flux. The main core of the experimental unit was represented by a stainless still pipe with the inside diameter of 20 mm and 1.8 m long. The experimental tube was separated into the initial 1.5 m stabilization section and 0.3 m measurement section. The down flowing of water (sugar solutions) film has been formed by means of overflowing over the tube's upper rim. In the event of steam-liquid flow modeling, dry saturated steam has been

supplied in co-current regime. The liquid falling film has been heated by dry saturated steam which was supplied into outside heating sections attached to the experimental tube. The heating chambers were designed in a such way as to provide an individual heating of the stabilization section and the experimental one. The said sections were hooked up to the individual vacuum-condensation sections which allowed for the keeping of different pressures in each chamber. Such arrangement allowed also maintaining vacuum down to 0.8 bars and thus, vary the temperature head between the heating steam temperature and evaporation temperature. Special probes for taking samples of liquid to determine its concentration and measurements of temperatures were positioned directly after the measurement section. A detailed description of the experimental unit is given in [21] and its schematic is given in fig.1.

Results and discussions

The model of temperature field relaxation has been designed aiming at the following: to develop an analytical expression for the calculation of heat transfer to the liquid film, containing a sought parameter, which can be determined by the comparison of experimental data with those found analytically.

Thus, the developed expression could be used for the generalization of the experimental data, obtained within a wide range of regime parameters, and for the engineering calculations of heat transfer coefficients, either.

The main suggestions of the development of the proposed model are following: the big waves rolling over the liquid film surface are associated with the vortexes, which mix the liquid in the film; therefore we assume that right after the passage of a strong wave the velocity profile will be constant and the temperature profile is curved in a such way, hat the bulk of the liquid will have temperature equal to the saturation temperature, but the layers adjacent to the wall will be heated and assume the wall temperature.

This mode of velocity distribution will keep existing along the first regime of flow. At the same time there will be the development of temperature field in the liquid film. It is clear, that since a bulk of the liquid will have the temperature equal to that of saturation, there will be no temperature gradient on the liquid film interface and thus there will be no evaporation on this section. Along with the liquid film movement the development of the temperature field will take place. The temperature front will move gradually from the heated wall towards the film interface. Than the time will come when the sensitive temperature gradient appears on the film interface. This moment signifies the beginning of the second regime. This period we assume that the velocity profile keeps parabolic and constant. And the further development of the temperature field takes place. Within this period of flow the heat flux on the wall will go down and that on the interface goes up, until the temperature profile becomes linear. Despite this simplification this model looks quite reasonable, because the relative lengths of the mentioned above regimes is quite small, especially of the first one, and heating of the film until it starts evaporate takes very short time due to the small film thickness. It is worth mentioning that these suggestions allowed a significant simplification of the differential equations describing heat transfer. Thus, the suggestions of the constant value of mean film velocity within the two regimes permitted to substitute the temperature-time derivatives by the temperature-longitudinal distance derivatives. The model developed under such suggestions might be turned as a quasi-transient.

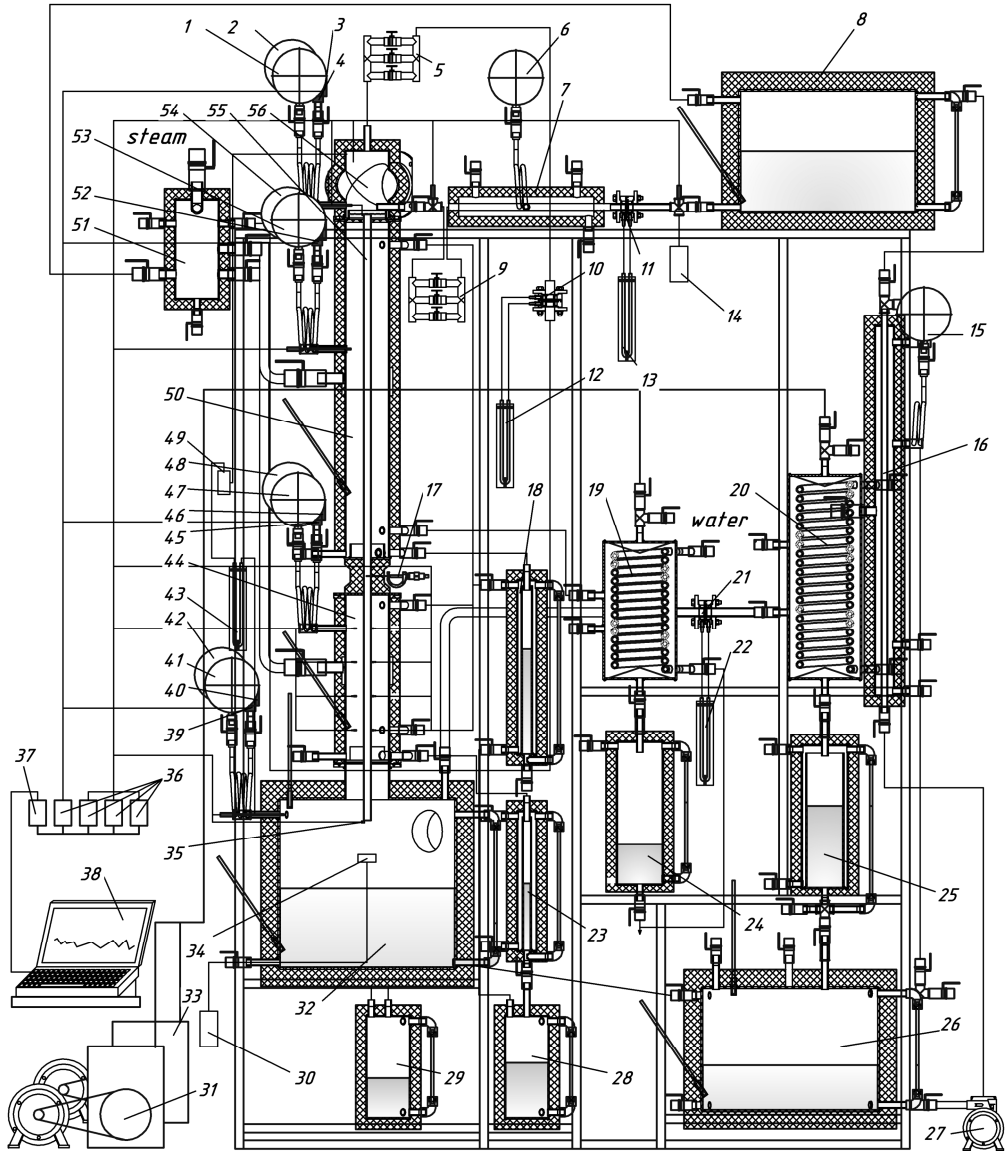


Fig.1. Scheme of the experimental stand:

1,6,15,41,47,53 – manometer; 2,42,48,54 – vacuum manometer; 3,39,45 – sensor for vacuum control; 4,40,46,52 – sensor for pressure control; 5,9 – collector flow control; 7,16 – heat exchanger; 8,26,32 – tank; 10,11,21 – diaphragm; 12,13,22,43 – differential pressure gage; 14,30 – sampler; 17 – gage probe; 18,23,24,25,28,29 – hotwell; 19,20 – condenser; 27 – pump; 31,33 – vacuum pump; 34 – tank for sampling; 35 – tank of solution's temperature measurement on the exit of pipe; 36 – module analog input 7018P; 37 – module 7520; 38 – computer; 44,50 – heating chamber; 49 – equaling tank; 51 – separator of heating steam; 55 – experimental pipe; 56 – top chamber.

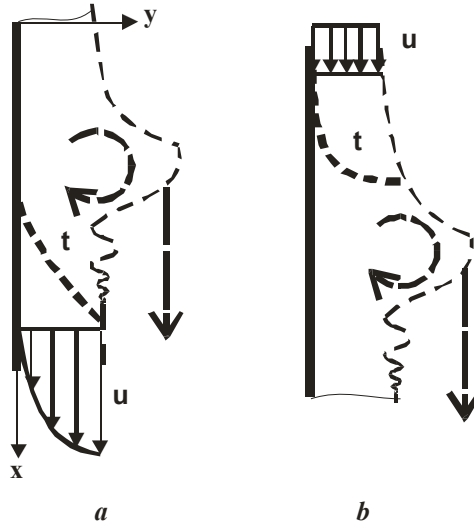


Fig. 2. Model of liquid film structure:
a – temperature and velocity distribution before a strong wave passage
b – after.

According to the fig. 2b which depicts the moment right after the passage of big wave assuming the constant velocity distribution which is $\bar{u} = \frac{g\delta^2}{3\nu}$, the heat transfer equation in the dimensionless form can be written as:

$$\frac{\partial\theta(\eta, \xi)}{\partial\xi} = \frac{4}{Pe} \frac{\partial^2\theta(\eta, \xi)}{\partial\eta^2}, \quad (1)$$

here $\theta(\eta, \xi) = \frac{t(\eta, \xi) - t_l}{t_w - t_l}$ – dimensionless temperature; t_w, t_l – temperature of wall and saturation, respectively; $\eta = \frac{y}{\delta}, \xi = \frac{x}{\delta}$ – dimensionless crossways and longitudinal coordinates; $Pe = \frac{4\tilde{A}_v}{a} = \frac{4\bar{u}\delta}{a}$ – the Peclet number; $\delta = \sqrt[3]{\frac{3\tilde{A}_v\nu}{g}}$ – liquid film thickness; \tilde{A}_v – volumetric flow rate; a – temperature conductivity; ν – cinematic velocity.

The solution of(1) with boundary conditions:

$$\theta(0,0) = 0, \theta(0,\xi) = 1, \theta(1,0) = 0, \frac{\partial\theta(\infty,\xi)}{\partial\eta} = 0, \quad (2)$$

will be

$$\theta(\eta, \xi) = \operatorname{erfc}\left(\frac{\eta}{4} \sqrt{\frac{Pe}{\xi}}\right). \quad (3)$$

The profile(3) will be developing until a significant temperature gradient on the interface, appears which corresponds to the ξ_m . Further on, as a result of evaporation, the temperature of the film surface will remain constant. In this case the temperature, which corresponds to the equation (3) at $\xi = \xi_m$ will be

$$\theta(\eta, \xi_m) = \operatorname{erfc}\left(\frac{\eta}{4} \sqrt{\frac{Pe}{\xi_m}}\right) \quad (4)$$

The curve (4) represents a limiting case, after which the boundary conditions (2) become meaningless. Thus the first regime ceases to exist and the second regime begins. As it was mentioned above for the second regime, we assume a parabolic velocity distribution. Then, the heat transfer equation will read:

$$\frac{g \delta^3}{2 \nu a} (2\eta - \eta^2) \frac{\partial \theta(\eta, \xi)}{\partial \xi} = \frac{\partial^2 \theta(\eta, \xi)}{\partial \eta^2}, \quad (5)$$

and the boundary conditions

$$\eta = 0, \theta = 1; \eta = 1, \frac{\partial \theta}{\partial \eta} = 0. \quad (6)$$

Substituting the left side of (5) by the mean velocity value, one obtains

$$\frac{g \delta^3}{2 \nu a} \int_0^1 (2\eta - \eta^2) \frac{\partial \theta(\eta, \xi)}{\partial \xi} d\eta = \frac{g \delta^3}{2 \nu a} \frac{2}{3} \frac{\partial \theta_{av}(\xi)}{\partial \xi} = \frac{Pe}{4} \frac{\partial \theta_{av}(\xi)}{\partial \xi},$$

and the equation (5) can be rewritten as:

$$\frac{Pe}{4} \frac{\partial \theta_{av}(\xi)}{\partial \xi} = \frac{\partial^2 \theta(\eta, \xi)}{\partial \eta^2}. \quad (7)$$

Double integration of (7) with boundary condition (6) yields

$$\theta(\eta, \xi) = \frac{Pe}{4} \frac{\partial \theta_{av}(\xi)}{\partial \xi} \left(\frac{\eta^2}{2} - \eta \right) + 1. \quad (8)$$

From the equation for mean temperature value

$$\theta_{av} = \int_0^1 \theta(\eta, \xi) \frac{u(\eta)}{\bar{u}} d\eta = \int_0^1 \theta(\eta, \xi) (2\eta - \eta^2) \frac{3}{2} d\eta = 1 - \frac{Pe}{10} \frac{\partial \theta_{av}(\xi)}{\partial \xi},$$

taking into account, that $\xi = 0$, $\theta_{av} = 0$ one obtains

$$\theta_{av} = 1 - \exp\left(-\frac{10}{Pe} \xi\right). \quad (9)$$

Substituting derivative of (9) by ξ into (8), one obtains limiting temperature curve at $\xi = \xi_m$

$$\theta(\eta, \xi_m) = \frac{5}{2} \exp\left(-\frac{10}{Pe} \xi_m\right) \left(\frac{\eta^2}{2} - \eta\right) + 1, \quad (10)$$

The coordinate ξ_m can be found from (10) at a condition, that at $\xi = \xi_m$ the dimensionless temperature equals 0 ($\theta(1, \xi_m) = 0$)

$$\xi_m = 0,0223 Pe \quad (11)$$

Within the region $\xi \geq \xi_m$ the liquid temperature on the interface remains constant, since evaporation takes place.

There fore the boundary conditions (6) will change in to

$$\eta = 0, \theta = 1; \eta = 1, \theta = 0, \quad (12)$$

and the initial conditions at $\xi = \xi_m$ will be as for equation (10).

Integration of (7) with the boundary conditions (12) yields

$$\theta(\eta, \xi) = \frac{Pe}{8} \frac{\partial \theta_{av}(\xi)}{\partial \xi} (\eta^2 - \eta) - \eta + 1. \quad (13)$$

In order to find the derivative $\frac{\partial \theta_{av}(\xi)}{\partial \xi}$, we determine a mean temperature within the region $\xi \geq \xi_m$

$$\theta_{av}(\xi) = \int_0^1 \theta(\eta, \xi) (2\eta - \eta^2) \frac{3}{2} d\eta = \frac{3}{8} - \frac{7}{320} Pe \frac{\partial \theta_{av}(\xi)}{\partial \xi}. \quad (14)$$

This, in turn, yields

$$\theta_{av} = \frac{3}{8} + C \exp\left(-\frac{320}{7Pe} \xi\right). \quad (15)$$

The integration constant C can be determined from the initial condition (10). Having determining the mean temperature $\theta_{m.av}$ at $\xi = \xi_m$

$$\theta_{m.av}(\xi_m) = \int_0^1 \theta(\eta, \xi_m) (2\eta - \eta^2) \frac{3}{2} d\eta = 1 - \exp\left(-\frac{10}{Pe} \xi_m\right),$$

and substituting this expression into (15), having θ_{av} in mind that $\xi \rightarrow \xi_m$, we obtain the constant C

$$C = \left(\frac{5}{8} - \exp\left(-\frac{10}{Pe} \xi_m\right) \right) \exp\left(\frac{320}{7Pe} \xi_m\right).$$

Then

$$\theta_{av} = \frac{3}{8} + \left(\frac{5}{8} - \exp\left(-\frac{10}{Pe} \xi_m\right) \right) \exp\left(\frac{320}{7Pe} (\xi_m - \xi)\right). \quad (16)$$

Substituting a derivative of (16) by ξ into (13), one obtains a temperature distribution in the film at $\xi \geq \xi_m$

$$\theta(\eta, \xi) = \left[\frac{40}{7} \exp\left(-\frac{10}{Pe} \xi_m\right) - \frac{25}{7} \right] \exp\left(\frac{320}{7Pe} (\xi_m - \xi)\right) (\eta^2 - \eta) - \eta + 1. \quad (17)$$

Thus obtained temperature profiles calculated by the equations (3, 10, 17) are given at fig. 3

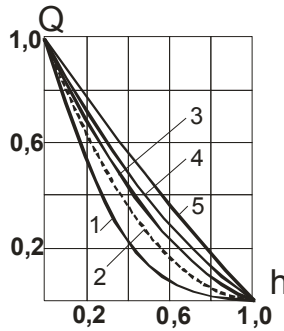


Fig. 3. Dimensionless temperature distribution across the water film as per (3, 10, 17) at $t = 100^\circ\text{C}$; $\Gamma_v = 0,5 \cdot 10^{-3} \text{ m}^2/\text{c}$; $\xi_m = 267$;

1 - $\xi = 167$, equation(3); 2 - $\xi = \xi_m$, equation(10); 3 - $\xi = 367$; 4 - $\xi = 467$;
5 - $\xi = 767$, 3, 4, 5 - equation (17).

The heat flux on the wall ($\eta = 0$) within the region $\xi \geq \xi_m$ is obtained from (17)

$$q_2(\xi)_{\eta=0} = -\lambda \frac{t_w - t_l}{\delta} \frac{d\theta}{d\eta}_{\eta=0} = \lambda \frac{t_w - t_l}{\delta} \left\{ \left[\frac{40}{7} \exp\left(-\frac{10}{Pe} \xi_m\right) - \frac{25}{7} \right] \exp\left(\frac{320}{7Pe} (\xi_m - \xi)\right) + 1 \right\} \quad (18)$$

and within the region $\xi \leq \xi_m$ from (3)

$$q_1(\xi)_{\eta=0} = -\lambda \frac{t_w - t_l}{\delta} \frac{d\theta}{d\eta}_{\eta=0} = \lambda \frac{t_w - t_l}{2\delta \sqrt{\pi}} \sqrt{\frac{Pe}{\xi}}. \quad (19)$$

As stated above, a cyclic process of heat transfer in the liquid film is subdivided into two characteristic zones: the first one $\xi \leq \xi_m$ which is characterized by the gradual development of the temperature field until the temperature curve reaches limiting curve ξ_m . It has been mentioned that within this region only heating at the liquid bulk in the film takes place. Since there is no temperature gradient on the interface in this region there is no evaporation from the film. Further on within the region $\xi \geq \xi_m$ the linearization of the temperature profile takes place. Here the process keeps going at a constant temperature on the interface. Respectively, we can determine the values of mean temperature fluxes within these regions $(\xi_m - \xi_o)$, $(\xi_v - \xi_m)$

$$q_{1av} = \frac{1}{\xi_m - \xi_o} \int_{\xi_o}^{\xi_m} q_1(\xi)_{\eta=0} d\xi = \lambda \frac{t_w - t_l}{\delta \sqrt{\pi} (\xi_m - \xi_o)} \left(\xi_m \sqrt{\frac{Pe}{\xi_m}} - \xi_o \sqrt{\frac{Pe}{\xi_o}} \right) \quad (20)$$

$$q_{2av} = \frac{1}{\xi_v - \xi_m} \int_{\xi_m}^{\xi_v} q_2(\xi)_{\eta=0} d\xi = \frac{\lambda(t_w - t_l)}{\delta(\xi_v - \xi_m)} \times$$

$$\left[\left(\frac{Pe}{8} \exp\left(-\frac{10\xi_m}{Pe}\right) \right) \left[\frac{5}{8} \exp\left(\frac{390\xi_m - 320\xi_v}{7Pe}\right) - \exp\left(-\frac{320(\xi_v - \xi_m)}{7Pe}\right) + 1 \right] \right] \left[-\frac{5Pe}{64} + \xi_v - \xi_m \right], \quad (21)$$

where $\xi_v - \xi_o = \frac{L_w}{\delta}$ is dimensionless distance between the consequent peaks of big waves;

L_w - is distance between the consequent peaks of big waves.

Thus ξ_o determines the depth of the temperature profile “lowering” right after the passage of a big wave. The lower value of ξ_o , the bigger will be temperature gradient and, respectively, the lower deviation of the temperature curve depicted on fig. 3.

The length of big waves on the surface of water films according [20] within the Re numbers range 40...400, remains constant at the level of 100...120 mm and at further growth of Re number to 4000 gradually increases to the value of 140 mm at a distance approximately 2.4 m from the film distributor.

Therefore, the mean temperature flux could be given as a mean weighted

$$q_{av} = \frac{q_{1av}(\xi_m - \xi_o) + q_{2av}(\xi_v - \xi_m)}{\xi_v - \xi_o}, \quad (22)$$

and the Nusselt number will be determined as:

$$Nu = \left(\frac{\sqrt{Pe\xi_m} - \sqrt{Pe\xi_o}}{\sqrt{\pi}(\xi_v - \xi_o)} \right) + \frac{1}{\xi_v - \xi_o} \left[\left(\frac{Pe}{8} \exp\left(-\frac{10\xi_m}{Pe}\right) \right) \left[\frac{5}{8} \exp\left(\frac{390\xi_m - 320\xi_v}{7Pe}\right) - \exp\left(-\frac{320(\xi_v - \xi_m)}{7Pe}\right) + 1 \right] \right] \left[-\frac{5Pe}{64} + \xi_v - \xi_m \right] \quad (23)$$

where $\alpha = \frac{q_{av}}{t_w - t_i}$, $Nu = \frac{\alpha \delta}{\lambda}$.

The temperature of interface surface t_i is determined as the saturation temperature at a mean liquid concentration in the film.

Comparison of the calculated by (23) Nusselt numbers with the experimental data of heat transfer to the saturated liquid films of water and sugar solutions [13] allows to determine the value of ξ_o

$$\begin{aligned} \xi_o &= 43Y + 0,2 \text{ at } Y \leq 0,115 \\ \xi_o &= 1150Y^{0,98} - 133, \text{ at } Y \geq 0,115, \end{aligned} \tag{24}$$

here $Y = \left(\frac{\delta}{\delta^*}\right)^{0,9} \left(\frac{\nu_{wat}}{\nu}\right)^{0,01}$, ν_{wat} – water cinematic viscosity; $\delta^* = \sqrt{\frac{\sigma}{g\rho}}$.

At $Pe \geq Pe_o$, $Y_o = \left(\sqrt[3]{\frac{3Pe_o a \nu}{4g}} / \delta^*\right)^{0,9} \left(\frac{\nu_{wat}}{\nu}\right)^{0,01}$, here $Pe_o = 10300$.

$$\begin{aligned} \xi_o &= 43Y_o + 0,2 \text{ at } Y \leq 0,115, \\ \xi_o &= 1150Y_o^{0,98} - 133, \text{ at } Y \geq 0,115 / \end{aligned}$$

The generalization of experimentally obtained heat transfer coefficients to the water and sugar solutions up to the concentration of 70% ($\nu = 3,77 \cdot 10^{-6} \text{ m}^2 / \text{sec}$) at free flowing down films with the results of calculations are given on fig. 4.

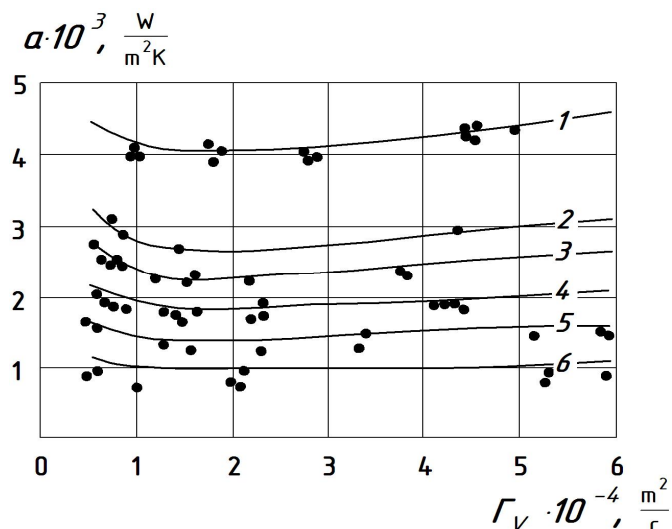


Fig. 4. Comparison of experimentally determined heat transfer coefficient to water and sugar solutions at free flowing down over the vertical surface films at a regime of evaporation from the interface with the calculated values. The lines are calculated as per (23, 24) at 120 mm. 1 – water, 2t = 100 C; 2 – sugar solution, DM = 30 %; 3 – 40 %; 4 – 50 %; 5 – 60 %; 6 – 70 %.

As it might be seen from the fig.4, the calculations as per (23, 24) closely correlate the experimental data within the volumetric flow rates from $0...6 \times 10^{-4}$ m²/sec. It is worth mentioning, that the close correlation takes place not only within the laminar regime of flow, but also in the turbulent region despite the fact that, whilst deriving the model the parameters of turbulence were not explicitly taken into consideration.

Conclusions

The proposed model of heat transfer with the cyclic relaxation of temperature field in the down flowing liquid films adequately displays the physical processes that take place. Particularly the proposed model allows to interpret the deviation of the experimental data on heat transfer within the laminar flow regime from the theoretical Nusselt curve.

It has been proven that the proposed model can be successfully applied the generalization of experimental data of heat transfer to the down flowing liquid films of water and viscose water solutions at a regime of evaporation from the free surface.

Equates(23, 24) are recommended to be used for the engineering calculations of heat transfer coefficients to the liquid films of water and sugar solutions in the regimes of industrial multi-effect industrial evaporators within the wide range of liquid mass flow rates.

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Development of the occupational safety in the food industry with regard for the risk-based approach

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Abstract

Keywords:

Safety
Labor
Traumatism
Risk

Article history:

Received 22.01.2016
Received in revised
form 16.03.2016
Accepted 24.03.2016

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Introduction. The prediction of occupational risks and making the conditions for the prevention of injuries based on it is one of the promising scientific directions of the workplace safety development in industry, as it is directly connected with the manufacturing process.

Materials and methods. The investigation was performed on the basis of the following methods: the methods of statistical analysis of accidents that occurred in the food industry within the last decade for the determination of tendencies of traumatism; the method of regression analysis; the method of principal components; the expert evaluation method for improving the method of prediction of injury risks; the method of apriori ranking of factors in the processing of the results of expert grades.

Results and discussion. Because of the performed investigation, the technique of increasing the occupational safety in the food industry was developed on the basis of the prediction of occupational injury risks. This technique is of great importance for preventing dangers and hazards with the aim of providing favorable working conditions, precluding failures and preventing occupational diseases and accidents. One of the most promising scientific directions of the safety development in the manufacturing process is the prediction of occupational injury risks, directly connected with the manufacturing process, and making the conditions for preventing traumatism basing these predictions. The results of the comparative analysis of retrospective prediction according to the methods of regression analysis (prediction) and the improved method of combined prediction based on the method of principal components combined with the expert evaluation method indicate that the statistical prediction of the number of injured employees at the food industry enterprises shows larger deviations from the actual number of injured employees (the standard error equal to 2.53) than the combined prediction (standard error equal to 0.85). Thus, it is possible to conclude that, on the average, the efficiency of prediction increases by 60% due to the combination of the method of principal components with the expert evaluation method.

Conclusions. The scientific results of the investigations are a contribution to the development of theoretical and applied fundamentals of labor protection in the part that concerns diagnostics, prediction, modeling of extreme situations, and evaluation of their consequences.

Introduction

The basic equipment of the food industry of Ukraine is predominantly morally and physically obsolete, and its operation time is already 1.5–2.5 times greater than its design life. More than 50% of employees work under the conditions that do not comply with the occupational safety norms and regulations. At the workplaces of food enterprises, increased level of noise and vibration is registered, illumination and microclimatic parameters do not comply with the sanitary requirements. Mandatory medical inspections are often performed formally and incompletely. A substantial level of traumatism, occupational deceases and accidents with severe and lethal consequences are the consequence. Within the last decade (2005–2014), more than 9.5 thousands of employees of the food industry were injured at enterprises, and 633 of them had a lethal outcome. All these facts indicate that the state of labor protection in the food industry of Ukraine cannot be considered as satisfactory [1].

Development of the level of safety of the manufacturing process is connected with substantial costs of its re-equipment, retraining of personnel, and introduction of modern systems of manufacture control. In this case, the contradiction appears that may lead to increase of occupational injuries. It is connected, on one hand, with the necessity of occupational safety development, necessarily reducing to additional expenses at the above-listed works and to the products' manufacturing cost increase. On the other hand, a decrease in the productions expenses may lead to the increase of occupational traumatism.

As there is the substantial number of scientific sources that consider the problem of organization of occupational safety and prevention of traumatism, however, most of them do not include deep investigation of the problem of complex development of the occupational safety and analysis of the occupational injuries in the food industry [6-10].

One of the promising scientific directions of the safety of the manufacturing process increasing is the prediction of occupational risks directly connected with the manufacturing process and making the conditions for the prevention of injuries on its basis. Analysis of the existing risk prediction methods enables us to draw the conclusion on the necessity of improvement for most of them, with the aim to adapt them to the features of enterprises of the food industry and to make complex evaluation of risks of occupational injuries at the enterprise.

Thus, the scientific-applied problems of developing a technique of increasing the level of occupational safety in the food industry based on the prediction of risks of occupational injuries are to be solved.

The aim of the investigation is to increase the efficiency of the preventive measures of occupational injuries at enterprises of the food industry due to the on-line prediction of injury risks.

To achieve the set aim, the following tasks of the investigation were specified.

1. To perform the statistical analysis of accidents in the food industry.
2. To develop methods of investigating the cause-and-effect relations of the injury processes in the food industry.
3. To improve the methods of predicting the risk of occupational injuries.
4. To investigate the cause-and-effect relations that lead to injuries in the food industry and the influence of preventive measures on them.
5. To develop algorithms of functioning of the information-analytic system for the on-line analysis of work conditions in the industry, determine the rational directions of preventive measures of occupational injuries, and justify the organizational measures of labor protection.

The object of investigation is the prediction methods of occupational injury risk at enterprises of the food industry.

Materials and methods

In the work, we used the following methods of investigation: the method of statistical analysis of accidents that occurred in the food industry between 2003 and 2011 for the determination of tendencies of traumatism; methods of regression analysis for the evaluation of the cause-and-effect relations of injury processes; the method of principal components for the determination of the main factors of injury of the employees in the food industry and the prediction of injury risks; the expert evaluation method for improving the method of prediction of injury risks; the method of a priori ranking of factors in the processing of the results of expert grades. Moreover, the experience of accidents analysis in branches of economy both in Ukraine and abroad was taken into account.

Results of discussion

Basing on the analysis of the statistics of occupational injuries in the food industry of Ukraine between 2003 and 2011, the existing methods of injury prediction were analyzed, and the problems of the investigation were stated.

The process of improving the functioning of the system of labor protection control (SLPC) calls for the rational organization and well-organized cooperation of experts and heads of all structural divisions of the enterprise, and for the efficient cooperation with the industry and corresponding state organs. The analysis and prediction of indices of the state of labor protection is the important function of labor protection control. Thus, the question of how to form an SLPC that will consider the problem of labor safety at the enterprises of the food industry complexly, with regard for its future state, arises.

The situation of taking a decision in the SLPC on reducing the level of occupational injuries is determined by the tuple $\{X, Y, Q, R, Z, S, E, C, T\}$, where X is the set of information data used in the formation of managerial decisions; Y is the set of indices proceeding from which the level of occupational injury is evaluated; Q is the set of managerial decisions admissible within the framework of the specified type of the problem; R is the formalized rule of choosing a managerial decision from the set of possible decisions; Z is the set of restrictions; S is the set of possible states of the external medium; E is the set of expected results of alternative managerial decisions realization; C is the cost of injury preventive measures; T is the time factor.

The results of execution of the managerial decision in the time interval $t + \Delta t$ depend on the values of the set of indices proceeding from which the state of labor protection is evaluated in the previous time interval and managerial decision is taken:

$$Y^{t+\Delta t} = f\left(\left[X^{t-n\Delta t}, X^t\right], Z, S^t, Q, C\right). \quad (1)$$

One of the promising scientific directions of increasing the general level of safety of the manufacturing process is the prediction of risks of occupational injuries $Y^{t+\Delta t}$ and making the conditions of injury prevention based on these predictions. The labor protection control is aimed at minimization of risks. The mathematical expression for choosing an optimal decision from the set of possible ones has the form

$$q_{i\bar{i}\delta} = q_j \cdot y_j^{t+\Delta t} = \min_i \left(y_i^{t+\Delta t} \right), \quad y_j^{t+\Delta t} \leq y_{\bar{A}}^{t+\Delta t}, \quad C_j \leq C_{\bar{A}}, \quad (2)$$

where $q_{i\bar{i}\bar{o}}$ is the optimal managerial decision; $y_j^{t+\Delta t}$, $y_{\bar{A}}^{t+\Delta t}$ are, consequently, the predicted and limiting (specified) values of the injury index at the moment $t + \Delta t$; $C_j, C_{\bar{A}}$ are, consequently, the predicted and limiting (admissible) expenses for the realization of the preventive measures of injuries. The values $y_j^{t+\Delta t}$, $y_{\bar{A}}^{t+\Delta t}$ will determine the degree of occupational injury risk at an enterprise.

The analysis of the existing methods of risk prediction enables us to draw the conclusion about the necessity of their improvement with the aim of their adaption to the features of enterprises of the food industry and complex evaluation of the risks of occupational injuries at the enterprise, which determines the necessity to solve the urgent scientific-applied problem of *the development of a technique for increasing the labor safety in the food industry basing on the prediction of risks of occupational injuries*.

To improve the methods of prediction of occupational injury risks in the food industry, a general model of risk [2] and a method of determination of the cause-and-effect relations of the phenomenon of occupational injuries [2] were developed, and the methods of labor protection control based on combined predictions of risks [2-3] were improved.

In general, the risk of occupational injuries can be determined as follows:

$$R = \sum_{i=1}^n S_i P_i, \quad (3)$$

where S_i are consequences of an accident; P_i is the probability (frequency) of accidents; n is the number of accidents.

For the analysis of the direct cause-and-effect relations that take place in a process of injury, we used a scheme of an accident emergence represented by the statistical data on the direct causes of occupational injury (Fig. 1) [3].

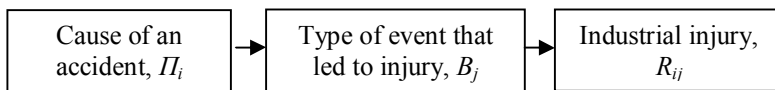


Fig. 1. The scheme of the emergence of an accident represented by statistical data on direct causes of occupational injury

For the calculation of conditional probability, the Bayes formula is used

$$P_{I_i}(B_j) = \frac{P(B_j)P(I_i)}{\sum_{i=1}^n P(I_i)}. \quad (4)$$

The matrix of injury risks in the industry is calculated by formula (4):

$$R_{ij} = \begin{bmatrix} R_{I_{1\bar{A}1}} & R_{I_{2\bar{A}1}} & \cdots & R_{I_{16\bar{A}1}} \\ R_{I_{1\bar{A}2}} & R_{I_{2\bar{A}2}} & \cdots & R_{I_{16\bar{A}2}} \\ \cdots & \cdots & \cdots & \cdots \\ R_{I_{1\bar{A}15}} & R_{I_{2\bar{A}15}} & \cdots & R_{I_{16\bar{A}15}} \end{bmatrix}, \quad (5)$$

where $R_{I_{1A1}, \dots, R_{I_{16A15}}$ are the values of injury risks for binary complexes “cause of injury risk – type of injury event”; $i = 1, \dots, 16$ is the number of the main causes of injury in the industry I_i , which, at present, is fixed in the valid classification of the form of mandatory statistical accounting No. 7-tnv; $j = 1, \dots, 15$ is the number of the main types of injury events.

For the prediction of occupational injuries in the work, we used the method of principal components, as a minimum error of prediction is provided due to its main properties. For example, let the initial investigated p -dimensional vector of observations X be replaced

by the vector $Z = (z^{(1)}, z^{(2)}, \dots, z^{(p')})^T$ of smaller dimensionality p' , in which each

component is a linear combination of p initial (or auxiliary) features without too much information loss. The informativeness of the new vector Z depends on the measure, to which p' introduced auxiliary variables make it possible to “restore” p initial features

with the help of corresponding linear combinations $z^{(1)}, z^{(2)}, \dots, z^{(p')}$. It can be imagined that the error σ of prediction of X from Z will be determined by the residual dispersion matrix of the vector X after subtraction of the best prediction from Z from it, i.e., by the

matrix $\Delta = [\Delta_{ij}]$, where $\Delta_{ij} = E \left\{ \left(x^{(i)} - \sum_{l=1}^{p'} b_{il} z^{(l)} \right) \left(x^{(j)} - \sum_{l=1}^{p'} b_{jl} z^{(l)} \right) \right\}$. Here,

$\sum_{l=1}^{p'} b_{il} z^{(l)}$ is the best prediction $x^{(i)}$ in the least-squares sense at the components $z^{(1)}, z^{(2)}, \dots, z^{(p')}$. The error of prediction of X from Z is given as a certain defined function

of the elements of the matrix $\Delta = [\Delta_{ij}]$, i.e., $\sigma = f(\Delta)$, where $f(\Delta)$ determines a certain criterion of prediction quality.

The following measures of prediction error can be used:

1. $f(\Delta) = Tr(\Delta) = \Delta_{11} + \Delta_{22} + \dots + \Delta_{pp}$ on the basis of the trace of the matrix

$$\Delta = [\Delta_{ij}];$$

2. $f(\Delta) = \|\Delta\| = \sqrt{\sum_{i=1}^p \sum_{j=1}^p \Delta_{ij}^2}$ on the basis of the Euclidean norm of the matrix

$$\Delta = [\Delta_{ij}].$$

It was proved that both measures simultaneously attain their maximums if only the first p' principal components of the vector X are chosen as $z^{(1)}, z^{(2)}, \dots, z^{(p')}$, and the value of the prediction error $\sigma = f(\Delta)$ is explicitly expressed in terms of the last $p - p'$ eigenvalues of the initial covariance matrix C or approximately in terms of the last $p - p'$ eigenvalues $\lambda_{p'+1}, \dots, \lambda_p$ of the sample covariance matrix \hat{n} constructed from the observations X_1, X_2, \dots, X_n . In particular,

$$\text{for } f(\Delta) = Tr(\Delta): \sigma \approx \lambda_{p+1} + \lambda_{p+2} + \dots + \lambda_p;$$

$$\text{for } f(\Delta) = \|\Delta\|: \sigma \approx \sqrt{\lambda_{p+1}^2 + \lambda_{p+2}^2 + \dots + \lambda_p^2}.$$

Thus, basing on the methods of regression and component analysis, the model of occupational injury risk is formed. It complexly relates the probability of emergence of the accident to the frequency of emergence of accidents in the industry for the whole range of reasons.

In the work, we improved the combined method of principal components and the regression analysis on principal components, with the linear model in the form

$$R = b_0 + b_1Y_1 + b_2Y_2 + \dots + b_kY_k + \varepsilon_k \quad (6)$$

where R is the dependent index or a characteristic of the process or phenomenon investigated, Y_k is the value of the first principal components for the objects of investigation, $k = 1, 2, 3, \dots, p$; $b_0 \dots b_k$ are the coefficients of the regression equation; ε_k is the normally distributed random quantity with a zero average and variance.

The necessity of improvement is caused by the correlation of indices, which determines the poor conditionality of the system of normal equations for the determination of the regression coefficients and by the presence of errors, which causes the shift of estimates. To prevent the indicated disadvantages, it is proposed to improve the combined method of regression analysis on principal components basing on the *expert evaluation method* usage, with the aim to evaluate the significance (weight) of each factor (component) and the consistency of opinions of experts (Table 1).

Table 1

Matrix of results of expert estimate of indices

Experts	Factors / components				
	X_1	X_2	X_i
1	a_{11}	a_{12}	a_{1i}
2	a_{21}	a_{22}	a_{2i}
j	a_{j1}	a_{j2}	a_{ji}

Further in the work, the methods of labor protection control based on the combining of the statistical analysis, expert evaluation with ranking of factors and combined prediction of risks of occupational injuries are considered. In the work, we propose an algorithm of formation of decisions (Fig. 2) about organization and providing safe work conditions basing on the risk prediction, where the main stages of the process of decisions formation on the basis of combined prediction of risks are presented.

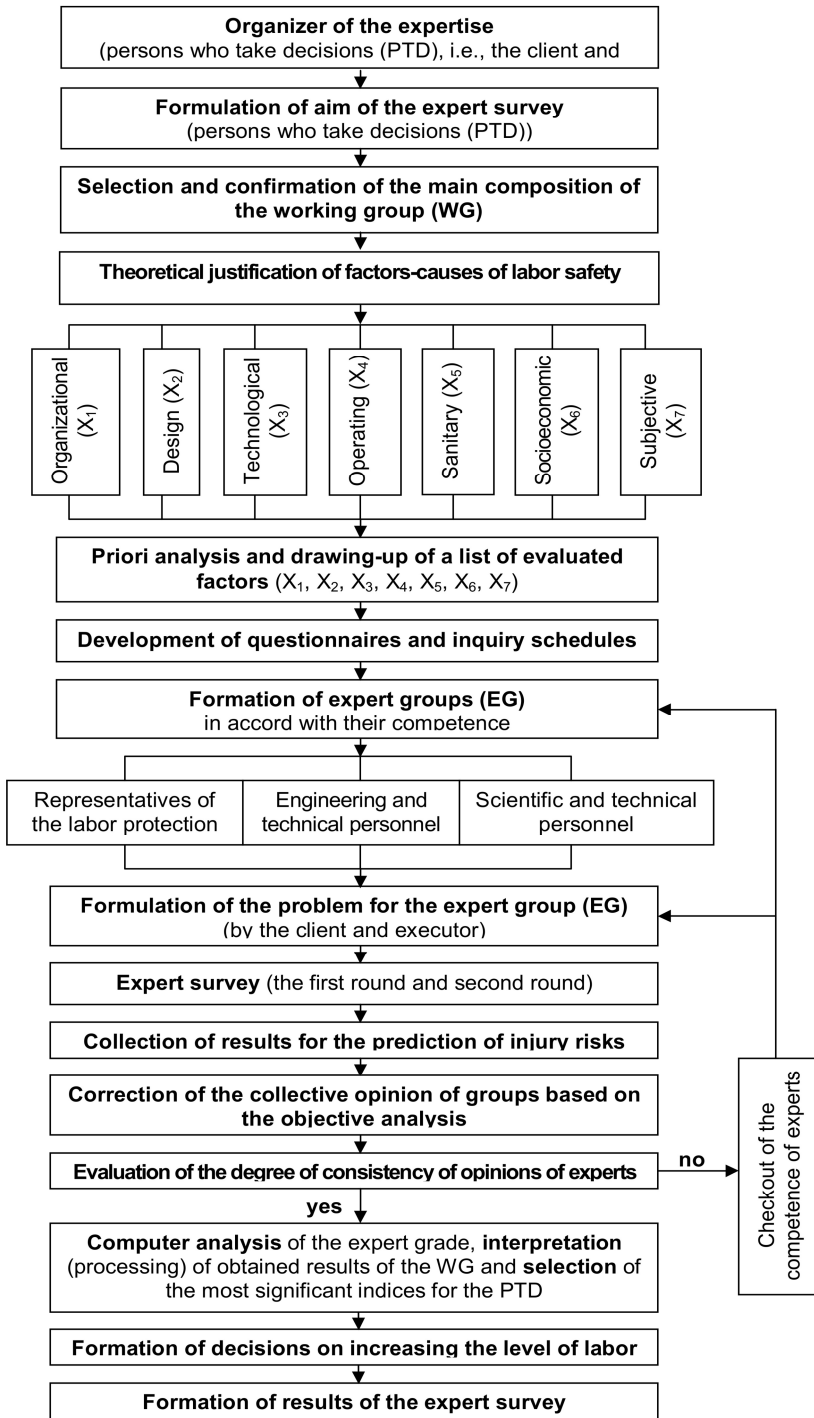


Fig. 2. Algorithm of formation of decisions on the organization and ensuring of safe working conditions based on the prediction of risks

Applying the models and methods developed using data for the period between 2001 and 2012, we investigated the statistics of occupational injuries with the methods of regression analysis, construction of multifactor regressive models and performed combined prediction for the period between 2012 and 2013.

Basing on the corresponding time series, mathematical models of trends and predictions of the future behavior of time series were constructed. In the Fig. 3, the dynamics of the number of injured employees for the different types of events is shown for an example. The dynamics of the number of injured employees for different professions and different shops was determined analogously. The mean error of prediction is equal to 10–12%, indicating the applicability of the proposed approach to the prediction of the dynamics of time series of occupational injury [4].

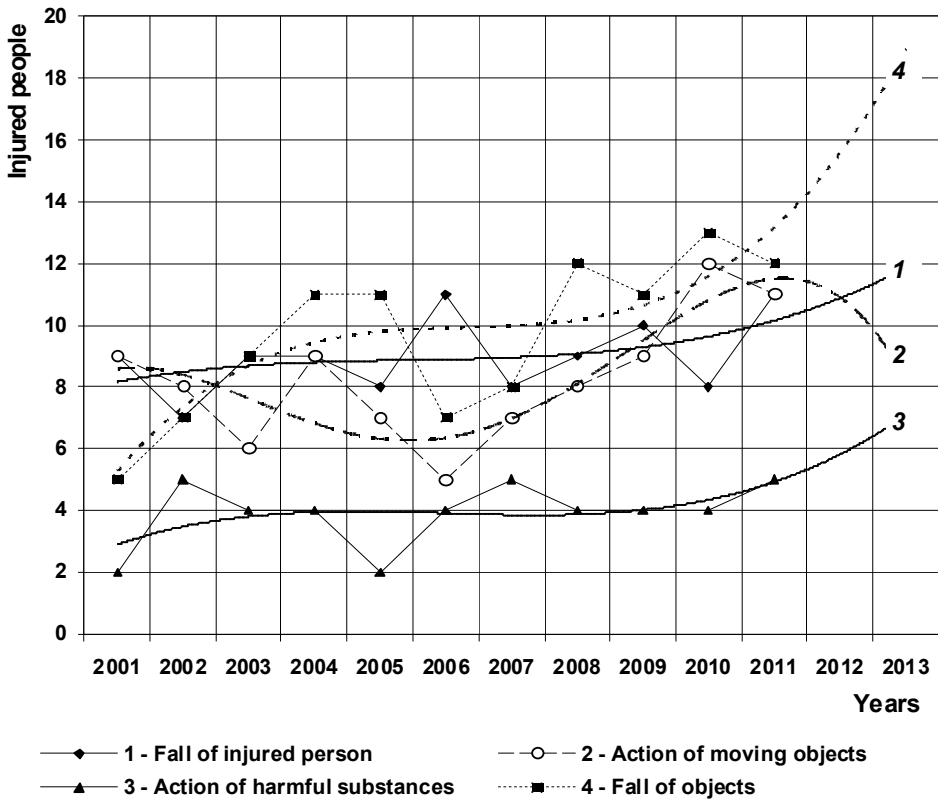


Fig. 3. Dynamics of the number of injured employees for different types of events

Fall of injured person:

$$y = 0,0062x^3 - 0,1014x^2 + 0,5894x + 7,6667, R^2 = 0,885$$

Action of moving objects:

$$y = -0,0061x^4 + 0,1455x^3 - 0,9962x^2 + 1,8522x + 7,6061, R^2 = 0,7172$$

Action of harmful substances:

$$y = 0,0105x^3 - 0,1888x^2 + 1,0734x + 2, R^2 = 0,8256$$

Fall of objects:

$$y = 0,0289x^3 - 0,549x^2 + 3,5282x + 2,2576, R^2 = 0,8474$$

To evaluate the efficiency of predicting the level of injuries basing on the method of combined prediction, we compared the prediction grades using multifactor models of the dependence of the number of accidents on the causes-factors that led to accidents, and the dependences of the number of accidents on the types of events that led to accidents.

$$A = -0,06112 + 0,6754X_{n1} + 0,8718X_{n2} + 1,5954X_{n3} - 0,8534X_{n4} + 0,2794X_{n5} + 0,2953X_{c1} + 0,3732X_{c2} + 0,3609X_{c3} + 0,4207X_{c4} + 0,7141X_{c5}$$

Errors of the prediction with this combined model are equal to 0.43–1.11%, which is a better result than those obtained individually from each of the previous models.

It is reasonable to complement the methods of the combined prediction by the refined grades based on expert evaluation, the aim of which is to refine the influence of the factors on the occupational injuries. With the results of processing, we constructed diagrams of ranks, which refine the values of the factors of influence on the occupational injuries (Fig. 4). Professions most susceptible to traumatism, causes of intentional violation of safety requirements, potential causes of traumatism, factors that cause injuries during the technological process performance, factors of the most dangerous (in terms of injury) equipment, and labor protection measures were investigated analogously.

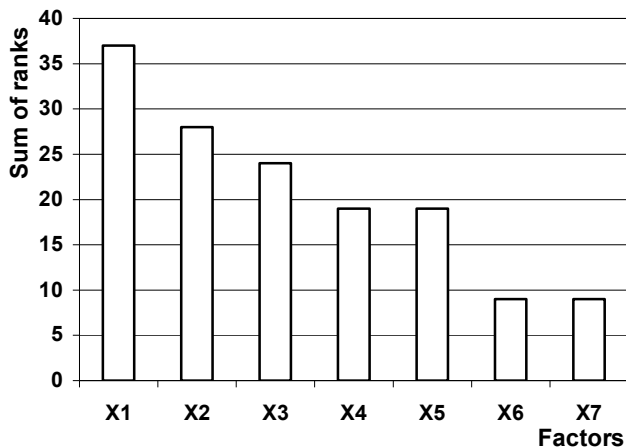


Fig. 4. Diagram of ranks of production and technical factors

Then we performed the general evaluation of the efficiency of the proposed theoretical results and justified measures and means for preventive measures of risk of occupational injuries.

The results of the comparative analysis of the retrospective prediction by the methods of regression analysis (prediction) and the improved method of combined prediction based on the method of principal components in combination with the expert evaluation method are shown in the Fig. 5.

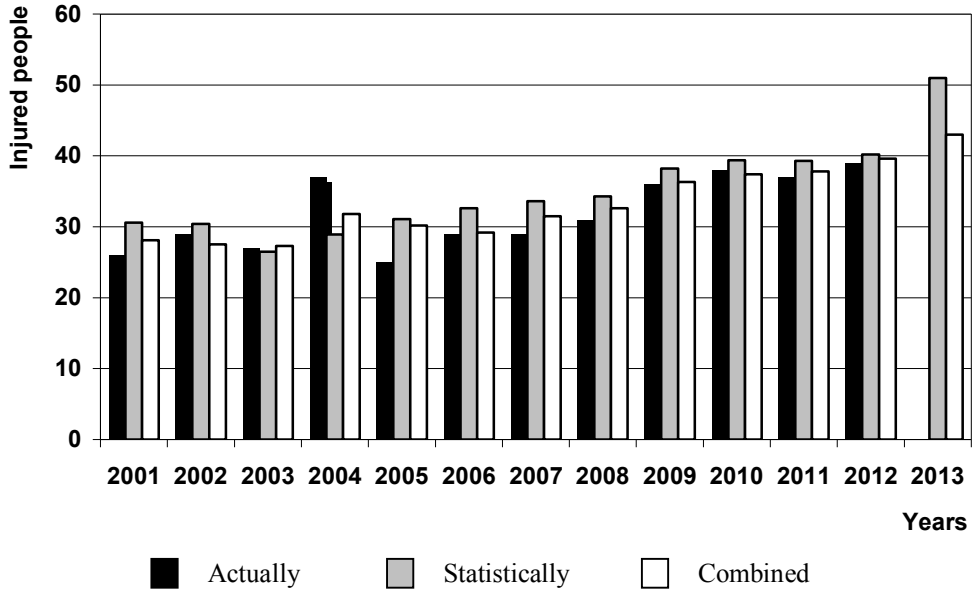


Fig. 5. Comparison of statistical and combined predictions of risk of occupational injuries in the food industry

As we see in the Fig. 5, the statistical prediction of the number of injured employees at enterprises of the food industry shows larger deviations from the actual number of injured employees (standard error equal to 2.53) than the combined prediction (standard error equal to 0.85). Thus, it can be concluded that, on the average, the efficiency of prediction increases by 60% due to the combination of the method of principal components with the expert evaluation method.

Basing on the obtained theoretical and practical results, in the work we justified measures and means for preventive measures of risk with the help of its prediction, and developed a project of a complex of means of automation of labor protection control for the food industry, which consists of two software tools: “Automated system of accounting, analysis, and evaluation of accidents at enterprises of the food industry” and “Control of knowledge on labor protection of production personnel” [5].

Conclusions

As a result of the performed investigations, we have developed a technique for increasing the level of labor safety in the food industry basing on the prediction of risks of occupational injuries, which is of great importance for prevention of dangers and hazards with the aim of providing favorable work conditions, preventing failures and precluding occupational diseases and accidents.

One of the promising scientific directions of enhancing the safety of the manufacturing process is the prediction of risks of occupational injuries, which is directly connected with the manufacturing process, and making the conditions for preventing traumatism using these predictions. The analysis of the existing risk prediction methods enables us to draw

the conclusion that they need improvement with regard for the features of the food industry and complex evaluation of risks of occupational injuries.

For the first time, the model of risk of occupational injury in the food industry has been developed. This model is based on taking into account complex influence of the whole range of manufacturing and socioeconomic factors on injuries and constructed on the fundament of a scheme of an accident emergence, in which each fact of the accident is related to the prerequisite of its emergence. The indicated approach enables us to carry out an analysis of direct cause-and-effect relations that take place in the process of injury and to reveal both main and hidden causes of occupational injuries, and types of events that lead to an accident.

The combined method of regression analysis on principal components has been improved. In contrast to the existing method, it additionally includes the results of refinement of the main influence factors taken from the expert evaluation method, which makes it possible to use it for predicting injury risks in case of the substantial correlation of the initial statistical data and insufficient conditionality of the system of normal equations in the determination of regression coefficients and in case of presence of errors in the determination of the initial indices and a shift of grades of traumatism.

The methods of labor protection control have been further developed basing on the combination of statistical analysis, expert evaluation with ranking of factors and combined prediction of risks of occupational injuries with the realization of the algorithm of formation of propositions to the improvement of the work conditions at enterprises of the food industry. Thus, it becomes possible to develop managerial decisions on providing safe work conditions for the personnel employed in the food industry on the basis of objective prediction of risks.

The proposed technique for increasing the level of safety in the food industry basing on the prediction of risks of occupational injuries formed the base of the algorithm of monitoring of causes and circumstances that lead to occupational injuries in the food industry and of the informational support formation for personnel training on urgent problems of labor protection. On the base of the indicated technique, recommendations were created on the analysis of the causes and circumstances that lead to injury of an employee at a specific working place and on the complex of the most reasonable antitraumatic measures was determined.

The developed models and methods have new and novel properties and make it possible to increase the efficiency (precision) of prediction, on the average, by 60%, based on the combination of the method of principal components with the expert evaluation method, which enables us to increase the total efficiency of preventive measures of occupational injuries at enterprises of the food industry, on the average, by 18–23%.

The scientific results of the investigations are a contribution to the development of theoretical and applied fundamentals of labor protection in the part that concerns diagnostics, prediction, and modeling of extreme situations, and evaluation of their consequences. The results of the investigations have been introduced at a number of enterprises of the food industry.

A wide range of problems on the development of the methods of determination of the cause-and-effect relations of occupational injuries, prediction of risks and development of efficient measures for improving the system of labor protection control in the food industry can be promising directions of further investigation.

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Food quality perception in the Czech Republic: trial study results

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Abstract

Keywords:

Food
Label
Quality
Consumer
Shopping

Article history:

Received 04.01.2016

Received in revised
form 08.02.2016

Accepted 24.03.2016

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Introduction. The article presents outputs of a trial which discovered consumers' opinions on food quality labels in the Czech Republic and their influence on shopping behaviour in the process of purchasing foodstuff.

Materials and methods. Questionnaire research is a basis for further research realized in 2016. In the trial, 36 respondents over 18 years of age were interviewed in front of grocery stores in the period from December 2015 to January 2016, their distribution roughly corresponded the Czech population sample. Individual semi-structured interviews were used where the respondents answered a set of 13 questions.

Results and discussion. Three most influential factors that affect consumers shopping food are price in the first place, origin of the product in the second, and its appearance as third. However, more accurate results will appear with a higher number of respondents in further research. So far the trial leads to the conclusion that quality labels are not the key factor having significant influence on shopping behaviour. In naming quality labels, Klasa appears to be the most recognized label of quality and shows the highest awareness. Other featured labels were Český výrobek and Bio. Nevertheless, consumers do not have an accurate understanding of quality labels meaning in general. In 95% of cases, the respondents agree that labelled products meet their expectations, but only 58% consider labelled products to have higher quality. Almost 61% of the respondents agree that quality labels are trustworthy, and 72% of the sample are willing to pay extra money for labelled food products.

Conclusion. About two thirds of consumers trust quality labels and nearly three quarters are willing to pay more for labelled products than unlabelled.

Introduction

After lifting food quality standards in 1993, quality of numerous products has decreased without attention on the side of the consumers. Gradually, information began appearing that it is necessary to monitor composition of products as they actually might not contain what they should. The problem was and still remains that while shopping consumers do not want to spend time reading unclear and often confusing information on composition of the products which is usually even missing at over-the-counter sale. Therefore, it is common that consumers purchase spurious products where name and packaging play the key role, not the actual composition or nutritional value.

Over time, price has become guidance for quality of food products. But nowadays even that is no guarantee for quality or content of the ingredients expected by the consumers. On the one hand, some product quality indicators are improving, on the other hand, use of substitutes in foodstuffs is growing. Quality labels the number of which is growing rapidly on the market are supposed to resolve the issue. These labels should guarantee quality of products in terms of composition, place, or method of production, and should help consumers choose fine quality, unadulterated food.

Due to the fact that quality is gradually becoming a significant factor in the choice of food by more and more consumers, quality labels are gaining importance as well. They are substantial not only for consumers but also manufacturers who are able to attract attention or differentiate from the competition thanks to products which meet the parameters for obtaining such label of quality.

Theoretical background

Quality labels, or the so called utility signs, are graphic symbols that appear on a product, its packaging, or enclosed information materials. They inform about particular parameters of a product (packaging), or its use [1]. They are a tool to reassure consumers about the quality through certification. Specific labels only cover certain aspects of quality. It means that the market offers product or service quality labels.

Quality labels can help producers to communicate their products with the value-adding characteristics, highlight the specific character of their products, and stimulate consumers' interest in such products. [2]

According to [3] there is great diversity within quality labels. Symbols are divided into several categories which may overlap with one another. Those are related to:

- Industry (sector) - e.g. HORECA Select,
- Working conditions - e.g. Fairtrade, Oké bananas,
- Production conditions - e.g. FSC certification, Rainforest Alliance,
- Recycling and organic products - e.g. Eco-O.K.,
- HR policies - e.g. Investor in People,
- Product - e.g. Klasa.

All utility signs are segmented in higher detail by [1] according to the following criteria: in terms of severity, content, extent, and geographic perspective.

There is no doubt that quality labels have undeniable importance for producers as well as consumers. To consumers they provide certain assurance as products marked by such labels must meet the established standards and requirements. They also contribute to simpler orientation on the market and help choose a quality product or service with minimal risk. Currently, one of the assumptions about today's consumer behaviour is the fact that people are increasingly buying products not because of their parameters but for the personal value they represent. Products are often evaluated according to their specific qualities (not the main benefit

it should deliver), but the so called enhanced product (a set of intangible elements which bring the perceived advantage to the consumer, e.g. image, service, consulting, etc.). Quality labels are part of the enhanced product which influences consumer behaviour. [4], [5]

Contribution of brands for manufacturers is often far greater than benefits for consumers. Quality labels can serve as an effective marketing tool which leads to an increase in sales (after being marked with a brand logo) and raise in awareness among consumers. Brands are therefore considered an important tool for manufacturer's sales support. The survey conducted by Focus Agency for an expert periodical Marketing Journal shows that 81% of companies see the main benefits of using quality labels in the expected increase in consumer confidence. Another benefit is the aforementioned increase in revenues and a way to differentiate from competition. Also, 39% of companies perceive quality labels as a guarantee of production stability and high quality of its products. [6], [7]

These labels can be an important factor in consumer choice. Consumers may prefer a product from a certain geographical area simply because they believe it to be better, or they may prefer a product from their own region or country due to consumer ethnocentrism, i.e. their loyalty to the region/country and their preference to support the local economy. [8]

Czech food market is flooded by a large number of quality labels which should function as a guide for consumers and at the same time guarantee quality and origin of products. Consumers may encounter labels used exclusively for food products (eg. Klasa or Regionální potravina), or labels given in other product categories (eg. CZECH MADE or Český výrobek).

Methodology

This paper aims to (1) identify the main factors in decision-making while purchasing food and their order stated by the respondents, (2) discover knowledge and recognition of quality labels that appear on the Czech food market, and (3) gain respondents' opinions on food products marked by quality labels.









The presented study is a trial for the research on quality labels. In the trial during the period from December 2015 to January 2016, 36 respondents were interviewed distribution whom roughly corresponds the distribution of the monitored categories of the population sample. The survey sample consisted of residents of the Czech Republic over 18 years of age addressed in front of grocery stores. The interviews were recorded for qualitative evaluation. Based on the information obtained from the trial study, the questionnaire will be adjusted for final interviews.






The research technique used were individual semi-structured interviews, the respondents answered a set of 13 questions with closed and open-ended answers and scales. Representative technique was used for the selection of respondents, namely simple random selection where respondents were interviewed in front of a grocery stores. The questions were focused on the attitude of respondents towards purchasing food labelled by quality labels and their knowledge of quality labels placed on food sold in the Czech Republic. Further segmentation questionnaire contained questions on household size, total net income of the respondent's household, the highest educational attainment of the respondent, and zip code for region identification. The aim of the survey was to get the majority of responses from women who have higher influence on shopping behaviour of food and stronger decision-making power than men. The respondents were willing to answer questions, and no significant number of respondents who would be reluctant to participate in the questioning was registered. The obtained data were then processed and classification of the first and second degree was conducted, followed by correlation analysis and hypotheses testing.

Previously conducted interesting researches describe the situation in the Black Sea area using critical analysis [11] and [12].

Table 1

Quality labels used in the Czech Republic

Logo	Name	Characteristics
	KLASA	Label awarded by the Ministry of Agriculture to food and agricultural products of finest quality.
	Český výrobek – guaranteed by Federation of the Food & Drink Industries of the Czech Republic	Products must be manufactured in the Czech Republic and must contain a certain share of Czech ingredients. The label is awarded by Federation of the Food & Drink Industries of the Czech Republic.
	Český výrobek (belongs to the Český výrobek fund)	Label for both food and non-food products whose production company is owned by Czech citizens and revenue is not transferred outside the country. Label is awarded by Český výrobek fund.
	Český výrobek (belongs to Český výrobek Ltd.)	Designation of safe products manufactured in the Czech Republic (where employees are Czech). The label is granted by Český výrobek Ltd.
	Czech made	The label which is part of the state program Česká kvalita reflects that the quality of designated goods and services has been objectively verified by a third party. This label is awarded by Sdružení pro Cenu České republiky za jakost.
	BIO – a product of eco agriculture	Nationwide trademark for organic food given awarded by organizations entrusted by the Ministry of Agriculture.
	BIO in EU	EU logo for organic packaged foods, which was introduced by the European Commission.
	Ekologicky šetrný výrobek (Eco-friendly product)	Goods and services that are proven environmentally and consumer health friendly, label is granted by the Ministry of the Environment.

	<p>Regionální potravina (Regional food)</p>	<p>Label awarded by the Ministry of Agriculture to finest-quality agricultural products that win in regional competitions.</p>
	<p>Vím, co jím (I know what I eat)</p>	<p>Designation of nutritionally balanced food granted by the non-profit organization Vím, co jím a piju.</p>
	<p>Zdravá potravina (Healthy food)</p>	<p>Labelled food must not contain controversial additives, artificial flavourings and E-additives, is awarded by Zdravá potravina.</p>
	<p>Certified e-friendly food (CEFF)</p>	<p>Food products without preservatives, artificial colourings and flavours, the label is awarded by an independent institution.</p>
	<p>Chráněné zeměpisné označení (Protected geographic trademark)</p>	<p>Designation of an exceptional agricultural product or foodstuff from a given region / location. At least one phase of production - production, processing, or preparation must take place in the designated area. Awarded by the European Commission.</p>
	<p>Chráněné označení původu (Protected origin trademark)</p>	<p>Designation of an exceptional agricultural product or foodstuff from a given region / location. All stages of production must take place in the designated area, it also applies to ingredients. Awarded by the European Commission.</p>
	<p>Zaručená tradiční specialita (Guaranteed traditional specialty)</p>	<p>Agricultural product or foodstuff produced or manufactured for at least 30 years specific nature of which is recognized by the EU. Awarded by the European Commission.</p>
	<p>Fair Trade</p>	<p>A certification system for products from the countries of the Third World where consumer buying this product helps disadvantaged producers (mainly from the Third World countries). Managed by Fairtrade Labelling Organisation International.</p>

Source: [9], [10]

Results and discussion

Responses were distributed evenly within the sample according to the number of members in the households, as well as in the category of total monthly net income of the households. In the category of gender a higher proportion of women was reached, which is advantageous as in most families women take decisions on food purchase. Unequal representation was achieved in the category of age where almost over 41% of respondents fall into the age group of 20-29 years. The territorial distribution of the respondents is that nearly 64% of respondents come from the Central Bohemian Region, the rest of the respondents from the regions of Olomouc and Plzen. Thus it is possible to say that the inquirers managed to ensure representation of respondents living in large cities and near such cities who usually have different lifestyle and therefore distinct shopping behaviour from people living in rural areas.

Table 1

Sample characteristics

Number of household members	1	8,3 %	Total monthly net income of households	up to 10 000 CZK	16,7 %
	2	22,2 %		10 000 – 20 000 CZK	19,4 %
	3	22,2 %		20 000 – 30 000 CZK	27,8 %
	4	22,2 %		30 000 – 40 000 CZK	13,9 %
	5	25 %		40 000 – 60 000 CZK	11,1 %
					over 60 000 CZK
Education	Primary	11,1 %	Age	20 -29 years	41,7 %
	Secondary	16,7 %		30 – 39 years	19,4 %
	Secondary (higher)	44,4 %		40 – 49 years	19,4 %
	College	2,8 %		50 – 59 years	11,1 %
	University	25,0 %		60 years and more	8,3 %
Gender	Female	63,9 %	Region	Central Bohemian	63,9 %
	Male	36,1 %		Plzen	2,8 %
				Olomouc	33,4 %

In the ranking of the factors that most affect food purchase, an earlier assumption was confirmed that price is the main criterion. Each respondent was asked to state three factors that most influence their purchase of food and, in addition to price, respondents placed great emphasis on the origin of products (whether it is a Czech or foreign products and whether it is a regional product, or a product imported from a greater distance).

Moreover, the demand for regional foods and specialties is powered by the growing consumer interest in product attributes such as origin, sustainability, traceability, and authenticity. [13]

Among other qualities, appearance of the product was considered important. Other factors that placed on the first to fifth position were quality, composition of the product, taste (which is the most subjective criterion), and recommendation. Quality label placed 7th in case of the first factor, 10th as the second factor, and 3rd in stating the third factor. This means that quality label is not one of the main selection criteria for the respondents.

There are some important factors driving consumers to buy or not to buy their local food products. These factors can be food quality, costs, lifestyle, and motivation to support local economic growth. In terms of food quality, local foods are believed to be a fresh due to the fact that they are grown near the consumer and distributed within a shorter transportation distance [14].

Table 2

Order of factors with most influence on food purchase

	First factor	Second factor	Third factor
1	Price	Price	Price
2	Origin	Origin	Appearance
3	Quality	Composition	Quality label
4	Composition	Appearance	Composition
5	Taste	Other	Recommendation
6	Appearance	Recommendation	Quality
7	Quality label	Habit	Freshness
8	Habit	Freshness	Taste
9	Freshness	Taste	Shelf-life
10	Other	Quality label	Appearance

The tests made on rank correlation (Kendall's tau) did not confirm dependency between the order of the factors cited meaning that it is impossible to say unequivocally which factor respondents generally consider as the most important as there is no trend of a single factor appearing on the first place. Values of Kendall's tau varied from -0.433 to 0.06 and are statistically significant at a significance level of $\alpha = 0.05$.

Like in the previous question on factors influencing food purchases, respondents were asked to name three quality labels they know. This confirmed the earlier assumption that Klasa holds the leading position on the Czech food market as the majority of respondents named it as the first option. Many respondents were not able to name a second label, however, Český produkt, Bio, and the response "Other" appeared among the answers. The respondents also named brands that do not belong among quality labels – e.g. private labels of retail chains. Therefore it is possible to conclude that the concept of quality labels is unclear for many respondents and, despite repeated campaigns to promote recognition and knowledge of quality labels, consumers are still unsure about what such labels represent. As the second option respondents named Český produkt, Regionální potravina, and Zdravá potravina. It is also interesting that some reported Chráněné zeměpisné označení a Chráněné označení původu as the second and third answer since those labels are not often known among Czech consumers.

Table 3

Order of labels by awareness

	First label	Second label	Third label
1	Klasa	Other	Regionální potravina
2	Český produkt	Český výrobek	Other
3	Other	Chráněné zeměpisné označení	Chráněné označení původu
4	Bio	Regionální potravina	Český produkt
5		Český produkt	
6		Zdravá potravina	

Even in this case the conducted rank correlation tests (Kendall's tau) did not confirm any dependency between awareness rankings of quality labels, which means that it is not possible to say unequivocally which labels are more significant than others, except for the Klasa label whose position is exceptional. Values of Kendall's tau vary from -0.501 to 0.229 and are statistically significant at a significance level of $\alpha = 0.05$, at the same time there is no visible trend.

Another aim of this paper was to find out opinions of the respondents on food products marked with quality labels. It is noteworthy that 94.4% of respondents rather agree that labelled foodstuffs meet their expectations, but only 58.3% of respondents consider these products actually better (13.9% absolutely agree and 44.4% rather agree) while 30.6% of respondents were neither concurring nor dissenting. Similarly, the respondents answered questions on whether the labelled products are trustworthy where 61.1% of respondents agree with such statement (19.4% absolutely agree and 41.7% rather agree) which may seem interesting for food producers who endeavour to obtain some of the quality labels. Willingness to pay extra money for the labelled food products was confirmed by 72.2% of the respondents which shows a positive trend that consumers are willing to pay more for products which are marked with quality labels and which are expected to have higher quality than unlabelled products. These findings may also be confirmed by one-sample t-test the value of which reached $t = 0$ at a significance level of $\alpha = 0.05$.

Table 4

Respondents' opinion on labelled food products

	1	2	3	4	5	T-test value
Do food products marked with quality labels meet your expectations?	0	94,4	5,6	0	0	Sig = 0, T = 53,09
In your opinion, are labelled food products of better quality?	13,9	44,4	30,6	2,8	2,8	Sig = 0, T = 15,43
In your opinion, are quality labels trustworthy?	19,4	41,7	22,2	8,3	2,8	Sig = 0, T = 13,36
Are you willing to pay more for labelled than unlabelled products?	27,8	44,4	13,9	5,6	2,8	Sig = 0, T = 12,21

Note: 1 – absolutely agree, 2 – rather agree, 3 – neither agree nor disagree, 4 – rather disagree, 5 – absolutely disagree

Conclusion

Based on the trial of quality labels where 36 respondents were interviewed several conclusions can be drawn. In identifying three factors that most affect consumers while shopping food, an aforementioned assumption was confirmed that the most important factor is price. However, the assumption cannot be unequivocally confirmed at this stage of the research. More accurate results will appear with a higher number of respondents and statistically significant correlation. In the opinion of consumers the second most important factor after price is origin of the food, where in addition respondents care what country a product comes from and, in case it is a domestic product, from which region. The third most influential factor according to the gathered information is appearance of the products. Visual characteristics therefore play an important role in food selection. It is also necessary to mention other factors which occurred on various positions from first to fifth, those are: quality, composition of the product, taste, and word of mouth or recommendation. Based on all the previously mentioned information it is possible to conclude that quality labels are not one of the key factors in food selection and thus do not have significant influence on consumer behaviour.

Respondents were also asked to name three labels of quality, most frequently they named Klasa as the first label. Based on the results of this study, Klasa seems to have the highest awareness on the Czech market. Other labels featured on the first place are Český výrobek and Bio. In addition, respondents also named brands which are not considered quality labels but private labels of retail chains. Several respondents were unable to name

any brand of quality. These facts reveal that brand awareness of quality labels in the Czech Republic is generally not high. Such labelling thus has low significance and consumers do not have an entirely clear and accurate idea of what quality labels mean.

The final part of the study examined consumers' views on food products marked with quality labels. Nearly 95% of the respondents rather agree that labelled foodstuffs meet their expectations, while only 58% of consumers actually consider labelled products to have higher quality in comparison with conventional products. As for the question whether quality labels are trustworthy, 61% of those surveyed responded approvingly, while 19% absolutely agree and 42% rather agree with this statement. At the same time, 72% of the respondents are willing to pay more for the labelled food products than for those unlabelled. In conclusion, approximately two thirds of consumers trust quality labels and almost three quarters are willing to pay extra money for such labelled products. However, somewhat paradoxically, the awareness of these labels and their meaning is not high. Therefore, the question remains whether and what actual value labels of quality have on the food market in the Czech Republic.

Acknowledgement

The research was supported by grant F2/94/2015 Analysis of quality labels utilization in the Czech food market and efficiency of campaigns supporting them of Faculty of International Relations, University of Economics, Prague.

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АНОТАЦІЇ

Харчові технології

Вплив попередньої мікрохвильової обробки насіння ріпаку на склад і антиоксидантні властивості пресової ріпакової олії

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Вступ. Досліджено вплив попередньої мікрохвильової обробки ріпакового насіння на вихід пресової олії, значення кислотного та пероксидного числа, жирнокислотний склад, вміст у ній фосфоровмісних сполук, токоферолів, каротиноїдів і стійкість до окиснення.

Матеріали і методи. Значення пероксидних та кислотних чисел встановлювали за допомогою методів IUPAC. Вміст загального фосфору і каротиноїдів визначали спектрофотометричними методами, склад жирних кислот і вміст токоферолів – хроматографічними методами. Тривалість індукційного періоду окиснення олії розраховували за кривою окиснення, ініційованого 2,2-азоізобутиронітрилом.

Результати і обговорення. Перевагою мікрохвильового нагрівання є висока швидкість зростання температури і, як наслідок, висока швидкість зменшення вологості матеріалу. Вологість насіння ріпаку зменшувалась від 13,0 до 7,2 % протягом 10 та 30 хв унаслідок мікрохвильового та звичайного нагрівання відповідно. Встановлено, що в результаті попередньої мікрохвильової обробки ріпакового насіння вихід пресової олії збільшувався на 16—90 %. Основним параметром, що визначав вихід пресової олії, була вологість насіння після обробки. Вихід пресової олії із насіння з однаковою вологістю збільшувався на 16 % за використання мікрохвильової обробки насіння.

Одержані дані свідчать, що жирнокислотний склад олії не зазнавав змін унаслідок мікрохвильової обробки насіння. Проте олія, одержана після такої обробки насіння, мала нижчі значення кислотного й пероксидного числа, більш високий вміст загального фосфору, токоферолів і каротиноїдів. Підвищення окиснювальної стабільності олії після мікрохвильової обробки насіння підтверджено збільшенням тривалості періоду індукції окиснення, ініційованого 2,2-азоізобутиронітрилом. Індукційний період окиснення контрольного зразка олії становив 27 хв, тоді як олії, вилученої із насіння після мікрохвильової обробки, — понад 90 хв.

Висновки. Попередня мікрохвильова обробка ріпакового насіння може бути використана для підвищення ефективності пресового вилучення олії, її біологічної цінності й окислювальної стійкості.

Ключові слова: мікрохвильова обробка, ріпак, олія, окислення, стійкість.

Вплив теплової обробки антиоксидантами на утилізацію активних форм кисню впродовж зберігання солодкого перцю

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Вступ. Попри визнану ефективність теплових обробок та антиоксидантів для зменшення окисного пошкодження індукованого охолодженням, їх сумісний вплив під час зберігання солодкого перцю в цьому аспекті не розглядався.

Матеріали і методи. Плоди перцю Нікіта F1 та Геркулес F1 з попередньою тепловою обробкою композицією антиоксидантів зберігали при $7 \pm 0,5$ °C. Вміст малонового діальдегіду

(МДА) визначали тіобарбітуровим методом. Активність супероксиддисмутази (СОД) визначали за її здатністю інгібувати реакцію автоокислення адреналіну з модифікацією у частині підготовки сировини. Активність пероксидази (ПО) та каталази (КАТ) визначали титруванням нерозкладеного залишку пероксиду водню.

Результати і обговорення. Без додаткових заходів запобігання холододивним пошкодженням, після 24 діб зберігання ураженими є третина плодів перцю. У плодів з тепловою обробкою композицією антиоксидантів холододивні дефекти з'являються лише на 21 добу незалежно від досліджуваного гібриду. Холодові травми скорочуються в 3,9...4,5 рази порівняно з плодами без обробки. Під час зберігання перцю при температурах, вищих за поріг чутливості до холоду, рівень МДА постійно зростає. Теплова обробка антиоксидантами змінює динаміку МДА плодів перцю. До 12 доби зберігання рівень перекисного окислення ліпідів в оброблених зразках залишається стабільним. На кожному наступному етапі зберігання рівень МДА збільшується на 5...15 % залежно від гібриду. На 18 добу (втрата товарної якості контрольних зразків) рівень МДА в оброблених плодах складає в 1,7...2 рази. Теплова обробка антиоксидантами сповільнює швидкість деактивування СОД на 25 %, КАТ на 30..50 %. Активність ПО при зберіганні перцю поступово знижується до моменту втрати товарної якості, а надалі зростає. В оброблених плодах зростання активності ПО відбувається на 12 діб пізніше, ніж у контрольних варіантах. Між активністю досліджуваних ферментів і вмістом МДА встановлено тісні обернені залежності ($r = -0,81 \dots \approx -1$), що свідчить про їх антиоксидантні функції.

Висновки. Сполучення теплової обробки й антиоксидантів для підготовки перцю до зберігання підвищує ефективність функціонування системи утилізації активних форм кисню, що дозволяє мінімізувати пошкодження холододив.

Ключові слова: зберігання, перець, антиоксиданти, обробка.

Розсіювання лазерного випромінювання частинками молока

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Вступ. Основною метою даної статті є теоретичне й експериментальне дослідження розсіювання лазерного випромінювання частинками молока і аналіз характеру розподілу частинок молока за розмірами залежно від концентрації та нагрівання зразків упродовж технологічних процесів.

Матеріали і методи. Обговорюються теорія розсіювання світла і залежність коефіцієнта розсіювання від довжини хвилі світла і параметрів частинок молока. Експериментальне дослідження розсіювання світла частками молока було виконано за допомогою фотонного кореляційного спектрометра 4700с.

Результати і обговорення. Розглядаються різні типи розсіювання, такі як розсіювання Релея і Релея-Ганса, аномальна дифракція, а також відповідні коефіцієнти розсіювання. Досліджені гістограми розподілу розмірів частинок розведеного ($C \leq 10^{-3}$ %) молока (температура 25 °С) і зразків молока з різним вмістом жиру (4,2 %; 5,2 %; 7,4 % при температурі 20 °С і 7,4 % при температурі 50 °С), а також залежність інтенсивності розсіяного світла від кута спостереження.

Метод розсіювання світла може бути використаний для аналізу впливу вмісту молока і технологічних процесів, які пов'язані з нагріванням (пастеризації, стерилізації, гомогенізації) на розподіл частинок молока за розмірами.

Висновки. Розсіювання лазерного випромінювання є передовим методом, який може бути використаний для визначення характеру розподілу часток молока за розмірами. Метод характеризується високою точністю, неруйнівною дією і не вимагає калібрувальних стандартів.

Ключові слова: молоко, жир, казеїн, сироватка, білок, лазер, випромінювання.

Високий тиск у технологіях молока і м'якого сиру

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Вступ. У статті досліджено використання технології високого тиску в молокопереробній промисловості, зокрема обґрунтовано раціональні параметри обробки високим тиском вітчизняної молочної сировини при виробництві молока питного та м'якого сиру.

Матеріали і методи. Об'єкти дослідження: молоко, оброблене високим тиском, сир кисломолочний, вироблений із застосуванням високого тиску. Мінеральний склад молока і сиру кисломолочного визначали методом атомно-абсорбційної спектрофотометрії на атомно-абсорбційному спектрофотометрі «С-115 ПК», реологічні властивості сиру визначали на електромеханічній універсальній випробувальній машині SANS CMT2503 виробництва «Shenzhen SANS Testing Co. Ltd.».

Результати і обговорення. Встановлено механізм впливу тиску, температури і тривалості обробки на мікрофлору вихідної сировини для виробництва молока питного та сиру м'якого, визначено параметри обробки, які забезпечують ефективну інактивацію мікрофлори вітчизняної молочної сировини.

Визначено раціональні параметри обробки: для молока — тиск 300—330 МПа, 40—45 °С, тривалість витримки 30·60¹с; для сиру — тиск 450—580 МПа при температурі 18°С і тривалості обробки 20—30·60¹с.

При оцінці фізико-хімічних показників молока і сиру порівняно з контрольними зразками встановлена незначна зміна вмісту загального білка, жиру, лактози і масової частки сухих речовин.

Вміст основних вітамінів у молоці і м'якому сирі свідчить про їх максимальне збереження. У молоці, обробленому високим тиском, зберігається в 4—6 разів більше жиророзчинних і 2—3 рази більше водорозчинних вітамінів, ніж у пастеризованому, виготовленому за традиційною високотемпературною обробкою. У м'якому сирі збереженість вітамінів в 1,5—2 рази більше, ніж виробленому за традиційною технологією.

За бальною оцінкою сенсорних характеристик найвищі показники отримали продукти, вироблені із застосуванням технології високого тиску: молоко — 98,6 бала, м'який сир — 96,4 бала. Протягом зберігання виражені ознаки псування в контрольних зразках молочних продуктів з'явилися значно швидше, ніж у дослідних зразках, що є наслідком активності ферментів мікроорганізмів, які вижили.

Висновки. Технологія виробництва молока і м'якого сиру із застосуванням високого тиску дозволяє забезпечити мікробіологічну стерильність продуктів, збільшити їх термін зберігання, максимально зберегти ферментно-вітамінний комплекс вихідної сировини.

Ключові слова: тиск, молоко, м'який сир.

Вплив пакування на якість м'яких розсільних сирів, фортифікованих добавкою з морських водоростей

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Вступ. Метою статті є дослідження впливу різних способів пакування і зберігання на споживні властивості м'яких розсільних сирів, фортифікованих добавкою з морських водоростей, а також зміни їх якості у процесі зберігання. За допомогою збагачення м'яких розсільних сирів біологічно активними добавками на органічно зв'язаний йод можна запобігти нестачі йоду в харчуванні людини [1—3].

Матеріали і методи. Об'єктами досліджень були нові м'які розсільні сири: “Зразок 1” — з додаванням добавки з бурої водорості у кількості 0,5 % до маси сиру; “Зразок 2” — з

одночасним додаванням добавки з бурої водорості у кількості 0,5 % і альбумінної маси отриманої з підсириної сироватки у кількості 0,3 % до маси сиру; “Зразок 3” виготовлений термокислотним способом із додаванням добавки з бурої водорості у кількості 0,5 % до маси сиру; “Контроль 1” м’який — розсільний сир, виготовлений традиційним методом; “Контроль 2” — розсільний сир, виготовлений термокислотним способом за традиційною рецептурою. У процесі досліджень використано загальноприйняті та сучасні інструментальні методи.

Органолептичну оцінку якості нових м’яких розсільних сирів здійснювали за розробленою нами 5-бальною шкалою, амінокислотний склад визначали методом іонообмінної рідинно-колункової хроматографії (автоматичний аналізатор амінокислот Т 339).

Результати і обговорення. Аналіз впливу способу пакування на зміни показників якості готових виробів під час зберігання за температури 2...5 °С протягом 60 діб підтвердив, що пакування розсільного сиру та дозрівання упродовж 14 діб у розсолі з подальшим пакуванням і зберіганням у полімерних пакетах “Саран” позитивно впливає на його якість. Якщо у сирі “Контроль 1” кількість вільних амінокислот збільшилась на 19,8 %, то у дослідних розсільних сирах “Зразок 1” - на 21,2 %, “Зразок 1” - на 20,8 %. Розсільний сир “Контроль 2” характеризувався збільшенням вмісту вільних амінокислот на 7,7 % при пакуванні в полімерну плівку, водночас у сирі “Зразок 3” їх кількість збільшилась на 22,2 %. Такий спосіб зберігання розсільних сирів сповільнює процес втрати вологи, сприяє інтенсифікації протеолізу та збільшенню вмісту вільних амінокислот, що позитивно впливає на органолептичні показники.

Висновки. Отримані результати спрямовані на вдосконалення способу дозрівання та пакування м’яких розсільних сирів фортифікованих добавкою з морських водоростей, покращення їх споживних властивостей.

Ключові слова: пакування, зберігання, якість, розсіл, сир.

Вивчення властивостей олії соняшникової високоолеїнового типу за умов гідротермічного впливу

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Вступ. Сучасний тренд життя обумовлює необхідність формування профілю споживчих властивостей виробів із заварного тіста за принципом: доступно, смачно, корисно, зручно. Вивчення властивостей жирового рецептурного компоненту заварного тіста в умовах моделювання технологічного процесу дозволить забезпечити одержання конкурентоспроможної продукції.

Матеріали і методи. Для оцінки перетворень, які відбуваються в модельних системах високоолеїнова соняшникова олія-вода протягом гідротермічного впливу, застосовували фізико-хімічні методи досліджень, а саме: стандартні методики визначення кислотного (КЧ), пероксидного чисел (ПЧ) та числа омилення (ЧО).

Результати і обговорення. Отримані дані підтверджують зростання швидкості гідролізу і накопичення вільних жирних кислот у модельних системах олія-вода внаслідок підвищення температури (від 20 до 100 °С) та рН середовища. За рН 4,5 величина КЧ знаходиться в інтервалі 0,22...0,41, рН 6,0 – 0,19...0,34, рН 8,0 – 0,32...0,38 мг КОН/г. Стрімке зростання значень пероксидного числа спостерігається за підвищення температури до 80...100 °С і становить 3,90...4,70 ммоль 1/2O/кг. Зразки олії, що зазнали гідротермічного впливу за лужної реакції середовища (рН=8,0) характеризуються практично незмінним складом ацилгліцеролів порівняно з необробленою олією і значенням ЧО, яке становить 191,0±06 мг КОН/г. Величина ЧО оброблених зразків дещо менша і становить за значень рН 4,5 – 186,0±0,5; рН 6 – 184,0±0,4 мг КОН/г. Отримані залежності КЧ, ПЧ, ЧО олії соняшникової високоолеїнової від реакції середовища модельної системи олія-вода (1,0:2,5) і температури гідротермічного впливу свідчать про її достатню стабільність. Значення цих показників знаходяться у межах 0,19...0,41 мг КОН/г, 0,95...4,70 ммоль 1/2O/кг, 184,0...191,0 мг КОН/г відповідно.

У модельній системі олія-вода з рН=6,0 і гідромодулем 1,0:2,5 спостерігається незначне накопичення вільних жирних кислот і первинних продуктів окиснення, а величини КЧ і ПЧ за 20° С не перевищують 0,17 мг КОН/г і 1,55 ммоль $1/2O$ /кг відповідно. Протягом перших 20·60 с гідротермічного впливу найбільша швидкість окиснення триацилгліцеролів спостерігається для модельної системи з рН 8,0, в якій ПЧ зростає у 2,3 раза. У системах з рН 4,5 і 6,0 ПЧ зростає у 1,4 і 1,2 раза.

Висновки. Експериментально доведено перспективність використання олії соняшникової високоолеїнового типу як джерела жиру в технологіях кулінарної продукції, зокрема технології виробів на основі заварного тіста.

Ключові слова: соняшник, олія, високоолеїновий, заварне тісто.

Вплив складу шоколадних глазурей на їх властивості і використання в харчовій промисловості

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Вступ. Метою дослідження є одержання поверхнево-активних речовин на основі жирів рослинного і тваринного походження для зниження в'язкості шоколадної глазури

Матеріали і методи. Шоколадні глазури, до складу яких входить, окрім какао-продуктів, суха молочна сироватка та фруктовий концентрат. Поверхнево-активні речовини одержані з відпрацьованої пальмової олії та свинячого жиру методом гліцеролізу.

Результати і обговорення. Додавання 0,4 % лецитину і суміші МГ, ДГ, ТГ свинячого жиру дозволило знизити в'язкість шоколадної глазури до оптимального рівня (2500—2900 мПа·с), а при додаванні суміші МГ, ДГ, ТГ пальмової олії такий показник досягається вже при 0,2 %. Додавання 0,4 % суміші МГ, ДГ, ТГ свинячого жиру дозволило знизити в'язкість шоколадної глазури з додаванням молочної сироватки до 2690 мПа·с, а при додаванні лецитину або суміші МГ, ДГ, ТГ відпрацьованої пальмової олії такий показник досягається вже при 0,3 % ПАР. При додаванні 1% ПАР найбільше зниження в'язкості глазури з додаванням фруктового концентрату було досягнуто з використанням суміші МГ, ДГ, ТГ свинячого жиру (3400 мПа·с). З використанням лецитину досліджувані показники досяг 3900 мПа·с, а з сумішшю МГ, ДГ, ТГ відпрацьованої пальмової олії склав 3600 мПа·с. Використані ПАР знижують в'язкість, але не впливають на смак, запах і відчуття плавлення в роті досліджуваних зразків глазури, а суха молочна сироватка і фруктовий концентрат збагатили смак і аромат шоколадних глазурей. Додавання суміші МГ, ДГ, ТГ відпрацьованої пальмової олії і свинячого жиру погіршило стійкість шоколадних глазурей до «посивіння», особливо глазури з сухою молочною сироваткою. Лецитин і ПАР, отримані на основі відпрацьованої пальмової олії і свинячого жиру, мають розріджувальну здатність, яка перевищує 0,8 %.

Висновки. Поверхнево-активні речовини, отримані на основі відпрацьованої пальмової олії та свинячого жиру, доцільно використовувати в рецептурах шоколадної глазури для зниження в'язкості готового продукту.

Ключові слова: ПАР, шоколад, глазури, в'язкість.

Антибактеріальні біодеградабельні плівки для харчових продуктів

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Вступ. Діоксид титану (TiO₂) має антибактеріальні властивості, але не використовується для створення антибактеріальних плівок. Необхідним є напрям досліджень його

антибактеріальних властивостей у складі пакувань, а також впливу такого пакування на якість харчових продуктів.

Матеріали і методи. Досліджували плівки на основі полівінілового спирту (ПВС) з різною концентрацією TiO_2 . Визначали відносне подовження, міцність при розриві, температуру склування та плавлення плівок з полімерних матеріалів. Крім того, проводили дослідження з визначення інгібуючої дії нанодисперсного порошку TiO_2 на деякі мікроорганізми поверхневим методом та визначення антимікробних властивостей плівки з TiO_2 методом агарових дисків.

Результати і обговорення. Введення додатково до плівки нанодисперсного порошку TiO_2 погіршує еластичність плівки на 20—45 % залежно від доданої кількості, але покращує її міцність на розрив. При додаванні порошку TiO_2 більше 1 % міцність на розрив перевищувала міцність поліетиленової плівки (46,7 МПа). Температура плавлення (текучості) (175 °С) і склування (78 °С) найкраща для зразка з 1 % наповнювача, для нього ж спостерігали найменшу деформацію, що свідчить про утворення найбільш зшитої системи з максимальною кількістю водневих зв'язків. Найоптимальнішим варіантом є оброблення TiO_2 УФ-випромінюванням, адже таким чином можна використовувати мінімальну концентрацію (2,5 %) суспензії TiO_2 . Якщо оброблення УФ не проводиться, то необхідним є використання 10—20 % суміші TiO_2 . Розчини TiO_2 не пригнічують дію грибів і дріжджів. TiO_2 , нанесений на плівку, пригнічує розвиток бактерій (*E. coli* IEM-1, *B. subtilis* БТ-2), також спостерігається затримка росту.

Висновки. Впровадження біодеградабельного пакування з антибактеріальними властивостями є доцільним, однак потребуватиме розроблення або перегляду існуючої нормативної документації. Невисока вартість запропонованої нанодисперсної добавки суттєво не вплине на собівартість пакування, що вкрай важливо в складних економічних умовах.

Ключові слова: біодеградабельність, плівка, упаковка, нанокмполит, діоксид титану.

Біотехнологія, мікробіологія

Вплив олеорезинів на мікробіологічну стабільність варених сосисок з м'яса птиці

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Вступ. Визначали вплив олеорезинів коріандру, мускатного цвіту та чорного перцю на мікробіологічну стабільність варених сосисок з м'яса птиці протягом охолодженого зберігання з метою встановлення термінів придатності.

Матеріали і методи. Досліджували варені сосиски з м'яса птиці з різною часткою нем'ясної сировини. Мікробіологічні показники, а саме: кількість мезофільних аеробних і факультативно-анаеробних мікроорганізмів (МАФАНМ), бактерії групи кишкової палички (БГКП), патогенна флора, в тому числі *Salmonella*, сульфитредукуючі клостридії, *Proteus*, *Listeria monocytogenes*, *Staphylococcus aureus*, плісеневі гриби та дріжджі, визначали загальноприйнятими методами.

Результати і обговорення. Значення МАФАНМ несуттєво відрізнялося для всіх зразків одразу після термообробки. МАФАНМ для зразків з олеорезином коріандру зростали протягом усього дослідження. Наприкінці зберігання значення МАФАНМ досягало $1,1-8,5 \times 10^5$ КУО/г і було значно вищим порівняно з іншими зразками, крім СО 40. Початкові значення МАФАНМ для зразків з олеорезином мускатного цвіту становили $1,0 \times 10^1-1,5 \times 10^2$ КУО/г і були стабільними протягом 7 днів зберігання. Проте після 13 діб зберігання відбулося стрімке зростання. Сосиски з олеорезином чорного перцю демонстрували стабільні значення МАФАНМ протягом усього

періоду досліджень. Після 13 діб зберігання значення МАФАНМ для зразків з олеорезином чорного перцю були значно нижчі порівняно зі зразками, які містять олеорезини коріандру та мускатного цвіту. Початкова кількість плісеньових грибів була <10 КУО/г, на 4 добу зберігання досягла значення $2,5\text{--}7,0 \times 10^1$ КУО/г для зразків з олеорезином коріандру. Зразки з олеорезином мускатного цвіту та чорного перцю демонстрували сталі значення плісеньових грибів, лише на 13 добу зберігання для зразків ВРО100 та МО60 показники становили $2,0 \times 10^1$ та $2,5 \times 10^1$ КУО/г відповідно. Кількість дріжджів для зразків з олеорезином і чорного перцю не відрізнялася після 7 діб зберігання, проте була значно нижчою порівняно зі зразками, які містили олеорезин коріандру. Кількість дріжджів для зразків з олеорезином коріандру зростала протягом усього періоду дослідження. Сосиски з олеорезином мускатного цвіту демонстрували стабільну кількість дріжджів протягом зберігання. Початкове значення показника для ВРО80 становило $3,0 \times 10^1$ КУО/г, проте на 7 добу зберігання спостерігалось пригнічення росту. Кількість дріжджів для зразків з олеорезином чорного перцю і мускатного цвіту були нижча порівняно зі зразками, які містили олеорезин коріандру протягом усього зберігання.

Висновки. Олеорезини мускатного цвіту та чорного перцю мають більшу антимікробну активність порівняно з олеорезином коріандру. Бактеріальний ефект при терміні зберігання більше 10 діб виявляє лише олеорезин чорного перцю, тому при виробництві м'ясомістких продуктів з використанням олеорезину необхідно надавати перевагу олеорезину чорного перцю.

Ключові слова: *сосиска, м'ясо, птиця, олеорезин, зберігання.*

Процеси і обладнання харчових виробництв

Математичне моделювання процесу розділення суспензії на фільтрі із самоочисним фільтрувальним елементом

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Вступ. Метою дослідження є побудова математичної моделі процесу фільтрування суспензії на фільтрі із самоочисним фільтрувальним елементом, що виконаний у вигляді циліндричної пружини стиску.

Матеріали і методи. Дослідження процесу фільтрування проводили на фільтрі із самоочисним фільтрувальним елементом. Як дослідну суспензію використовували молочну сироватку, отриману при виробництві сиру кисломолочного. Концентрацію дисперсної фази в сироватці визначали шляхом центрифугування проб з подальшим висушуванням осаду в сушильний шафі.

Результати і обговорення. Отримана математична модель ґрунтується на моделі процесу фільтрування із закупорюванням кожної пори окремою частинкою.

У моделі враховується, що не всі частинки дисперсної фази, розмір яких перевищує ширину фільтрувальних отворів, будуть їх закупорювати, а лише їх частка, що прямо пропорційна відношенню площі живого перерізу до загальної площі фільтрувальної поверхні.

Математична модель дозволяє визначати тривалість процесу фільтрування з урахуванням об'єму суспензії та встановлювати раціональний період між регенераціями самоочисного фільтрувального елемента.

Порівняння параметрів, отриманих шляхом математичного моделювання із реальним процесом фільтрування молочної сироватки свідчить, що математична модель адекватно відображає процес розділення суспензії на фільтрі із самоочисним фільтрувальним елементом при об'ємі фільтрату від 0 до 5 м³ на 1 м² фільтрувальної поверхні. Середнє відносне відхилення результатів, отриманих з допомогою математичної моделі від експерименту, становить 11 %.

Висновки. Математична модель може бути застосована при розрахунку параметрів процесу фільтрування суспензії на фільтрі із самоочисним фільтрувальним елементом.

Ключові слова: *фільтрування, суспензія, пори, закупорювання, капіляри, регенерація.*

Кінетичні закономірності процесу гранулоутворення комплексних гуміново-органомінеральних добрив у грануляторі з псевдозрідженим шаром

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Вступ. Основну частину відходів виробництва соняшникової олії складає соняшникова зола, яка містить корисні речовини. Метою дослідження є визначення кінетичних закономірностей процесу одержання комплексних гранульованих органо-мінеральних добрив із використанням золи соняшника.

Матеріали і методи. Зневоднення й грануляція гетерогенних рідких систем, що містили мінеральні, гумінові речовини та соняшникову золу, проводилися в апараті з псевдозрідженим шаром, спорядженим спеціальним газорозподільним пристроєм для створення струменево-пульсаційного режиму псевдозрідження при підведенні нагрітого газового теплоносія.

Результати і обговорення. Стійку кінетику процесу гранулоутворення гуміново-органомінеральних добрив складу К:N:Ca:P:Mg:S:Г=23:9:5:2:6:15:2 із коефіцієнтом гранулоутворення $\psi \geq 90$ % досягнуто при середньому значенні різниці температур теплоносія на вході в гранулятор та у шарі $\Delta T = 117^\circ\text{C}$. Одержаний продукт має сфероподібну форму, рівномірне розподілення компонентів на мікрорівні по всьому об'ємі гранул, міцність $\sigma \geq 35$ Н на гранулу, що більш ніж у 3 рази перевищує нормативний показник. При реалізації струменево-пульсаційного гідродинамічного режиму псевдозрідження досягнуто збільшення приведенного питомого навантаження поверхні шару за вологою, віднесеного до корисної різниці температур – $A_f = 0,006 \pm 0,0066$ кг_{вол.}/(м²·год·град).

Результати досліджень можуть бути застосовані при створенні промислового обладнання для виробництва гуміново-органомінеральних добрив із використанням поживних речовин мінерального й органічного походження. Застосування соняшникової золи при створенні нових гуміново-органомінеральних добрив забезпечить раціональне використання природних ресурсів зі збереженням природного ланцюга харчування та покращить екологічну безпеку в результаті утилізації відходів масложирового виробництва.

Висновки. Розроблений спосіб дозволяє утилізувати відходи виробництва соняшникової олії шляхом використання їх при виготовленні нових комплексних гуміново-органомінеральних добрив.

Ключові слова: *кінетика, добриво, псевдозрідження, грануляція, гумат, зола, утилізація.*

Впливи осмотичних тисків середовищ на рівень летальних ефектів в умовах вакуумування

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Вступ. Проведено експериментальні дослідження з визначення дії осмотичних тисків середовищ на рівень летальних ефектів за різних фізичних впливів.

Матеріали і методи. Дослідження виконувались на камерній вакуумпакувальній машині Easy PASC фірми WEBOMATIC (Німеччина). При цьому температури середовищ в усіх випадках витримувалися з обмеженням по максимальним значенням $t \leq 38^\circ\text{C}$, що виключало летальні ефекти по цьому показнику. Вакуум-насос у складі лабораторної установки забезпечував залишковий тиск у вакуумній камері на рівні 0,002—0,004 МПа, що відповідає діапазону температур кипіння середовищ $\sim 20\dots 30^\circ\text{C}$, тому за початкових температур середовищ $t_{(n)} \leq 15^\circ\text{C}$ кипіння не відбувалося, а перехід до початкових температур середовищ $20 < t_{(n)} \leq 38^\circ\text{C}$ забезпечував режим адіабатного кипіння

Результати і обговорення. Відомо, що витримка дріжджів у воді супроводжується підвищенням в клітинах фізичного тиску на рівні осмотичного тиску клітинного соку. Передавання дріжджів в осмотично активний розчин призводить до зменшення фізичного тиску, що повинно мати відповідне відображення за вакуумування. Збільшення осмотичного тиску розчину послаблює рівень летальних ефектів від вакуумування, але разом з тим суттєвий вплив на досягнення летальних ефектів має організація адіабатного кипіння середовищ.

Визначено вплив на рівень летальних ефектів по мікрофлорі таких показників, як час витримки середовища та динаміка зміни тиску у вакуумній камері, кількість сухих речовин у середовищі та значення температурних режимів, а також наявність чи відсутність адіабатного кипіння середовища за умов вакуумування.

Висновки. Вакуумування біологічного середовища за рахунок зниження сталих насичення газами рідиною фази створює обмеження в масообміні між мікробіологічними клітинами і середовищем, а рівень цих обмежень залежить від осмотичного тиску розчину.

Досягнута можливість отримання летальних ефектів по мікрофлорі, що супроводжує харчові середовища, за рахунок сукупності впливів осмотичних тисків і вакуумування.

Ключові слова: *середовище, вакуумування, клітина, кипіння, мікроорганізм.*

Моделювання теплообміну у вільно стікаючих ламінарних плівках з розвинутою хвильовою структурою в режимі випаровування з вільної поверхні

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Вступ. У довгих вертикальних кип'ятильних трубах тепло-гідродинамічні процеси в плівках відбуваються в режимі циклічного перемішування великими хвилями, що не відображено в існуючих моделях теплообміну в плівках.

Матеріали і методи. Досліджуються тепло-гідродинамічні процеси в плівках води та цукрових розчинах у режимі випаровування з вільної поверхні методами математичного й фізичного моделювання на модельних установках з відтворенням реально плинних процесів теплообміну в трубах при концентруванні розчинів.

Результати і обговорення. Розроблена модель теплообміну в ламінарних, догрітих до температури насичення плівках рідини з розвинутою хвильовою структурою як циклічного процесу релаксації нестационарного температурного поля після проходження великих хвиль. Математична модель описує процес розвитку двовимірного температурного поля залежно від числа Пекле та характеристики хвильового руху – довжини великих хвиль. На основі запропонованої моделі отримані кореляції, які пропонуються для узагальнення даних з теплообміну в догрітих до температури кипіння плівках у режимі випаровування з вільної поверхні. Надано узагальнююче рівняння для розрахунку інтенсивності тепловіддачі до насичених плівок цукрових розчинів, яке містить хвильові характеристики плівкової течії, в діапазоні зміни режимних параметрів, характерних для роботи випарних установок цукрової промисловості, а саме: концентрації від 0 до 70 %, щільності зрощення від $0,01 \cdot 10^{-3}$ до $0,6 \cdot 10^{-3}$ м²/с, чисел Пекле від 400 до 25000. Математична модель циклічної релаксації температурного

поля виявилась ефективною для узагальнення даних з теплообміну не тільки ламінарних, а й турбулентних плівок, незважаючи на відсутність у рівняннях перенесення параметрів турбулентності.

Висновки. Встановлено зв'язок хвильової структури плівки з інтенсивністю теплообміну на основі моделі теплообміну з циклічною релаксацією температурного поля в результаті проходження великих хвиль, надані відповідні розрахункові співвідношення.

Ключові слова: *плівка, хвилі, теплообмін, температура, профіль, випаровування.*

Безпека життєдіяльності

Підвищення рівня безпеки праці в харчовій промисловості з урахуванням ризико-орієнтованого підходу

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Вступ. Одним із перспективних наукових напрямків підвищення рівня безпеки праці на виробництві є прогнозування професійних ризиків, безпосередньо пов'язаного з процесом виробництва, і створення на його основі умов для профілактики та запобігання травматизму.

Матеріали і методи. Дослідження проведене на основі методу статистичного аналізу нещасних випадків, які виникли в харчовій галузі за останнє десятиріччя; методу регресійного аналізу; методу головних компонент; методу експертних оцінок; методу апріорного ранжування факторів при обробці результатів експертних оцінок.

Результати і обговорення. У результаті проведених досліджень створено методику підвищення рівня безпеки праці в харчовій галузі на основі прогнозування ризиків виробничого травматизму, яка має суттєве значення для запобігання небезпек і шкідливостей з метою забезпечення сприятливих умов праці, недопущення аварій й усунення професійних захворювань і нещасних випадків. Одним з перспективних наукових напрямків підвищення безпеки виробництва є прогнозування ризиків виробничого травматизму, безпосередньо пов'язаного з процесом виробництва та створення умов уникнення травматизму на основі таких прогнозів. Результати порівняльного аналізу ретроспективного прогнозування методами регресійного аналізу (прогнозу) й удосконаленим методом комбінованого прогнозування на основі методу головних компонент у поєднанні з методом експертного оцінювання свідчать про те, що статистичне прогнозування кількості травмованих на підприємствах харчової промисловості показує більші відхилення від фактичної кількості травмованих осіб (середньоквадратична похибка дорівнює 2,53), ніж комбіноване прогнозування (середньоквадратична похибка складає 0,85). Таким чином, можна зробити висновок про підвищення ефективності прогнозування у середньому на 60 % за рахунок поєднання методу головних компонент з методом експертного оцінювання.

Висновки. Розроблені моделі та методи мають якісно нові властивості і дозволяють підвищити ефективність (точність) прогнозування у середньому на 60 % на основі поєднання методу головних компонент з методом експертного оцінювання, що надає можливість підвищити загальну ефективність профілактики виробничого травматизму на підприємствах харчової промисловості у середньому на 18–23 %.

Ключові слова: *безпека, праця, травматизм, ризик.*

Аннотации

Пищевые технологии

Влияние предварительной микроволновой обработки семян рапса на состав и антиоксидантные свойства прессового рапсового масла

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Введение. Исследовано влияние предварительной микроволновой обработки семян рапса на выход прессового масла, значения кислотного и пероксидного чисел в нем, жирнокислотный состав, содержание фосфоросодержащих соединений, токоферолов, каротиноидов и его стойкость к окислению.

Материалы и методы. Значения пероксидных и кислотных чисел устанавливали методами IUPAC. Содержание общего фосфора и каротиноидов определяли спектрофотометрическими методами, состав жирных кислот и содержание токоферолов – хроматографическими методами. Длительность индукционного периода окисления масла рассчитывали по кривой окисления, инициированного 2,2-азоизобутиронитрилом.

Результаты и обсуждение. Преимуществом микроволнового нагревания является высокая скорость увеличения температуры и, как следствие, высокая скорость снижения влажности материала. Влажность семян рапса снижалась от 13,0 до 7,2 % в течение 10 и 30 мин вследствие микроволнового и обычного нагревания соответственно. Установлено, что вследствие предварительной микроволновой обработки рапсовых семян выход прессового масла увеличивался на 16–90 %. Основным параметром, определяющим выход прессового масла, была влажность семян после предварительной обработки. Выход прессового масла из семян с одинаковой влажностью увеличивался на 16 % при использовании микроволновой обработки семян.

Полученные данные свидетельствуют, что жирнокислотный состав масла не изменялся вследствие микроволновой обработки семян. Однако масло, полученное после такой обработки семян, характеризовалось более низкими значениями кислотного и пероксидного чисел, высшим содержанием общего фосфора, токоферолов и каротиноидов. Увеличение окислительной стабильности масла после микроволновой обработки семян подтверждено более высокой длительностью периода индукции окисления, инициированного 2,2-азоизобутиронитрилом. Индукционный период окисления контрольного образца масла составил 27 мин, тогда как масла, извлеченного после микроволновой обработки семян, — более 90 мин.

Выводы. Предварительная микроволновая обработка рапсовых семян может быть использована для увеличения эффективности прессового извлечения масла, его биологической ценности и окислительной стойкости.

Ключевые слова: *микроволновая обработка, рапс, масло, окисление, стойкость.*

Влияние тепловой обработки антиоксидантами на утилизацию активных форм кислорода при хранении сладкого перца

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Введение. Несмотря на признанную эффективность тепловых обработок и антиоксидантов для уменьшения окислительного повреждения индуцированного охлаждением, их совместное влияние во время хранения сладкого перца в этом аспекте не рассматривалось.

Материалы и методы. Плоды перца Никита F1 и Геркулес F1c предварительной тепловой обработкой композицией антиоксидантов хранили при $7 \pm 0,5$ °С. Содержание малонового диальдегида (МДА) определяли тиобарбитуровым методом. Активность супероксиддисмутазы (СОД) определяли по ее способности ингибировать реакцию аутоокисления адреналина с модификацией в части подготовки сырья. Активность пероксидазы (ПО) и каталазы (КАТ) определяли титрованием неразложенного остатка пероксида водорода.

Результаты и обсуждение. Без применения дополнительных мер при хранении перца после 24 суток треть всех плодов повреждается холодом. У плодов с тепловой обработкой композицией антиоксидантов повреждения холодом появляются только на 21 сутки независимо от исследуемого гибрида. Холодовые травмы сокращаются в 3,9 ... 4,5 раза по сравнению с плодами без обработки. Во время хранения перца при температурах выше порога чувствительности к холоду, уровень МДА постоянно растет. Тепловая обработка антиоксидантами меняет динамику МДА плодов перца. До 12 суток хранения уровень перекисного окисления липидов в обработанных образцах остается стабильным. На каждом следующем этапе хранения уровень МДА увеличивается на 5 ... 15 % в зависимости от гибрида. На 18 сутки (потеря товарного качества контрольных образцов) уровень МДА в обработанных плодах ниже в 1,7 ... 2 раза. Тепловая обработка антиоксидантами замедляет скорость инактивирования СОД на 25 %, КАТ на 30..50 %. Активность ПО при хранении перца постепенно снижается до момента потери товарного качества, а потом возрастает. В обработанных плодах рост активности ПО происходит на 12 суток позже, чем в контрольных вариантах. Между активностью исследуемых ферментов и содержанием МДА установлены тесные обратные корреляционные зависимости ($r = -0,81 \dots -1$), что свидетельствует об их антиоксидантных функциях.

Выводы. Сочетание тепловой обработки и антиоксидантов для подготовки перца к хранению повышает эффективность функционирования системы утилизации активных форм кислорода, что позволяет минимизировать повреждения холодом.

Ключевые слова: хранение, перец, антиоксиданты, обработка.

Рассеивание лазерного излучения частицами молока

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Введение. Основной целью данной статьи является теоретическое и экспериментальное исследование рассеивания лазерного излучения частицами молока и анализ характера распределения частиц молока по размерам в зависимости от концентрации и нагрева образцов в ходе технологических процессов.

Материалы и методы. Обсуждаются теория рассеивания света и зависимость коэффициента рассеивания от длины волны света и параметров частиц молока. Экспериментальное исследование рассеивания света частицами молока было выполнено с помощью фотонного корреляционного спектрометра 4700с.

Результаты и обсуждение. Рассматриваются различные типы рассеивания, такие как рассеивание Релея и Рэлея-Ганса, аномальная дифракция, а также соответствующие коэффициенты рассеивания. Исследованы гистограммы распределения размеров частиц разбавленного ($C \leq 10^{-3}$ %) молока (температура 25 °С) и образцов молока с различным

содержанием жира (4,2 %; 5,2 %; 7,4 % при температуре 20 °С и 7,4 % при температуре 50 °С), а также зависимость интенсивности рассеянного света от угла наблюдения.

Метод рассеивания света может быть использован для анализа влияния содержания молока и технологических процессов, которые связаны с нагреванием (пастеризации, стерилизации, гомогенизация) на распределение частиц молока по размерам.

Выводы. Рассеивание лазерного излучения является передовым методом, который может быть использован для определения характера распределения частиц молока по размерам. Метод характеризуется высокой точностью, неразрушающим действием и не требует калибровочных стандартов.

Ключевые слова: молоко, жир, казеин, сыворотка, белок, лазер, излучение.

Высокое давление в технологиях молока и мягкого сыра

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Введение. Представленная работа посвящена использованию технологии высокого давления в молокоперерабатывающей промышленности, в частности обоснованию рациональных параметров обработки высоким давлением молочного сырья при производстве молока питьевого и мягкого сыра.

Материалы и методы. Объекты исследования: молоко, обработанное высоким давлением, сыр, произведенный с применением ВД. Минеральный состав молока и сыра определяли методом атомно-абсорбционной спектrophотометрии на атомно-абсорбционном спектрофотометре «С-115 ПК», реологические свойства сыра определяли на электромеханической универсальной испытательной машине SANS CMT2503 производства «Shenzhen SANS Testing Co. Ltd. ».

Результаты и обсуждение. Установлен механизм воздействия давления, температуры и продолжительности обработки на микрофлору исходного сырья для производства молока питьевого и сыра мягкого, определены параметры обработки, которые обеспечивают эффективную инактивацию микрофлоры молочного сырья. Определены рациональные параметры обработки: для молока – давление 300—330 МПа, 40—45 °С, продолжительность выдержки 30-60¹с; для сыра – давление 450—580 МПа при температуре 18 °С и продолжительности обработки 20—30-60¹ с. При оценке физико-химических показателей молока и сыра по сравнению с контрольными образцами установлено незначительное изменение содержания общего белка, жира, лактозы и массовой доли сухих веществ. Содержание основных витаминов в молоке и мягком сыре свидетельствует об их максимальной сохраняемости. В молоке, обработанном высоким давлением сохраняется в 4—6 раз больше жирорастворимых и 2—3 раза больше водорастворимых витаминов, чем в пастеризованном, изготовленном с применением традиционной высокотемпературной обработки. В мягком сыре сохранность витаминов в 1,5—2 раза больше, чем произведенном по традиционной технологии. Согласно балловой оценке сенсорных характеристик высокие показатели получили продукты, произведенные с применением технологии высокого давления: молоко — 98,6 балла, мягкий сыр — 96,4 балла. В период хранения выраженные признаки порчи в контрольных образцах молочных продуктов появились значительно быстрее, чем в опытных образцах, что является следствием активности ферментов выживших микроорганизмов.

Выводы. Технология производства молока и мягкого сыра с применением высокого давления позволяет обеспечить микробиологическую стерильность продуктов, увеличить

их срок хранения, максимально сохранить ферментно-витаминный комплекс исходного сыра.

Ключевые слова: *давление, молоко, мягкий сыр.*

Влияние упаковки на качество мягких рассольных сыров, фортифицированных добавкой из морских водорослей

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Введение. Целью статьи является исследование влияния различных способов упаковки и хранения на потребительские свойства мягких рассольных сыров, фортифицированных добавкой с морских водорослей, а также изменения их качества в процессе хранения. С помощью обогащения мягких рассольных сыров биологически активными добавками органически связанным йодом можно предотвратить недостаток йода в питании человека [1-3].

Материалы и методы. Объектами исследований были новые мягкие рассольные сыры: “Образец 1” с добавлением добавки с бурой водоросли в количестве 0,5 % к массе сыра; “Образец 2” с одновременным добавлением добавки с бурой водоросли в количестве 0,5 % и альбуминной массы, полученной с подсырной сыворотки в количестве 0,3 % к массе сыра; “Образец 3” изготовлен термокислотным способом с добавлением добавки с бурой водоросли в количестве 0,5 % к массе сыра; “Контроль 1” — мягкий рассольный сыр, изготовленный традиционным методом; “Контроль 2” — рассольный сыр, изготовленный термокислотным способом по традиционной рецептуре. Органолептическую оценку качества новых мягких рассольных сыров осуществляли по разработанной нами 5-бальной шкале. Аминокислотный состав определяли методом ионообменной жидкостно-колоночной хроматографии (автоматический анализатор аминокислот Т 339).

Результаты и обсуждение. Результаты анализа способа упаковки на изменения показателей качества готовых изделий при температуре 2 ... 5 °С в течение 60 суток свидетельствуют о том, что созревание рассольного сыра в течение 14 суток в рассоле с последующей упаковкой и хранением в полимерных пакетах «Саран» положительно влияет на его качество. В сыре “Контроль 1” количество свободных аминокислот увеличилась на 19,8 %, в опытных рассольных сырах “Образец 1” — на 21,2 %, “Образец 2” — на 20,8 %. “Контроль 2” характеризовался увеличением содержания свободных аминокислот на 7,7 % при упаковке в полимерную пленку. В то же время в сыре “Образец 3” их количество увеличилось на 22,2 %. Такой способ хранения рассольных сыров замедляет процесс потери влаги, способствует интенсификации протеолиза и увеличению содержания свободных аминокислот, что положительно влияет на органолептические показатели.

Выводы. Полученные результаты направлены на совершенствование способа созревания и упаковки мягких рассольных сыров, улучшение их потребительских свойств.

Ключевые слова: *упаковка, хранение, качество, рассол, сыр.*

Изучение свойств масла подсолнечного высокоолеинового типа в условиях гидротермического воздействия

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Введение. Современный тренд жизни обуславливает необходимость формирования профиля потребительских свойств изделий из заварного теста по принципу: доступно, вкусно, полезно, удобно. Изучение свойств жирового рецептурного компонента заварного теста в условиях моделирования технологического процесса позволит управлять и обеспечить получение конкурентоспособной продукции.

Материалы и методы. Для оценки преобразований, которые происходят в модельных системах высокоолеиновое подсолнечное масло-вода в течение гидротермического воздействия, применяли физико-химические методы исследований, а именно стандартные методики определения кислотного (КЧ), пероксидного (ПЧ) чисел, числа омыления (ЧО).

Результаты и обсуждение. Полученные данные подтверждают возрастание скорости гидролиза и накопление свободных жирных кислот в модельных системах масло-вода вследствие повышения температуры (от 20 до 100° С) и рН среды. При рН 4,5 величина КЧ находится в интервале 0,22...0,41, рН 6,0 – 0,19...0,34, рН 8,0 – 0,32...0,38 мг КОН/г. Стремительное возрастание значений пероксидного числа наблюдается при повышении температуры до 80...100° С и составляет 3,90...4,70 ммоль 1/2О/кг. Образцы масла, подвергнутые гидротермическому воздействию в щелочной среде (рН=8,0), характеризуются практически неизменным составом ацилглицеролов в сравнение с необработанным маслом и значением ЧО, составляющим 191,0±0,6 мг КОН/г. Величина ЧО обработанных образцов несколько меньшая и составляет при рН 4,5 – 186,0±0,5; рН 6 – 184,0±0,4 мг КОН/г. Полученные зависимости КЧ, ПЧ, ЧО масла подсолнечного высокоолеинового от реакции среды модельной системы масло-вода (1,0:2,5) и температуры гидротермического воздействия свидетельствуют о ее достаточной стабильности. Значения этих показателей находятся в интервале 0,19...0,41 мг КОН/г, 0,95...4,70 ммоль 1/2О/кг, 184,0...191,0 мг КОН/г соответственно.

В модельной системе масло-вода с рН=6,0 и гидромодулем 1,0:2,5 наблюдается незначительное накопление свободных жирных кислот и первичных продуктов окисления, а величины КЧ и ПЧ при 20° С не превышают 0,17 мг КОН/г и 1,55 ммоль1/2О/кг соответственно. В течение первых 20-60 с гидротермического воздействия наибольшая скорость окисления триацилглицеролов наблюдается для модельной системы с рН 8,0, в которой ПЧ возрастает в 2,3 раза. В системах с рН 4,5 и 6,0 ПЧ возрастает в 1,4 и 1,2 раза.

Выводы. Экспериментально доказана перспективность использования масла подсолнечного высокоолеинового типа в качестве источника жира в технологиях кулинарной продукции, в том числе технологии изделий на основе заварного теста.

Ключевые слова: *подсолнух, масло, высокоолеиновый, заварное тесто.*

Влияние состава шоколадных глазурей на их свойства и использование в пищевой промышленности

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Введение. Целью работы было получение поверхностно-активных веществ на основе жиров растительного и животного происхождения для снижения вязкости шоколадных глазурей

Материалы и методы. Шоколадные глазури, в состав которых входит, кроме какао-продуктов, сухая молочная сыворотка и фруктовый концентрат. Поверхностно-активные

вещества получены из отработанного пальмового масла и свиного жира методом глицеролиза.

Результаты и обсуждение. Добавление 0,4 % лецитина и смеси МГ, ДГ, ТГ свиного жира позволило снизить вязкость шоколадной глазури до оптимального уровня (2500–2900 мПа•с), а при добавлении смеси МГ, ДГ, ТГ пальмового масла такой показатель достигается уже при 0,2 %. Добавление 0,4 % смеси МГ, ДГ, ТГ свиного жира позволяет снизить вязкость шоколадной глазури с добавлением молочной сыворотки до 2690 мПа•с, а при добавлении лецитина или смеси МГ, ДГ, ТГ отработанного пальмового масла такой показатель достигается уже при 0,3 % ПАВ. При добавлении 1 % ПАВ наибольшее снижение вязкости глазури с добавлением фруктового концентрата достигалось с использованием смеси МГ, ДГ, ТГ свиного жира (3400 мПа•с). С использованием лецитина исследуемый показатель достигал 3900 мПа•с, а со смесью МГ, ДГ, ТГ отработанного пальмового масла составил 3600 мПа•с. Используемые ПАВ снижают вязкость, но не влияют на вкус, запах и ощущение плавления во рту исследуемых образцов глазури, а сухая молочная сыворотка и фруктовый концентрат обогатили вкус и аромат шоколадных глазурей. Добавление смеси МГ, ДГ, ТГ отработанного пальмового масла и свиного жира ухудшило устойчивость шоколадных глазурей к «поседению», особенно глазури с сухой молочной сывороткой. Лецитин и ПАВ, полученные на основе отработанного пальмового масла и свиного жира, имеют разжижающую способность выше 0,8 %.

Выводы. Поверхностно-активные вещества, полученные на основе отработанного пальмового масла и свиного жира, целесообразно использовать в рецептурах шоколадной глазури для снижения вязкости готового продукта.

Ключевые слова: ПАВ, шоколад, глазурь, вязкость.

Антибактериальные биоразлагаемые пленки для пищевых продуктов

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Введение. Диоксид титана (TiO₂) обладает антибактериальными свойствами, но не используется для создания антибактериальных пленок. Необходимым является исследование его антибактериальных свойств в составе упаковки, а также влияние такой упаковки на качество пищевых продуктов.

Материалы и методы. Исследовали пленки на основе поливинилового спирта (ПВС) с различной концентрацией TiO₂. Определяли относительное удлинение, прочность при разрыве, температуру стеклования и плавления пленок из полимерных материалов. Микробиологические исследования по установлению ингибирующего действия нанодисперсного порошка TiO₂ на некоторые патогенные микроорганизмы проводили поверхностным методом. Антибактериальные свойства пленки с TiO₂ изучали методом агаровых дисков.

Результаты и обсуждение. Введение дополнительно к пленке нанодисперсного порошка TiO₂ ухудшает эластичность пленки на 20—45 % в зависимости от добавленного количества, но улучшает ее прочность на разрыв. При добавлении порошка TiO₂ более 1 % масс. прочность на разрыв превышала прочность полиэтиленовой пленки (46,7 МПа). Температура плавления (текучести) (175 °С) и стеклования (78 °С) лучшая для образца с 1 % наполнителя, для него же наблюдали наименьшую деформацию, что свидетельствует об образовании наиболее сшитой системы с максимальным количеством водородных связей. Самым оптимальным вариантом является обработка TiO₂ УФ-излучением, ведь

таким образом можно использовать минимальную концентрацию (2,5 %) суспензии TiO_2 . Если обработка УФ не проводится, то необходимым является использование 10—20 % смеси TiO_2 . Растворы с TiO_2 не подавляют действие грибов и дрожжей. TiO_2 , нанесенный на пленку, подавляет развитие бактерий (*E. coli* IEM-1, *B. subtilis* БТ-2), наблюдается задержка роста.

Выводы. Внедрение биоразлагаемой упаковки с антибактериальными свойствами целесообразно, однако требует разработки или пересмотра существующей нормативной документации. Невысокая цена предложенной нанодисперсной добавки существенно не повлияет на себестоимость упаковки, что крайне важно в сложных экономических условиях.

Ключевые слова: биоразлагаемость, пленка, упаковка, нанокompозит, диоксид титана.

Биотехнология, микробиология

Влияние олеорезинов на микробиологическую стабильность вареных сосисок с мяса птицы

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Введение. Определяли влияние олеорезинов кориандра, мускатного цвета и черного перца на микробиологическую стабильность вареных сосисок из мяса птицы во время охлажденного хранения.

Материалы и методы. Исследовали вареные сосиски из мяса птицы с разным количеством немясного сырья. Микробиологические показатели, а именно: количество мезофильных аэробных и факультативно-анаэробных микроорганизмов (МАФАНМ), бактерии группы кишечной палочки (БГКП), патогенная флора, в том числе *Salmonella*, сульфитредуцирующие клостридии, *Proteus*, *Listeria monocytogenes*, *Staphylococcus aureus*, плесневые грибы и дрожжи, определяли общепринятыми методами.

Результаты и обсуждение. Значения МАФАНМ незначительно отличались для всех образцов сразу после термообработки. МАФАНМ для образцов с олеорезином кориандра увеличивались на протяжении всего исследования. В конце хранения значение МАФАНМ достигало $1,1-8,5 \times 10^5$ КОЕ/г и было значительно выше по сравнению с другими образцами, кроме СО 40. Начальные значения МАФАНМ для образцов с олеорезином мускатного цвета составляли $1,0 \times 10^1 - 1,5 \times 10^2$ КОЕ/г и были стабильными на протяжении 7 суток хранения. Однако после 13 суток хранения произошел стремительный рост. Сосиски с олеорезином черного перца демонстрировали стабильное значение МАФАНМ на протяжении всего периода исследований. После 13 суток хранения значения МАФАНМ для образцов с олеорезином черного перца были значительно ниже по сравнению с образцами, которые содержали олеорезины кориандра и мускатного цвета. Начальное количество плесневых грибов составило <10 КОЕ/г, на 4 сутки хранения достигло значения $2,5-7,0 \times 10^1$ КОЕ/г для образцов с олеорезином мускатного цвета. Образцы с олеорезинами мускатного цвета и черного перца демонстрировали стабильное значение для плесневых грибов, только на 13 сутки хранения для образцов ВРО100 и МО60 показатели составили $2,0 \times 10^1$ и $2,5 \times 10^1$ КОЕ/г соответственно. Количество

дрожжей для образцов с олеорезинами мускатного цвета и черного перца не отличалось после 7 суток хранения, однако было значительно ниже в сравнении с образцами, которые содержали олеорезин кориандра. Количество дрожжей для образцов с олеорезином кориандра увеличивалось на протяжении всего периода исследований. Сосиски с олеорезином мускатного цвета демонстрировали стабильное количество дрожжей на протяжении хранения. Начальное значение показателя для ВРО80 составило $3,0 \times 10^1$ КОЕ/г, однако на 7 сутки хранения наблюдалось угнетение роста. Количество дрожжей для образцов с олеорезином черного перца и мускатного цвета было ниже в сравнении с образцами, содержащими олеорезин кориандра, на протяжении всего исследования.

Выводы. Олеорезины мускатного цвета и черного перца имеют большую антимикробную активность в сравнении с олеорезином кориандра. Бактериальный эффект при сроке хранения больше 10 суток проявляет только олеорезин черного перца, потому при производстве мясосодержащих продуктов с использованием олеорезинов необходимо использовать олеорезин черного перца.

Ключевые слова: *сосиска, мясо, птица, олеорезин, хранение.*

Процессы и оборудование пищевых производств

Математическое моделирование процесса разделения суспензии на фильтре с самоочищающимся фильтрующим элементом

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Введение. Целью работы было построение математической модели процесса фильтрации суспензии на фильтре с самоочищающимся фильтрующим элементом, который выполнен в виде цилиндрической пружины сжатия.

Материалы и методы. Исследование процесса фильтрации проводили на фильтре с самоочищающимся фильтрующим элементом. В качестве исследовательской суспензии использовали молочную сыворотку, полученную при производстве творога. Концентрацию дисперсной фазы в сыворотке определяли путем центрифугирования проб с последующим высушиванием осадка в сушильном шкафу.

Результаты и обсуждение. Полученная математическая модель основывается на модели процесса фильтрации с закупориванием каждой поры отдельной частицей.

В модели учитывается, что не все частицы дисперсной фазы, размер которых превышает ширину фильтровальных отверстий, будут их закупоривать, а только их часть, прямо пропорциональна отношению площади живого сечения к общей площади фильтрующей поверхности.

Математическая модель позволяет определять продолжительность процесса фильтрации, исходя из объема суспензии, и устанавливать оптимальный период между регенерациями самоочищающегося фильтрующего элемента.

Сравнение параметров, полученных путем математического моделирования, с реальным процессом фильтрации молочной сыворотки свидетельствует, что математическая модель адекватно отражает процесс разделения суспензии на фильтре с самоочищающимся фильтрующим элементом при объеме фильтрата от 0 до 5 м^3 на 1 м^2 фильтрующей поверхности. Среднее относительное отклонение

результатов, полученных с помощью математической модели, от эксперимента составляет 11 %.

Выводы. Математическая модель может быть применена при расчете параметров процесса фильтрования суспензии на фильтре с самоочищающимся фильтрующим элементом.

Ключевые слова: *фильтрование, суспензия, укупорки, капилляры, регенерация.*

Кинетические закономерности процесса гранулообразования комплексных гуминово-органоминеральных удобрений в грануляторе с псевдооживленным слоем

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Введение. Основную часть отходов производства подсолнечного масла составляет подсолнечная зола, которая содержит полезные вещества. Целью работы является определение кинетических закономерностей процесса получения комплексных гранулированных органоминеральных удобрений с использованием золы подсолнечника.

Материалы и методы. Обезвоживание и грануляция гетерогенных жидких систем, содержащих минеральные, гуминовые вещества и подсолнечную золу, проводились в аппарате с псевдооживленным слоем, оснащенным специальным газораспределительным устройством для создания струйно-пульсационного режима псевдооживления при подведении нагретого газового теплоносителя.

Результаты и обсуждение. Устойчивую кинетику процесса гранулообразования гуминово-органоминеральных удобрений состава К:N:Ca:P:Mg:S:Г=23:9:5:2:6:15:2 с коэффициентом гранулообразования $\psi \geq 90\%$ достигнуто при среднем значении разности температур теплоносителя на входе в гранулятор и в слое $\Delta T = 117^\circ\text{C}$. Полученный продукт имеет сферообразную форму, равномерное распределение компонентов на микроуровне по всему объему гранул, прочность $\sigma \geq 35$ Н на гранулу, что более чем в 3 раза превышает нормативный показатель. При реализации струйно-пульсационного гидродинамического режима псевдооживления достигнуто увеличение приведенной удельной нагрузки поверхности слоя по влаге, отнесенного к полезной разности температур $-A_f = 0,006 \div 0,0066$ кг_{вол.}/(м²·час·град). Результаты исследований могут быть применены при создании промышленного оборудования для производства гуминово-органоминеральных удобрений с использованием питательных веществ минерального и органического происхождения. Применение подсолнечной золы при создании новых гуминово-органоминеральных удобрений обеспечит рациональное использование природных ресурсов с сохранением природной цепи питания и улучшит экологическую безопасность в результате утилизации отходов масложирового производства.

Выводы. Разработанный способ позволяет утилизировать отходы производства подсолнечного масла путем использования при изготовлении новых комплексных гуминово-органоминеральных удобрений.

Ключевые слова: *кинетика, удобрение, псевдооживление, грануляция, гумат, зола, утилизация.*

Влияние осмотических давлений сред на уровень летальных эффектов микроорганизмов в условиях вакуумирования

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Введение. Проведены экспериментальные исследования по определению действия осмотических давлений среды на уровень летальных эффектов при различных физических воздействиях.

Материалы и методы. Исследования выполнялись на камерной вакуумупаковочной машине Easy PASC фирмы WEBOMATIC (Германия). При этом температуры сред во всех случаях выдерживались с ограничением по максимальным значениям $t \leq 38$ °C, что исключало летальные эффекты по этому показателю. Вакуум-насос в составе лабораторной установки обеспечивал остаточное давление в вакуумной камере на уровне 0,002-0,004 МПа, что соответствует диапазону температур кипения сред $\sim 20...30$ °C, поэтому при начальных температурах сред $t_{(н)} \leq 15$ °C кипение не имело места, а переход к исходным температур сред $20 < t_{(н)} \leq 38$ °C обеспечивал режим адиабатного кипения.

Результаты и обсуждение. Известно, что выдержка дрожжей в воде сопровождается повышением в клетках физического давления на уровне осмотического давления клеточного сока. Передача дрожжей в осмотически активный раствор приводит к уменьшению физического давления, что должно иметь соответствующее отражение при вакуумировании. Увеличение осмотического давления раствора ослабляет уровень летальных эффектов от вакуумирования, но вместе с тем существенное влияние на достижение летальных эффектов имеет организация адиабатного кипения сред. Определено влияние на уровень летальных эффектов по микрофлоре таких показателей, как время выдержки среды и динамика изменения давления в вакуумной камере, количество сухих веществ в среде и значения температурных режимов, а также наличие или отсутствие адиабатного кипения среды в условиях вакуумирования.

Выводы. Вакуумирование биологической среды за счет снижения постоянных насыщения газами жидкой фазы создает ограничения в массообмене между микробиологическими клетками и средой, а уровень этих ограничений зависит от осмотического давления раствора.

Достигнута возможность получения летальных эффектов по микрофлоре, которая сопровождает пищевые среды, за счет совокупности воздействий осмотических давлений и вакуумирования.

Ключевые слова: *среда, вакуумирование, клетка, кипение, микроорганизм.*

Моделирование теплообмена в свободно стекающих ламинарных пленках с развитой волновой структурой в режиме испарения со свободной поверхностью

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Введение. В длинных вертикальных кипятильных трубах тепло- гидродинамические процессы в пленках протекают в режиме циклического ее перемешивания большими волнами, что не отражено в существующих моделях теплообмена в пленках.

Материалы и методы. Исследуются тепло- гидродинамические процессы в пленках воды и сахарных растворах в режиме испарения со свободной поверхности методами математического и физического моделирования на модельных установках с воспроизведением реально изменяющихся процессов теплообмена в трубах при концентрировании растворов.

Результаты и обсуждение. Разработана модель теплообмена в ламинарных, догретых до температуры насыщения пленках жидкости с развитой волновой структурой как циклического процесса релаксации нестационарного температурного поля после прохождения больших волн. Математическая модель описывает процесс развития двумерного температурного поля в зависимости от числа Пекле и характеристики

волнового движения — длины больших волн. На основе предложенной модели получены корреляции, которые предлагаются для обобщения данных по теплообмену в догретых до температуры кипения пленках в режиме испарения со свободной поверхности. Предоставлено обобщающее уравнение для расчета интенсивности теплоотдачи к насыщенным пленкам сахарных растворов, которое содержит волновые характеристики пленочного течения, в диапазоне изменения режимных параметров, характерных для работы испарительных установок сахарной промышленности, а именно: концентрации от 0 до 70 %, плотности орошения от $0,01 \cdot 10^{-3}$ до $0,6 \cdot 10^{-3} \text{ м}^2/\text{с}$, чисел Пекле от 400 до 25000. Математическая модель циклической релаксации температурного поля оказалась эффективной для обобщения данных по теплообмену не только ламинарных, а и турбулентных пленок, несмотря на отсутствие в уравнениях переноса параметров турбулентности.

Выводы. Установлена связь волновой структуры пленки с интенсивностью теплообмена на основе модели теплообмена с циклической релаксацией температурного поля в результате прохождения больших волн, предоставлены соответствующие расчетные соотношения.

Ключевые слова: пленка, волны, теплообмен, температура, профиль, испарение.

Безопасность жизнедеятельности

Повышение уровня безопасности труда в пищевой промышленности с учетом риск-ориентированного подхода

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Введение. Одним из перспективных научных направлений повышения уровня безопасности труда на производстве является прогнозирование профессиональных рисков, непосредственно связанного с процессом производства, и создание на его основе условий для профилактики и предотвращения травматизма.

Материалы и методы. Исследования проведено на основе метода статистического анализа несчастных случаев, которые возникли в пищевой отрасли за последнее десятилетие; метода регрессионного анализа; метода главных компонент; метода экспертных оценок; метода априорного ранжирования факторов при обработке результатов экспертных оценок.

Результаты и обсуждение. В результате проведенных исследований создана методика повышения уровня безопасности труда в пищевой отрасли на основе прогнозирования рисков производственного травматизма, которая имеет существенное значение для предотвращения опасностей с целью обеспечения благоприятных условий труда, недопущения аварий и устранения профессиональных заболеваний и несчастных случаев. Одним из перспективных научных направлений повышения безопасности производства является прогнозирование рисков производственного травматизма, непосредственно связанного с процессом производства и создание условий предотвращения травматизма на основе таких прогнозов. Результаты сравнительного анализа ретроспективного прогнозирования методами регрессионного анализа (прогноза) и усовершенствованным методом комбинированного прогнозирования, на основе метода главных компонент в сочетании с методом экспертной оценки свидетельствуют о том, что статистическое прогнозирование количества травмированных на предприятиях пищевой промышленности показывает большие отклонения от фактического количества травмированных (среднеквадратичная погрешность равна 2,53), чем комбинированное

прогнозирование (среднеквадратичная погрешность составляет 0,85). Таким образом, можно сделать вывод о повышении эффективности прогнозирования в среднем на 60 % за счет сочетания метода главных компонент с методом экспертной оценки.

Выводы. Разработанные модели и методы имеют качественно новые свойства и позволяют повысить эффективность (точность) прогнозирования в среднем на 60 % на основе сочетания метода главных компонент с методом экспертной оценки, что дает возможность повысить общую эффективность профилактики производственного травматизма на предприятиях пищевой промышленности в среднем на 18—23 %.

Ключевые слова: *безопасность, труд, травматизм, риск.*

Instructions for authors



Dear colleagues!

The Editorial Board of scientific periodical
«**Ukrainian Food Journal**»
invites you to publication of your scientific research.

Requirements for article:

Language – English, Ukrainian, Russian

Size of the article – 8-15 pages in Microsoft Word 2003 and earlier versions with filename extension *.doc (!)

All article elements should be in Times New Roman, font size 14, 1 line intervals, margins on both sides 2 cm.

The structure of the article:

1. The title of the article
2. Authors (full name and surname)
3. Institution, where the work performed.
4. Abstract (2/3 of page). The structure of the abstract should correspond to the structure of the article (Introduction, Materials and methods, Results and discussion, Conclusion).
5. Key words.

Points from 1 to 5 should be in English, Ukrainian and Russian.

6. The main body of the article should contain the following obligatory parts:

- Introduction
- Materials and methods
- Results and discussing
- Conclusion
- References

If you need you can add another parts and divide them into subparts.

7. The information about the author (Name, surname, scientific degree, place of work, email and contact phone number).

All figures should be made in graphic editor, the font size 14.

The background of the graphs and charts should be only in white color. The color of the figure elements (lines, grid, text) - in black color.

Figures and EXCEL format files with graphs additionally should submit in separate files.

Photos are not appropriate to use.

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Шановні колеги!

Редакційна колегія наукового періодичного видання «**Ukrainian Food Journal**» запрошує Вас до публікації результатів наукових досліджень.

Вимоги до оформлення статей

Мови статей – англійська, українська, російська
Рекомендований обсяг статті – **8-15 сторінок** формату А4.
Стаття виконується в текстовому редакторі Microsoft Word 2003, в форматі *.doc.
Для всіх елементів статті шрифт – **Times New Roman**, кегль – **14**, інтервал – 1.
Всі поля сторінки – по 2 см.

Структура статті:

1. УДК.
2. **Назва статті.**
3. Автори статті (ім'я та прізвище повністю, приклад: Денис Озеряно).
4. *Установа, в якій виконана робота.*
5. Анотація. Обов'язкова структура анотації:
 - Вступ (2-3 рядки).
 - Матеріали та методи (до 5 рядків)
 - Результати та обговорення (пів сторінки).
 - Висновки (2-3 рядки).
6. Ключові слова (3-5 слів, але не словосполучень).

Пункти 2-6 виконати англійською, українською та російською мовами.

7. Основний текст статті. Має включати такі обов'язкові розділи:
 - Вступ
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За необхідності можна додавати інші розділи та розбивати їх на підрозділи.

8. Авторська довідка (Прізвище, ім'я та по батькові, вчений ступінь та звання, місце роботи, електронна адреса або телефон).
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Рисунки виконуються якісно. Скановані рисунки не приймаються. Розмір тексту на рисунках повинен бути **співрозмірним (!)** тексту статті. **Фотографії бажано не використовувати.**

Фон графіків, діаграм – лише білий. Колір елементів рисунку (лінії, сітка, текст) – чорний (не сірий).

Рисунки та графіки EXCEL з графіками додатково подаються в окремих файлах.

Скорочені назви фізичних величин в тексті та на графіках позначаються латинськими літерами відповідно до системи СІ.

В списку літератури повинні переважати статті та монографії іноземних авторів, які опубліковані після 2000 року.

Правила оформлення списку літератури

В Ukrainian Food Journal взято за основу загальноприйняте в світі спрощене оформлення списку літератури згідно стандарту Garvard. Всі елементи посилання розділяються **лише комами**.

1. Посилання на статтю:

Автори А.А. (рік видання), Назва статті, Назва журналу (курсивом), Том (номер), сторінки.

Ініціали пишуться після прізвища.

Всі елементи посилання розділяються комами.

1. Приклад:

Popovici C., Gitin L., Alexe P. (2013), Characterization of walnut (*Juglans regia* L.) green husk extract obtained by supercritical carbon dioxide fluid extraction, *Journal of Food and Packaging Science, Technique and Technologies*, 2(2), pp. 104-108.

2. Посилання на книгу:

Автори (рік), Назва книги (курсивом), Видавництво, Місто.

Ініціали пишуться після прізвища.

Всі елементи посилання розділяються комами.

Приклад:

2. Wen-Ching Yang (2003), *Handbook of fluidization and fluid-particle systems*, Marcel Dekker, New York.

Посилання на електронний ресурс:

Виконується аналогічно посиланню на книгу або статтю. Після оформлення даних про публікацію пишуться слова **available at:** та вказується електронна адреса.

Приклади:

1. (2013), *Svitovi naukovometrychni bazy*, available at:

http://www1.nas.gov.ua/publications/q_a/Pages/scopus.aspx

2. Cheung T. (2011), *World's 50 most delicious drinks [Text]*, available at:

<http://travel.cnn.com/explorations/drink/worlds-50-most-delicious-drinks-883542>

Список літератури оформлюється лише латиницею. Елементи списку українською та російською мовою потрібно транслітерувати. Для транслітерації з українською мови використовується паспортний стандарт, а з російської - стандарт МВД (в цих стандартах використовуються символи лише англійського алфавіту, без хвостиків, апострофів та ін).

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Тематика публікацій в Ukrainian Food Journal:

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Результати досліджень, представлені в журналі, повинні бути новими, мати чіткий зв'язок з харчовою наукою і представляти інтерес для міжнародного наукового співтовариства.

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Наукове видання

Ukrainian Food Journal

Volume 5, Issue 1
2016

Том 5, № 1
2016

Підп. до друку 24.03.2016 р. Формат 70x100/16.
Обл.-вид. арк. 12.24. Ум. друк. арк. 12.46.
Гарнітура Times New Roman. Друк офсетний.
Наклад 100 прим. Вид. № 26н/15.

НУХТ. 01601 Київ-33, вул. Володимирська, 68

Свідоцтво про державну реєстрацію
друкованого засобу масової інформації
КВ 18964-7754Р
видане 26 березня 2012 року.