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Research of Dry Mix Quality Indices based on Vegetable Components for Soft Ice Cream Production

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Abstract

Background/Objectives: The article studies the process of identification of fat oxidation degree and pattern in the process of dry mix sample storage for soft ice cream production containing milk fat in its formula which by 100% can be replaced by vegetable fat. **Method**: In the process of storage the odor of test samples was studied on odor analyzer using piezo-quartz resonators with different film sorbents on the electrodes. Titratable and active acidity was analyzed using both protolytometric and potentiometric methods. Quality microbiological indices in the process of storage were determined by taking a culture in relevant media using standard methods. **Findings:** Dry mix for soft ice cream with 100% content of milk fat was used as a reference sample. It has been revealed that in equilibrium gas-phase over test samples upon completion of 9 months of storage total change of highly volatile organic compounds made 83% and 51%, respectively, i.e. sample spoilage with milk fat is by 1.6 times more intensive. It has also been established that mix with vegetable fat upon completion of storage period had less values of titratable (by 1.7 times) and active (by 1.2 times) acidity compared with a sample containing milk fat. Based on the results of analysis of microbiological indices of test samples in the process of storage it has been determined that the shelf life of a dry mix with vegetable fat is 9 months at the storage temperature of 10±2ºC. **Improvements**: Multi-component dry mix developed is particularly intended for soft ice cream production in health and leisure institutions, health resorts, holiday hotels, recreation camps and in the areas remote from raw material sources.

Keywords: Dry Mix, Odor Analyzer, Soft Ice Cream

1. Introduction

The research is relevant due to the necessity to produce soft ice cream, including the products with improved quality characteristics based on multi-component dry mixes. One of the most promising directions in production of a wide range of soft ice cream with enhanced nutrition and biological values is inclusion of non-diary fats with improved quality and extended shelf life in dry mix composition.

In view of the problem associated with seasonal milk processing and raw material shortage in the districts with low developed resources for stock-breeding it is expedient to use multi-component dry mixes as a basis for soft ice cream production. One of the components of a dry mix for soft ice cream production is fat of animal origin having high assimilability and compatibility with almost all food products. Nutrition value of milk fat is caused by a peculiar variety of saturated and unsaturated fatty acids, presence of phospholipids, particularly lecithin, fat-soluble vitamins A, D, E and K. However milk fat in its glyceride composition has a low content of essential polyunsaturated fatty acids and increased content of cholesterol. Furthermore, none of natural fats is perfect in terms of its fatty acid composition and properties. Therefore all leading companies are using different fats of

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non-diary origin for expansion of product line, enhancing of both nutrition and biological value of food products, including ice cream, as wells as for increase of production profitability[1,2].

According to the recommendations of the Institute of Nutrition RAMS from standpoint of nutrition hygiene, replacement of milk fat with vegetable one improves the quality of a product due to the change of proportions of saturated and unsaturated fatty acids. Combination of vegetable fats and milk components improves the properties of vegetable fats[3-5].

2. Concept Headings

The objective of the present work is to develop a formula of multi-component dry mix based on vegetable fat with improved quality and extended shelf life.

Composition of a dry mix formula for soft ice cream production includes as follows: skimmed milk powder, soy protein isolate, dry vegetable fat, sand sugar and stabilizer[6-7]. Dry mix with milk fat for soft ice cream was used as a reference sample. Finished products were stored at the temperature range of 4±2, 10±2 and 20±2ºC at a relative air humidity of not more than 96 % and absence of a direct strong lighting. The research was conducted during nine- months' period.

For the time being a standard method of lipid oxidative spoilage control and correlation with presence of a foreign flavor in food products presents the analysis of headspace volatiles using an "electronic nose". This device is used for non-destructive analysis of both the taste and odor of products in the real time mode including the analysis of rancidification of food products at their storage[1].

Seven sensors were used as a sensor array based on piezo-quartz resonators of VAW type with base oscillation frequency of 10.0 MHz with different sorbents on the electrodes. The coating is selected in line with test tasks (emission of different organic compounds from test samples is possible): 4 – polar ones (sensitive to alcohols, aldehydes, esters, phenols and another organic compounds); 2 – polar ones sensitive to highly volatile acids and water; 1– propolis, selective to mid and low polar organic compounds, phenols, ammonia, amines and ketones.

For the purpose of preparation for analysis, dry mix samples with full replacement of milk fat with vegetable one as well as dry mix samples with milk fat (fresh samples and samples after nine months of storage) were thermo-stated up to the room temperature, then there was selected an average sample of the same size with weight of 2.00 g which was put into hermetically-sealed glass vessel with polymeric soft membrane. The samples were kept at a constant temperature (20°C) for not less than 30 minutes. For each test sample 3 cm^3 of equilibrium gas phase were selected using a separate syringe and injected into detection cell.

Measurement time made 60 sec, sensor response pick up mode was a steady one with a span of 1 sec and an optimal algorithm of sensor response presentation was a gradient one: 3/2 sec ¸ 3/20 sec, accuracy made 5–7%. For determination of distinctions and similarity of test samples the optimal kinetic "visual prints" (based on the signals of 7 sensors over the entire measurement period) and a "visual print" of maxima (maximum responses from sensors) was used, correspondently.

The following factors have been selected as criteria for the assessment of distinctions in the odor of test samples and difference in signals in Equilibrium Gas Phase (EGP) of a sampling unit:

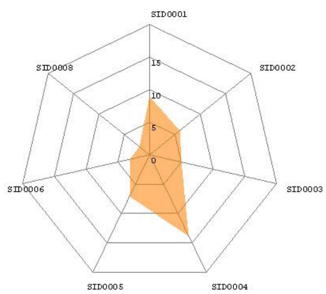
- Qualitative characteristics: 1. A shape of a "visual print" with typical response axis distribution is determined by a set of compounds in EGP; 2. A correlation of two maximum signals from two sensors which enables to identify (recognize) the main sorbing substances or to distinguish them from a standard (for example, water);
- Quantitative characteristics: 1. $\mathbf{S}_S$ (Hz/sec) – a total area of a full "visual print" built based on all signals from all sensors over the full time of measurement. This total area evaluates the total odor intensity and is proportional to the concentration of highly volatile substances; 2. **S** (Hz/sec) area of an optimized kinetic "visual print" evaluates the content of highly volatile substances in equilibrium gas phase. From a sensor array there can be excluded sensor(s) with active polar sorbent(s), namely PVP, crown ether and specific sorbent with response on the noise level; 3. Maximum signals from sensors with the most active and specific film of sorbents DF_i, Hz; 4. Odor stability index $\boldsymbol{A}_{i/j}$ – correlation of signals from certain sensors in array enables to observe the change of concentration correlation of certain substance classes in equilibrium gas phase

over test samples. For example, if correlation of sensor signals in case with a test sample differs from correlation of the same signals in case with a reference sample, then the content of certain organic compounds changes due to product matrix connections or due to enrichment of equilibrium gas phase with native substances of the additives in consequence of spoilage or other changes, however responses from sensors or total area of a "visual print" can change insignificantly.

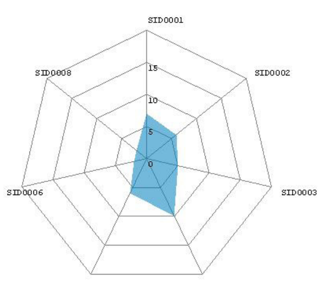
Laboratory air after long-continued ventilation was used as a test sample for the purpose of checking of correctness of measurements and responses from sensors.

3. Results

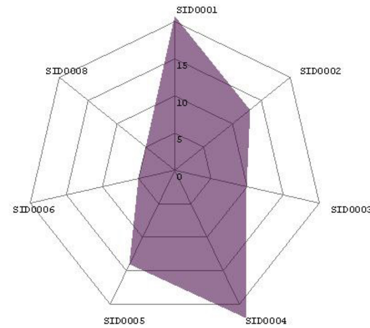
For determination of similarity degree of composition of equilibrium gas phases over test samples, there were analyzed the peculiarities of a geometrical pattern of visual prints of maxima (Figure1). It has been established that test samples have different qualitative and quantitative composition of EGP before and after nine months of storage.



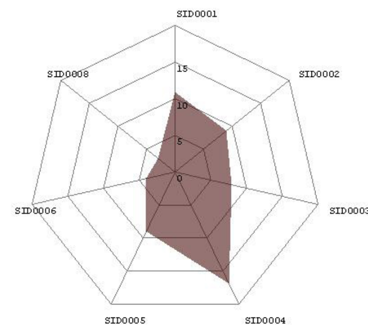
a) Equilibrium gas phase over mix with milk fat (fresh sample)



b) Equilibrium gas phase over mix with vegetable fat (fresh sample)



c) Equilibrium gas phase over mix with milk fat (after nine months of storage)



d) Equilibrium gas phase with vegetable fat (after nine months of storage)

Figure 1. "Visual prints" of maximum signals from sensors in equilibrium gas phase over test samples. (Numbers of sensors in array are specified along axis)

"Visual prints" of maxima differ in sizes, i.e. in figure area which depends on the content of substances in equilibrium gas phase over test samples and shape. The shape of a "visual print" is determined by correlation in a test sample and, as a consequence in equilibrium gas phase, of concentration of main organic compound classes for detection of which a sensor array is tuned to, namely: highly volatile acids, aliphatic alcohols, aldehydes, esters, ketones and amines. The amount of aldehydes over a test sample of mix with milk fat is by 39% bigger than in mix with vegetable fat circled in Figure 1. After storage the distinctions make 32%. The content of moisture, aliphatic acids and of the other high polar organic compounds is insignificant shown in Table 1[7].

Spoilage pattern of samples analyzed is different. Fine distinctions in the composition of equilibrium gas phase

Table 1. Total area of "visual print" of EGP and signals from selective sensors over test samples and to air blank sample

Sample type	Sensor 1 Twin 21	Sensor 2 PEG 2000	Sensor 4 TX-100	Sensor 5 PEG sk	S_{Σ},Hz·sec
Sample bottle background	2	-1	-1	-1	199
Laboratory air	-2	1	4	1	342
Mix with milk fat (fresh one)	9	6	14	8	1546
Mix with milk fat (after nine months of storage)	21	13	22	14	2822
Mix with vegetable fat (fresh one)	8	6	10	6	1399
Mix with vegetable fat (after nine months of storage)	11	9	17	9	2108

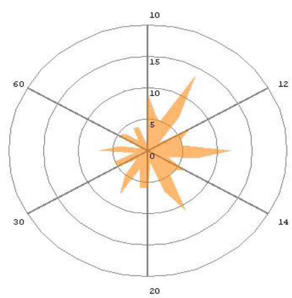
Table 2. Correlation of signals from several sensors in array with regard to all test and reference samples

Sample type	Odor stability index $A_{1/2}$		Odor stability index $A_{4/3}$	
	Fresh sample	Sample after storage	Fresh sample	Sample after storage
Sample bottle background	2.0	-	2.0	-
Laboratory air	2.0	-	2.0	-
Mix with milk fat	1.50	1.62	2.8	2.2
Mix with vegetable fat	1.33	1.22	2.0	2.2

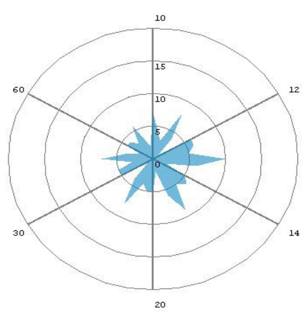
Table 3. Change of fatty acid composition of a dry mix for soft ice cream in the process of storage

Fatty acid	Fatty acid content, %			
	Mix with 100% replacement of milk fat with vegetable one	Mix with milk fat	Mix with 100% replacement of milk fat with vegetable one	Mix with milk fat
	0 months		9 months	
Saturated acids:	50.05	45.74	52.15	48.19
Octanoic acid ($C_{8:0}$)	-	5.2±0.01	-	6.3±0.03
Lauric acid ($C_{12:0}$)	0.31±0.01	0.056±0.01	0.36±0.01	0.057±0.01
Myristic acid ($C_{14:0}$)	1.47±0.02	11.0±0.03	1.05±0.03	10.2±0.01
Palmitic acid ($C_{16:0}$)	43.09±0.04	17.8±0.04	45.57±0.05	18.2±0.01
Stearic acid ($C_{18:0}$)	5.18±0.02	11.68±0.02	5.17±0.03	12.40±0.02
Pentadecanoic acid ($C_{15:0}$)	-	1.04±0.01	-	1.03±0.01
Unsaturated acids:	46.95	27.44	47.97	27.93
Arachic acid ($C_{20:0}$)	-	0.96±0.02	-	0.97±0.01
Oleic acid ($C_{18:1}$)	37.17±0.03	23.68±0.01	38.57±0.04	24.50±0.01
Linolic acid ($C_{18:2}$)	9.30±0.02	2.0±0.02	8.96±0.03	1.72±0.02
Llinolenic acid $C_{18:3}$)	0.48±0.01	0.8±0.03	0.44±0.01	0.74±0.01

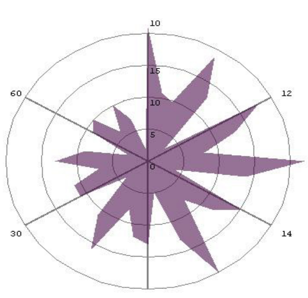
over test samples enable to establish optimized kinetic "visual prints" shown in Figure 2. The signals from intensively responding sensors (sensor 7) were excluded from response array. All test samples are characterized by "visual prints" with attenuating sorption and spontaneous desorption of the components from the film of sorbents. Such process pattern is possible at a low concentration of highly volatile components in equilibrium gas phase over test samples.



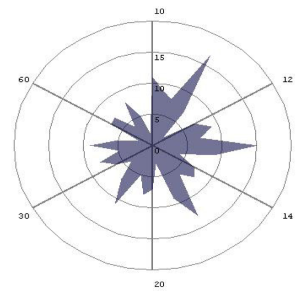
a) Equilibrium gas phase over mix with milk fat (fresh sample)



b) Equilibrium gas phase with vegetable fat (fresh sample)



c) Equilibrium gas phase over mix with milk fat (after nine months of storage)

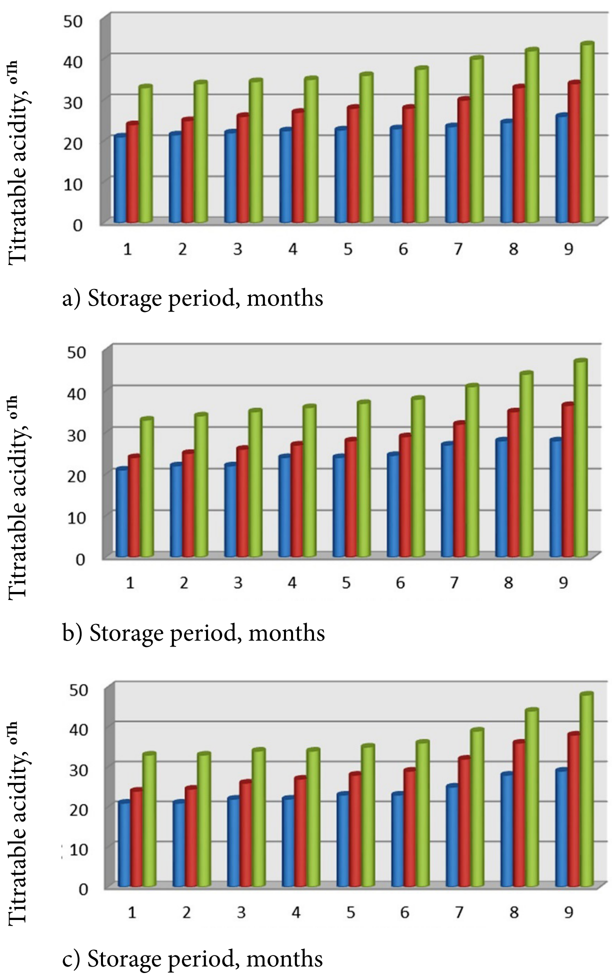


d) Equilibrium gas phase over mix with vegetable fat (after nine months of storage)

Figure 2. Kinetic "visual prints" of test samples odor (measurement time is specified circle-wise, c; response values of sensor array (7 sensors, Hz) are specified along vertical axis.

Index $A_{i/j}$ enables to observe the changes in the qualitative composition of equilibrium gas phase over samples. The aforementioned index shows a constancy of concentration correlation of certain highly volatile compound classes in the odor shown in Table 2. It has been determined that index increases in case of storage of mix test sample with milk fat and that such index decreases in case of storage of mix test sample with vegetable fat. All this reflects the process of accumulation of highly volatile acids and water at spoilage of compounds being different in nature in case with the first sample, and in case with the second sample this reflects a decrease of alcohols content.

It can be considered that the content of polar organic compounds, such as ketones, alcohols, esters and aldehydes is the same in test samples. It has been established that the content of the aforementioned group of substances decreases in case with mix with milk fat at its storage. Based on $A_{1/2}$ index it has been established that the content of acids and other polar compounds in equilibrium gas phase over mix with milk fat significantly increases (by 39%), consequently the decrease of $A_{4/3}$ index could be caused mainly by acidification and by rancidification in a less degree. And this index increases in case with the second sample insignificantly. Taking into consideration that $A_{1/2}$ index of accumulation of acids and water also decreases in case with the second sample, then in equilibrium gas phase the accumulation of ketones occurs including methylallkylketones, aldehydes and ester. These are the substances the rancidification of fat is characterized by. Oozing in a bigger amount takes place at spoilage of mix with milk fat.



- mix with 100 % replacement of milk fat with vegetable one

- mix with 50 % replacement of milk fat with vegetable one

- mix with milk fat

Figure 3. Change of titratable acidity of dry mix in the process of storage at temperature range of: a) 4±2 °C; b) 10±2 °C; c) 20±2 °C

Thereby a total change (increase) of the content of highly volatile organic compounds in equilibrium gas phases over test samples makes 83% and 51%, respectively, in the process of storage, i.e. spoilage of a test sample with milk fat is more intensive by 1.6 times.

Fat oxidation rate primarily depends on the composition of fatty acids of triacylglycerols, and oxidation of free fatty acids occurs more rapidly than oxidation of combined acids. Oxidation of saturated fatty acids occurs slower compared to unsaturated ones and oxidation of polyunsaturated fatty acids occurs more rapidly than oxidation of monounsaturated fatty acids which is explained by different rate of formation of free radicals by these acids[8].

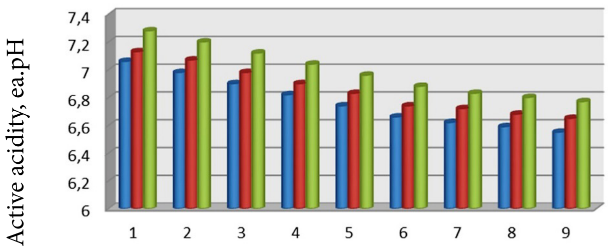
Analyzing a fatty acid composition of a product it is possible to describe both the pattern and degree of fat oxidation in the process of storage of a dry mix for soft ice cream shown in Table 3.

At oxidation of oleic acid and polyunsaturated fatty acids there can form low-molecular saturated aldehydes, namely pentanal, hexanal, octanal, nonanal, malondialdehyde and other monounsaturated and di-unsaturated aldehydes, namely peneten-2-al, octane-2-al, heptadiene-2,4-al, decadiene-2,4-al as well as saturated and unsaturated ketones. Many of the listed above aldehydes and ketones have unpleasant taste and in various combinations might add a foreign flavour to dairy products. Thus, a fish flavour is caused by saturated and unsaturated aldehydes, mainly hexanal, heptanal and heptadiene-2,4-al; a rancid flavour is caused by heptanal and nonanal; tallowy flavour is caused by pentanal, hexanal and pentene-2-al (dihydroxystearic acid). Unsaturated ketone, namely octene-1-on-3 (vinylamylketone) adds a metallic off-flavour to dairy products and ketone - penten-1-on-3 adds an oily flavour to dairy products[9-10].

Product storage stability is significantly affected by its acidity. Acidification facilitates the development of product defects[8,10]. Active and titratable acidity was measured in the process of mix storage. Dependence diagrams of dry mix titratable acidity on storage duration at different temperatures are given in Figure 3.

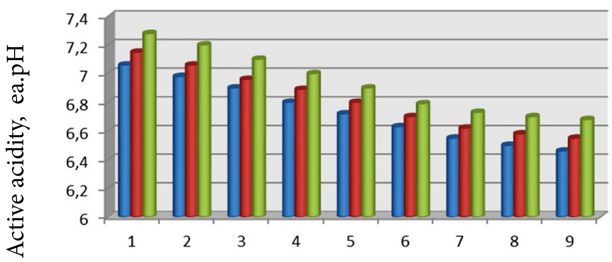
In as-prepared mixes with 100 % replacement of milk fat with vegetable one, a titratable acidity made 21.0°Th, with 50 % replacement of milk fat with vegetable one it made 24.2°Th and with milk fat a titratable acidity made 33.0°Th. After six months of mix storage with 100% replacement of milk fat with vegetable one at the temperature of 4±2 °C shown in Figure 3a an acidity index increased up to 22.4°Th, with 50% replacement of milk fat with vegetable one an acidity index increased up to 28.2°Th and with milk fat an acidity index increased up 37.5°Th. After nine months of storage an acidity index increased up to 26.0°Th, 34.3°Th and 43.2°Th, respectively. At the other storage temperatures (Figure3b and c) a titratable acidity index has bigger values which evidences of a rapid spoilage of a dry mix.

Based on data analysis given in Figure 4, it follows that in the process of product storage an active acidity, which is characterized by concentration of free ions in solution, intensively decreases in the first six months of storage and then the process slows down. Mix with 100% replacement of milk fat with vegetable one has less values of an active acidity at the following temperature range under study: 6.31 – 6.55 ea. pH.



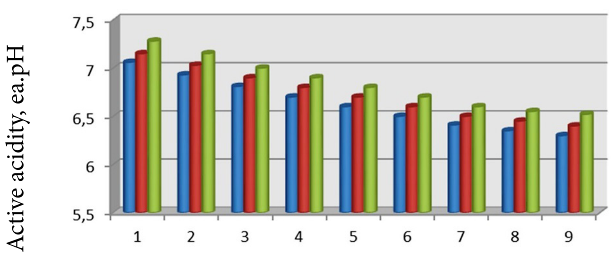
Storage period, months.

a)



Storage period, months

b)



Storage period, months

c)

- mix with 100 % replacement of milk fat with vegetable one

- mix with 50 % replacement of milk fat with vegetable one

- mix with milk fat

Figure 4. Change of dry mix acidity in the process of storage at temperature range of: (a) 4±2 ºC; (b) 10±2 ºC; (c) 20±2 ºC.

Decrease of an active acidity is explained by interaction of hydrogen cations with protein substances. At storage at partial hydrolysis of proteins there occurs their noticeable deacidification accompanied by a certain polypeptide chain unfolding. Low temperature also facilitates the decrease of an active acidity. Consequently, the temperature of not more than 10°C is required for storage of canned milk-containing products.

In the course of an experimental research, microbiological indices were studied in the process of storage within the following temperature range: 4±2ºC, 10±2ºC and 20±2ºC. The main criterion was the absence in test samples of a negative trend of a complex of indices, i.e. a discrepancy in values established by regulatory documentation.

At storage of dry mix test samples there was studied the number of aerobic и facultative anaerobic microorganisms, to which Escherihia coli, Staphylococcus aureus, Clostridium perfringens and Listeria monocyto genes refer to. The results of this study are given in Tables 4-6[10]. It has been established that mixes with a partial or full replacement of milk fat with vegetable one have better storage stability; a decrease of the total microbial population by 4-6% has also been noticed. It has been revealed that microbiological indices of a dry mix with a partial or full replacement of milk fat with vegetable one correspond to regulatory requirements upon completion of nine months of storage within the temperature range under study. Escherihia coli, Staphylococcus aureus, Clostridium perfringens and Listeria monocytogenes have not been revealed in test dry mixes during storage periods under study.

4. Discussion

Data obtained correlate with the results of experimental study given in[11-12] and aimed at the development of canned milk-containing products with sorghum sugar syrup addition. There has been established a decrease in concentration of carbon degradation products in equilibrium gas phase of the newly developed food products. Increased nutrition and biological value and better balance of protein acid composition have also been noticed.

Data obtained have a good correlation with the results of study given in[13-14] and aimed at the development of particular technologies of products made of dairy raw materials with the involvement of the products made of vegetable feedstock deep conversion based on milk or cream composite, fermented with lactic-acid bacterial flora as well with curd-based products, i.e. floury and culinary ones (cottage cheese pancakes, dumplings and pies). Developed products have an optimal ω-6 to ω-3 fatty acid ratio 5-6:1. The aforementioned products are also enriched with vegetable stock essential components

Table 4. Change of microbiological indices of a dry mix with 100% replacement of milk fat with vegetable one in the process of storage

Mix °T	Storage duration	QMAFAnM CFU/g, not more	Product weight (g, cm^3), in which the presence of the following bacteria is not permissible		
			Coliform bacteria (coliforms)	Pathogenic including salmonella	Staph. aureus
4±2 °C	1	$0.8 \cdot 10^5$	-	25	0.2
	3	$1.6 \cdot 10^5$	-	25	0.3
	6	$2.4 \cdot 10^4$	0.05	25	0.4
	9	$3.2 \cdot 10^4$	0.06	25	0.6
10±2 °C	1	$1.5 \cdot 10^5$	-	25	0.2
	3	$2.3 \cdot 10^5$	-	25	0.4
	6	$3.2 \cdot 10^4$	0.06	25	0.5
	9	$4.3 \cdot 10^4$	0.07	25	0.7
20±2 °C	1	$2.4 \cdot 10^5$	-	25	0.3
	3	3.010^5	-	25	0.4
	6	$3.7 \cdot 10^4$	0.07	25	0.6
	9	$4.5 \cdot 10^4$	0.08	25	0.8

Table 5. Change of microbiological indices of a dry mix with 50% replacement of milk fat with vegetable one in the process of storage

Mix storage temperature, °C	Storage period, months	QMAFAnM CFU/g, not more	Product weight (g, cm^3) in which the presence of the following bacteria is not permissible		
			Coliform bacteria (coliforms)	Pathogenic including salmonella	Staph. aureus
4±2 °C	1	$0.9 \cdot 10^5$	-	25	0.2
	3	$1.7 \cdot 10^5$	-	25	0.3
	6	$2.5 \cdot 10^4$	0.06	25	0.5
	9	$3.4 \cdot 10^4$	0.07	25	0.7
10±2 °C	1	$1.6 \cdot 10^5$	-	25	0.3
	3	$2.4 \cdot 10^5$	-	25	0.4
	6	$3.3 \cdot 10^4$	0.07	25	0.6
	9	$4.5 \cdot 10^4$	0.08	25	0.8
20±2 °C	1	$2.5 \cdot 10^5$	-	25	0.4
	3	$3.1 \cdot 10^5$	-	25	0.5
	6	$3.9 \cdot 10^4$	0.08	25	0.7
	9	$4.8 \cdot 10^4$	0.09	25	1.0

Table 6. Change of microbiological indices of a dry mix with whole milk in the process of storage

Temperature of mix storage, °C	Storage period, months	QMAFAnM CFU/g, not more	Product weight (g, cm^3) in which the presence of the following bacteria is not permissible		
			Coliform bacteria (coliforms)	Pathogenic including salmonella	Staph. aureus
4±2 °C	1	$1.1 \cdot 10^5$	-	25	0.3
	3	$1.9 \cdot 10^5$	-	25	0.4
	6	$2.7 \cdot 10^4$	0.07	25	0.5
	9	$3.8 \cdot 10^4$	0.08	25	0.8
10±2 °C	1	$1.8 \cdot 10^5$	-	25	0.4
	3	$2.6 \cdot 10^5$	-	25	0.5
	6	$3.5 \cdot 10^4$	0.08	25	0.7
	9	$4.7 \cdot 10^4$	0.09	25	0.9
20±2 °C	1	$2.7 \cdot 10^5$	-	25	0.5
	3	$3.3 \cdot 10^5$	-	25	0.6
	6	$4.1 \cdot 10^4$	0.09	25	0.8
	9	$5.0 \cdot 10^4$	0.1	25	1.1

and have a high biological value as well as a good balance of essential amino acids.

5. Conclusion

As can be seen from the above, the research has shown the expediency of developing multi-component dry mixes for soft ice cream production based on vegetable fats. In the course of the study a sensor array has been selected for determination of fine distinctions in the composition of equilibrium gas phases of test samples of dry mixes with milk or vegetable fats and the algorithms for comparison of signals from sensors have been developed to establish the degree of both similarity and distinction in the odor of test samples. It has been determined that total changes in the content of highly volatile organic compounds in equilibrium gas phases over test samples with milk or vegetable fats in the process of storage make 83% and 51%, respectively, which evidences of more intensive spoilage of a sample with milk fat (by 1.6 times). It has been revealed that the least values of a titratable and active acidity upon completion of nine months of storage within the temperature range under study are achieved in samples with 100% content of vegetable fat, namely: 26.0–29.2°Th and 6.31–6.55 ea.pH, respectively. It has been established that duration of the achievement of critical values of microbial population in samples of dry mixes with 100% content of vegetable fat exceeds nine months within a temperature range under study.

6. References

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