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CONTENT

THE STUDY OF CHEMICAL COMPOSITION FOR ANIMAL FATS DURING STORAGE

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Abstract

In this article the chemical composition for 3 types of animal fats (pork fat, beef tallow and buffalo tallow), following the variation of saturated and unsaturated fatty acids proportion during freezing storage was studied. Determination of chemical composition of animal fats is important in establishing organoleptic and physico-chemical parameters, the variation of them in time, nature and proportion of fatty acids conferring specific characteristics to them. For pork fat was determined the following chemical composition: saturated fatty acids 48.32%, monounsaturated fatty acids 36.78%, polyunsaturated fatty acids 14.89%. After 4 months of storage under freezing there was a change in fatty acids proportion, saturated fatty acid content increased slightly to 48.83%, due to installation of hydrolysis leading to release of fatty acids from triglycerides, monounsaturated fatty acids content decreased to 35.99%, and polyunsaturated fatty acids content increased to 15.18%. In the case of beef tallow there was an increasing in saturated and monounsaturated fatty acids content and a decreasing in polyunsaturated fatty acids content. Buffalo tallow presented a higher concentration of saturated and monounsaturated fatty acids than beef tallow and a lower concentration of polyunsaturated fatty acids.

Keywords: *fatty acids, animal fats, storage*

1. Introduction

In chemical terms fats are glycerol esters with fatty acids. Theoretically there is the possibility that one group of alcoholic glycerine is esterificated with a fatty acid molecule (monoglyceride), or two alcoholic groups are esterificated with two fatty acids molecules (diglyceride). In nature we meet only triglycerides. There are opinions that fats are made from simple triglycerides such as tripalmitin, tristearin, triolein, etc. But it turned out that in most of cases, fats are glycerin esters with 2 or 3 different fatty acids [1, 5]. Fatty acids represent the variable structure of lipids, the characteristics of the fats being conferred by the nature and proportion of fatty acids that enters into their composition [11].

Glycerine is the second component of the fats, which holds a proportion of 6-10.5%. From the chemical can be considered as a derivative of propane, in which a hydrogen atom from each carbon atom is replaced with an alcoholic group (-OH):

CH_3 $CH_2–OH$
| |
CH_2 → $CH–OH$
| |
CH_3 $CH_2–OH$

Major fatty acids from animal fats composition are those who have a number of 4 to 18 carbon atoms in the molecule, namely [5, 7]:

Butyric acid	$CH_3–(CH_2)_2–COOH$
Caproic acid	$CH_3–(CH_2)_4–COOH$
Caprylic acid	$CH_3–(CH_2)_6–COOH$
Caprynic acid	$CH_3–(CH_2)_8–COOH$
Lauric acid	$CH_3–(CH_2)_{10}–COOH$
Miristic acid	$CH_3–(CH_2)_{12}–COOH$
Palmitic acid	$CH_3–(CH_2)_{14}–COOH$
Stearic acid	$CH_3–(CH_2)_{16}–COOH$

The main unsaturated fatty acids in animal fats are oleic and linoleic acid. Oleic acid has 18 carbon atoms and one double link located between C9 and C10: $CH_3–(CH_2)_7–CH=CH–(CH_2)_7–COOH$. Linoleic acid has 18 carbon atoms, but two double links located between C9 and C10, C12 and C13: $CH_3–(CH_2)_4–CH=CH–CH_2–CH=CH–(CH_2)_7–COOH$ [28].

Animal fats has nutritional-biological and sensory value through: the provision of essential fatty acids for human body (linoleic, linolenic and arachidonic acids); concentrated sources of energy (cca.9 kcal/g, proteins and carbohydrates providing approx. 4 kcal/g); medium for transport /storage of liposoluble vitamins (A, D, E, K); formation of phospholipids with essential role in proper functionation of membranes; precursors of prostaglandins, essential hormones for the body; texture formers; structure formers in certain products (fillings for bakery products and confectionery); lipids confer softness (a slight bite and mastication), smaller the dry and granular feeling of food consumption (due to the lubrication effect and the liquid part of the fat); flavor providers (with positive or negative effect on the overall flavor of the product) and medium for hydrophobic flavour compounds [2, 3].

2. Materials and methods

Pork fat was obtained by fresh bacon and lard pork melting, beef tallow was obtained by raw tallow melting, collected from Baltata Romaneasca race, female, age of 8 years, and buffalo tallow was obtained by raw tallow melting collected from female, age of 6 years, which were determined the chemical composition in fresh state and after 4 months of storage under freezing (-15 ...- 18^{o}C).

Physicochemical examination

Fatty acid composition was determined using gas chromatography (GC) Shimadzu GC-17 A coupled with flame ionisation detector FID. Gas chromatography

column is Alltech AT-Wax, 0.25 mm I.D., 0.25 μm thick stationary phase (polyethylene), used helium as carrier gas at a pressure of 147 kPa, temperature of the injector and detector was set to 260^{o}C, the oven programm was the following: 70^{o}C for 2 min., then the temperature was raised up to 150^{o}C with a gradient of 10^{o}C/min., a level of 3 min. and the temperature was raised up to 235^{o}C with a gradient of 4^{o}C/min.

The method consists in transforming of fatty acids in methyl esters in the sample under analysis, followed by separation of components on a chromatography column, their identification by comparison with standard chromatograms and quantitative determination of fatty acids. Esterificated analyzed sample is introduced in column chromatography. By chromatography separation the sample chromatogram is obtained in which fatty acids are recorded in the form of peaks separated from each other by increasing the length chain, and at the same length chain by increasing of unsaturated degree. By comparing the distances of each peak from analyzed sample chromatogram with peaks distances from standard chromatograms, we identify each fatty acid present in the analyzed sample [14, 15, 16].

3. Results and discussions

The content of saturated fatty acids in pork fat was higher (48.32%) than monounsaturated (36.78%) and polyunsaturated (14.89%) fatty acids, the major fatty acids presented in pork fat were palmitic, stearic, oleic and linoleic acids. Oleic acid was determined in the largest proportion (33.51%), these results are in agreement with previous studies on this type of fat [4, 6]. The proportion of major fatty acids is indicated in table 1.

Figure 1 illustrates sample chromatogram for pork fat in witch fatty acids are registered in the form of peaks separated from each other by increasing the length chain, and at the same length chain by increasing of unsaturated degree.

In pork fat sample to 4 months freezing there were some differences from the fresh sample: miristic acid content decreased to 1.29%, palmitic acid increased to 27.15%, palmitoleic acid increased to 1.35%, margaric acid decreased to 0.35%, *cis*-10-heptadecanoic acid remained constant, stearic acid increased to 20.03%, oleic acid decreased to 32.66%, vaccenic acid remained constant, linoleic acid increased to 14.41% and alfalinoleic acid increased to 0.76%. In general, the saturated fatty acids content increased to 48.83%, the monounsaturated fatty acids content decreased to 35.99% and the polyunsaturated fatty acids content

increased to 15.18% (fig.2). The increas of saturated and polyunsaturated fatty acids content is due to the installation of hydrolysis leading to the release of acids from glycerides structure, which translates through the increas of titrable acidity.

The content of saturated fatty acids in beef tallow was higher (57.13%) than mono (34.47%) and polyunsaturated (8.4%) fatty acids, the major fatty acids present in beef tallow ware palmitic, stearic and oleic acids [8, 10, 12]. Oleic acid was determined in the largest proportion (30.14%). The proportion of major fatty acids is indicated in table 2.

Figure 3 illustrates sample chromatogram for beef tallow in witch fatty acids are registered in the form of peaks separated from each other by increasing the length chain, and at the same length chain by increasing of unsaturated degree.

In beef tallow sample at 4 months freezing there were some differences from the fresh sample: miristic acid content increased to 3%, pentaedecanoic and miristoleic acid were not detected, palmitic acid increased to 27.03%, palmitoleic acid increased to 1.77%, margaric acid decreased to 1.19%, *cis*-10-heptadecanoic acid decreased to 0.35%, stearic acid increased to 30.09%, oleic acid decreased to 30.09%, vaccenic acid increased to 4.64% and linoleic acid decreased to 1.32%. In general, the content of saturated fatty acids increased to 61.3%, the monounsaturated fatty acids content to 36.86% and the content of polyunsaturated fatty acids decreased to 1.84% (fig.4). In the case of beef tallow at 4 months congelation, monounsaturated fatty acids recorded an increase and polyunsaturated fatty acids a decrease, for pork fat at 4 months freezing we found the opposite.

Pork fat presents a highest proportion of mono and polyunsaturated fatty acids, than beef tallow, having aspect of alifios and homogeneous mass, beef tallow is presented as a compact and dense mass, fine granulated, with hard consistence, brittle to break or jam due to the higher content of saturated fatty acids [9, 11, 13].

The content of saturated fatty acids in buffalo tallow is higher (62.67%) than mono (36.35%) and polyunsaturates (0.97%) fatty acids, the major fatty acids present in buffalo tallow were palmitic, stearic and oleic acids. Stearic acid was determined in the largest proportion (36.24%). The proportion of major fatty acids is indicated in table 3.

There were some differences between buffalo and beef tallow. For buffalo tallow was determined a lower content of miristic, pentaedecanoic, palmitic, palmitoleic, linoleic and margaric acids and a higher content of stearic, oleic and vaccenic acids from beef tallow (fig.5).

There was determined a higher content of saturated (62.67%) and monounsaturated fatty acids (36.35%) in buffalo tallow, polyunsaturated fatty acids content was much lower (0.97%), which shows the highest resistance to alterative processes (hydrolysis and oxidation).

Table 1. Fatty acid composition of pork fat

No.	Fatty acid name	Abbreviation	Mg/g sample	Percentage %
1.	Miristic	14:0	13,480	1,44
2.	Palmitic	16:0	252,238	26,92
3.	Palmitoleic	16:1	12,077	1,29
4.	Margaric	17:0	3,455	0,36
5.	*Cis*-10-hepdecanoic	17:1	1,830	0,19
6.	Stearic	18:0	183,707	19,6
7.	Oleic	18:1	314,058	33,51
8.	Vaccenic	18:1 Isomer	16,696	1,78
9.	Linoleic	18:2	132,657	14,15
10.	Alfalinolenic	18:3	6,898	0,73

Table 2. Fatty acid composition of beef tallow

No.	Fatty acid name	Abbreviation	Mg/g sample	Percentage %
1.	Miristic	14:0	20,997	2,44
2.	Miristoleic	14:1	1,713	0,2
3.	Pentaedecanoic	15:0	3,389	0,4
4.	Palmitic	16:0	229,762	26,72
5.	Palmitoleic	16:1	13,726	1,6
6.	Margaric	17:0	11,709	1,36
7.	*Cis*-10-heptadecanoic	17:1	3,806	0,44
8.	Stearic	18:0	225,386	26,21
9.	Oleic	18:1	259,159	30,14
10.	Vaccenic	18:1 Isomer	17,964	2,09
11.	Linoleic	18:2	72,236	8,4

Table 3. Fatty acid composition of buffalo tallow

No.	Fatty acid name	Abbreviation	Mg/g sample	Percentage %
1.	Miristic	14:0	14,119	1,46
2.	Pentaedecanoic	15:0	3,568	0,36
3.	Palmitic	16:0	221,972	22,9
4.	Palmitoleic	16:1	7,819	0,81
5.	Margaric	17:0	16,594	1,71
6.	Stearic	18:0	351,210	36,24
7.	Oleic	18:1	315,024	32,5
8.	Vaccenic	18:1 Isomer	29,345	3,03
9.	Linoleic	18:2	9,440	0,97

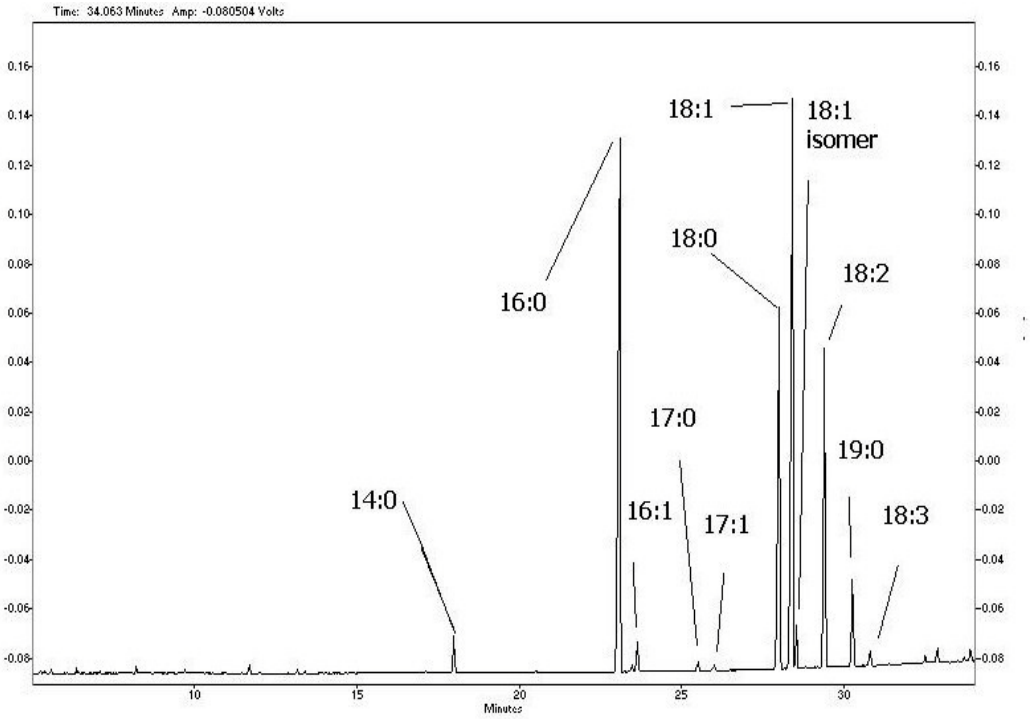

Fig.1 Chromatogram for fresh pork fat

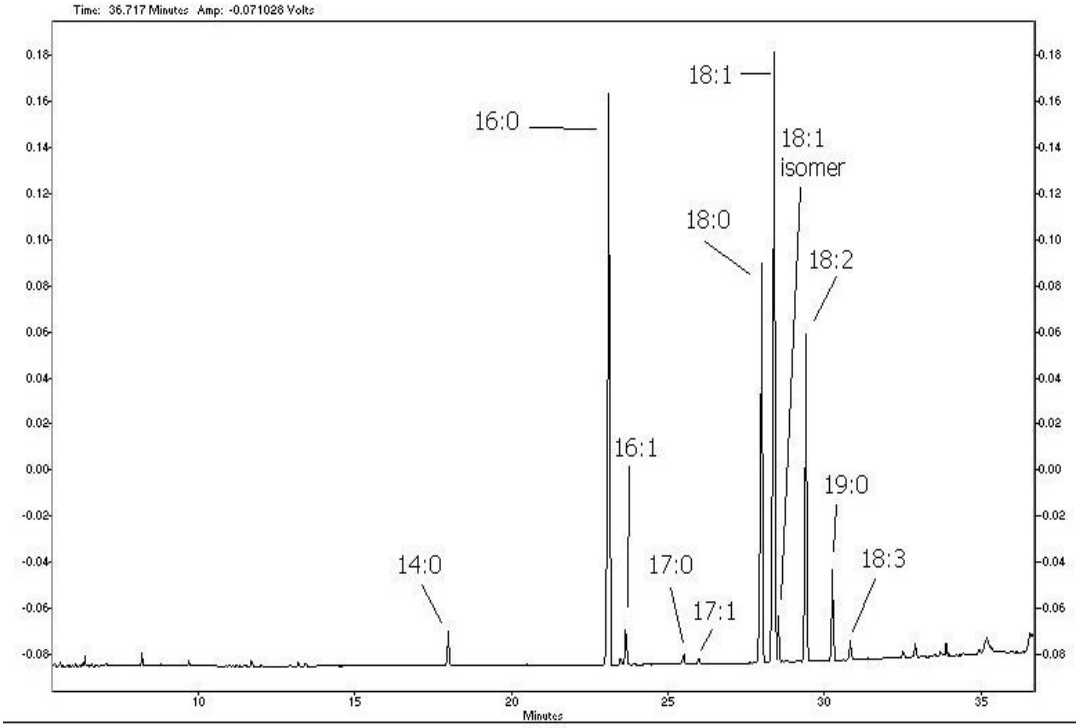

Fig.2 Chromatogram of pork fat to 4 months freezing

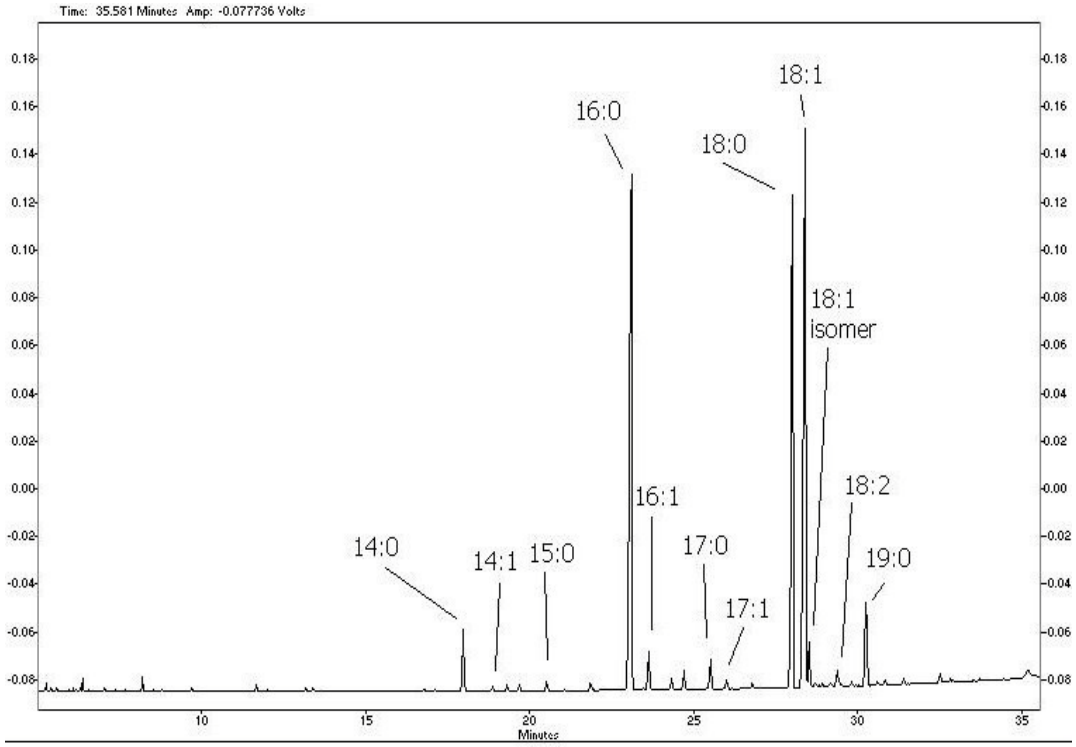

Fig.3 Chromatogram of fresh beef tallow

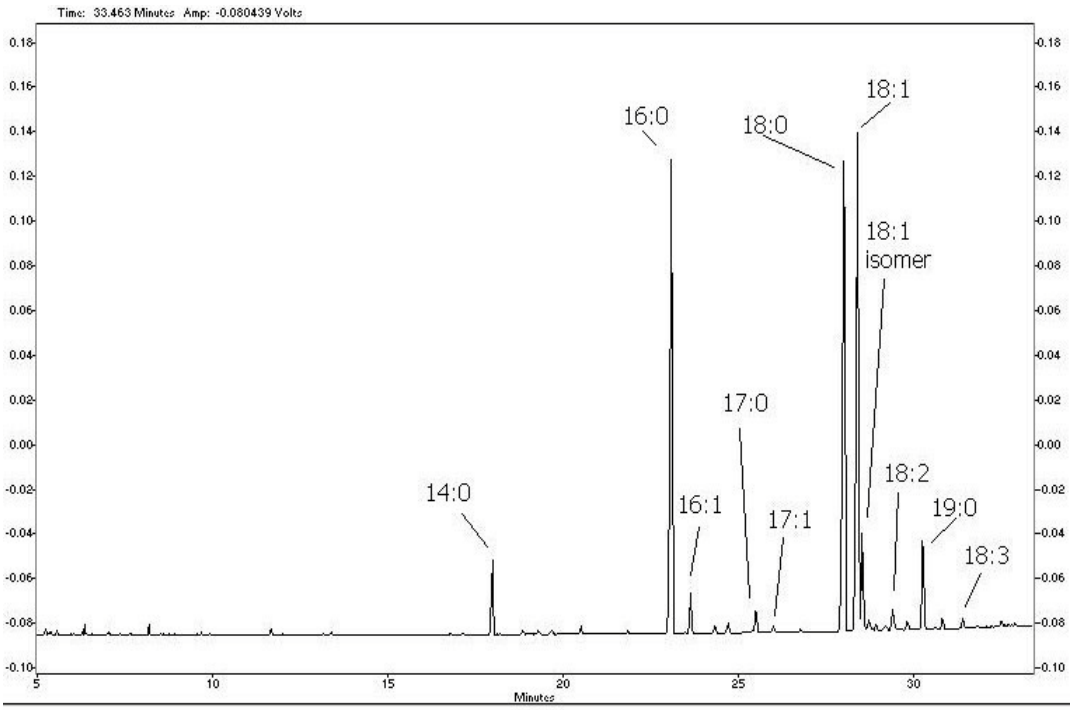

Fig.4 Chromatogram of beef tallow to 4 months freezing

4. Conclusions

Determination of chemical composition of animal fats is important in establishing organoleptic and physico-chemical parameters, the variation of them in time, being an indicator of their stability compared to alterative processes.

Buffalo tallow presented the lowest content of polyunsaturated fatty acids (0.97%), which are the most susceptible to autooxidation, it can be kept for a long period of time under refrigeration and freezing. Of studied fats, the most susceptible to altering is pork fat because its high content of polyunsaturated fatty acid (14.89%).

Chemical composition of fats influence their consistency, pork fat having aspect of alifios and homogeneous mass, beef tallow is presented as a compact and dense mass, fine granulated, with hard consistence, brittle to break or jam, with a higher value for melting point and a lower value for refractive and iodine index.

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PHYSICAL - CHEMICAL DETERMINATIONS ON PROTEINIZED MILK

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Abstract

The purpose of this work was to establish if the proteinized milk studied by us has the right physical-chemical characteristics and to compare some of these characteristics with the ones of the hyperproteic milk, a type of milk used to be produced in the past. The determinations showed that the proteinized milk has the right content of proteic substance and that it has a higher content of fat and a lower content of dry subctance.

Keywords: *proteinized milk, normalization, proteic substance, milk fats, dry substance*

1. Introduction

The proteinized milk can be distinguished from other sorts of consumption milk through its grown content of lactic proteins, the result of the enrichment of raw milk with lactic proteins. The proteinized milk is manufactured through the technology of the traditional consumption milk. The technologycal characteristic consists in the normalization of raw material not only by the fats content, but also by the proteins concentration until the established value of the standard.

In our case of the standardization using skimmed-milk powder, the calculated quantity of powder-milk will be dissolved into a smal quantity of raw milk, previously warmed up until 38 – 42 °C. This sort of milk will be mixed up with the whole quantity of milk stipulated for the manufacture of proteinized milk and the whole quantity of milk will be filtered, pasteurized, cooled, packed up and stored acording to the technological manufacture regime of the proteinized milk.

2. Materials and methods

2.1 The determination of the proteic substance

In a 100 ml Erlenmeyer glass we poured out 10 ml of milk and 10 drops of phenolphthalein. We titrated the mixture with sodium hydroxide (NaOH) N/10 until the pink – pal colour obtained persisted for 30 seconds. Then, we added 2 ml of formaldehyde solution which

decoloured the mixture and titrated again with sodium hydroxide (NaOH) N/10 until the pink – pal colour.

2.2 The determination of the milk fats by the acid-butirometric method (Gerber)

The fat content of the milk can be read in percentage directly on the graded scale of the butirometer after a previous dissolution of the proteic substances from milk under the sulphuric acid action and after the separation of the fat by the centrifugation in the presence of a small amount of isoamyl alcohol .

The proteinized milk has a fat content of 3,1 %.

2.3 The determination of the dry substance of the milk

The dry substance content in milk was determined through the evaporation of the water existing in the milk sample, using the drying oven with a 102°C temperature, until the sample had a constant mass.

$$\text{S.U.\%} = \frac{m_2 - m_0}{m_1 - m_0} \bullet 100$$

Where:

m_0 – the capsule's quantity, in grams (g);

m_1 – the capsule's quantity and the product's quantity before drying, in g;

m_2 – the capsule's quantity and the product's quantity after drying, in g .

Results:

$m_0 = 50,792$ g

$m_1 = 60,898$ g

$m_2 = 52,282$ g

$$\text{S.U.\%} = \frac{m_2 - m_0}{m_1 - m_0} \bullet 100$$

$$\text{S.U.\%} = \frac{52,282 - 50,792}{60,898 - 50,792} \cdot 100 = 12$$

3. Results and discussion

The protein concentration in milk can be calculated with the formula:

The total quantity of protein: P = V• 1, 95, %

Where: V = the NaOH N/10 volume used for the second titration.

Results:

Raw milk

V = 1, 74 ml NaOH.

Protein: P = V•1, 95 =1,74•1,95 = 3,4 %

Rehydrated powder milk

V = 2 ml NaOH.

Protein: P = V•1,95 = 2•1,95 = 3,9 %

Proteinized milk

V = 1, 4 ml NaOH.

Protein: P = V•1,95 = 2•1,95 = 3,9 %

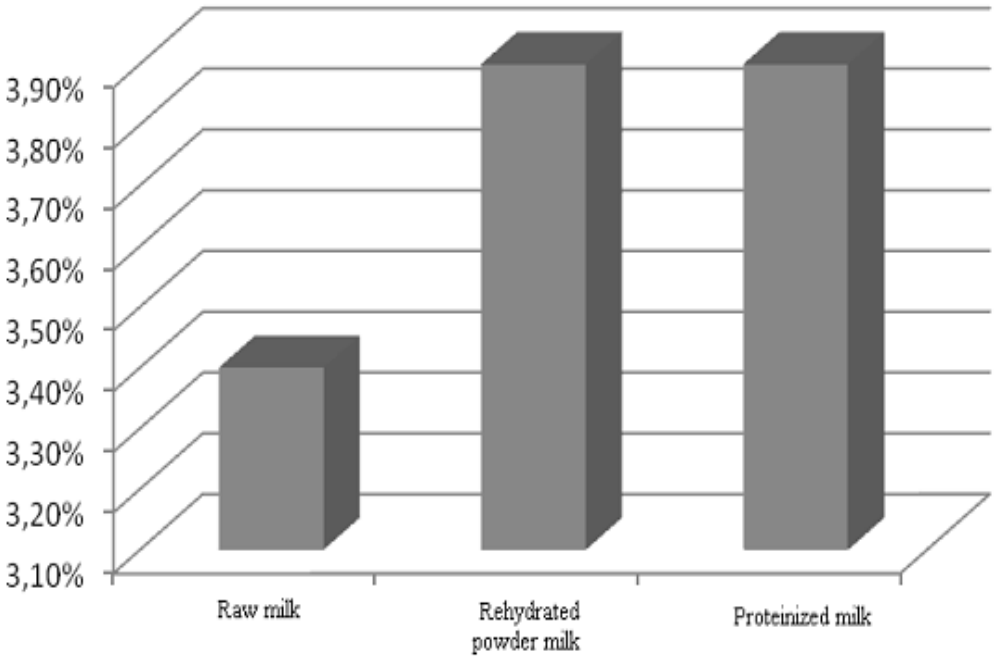

Fig. 1. The quantity of proteins for all the three types of milk

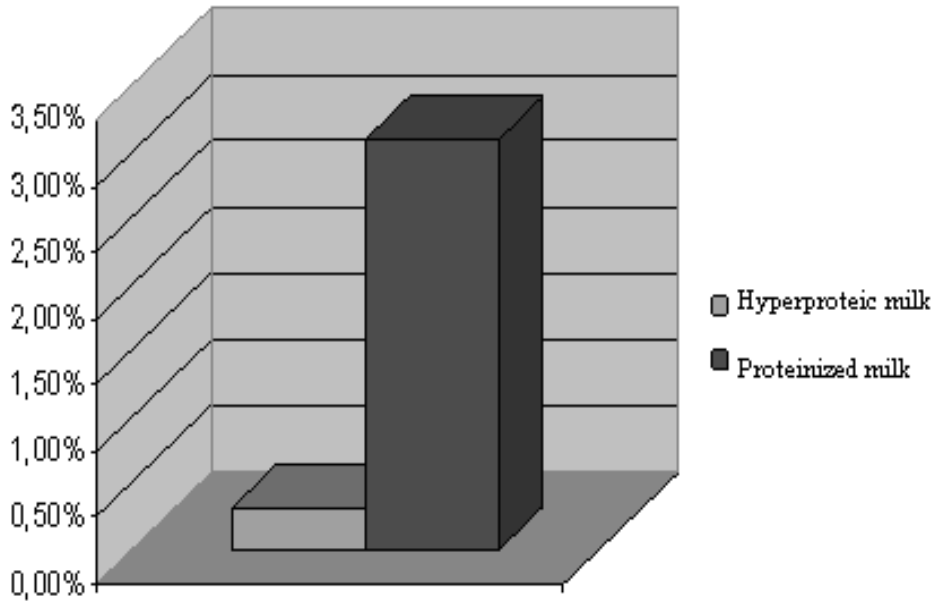

Fig. 2. Comparison between the fat content of the proteinized milk and the fat content of the hyperproteic milk, a sort of milk that used to be produced in the past

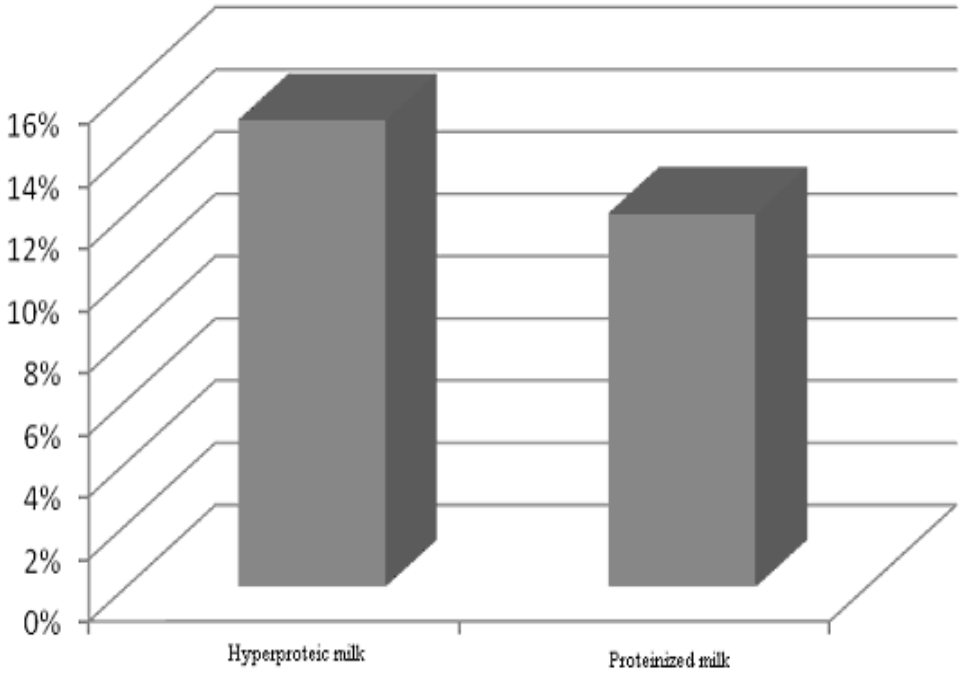

Fig. 3. Comparison between the dry substance content of the proteinized milk and of the hyperproteic milk

4. Conclusions

The casein is an amorphous substance, therefore it has acid groups (- COOH) and also alkaline groups (-NH_2). By this method the alkaline groups are blocked by formaldehyde and the acid groups are titrated with sodium hydroxide (NaOH). As we can see the NaOH N/10 volume used for the titration of the proteinized milk sample is higher than that used for the raw milk sample confirming that the protein content in the proteinized milk is bigger than the protein content in the raw milk.

The fat cells of the milk are covered with a pellicle of adsorbed proteinic substances, which prevents their fusion. This adsorbtion is reduced by the sulphuric acid (H_2SO_4) (d =1,820 – 1,825), which transforms the caseinic – calcic salts in soluble sulphate of casein, also precipitating the calcium sulphate. These soluble compounds reduce the adsorbtion phenomenon and enable the fusion of the fat cells. This cohesion process is hurried by the heatind and the centrifugation of the solution. Also, the isoamyl alcohol

favoures this process by reducing the superficial tension of the fat cells.

The dry substance of the milk is constituit of fat, proteic substance, lactose and mineral salts and varies between 12 – 14 % for the cow milk. The variation of the dry substance of the milk is determined by the variation of the milk components. By the determination of this characteristic we can estimated the quality of the milk and also can establish milk falsifications. As we can see the content of the dry substance which remains after the water evaporation from our proteinized milk is 12 %.

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VARIATION IN PHYSICO-CHEMICAL PARAMETERS OF PROBIOTIC YOGURT DURING REFRIGERATION STORAGE

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Abstract

The obtained products (clasic and probiotic yogurt) were analyzed under the following aspects: organoleptic characteristics: clot appearance, aroma and taste and physicochemical characteristics: acidity, pH, fat, dry matter, ash. At the end of the analyzed period classic yogurt had an acidity of 166°T, with 9°T more than in the first day so the rate of acidity increase was 0.31°T/day during refrigeration and probiotic yogurt had an acidity of 177°T, with 12°T more than in the first day so the rate of acidity increase was 0.41°T/day during refrigeration storage.Acidity of probiotic yogurt evolved faster than that of classic yogurt, due to addition of lactic bacteria specific cultures which hydrolysis lactose to lactic acid. Both products, classic and probiotic yogurt had a preservation period of 21 days in witch organoleptic characteristics were classified as normal issues.

Keywords: *probiotic yogurt, physico-chemical parameters, refrigeration, storage*

1. Introduction

In general, foods are characterized by safety quality, which implies all contaminants absence, physical, chemical or microbiological. Moreover, it is important for probiotic dairy products, as they are addressed to a large group of population, some segments of it being very vulnerable (children, elderly). Lactic fermentation using certain strains of lactobacillus and bifidobacteria cause nutritional properties increas, facilitating the digestion and intestinal functions regulation. It was demonstrated that the fermentation with lactic bacteria selective species has many favorable consequences for human health, which were emphasized in the literature (Costin, 1998). Probiotic is a food supplement with living organisms that beneficial influence the host by improving the intestinal microbial balance (Fuller, 1992). The use of antimicrobial compounds from natural sources is seen as a mean of improving the safety and stability of food at the same time with the maintenance of the natural character, high quality and a healthy product.

The optimization of the safety of probiotic yogurt, had in mind the selection of a bacterial strain with a pronounced

antimicrobial activity on pathogens, due to a inhibitory substance of protein nature - bacteriocin. Through a method, which consisted in antimicrobial substance concentration produced by the selected strain, and the addition of obtained concentrate in the two developed probiotic cultures, two biopreparations with antimicrobial activity on pathogens were made. They were used to probiotic yogurt production, emphasizing the antimicrobial potential on pathogens from obtained yogurt. This concept, created in this work, that was obtained the biopreparation with antimicrobial activity on pathogens, creates the premise of safety probiotic dairy products manufacturing. Probiotic yogurt was obtained by bacterial cultures of romanian conception, with a pronounced safety character and a high level of probiotic microorganisms.

The optimization of probiotic culture was followed by works selection of lactic bacteria strains, on the basis of rigorous microbiological and biotechnological criterions, and the development of probiotic cultures composition formula. Bianchi-Salvador (1996) presents the criteria of probiotic bacteria selection, for use in fermented dairy products manufacture. The basis criteria of bacterial strains selection were: organoleptic characteristics of reactivate pure culture, acidification capacity, the biotechnological potential, the potential of beneficial interrelations developing with other starter microorganisms, antimicrobial activity presence against pathogens.

Brunner, Huzan and Spillmann (1993) have emphasized the factors that influence bifidobacteria survival (*Bf. bifidum, Bf. Breve, Bf. Longum*) in fermented milk stored for 28 days at 4 and 8^{o}C: pH, dissolved oxygen, acidity and the number of live cells. pH has major influence on survival. At temperatures of 4 and 8^{o}C, survival duration was 16.6 respectively 15.3 days at 4.9 pH, 6.6 respectively 6 days at 4.5 pH and 1.5 respectively 1.2 days at 4.1 pH (Brunner et al., 1993). Rada (1997) found a positive effect of *Kluyveromices marxianus* yeast, which significantly extends the duration of bifidobacteria survival in milk at 4^{o}C. Mantere (1995) showed that besides lactic bacteria and bifidobacteria, propionic bacteria have probiotic effects, due to propionic acid production, bacteriocins and B_{12} vitamin, growth stimulation of other beneficial bacteria and ability to survive during stomach digestion.

Research motivation is the determination of physico-chemical parameters during refrigeration storage (2...4^{o}) of yogurt

obtained by classical method and yogurt fermented with lactic bacteria cultures with antimicrobial activity on pathogens.

2.1 Samples

To obtain the yogurt with thermophile organisms, we applied the technological process which comprises the following operations: quality and quantity reception of raw material milk by determining the following physicochemical parameters: acidity, pH, density, protein titre, lactose, dry matter and fat; standardization of raw material milk by bringing the fat content to 2.8% by whole milk creaming and the dry matter at least 11% by milk powder adding; milk pasteurization, by heating at 90^{o}C for 30 minutes; milk cooling to 44^{o}C; milk inoculation with thermophile culture; inoculated milk packaging in specific packages for fermented milk (polyethylene, polypropylene, glass) of different weights; inoculated milk thermostatation at a temperature of 42^{o}C until in the milk is obtained a 80-90^{o}T acidity (2.5-3 hours); the cooling for 30-40 minutes up to approx. 20^{o}C; the cooling to a temperature of 4-6^{o}C; the storage at 4-6^{o}C (Costin & Segal, 1999).

2. Materials and methods

2.2 Titrable acidity

Determination of acidity is the basic criterion for assessing the installation and intensity of hydrolysis. The method consists in neutralizing acidity with sodium hydroxide 0.1 N, using phenophtaleine, as an indicator. Acidity was expressed in ^{o}T (SR EN 14082, 1998, 2003).

2.3 Determination of fat content

In milk butirometre we placed 10 mL of sulfuric acid, 5 mL of acid milk product and with the same pipette 6 mL of distilled water and 1 mL izoamilic alcohol. The butirometre was cleaned with cotton, was put the rubber stopper and was homogenize. After homogenization, butirometre was centrifugated for 5 minutes at 1000 - 1200 rpm, then put on water bath at temperature of 65 °C, and then was read the fat content.

3. Results and discussion

The obtained products were analyzed under the following aspects: organoleptic characteristics: clot appearance, aroma and taste (a panel of external judges were selected beforehand and trained for the sensorial tests, Duo-Trio and Pairs

comparison tests were applied accordingly, as reported elsewhere (Chavez-Servin, Romeu-Nadal, Castellote, and López-Sabate, 2006); physicochemical characteristics: acidity, pH, fat, dry matter (Romanian Standard SR EN 14082, 2003).

Organoleptic parameters of fresh classic yogurt, immediately after obtaining were:

- clot appearance: strong, porcelain
- flavor: specific of lactic fermentation
- taste: nice, low tartish

For determining the period of validity has pursued the preservation of classical yogurt kept under refrigeration (2 - 4°C) for a 29 days period, during which was followed acidity variation.

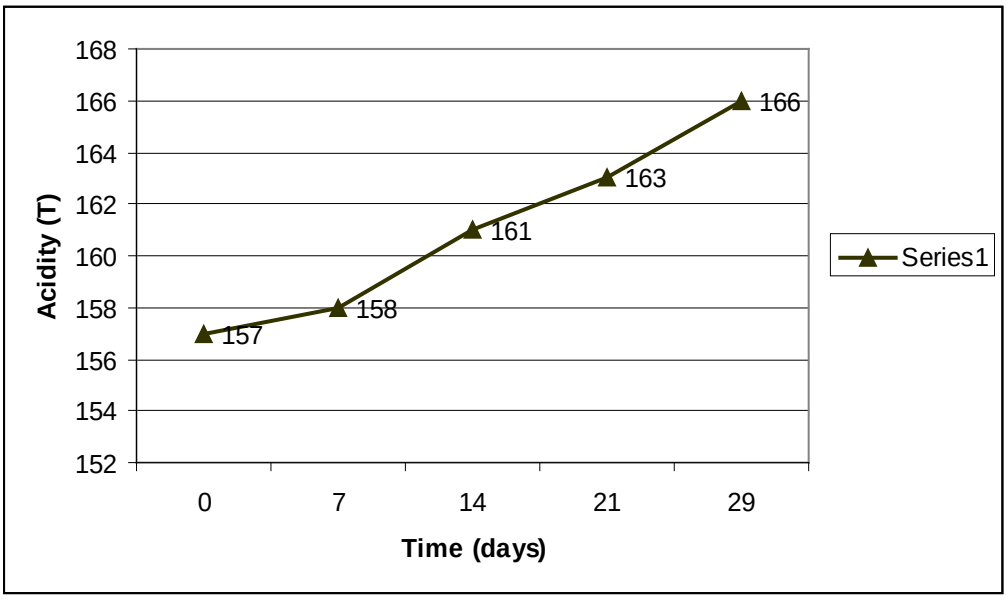

Fig.1 Acidity variation of classic yogurt

At the end of the analyzed period classic yogurt had an acidity of 166°T, with 9°T more than in the first day so the rate of acidity increase was 0.31°T/day during refrigeration (fig.1) and presented the following organoleptic characteristics:

- clot appearance: with removal of whey
- flavor: specific of lactic fermentation
- taste: tartish strong, bitter

The preservation period of classic yogurt was a period of 21 days, during this period acidity ranged in normal limits.

The content of dry matter and ash was a little higher in probiotic yogurt than in classic yogurt (fig.2, fig.3).

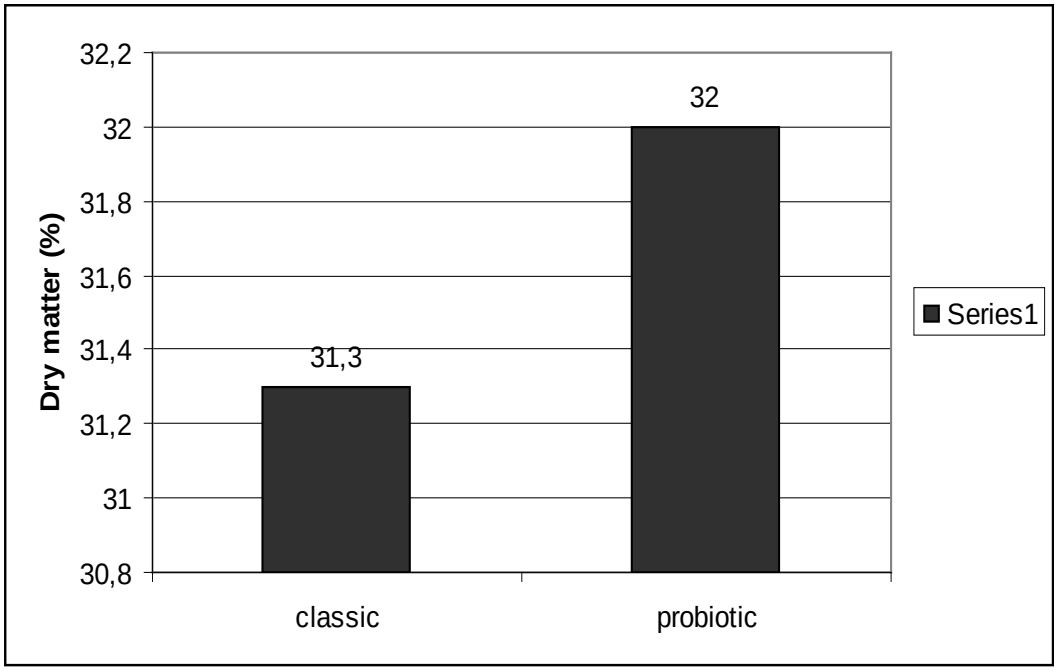

Fig.2 Dry matter variation of yogurt

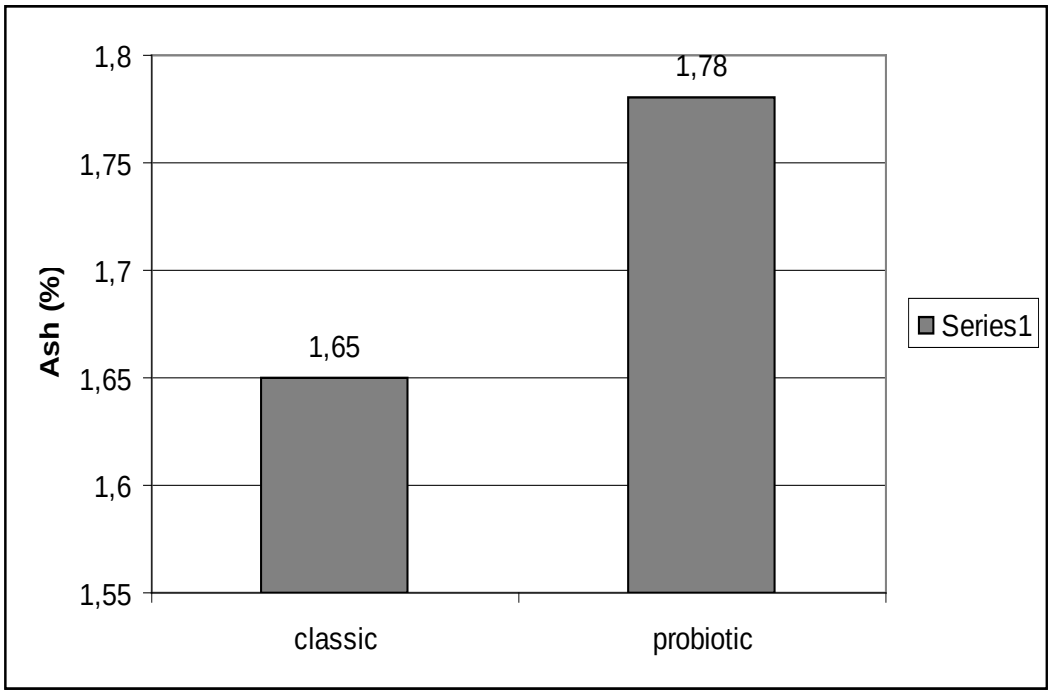

Fig.3 Ash variation of yogurt

Organoleptic parameters of fresh probiotic yogurt, immediately after obtaining were:

- clot appearance: compact, fine, creamy
- flavor: pleasant of lactic fermentation
- taste: specific of yogurt, low tartish, well expressed

For determining the period of validity has pursued the preservation of probiotic yogurt kept under refrigeration (2 - 4°C) for a 29 days period, during which was followed acidity variation.

At the end of the analyzed period, probiotic yogurt had an acidity of 177°T, with 12°T more than in the first day so the rate of acidity increase was 0.41°T/day during refrigeration (fig.4) and presented the following organoleptic characteristics:

- clot appearance: with removal of whey
- flavor: specific of lactic fermentation
- taste: tartish strong, bitter

The preservation period of probiotic yogurt was also a period of 21 days, during this period acidity ranged in normal limits but its evolution was more pronounced than in the case of classic yogurt.

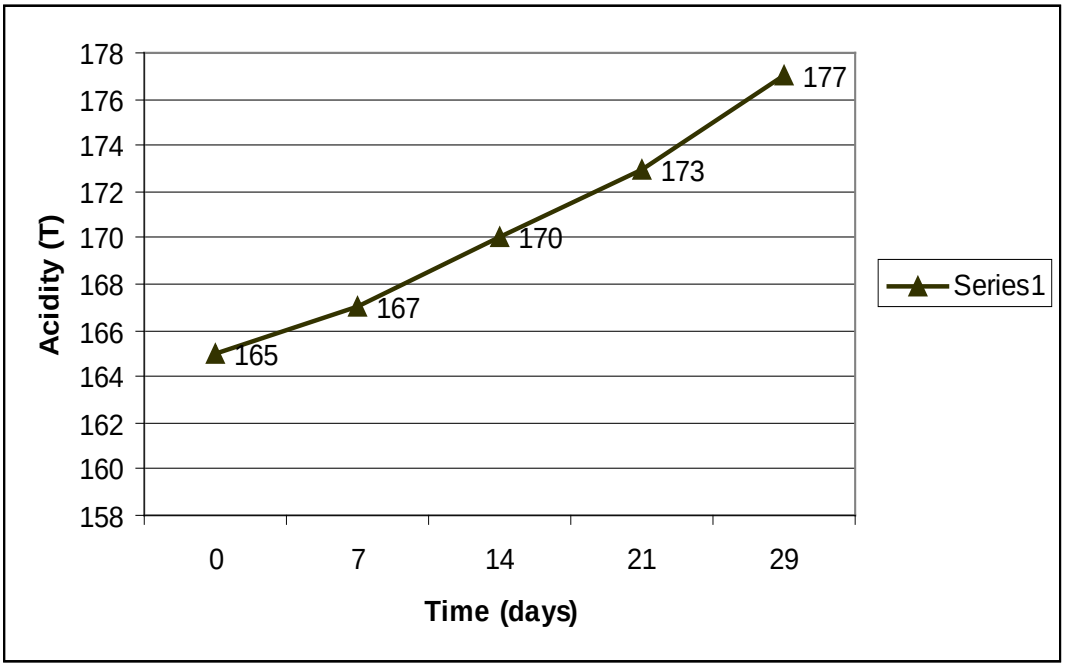

Fig.4 Acidity variation of probiotic yogurt

4. Conclusions

Acidity of probiotic yogurt evolved faster than that of classic yogurt, because the addition of lactic bacteria specific cultures which hydrolysis lactose to lactic acid. Both products, classic and probiotic yogurt had a preservation period of 21 days in witch organoleptic characteristics were

classified as normal issues, and can be translate that the increase of acidity in probiotic yogurt has antimicrobial activity on pathogens.

At the end of the analyzed period classic yogurt had an acidity of 166°T, with 9°T more than in the first day so the rate of acidity increase was 0.31°T/day during refrigeration and probiotic yogurt had an acidity of 177°T, with 12°T more than in the first day so the rate of acidity increase was 0.41°T/day during refrigeration storage.

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STUDY REGARDING THE MODIFICATION OF THE FAT GLOBULES STRUCTURE IN THE UNPASTEURIZED AND PASTEURIZED MILK DURING THE CONGELATION AND REFRIGERATION PROCESSES

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Abstract

The study presents pictures with microscope images of the fat globules and conclusions about the structure of these globules from the unpasteurized and pasteurized milk which suffered the processes of congelation or refrigeration. After the congelation, the milk was defrosted at the room temperature or with the microwaves.

Keywords: *fat globule structure, congelation, refrigeration, microwaves*

1. Introduction

The role of milk in nature is to nourish and provide immunological protection for the mammalian young. Milk has been a food source for humans since prehistoric times; from human, goat, buffalo, sheep, yak, to the focus of this section - domesticated cow milk (genus Bos). Milk and honey are the only articles of diet whose sole function in nature is food. It is not surprising, therefore, that the nutritional value of milk is high. Milk is also a very complex food with over 100,000 different molecular species found.

Bovine milk contains about 3.5 to 5% total lipid, existing as emulsified globules 2 to 4 μm in diameter and coated with a membrane derived from the secreting cell. In homogenized milk, the coat is mostly casein. About 98 % or more of the lipid is triacylglycerol, which is found in the globule. Phospholipids are about 0.5 to 2 % of total lipids and sterols are 0.2 to 0.5 %. These are mostly located in the globule membrane. Cholesterol is the major sterol at 10 to 20 mg/dl.

The physical properties of milk fat can be summarized as follows:

- density at 20° C is 915 kg m(-3);
- refractive index (589 nm) is 1.462 which decreases with increasing temperature;
- solubility of water in fat is 0.14% (w/w) at 20° C and increases with increasing temperature;

- thermal conductivity is about 0.17 J m(-1) s(-1) K(-1) at 20° C;
- specific heat at 40° C is about 2.1kJ kg(-1) K(-1);
- electrical conductivity is <10(-12) ohm(-1) cm(-1) ;
- dielectric constant is about 3.1.

2. Materials and methods

For this determination we used samples from the 2 types of milk, pasteurized and unpasteurized and examined them on the microscope. Using the photo camera attached to the microscope we took pictures of the milk fat globules in the following circumstances:

3. Results and discussion

The protein concentration in milk can be calculated with the formula:

The total quantity of protein: P = V• 1, 95, %

Where: V = the NaOH N/10 volume used for the second titration.

Results:

Raw milk

V = 1, 74 ml NaOH.

Protein: P = V•1, 95 =1,74•1,95 = 3,4 %

Rehydrated powder milk

V = 2 ml NaOH.

Protein: P = V•1,95 = 2•1,95 = 3,9 %

Proteinized milk

V = 1, 4 ml NaOH.

Protein: P = V•1,95 = 2•1,95 = 3,9 %

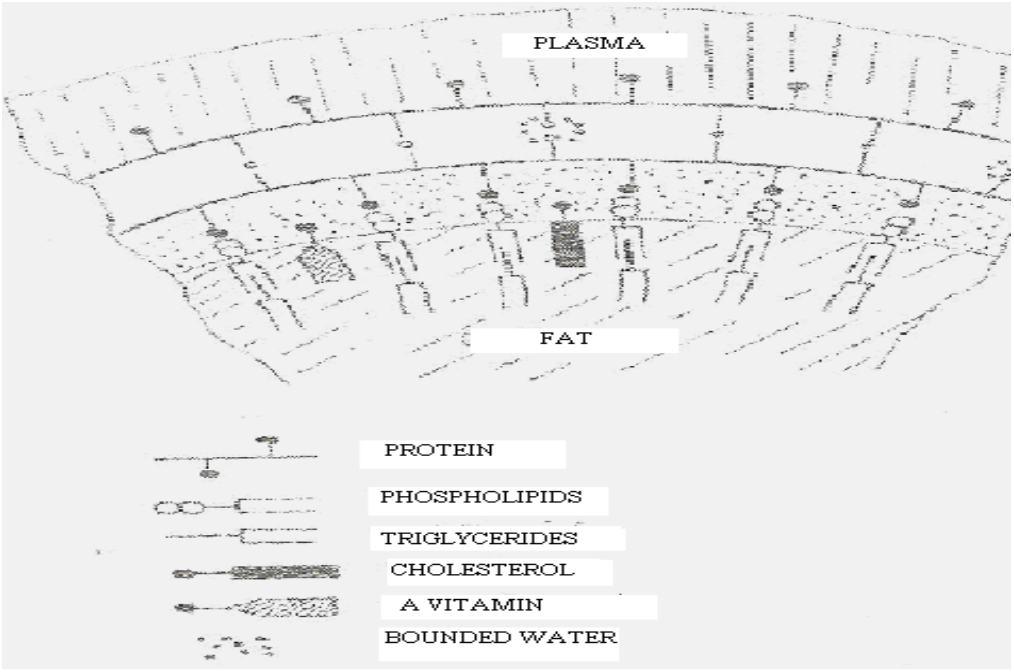

Fig. 1. Fat globule structure in the raw milk

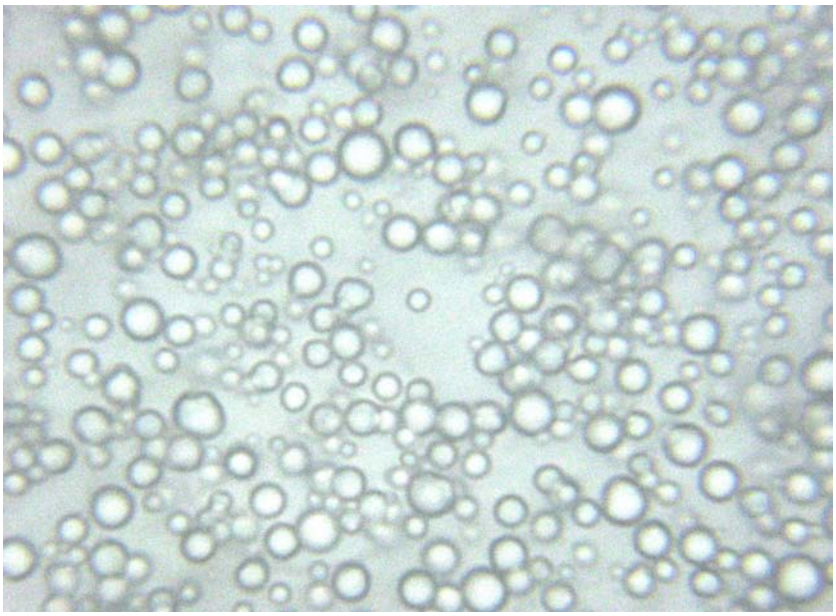

Fig. 2. Fat globule structure of the unpasteurized milk recently milked

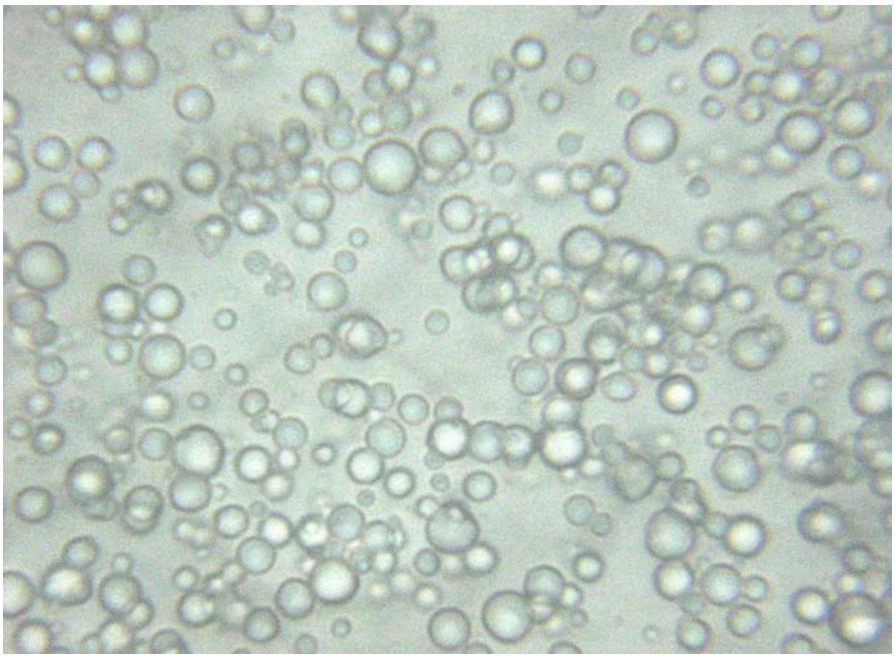

Fig. 3. Fat globule structure of the unpasteurized milk congelated and defrosted at the room temperature

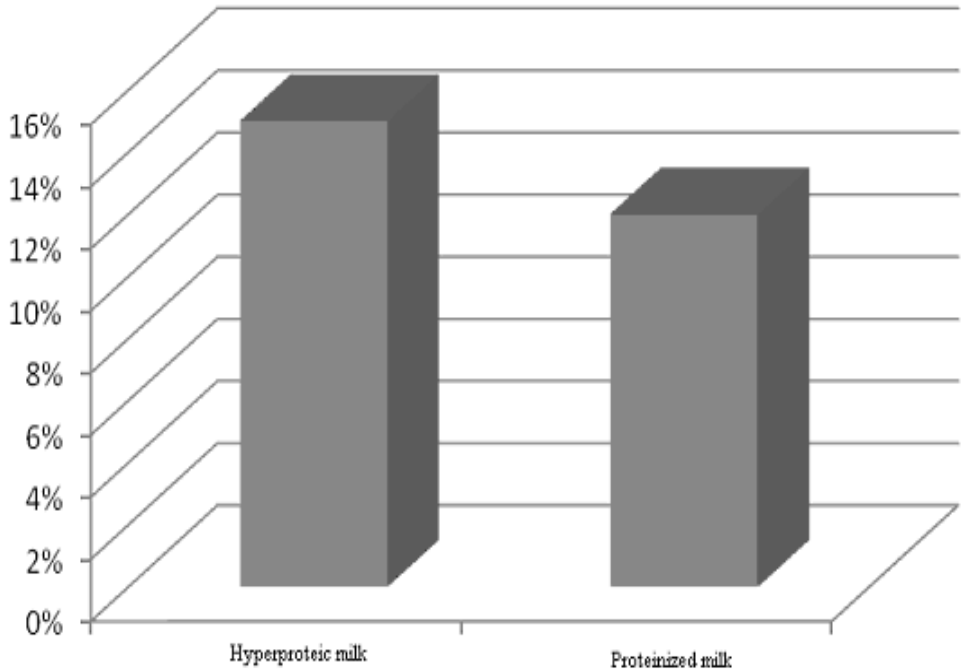

Fig. 3. Comparison between the dry substance content of the proteinized milk and of the hyperproteic milk

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RESEARCH CONCERNING PHYSICOCHEMICAL CHANGES AND FRESHNESS INDICATORS OF MILK FAT DURING STORAGE

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Abstract

Physicochemical characteristics and freshness indicators of cow butter during refrigeration (2 ... 4^{o}C) and freezing (-15 ...- 18^{o}C) storage were studied. Alteration (hydrolysis and oxidation) of food is responsible for the degradation of sensory quality, nutritional value and even the formation of toxic substances such as peroxides, which requires intimate knowledge of these processes and taking appropriate measures to avoid losses that can be registered. Research motivation was the determination of physicochemical indicators in fresh milk fat, and the moment when occur changes in organoleptic and physicochemical parametres of butter stored under refrigeration and freezing, making it unsuitable for human consumption. Changes in freshness parameters and the installation of alterative process when butter becomes improperly for consumption were studied inducing fatty acid content, acidity, peroxide index, iodine index and the presence of epyhidrinic aldehyde. The content of saturated fatty acids was higher (71.84%) than that of unsaturated fatty acids (27.09%), the main fatty acids present in butter were butyric, miristic, palmitic, oleic and stearic acids. There was an increase of titrabile acidity during storage, butter hydrolysis was install after 15 days under refrigeration and after one month under freezing conditions. Results showed that butter is resistant to oxidation, epyhidrinic aldehyde was shown after 6 months of storage under refrigeration and after 9 months in freezing conditions.

Keywords: *milk fat, physicochemical characteristics, refrigeration, freezing, freshness*

1. Introduction

Butter is considered one of the most popular concentrated milk products. Its nutritive value is high and is based on fat content. Digestibility of butter is 97% for fat and 94% for dry plasma, represents an important source of vitamin E [7].

Hydrolysis and oxidation occurring in animal fats during their storage have resulted in the depreciation of their quality and their exclusion from the diet.

Hydrolysis is the type of alteration which is finalized with the release of the two primary components: fatty acids and glycerine [1 , 4, 12].

The first factor which requires hydrolysis is the water content of fat, the other factor being hydrolitic specific enzymes [12].

Lipid oxidation includes fatty acid oxidation and generates compounds that affect food quality, due to changes in

color, flavor, texture and even nutrition and food safety [9, 14].

Autooxidation is the reaction of atmospheric oxygen and lipids, because unsaturated ties, after this process being irreversibly compromised the quality of fatty substances, not only in terms of organoleptic (taste and aroma) but also in terms of toxicology. Lipid oxidation involves radicals reactions which unfolds in three steps [14]:

- initiation (1)

fotosensibilization

$$R - H \rightarrow R\blacksquare + H\blacksquare \quad (1)$$

- propagation (2 and 3)

$$R\blacksquare + \blacksquare O - O\blacksquare \rightarrow R - O - O\blacksquare \quad (2)$$

$$R - O - O\blacksquare + R - H \rightarrow ROOH + R\blacksquare \quad (3)$$

- interruption or end (4 and 5)

$$R\blacksquare + R\blacksquare \rightarrow R - R \quad (4)$$

$$R - O\blacksquare + R\blacksquare \rightarrow R - O - R \quad (5)$$

In the initial phase of oxidation, oxygen is fixed in the peroxidic form at double links of several molecules of unsaturated fatty acids [12, 14]:

In an advanced stage, the peroxidic bound is breaking, after which results a lot of chemical compounds of decomposition: aldehydes, ketones, alcohols, inferior acids, acids-alcohols, acids –aldehydes, acids-ketones, etc. [14]. Reached at this stage fat becomes unfit for consumption. Of the chemical, specific reaction for aldehydes identification (Kreis) will be positive and regardless of the intensity of the reaction (weak positive, positive or mostly positive), fat should be excluded from the food circuit. In this stage of oxidation are installed organoleptic changes, easily discernible using the senses: yellow color, smell and taste of ranced [9.14]. Peroxide index provides us information on the incipient oxidation, and Kreis reaction illustrates advanced oxidation.

Research motivation is the determination of physicochemical indicators in fresh milk fat, and the moment when occur changes in the organoleptic and physicochemical parametres of butter stored under refrigeration and freezing, making it unsuitable for human consumption.

2. Materials and methods

Samples

Butter with a content of 80% fat and 16% water was collected immediately after obtaining in a processing milk unit and stored under refrigeration (2 ... 4^{o}C) and freezing (-15 ...- 18^{o}C), following the installation of altering processes (hydrolysis and oxidation).

Physicochemical examination

Fatty acids content was determined using Hp - Hewlett Packard 5890 GC, chromatography column with high resolution: DB-5, code 123-5011, L = 15 m, D_{int} = 0.320 mm, Film = 0.10 μm; temperature limit in the column: - 60 ... 350^{o}C; flame ionisation detector, by transformation in methyl esters of fatty acids in the sample under analysis, followed by components separation on a chromatography column, their identification by comparison with standard cromatograms and quantitative determination of fatty acids [6, 11, 16] .

Determination of acidity is the basic criterion for assessing the installation and intensity of hydrolysis. The method consists in neutralizing acidity with sodium hydroxide 0.1 N, using phenophtaleine, as an indicator. Acidity was expressed in oleic acid grams to 100 grams of fat [16, 17].

Peroxide index was determined using UV - VIS T60U spectrophotometer (England): operating temperature 5 – 45^{o}C; field wavelength 190 - 1100 nm; wave length accuracy 0.1 nm.

This protocol was based on the spectrophotometric determination of ferric ions (Fe^{3+}) derived from the oxidation of ferrous ions (Fe^{2+}) by hydroperoxides, in the presence of ammonium thiocyanate (NH_4SCN). Thiocyanate ions (SCN^-) react with Fe^{3+} ions to give a red-violet chromogen that can be determined spectrophotometrically, the absorbance of each solution was read at 500 nm. To quantify peroxide value, a calibration curve (absorbance at 500 nm vs. Fe^{3+} expressed in μg) was constructed and peroxide value was expressed as meq O_2/kg of fat.

Iodine index was determined using Hanus [16]. There have been weighing on analytical balance, 0.5 g fat, melted in advance, were added 10 ml chloroform under continuous agitation for complete dissolution and 25 ml solution Hanus. After homogenization, was plugged with cork and let in the darkness 30-60 minutes. Then were added 20 ml solution of potassium iodide 15%, fresh prepared, and 100 ml distilled water, that were well washed stopper and neck vessel, not to remain traces of iodine on them. In parallel, has been a witnessed sample, in the same conditions, but without fat. Were titrated with sodium thiosulphate 0.1 N. Around the end of titration (straw-yellow color), was add 1 ml starch solution 1% and continued titration, drop by drop, until the sudden disappearance of the color blue. Iodine index was calculated as the amount of iodine in g, in addition to 100 g fat.

By Kreis reaction we identify aldehydes results in advanced stages of fat oxidation. Epihidrinic aldehyde, formed during advanced oxidation of fats, released in an acid environment, reacts with phluoroglucine, giving a colored compound. Color intensity is proportional to the quantity of epihidrinic aldehyde, and so with the oxidation process [16].

3. Results and discussion

The content of saturated fatty acids of butter was higher (71.84%) than that of unsaturated fatty acids (27.09%), major fatty acids present in butter were butyric, miristic, palmitic, stearic and oleic acids (fig.1). Palmitic acid was determined in the largest proportion (32.04%) these results are in agreement with previous studies on different types of milk cow butter. Glew, Okolo, Chuang and Vanderjagt said that the content of palmitic acid in the „Fulani butter" was 30.2%, and Sagdic et al. presented a content of palmitic acid in the „Turkish butter" of 33.72% [5, 13].

To watch the acid hydrolysis were determined following values of titrable acidity of butter stored under refrigeration (2 ... 4^{o}C), determinations are executed at intervals of 5 days: for fresh butter acidity was 1% (g oleic acid); for butter to 5 days refrigerated 1.1%; for butter to 10 days refrigerated 1.3%; for butter to 15 days refrigerated 1.7%, and for butter to 20 days refrigerated 2.1%.

Results showed that for the butter with 16% water content, hydrolysis is triggered early and developed rapidly, after 5 days of refirgeration has registered a moderate increase of acidity, and this enhance during storage. It was found that advanced hydrolysis process appears after 15 days of refrigeration, acidity exceeds 2% (g oleic acid), the maximum permitted value, because it releases saturated fatty acids which are volatile, there are changes in color (yellow), taste (sour, rancid) and odor (butyric), butter becomes improper consumption [5, 13].

In assessing the degree of freshness and intensity of oxidation process for chilled butter were determined iodine index, peroxide index as an indicator of incipient oxidation [10, 15] and epihidrinic aldehyde which is an indicator of advanced oxidation [12], determinations were performed at intervals of one months until it was pointed Kreis positive feedback, when determining the installation of advanced oxidation process.

Were determined the following values for iodine index: for fresh butter 34 g I_2 / 100 g butter, butter at 1 month refrigeration 33.6; butter at 2 months refrigeration 33;

butter at 3 months refrigeration 32.3; butter at 4 months refrigeration 27.9; butter at 5 months refrigeration 27.2 and butter at 6 months refrigeration 26.4. In the first 3 months iodine index values falls slightly, in month 4 the decrease is more pronounced, in line with the propagation phase of lipid oxidation that forms the largest quantity of hydroperoxides, then the decrease represents a slow slope as presented in fig.2 . During the storage there is a fall of iodine index values, because with the beginning of oxidation processes decreases the degree of unsaturation due to unsaturated fatty acids oxidation [15].

For fresh butter the peroxide index was determined to be 0.4 meq O_2/kg butter, followed an upward slope. In the first 4 months of storage under refrigeration there was a slow increase of the peroxide index, which corresponds to the initiation phase of oxidation [10, 15], followed by a sharp increase corresponding to propagation phase in which are formed the largest amount of hydroperoxides as primary compounds of oxidation, value reached at 3.4 meq O_2/kg, in month 6 the growth is relatively constant, up from 3.9 meq O_2/kg because the balance formed between peroxides and secondary compounds, after which the peroxide index decreases as a result of the split of hydroperoxides in secondary compounds, in this moment Kreis reaction is positive indicating epihidrinic aldehyde presence (fig.3).

To follow the acid hydrolysis of butter stored in freezing conditions (-15 ...- 18^{o}C) were determined following values of titrable acidity, determinations were executed at intervals of one month: the fresh butter had 1% (g oleic acid) acidity; butter 1 month frozen 1.6% and butter 2 months frozen 2.1% . The results show that the butter acidity reach 2.1% (g oleic acid) after 2 months of freezing, exceeding the maximum permitted limit and butter become unsuitable for consuption.

To watch the installation of oxidation process were determined the following values of iodine index for butter store under freezing conditions: for fresh butter 34 g I_2/100 g of fat; butter at 1 month freezing 33.7; butter at 2 months freezeing 33.2; butter at 3 months freezing 32.5; butter at 4 months freezing 31.8; butter at 5 months freezing 30.9; butter at 6 months freezing 28.7; butter at 7 months freezing 24.4; butter at 8 months freezing 23.6 and butter at 9 months freezing 22.3. Results showed that during storage there is a fall of iodine index values, because with the beginning of oxidation processes decreases

the degree of unsaturation due to unsaturated fatty acids oxidation (fig.4).

Fig.4 shows that in the first 6 months of storage under freezing there was a slow increase of the peroxide index, which corresponds to the initiation phase of oxidation, followed by a sharp increase corresponding to propagation phase [10, 15] in which are formed the largest amount of hydroperoxides as primary compounds of oxidation, value reached at 3.5 meq O_2/kg, in the next 3 months the growth is relatively constant because the balance formed between peroxides and secondary compounds, and after 9 months the peroxide index decreases as a result of the split of hydroperoxides in secondary compounds, in this moment Kreis reaction is positive indicating epihidrinic aldehyde presence.

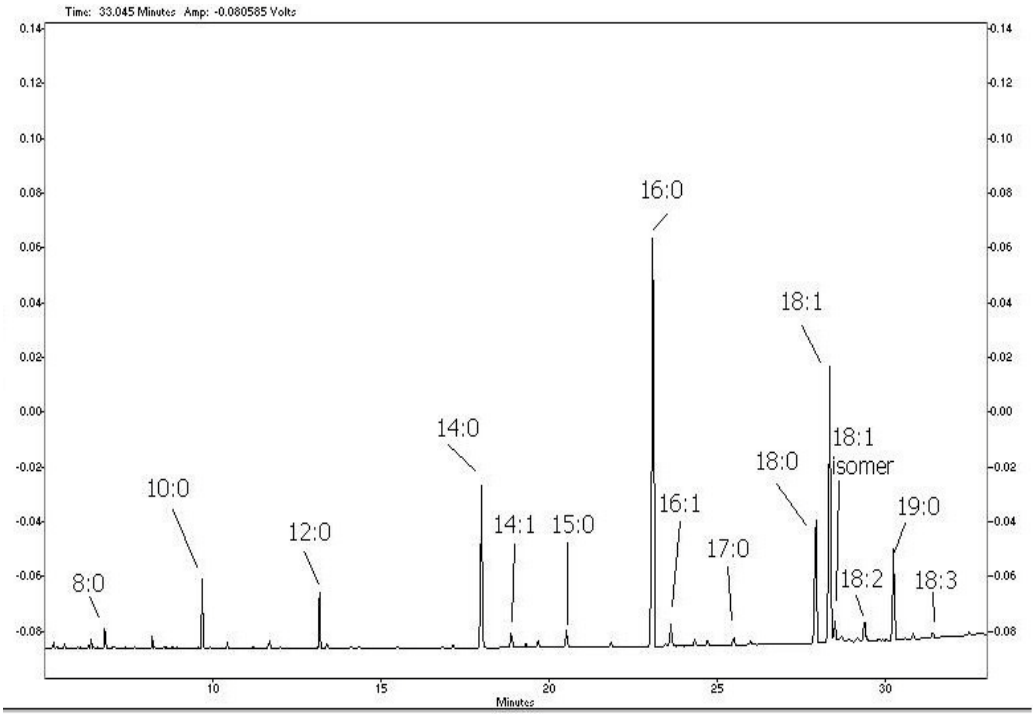

Fig. 1 Fatty acids composition of fresh cow milk fat

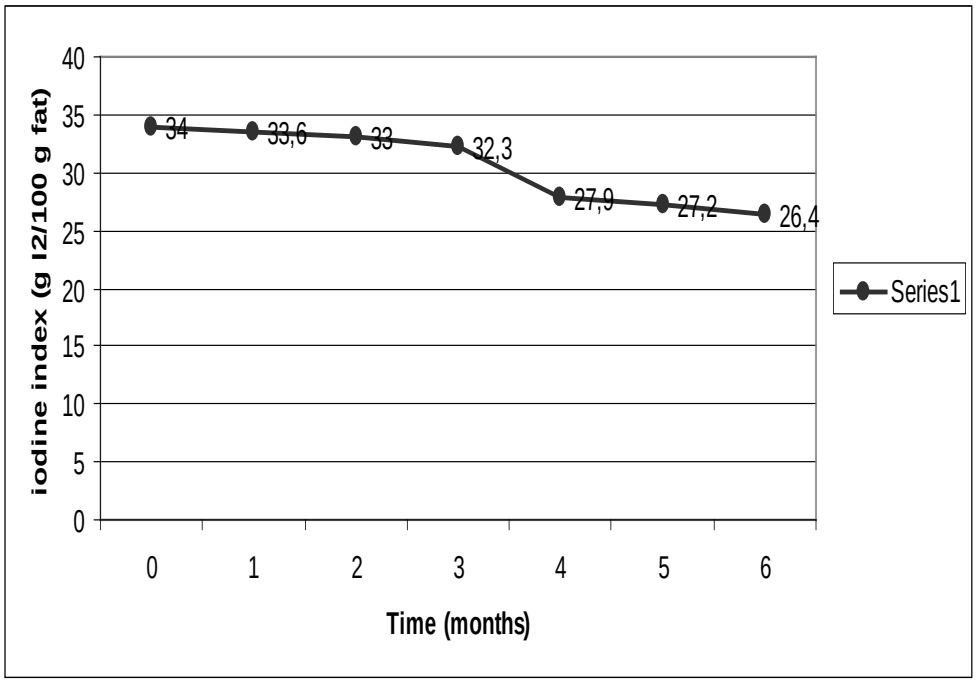

Fig.2 Iodine index variation of refrigerated butter

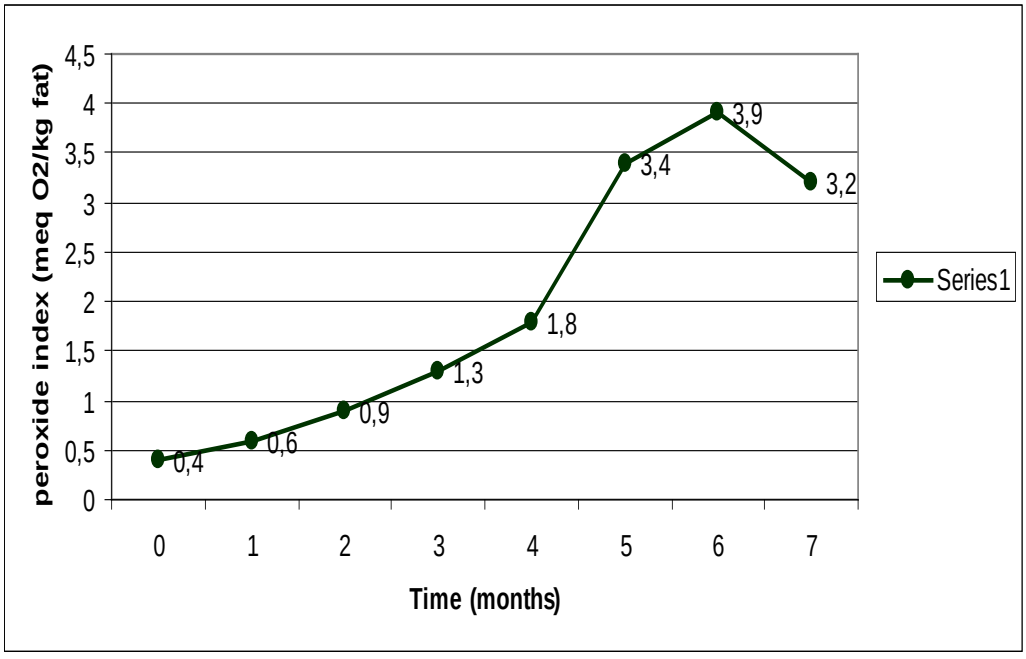

Fig.3 Peroxide index variation of refrigerated butter

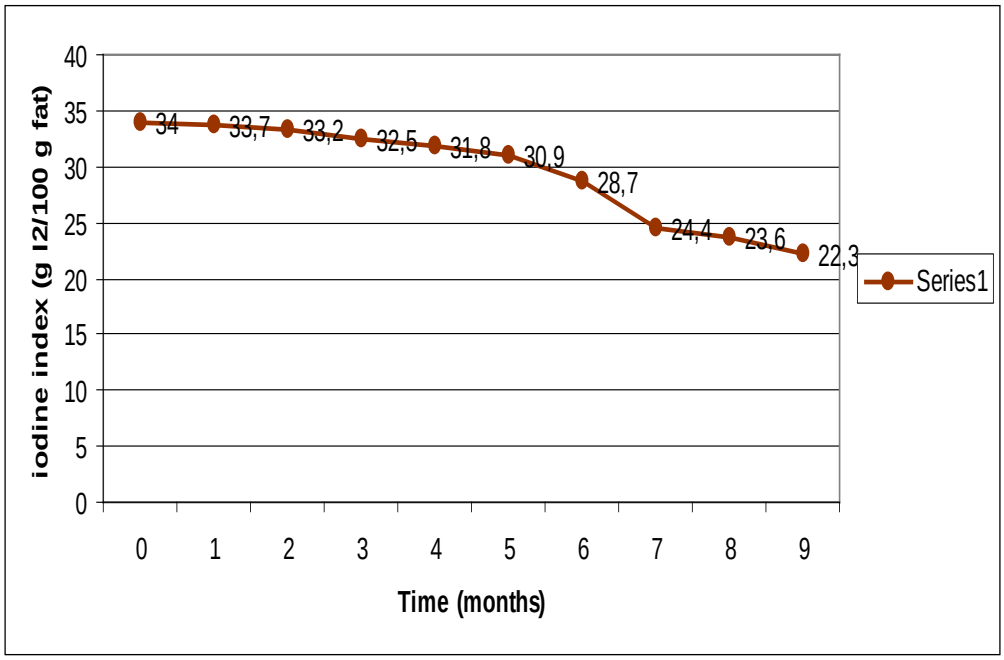

Fig.4 Iodine index variation of frozen butter

4.Conclusions

The timing of changes occurring in hydrolysis and oxidation processes of milk fat has particular importance in assessing the quality and its validity.

In frozen butter altering processes take place more slowly than in that stored under refrigeration. Hydrolysis process is installed more quickly in terms of refrigeration and freezing than oxidative processes, being intensified by a higher water content in product and by hydrolitic enzymes presence. Results showed that butter is likely to acid hydrolysis due to the high water content (16%), which favors glycerides hydrolysis translated by increasing of titrable acidity until it exceeds 2%, and is resistant to oxidation due to low composition in unsaturated fatty acids, advanced oxidation is installing after 6 months in chilled butter and after 9 months if frozen butter.

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27.*** International Standard ISO 3976, 2006.

28.*** Romanian Standard SR ISO 14082, 2003.

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STUDY REGARDING SOME PHYSICAL - CHEMICAL CHARACTERISTICS OF THE YOGHURT WITH RED BEETROOT JUICE

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Abstract

The study presents some physical - chemical characteristics of the beetroot juice yoghurt. The following determinations were carried out: the determination of the acidity by titration with sodium hydroxide (NaOH) N/10, the determination of the fat content by the acid-butirometric method and the determination of the dry substance using the drying oven with a 102 °C temperature.

Keywords: **:** *beetroot juice yoghurt, acidity, fat, dry substance*

1. Introduction

Yoghurt is produced by the controlled fermentation of milk by two species of bacteria, Lactobacillus and Streptococcus. The sugar in milk (called lactose) is fermented to acid (lactic acid) and it is this that causes the characteristic curd to form. The acid also restricts the growth of food poisoning bacteria and some spoilage bacteria. So, whereas milk is a potential source of food poisoning and only has a shelf life of a few days, yoghurt is safer and can be kept for up to ten days, under proper storage conditions.

Beet was looked upon by the Romans as being good for cooling the blood. Red beetroot is also found to benefit against feverish conditions, especially in growing children. Later generations found that beetroot was also beneficial for a very strong specific action to regulate the digestive system. The chlorine present in the red beetroot juice acts to regulate digestion by controlling the peristalsis, or natural rhythmical contractions of the gut and, because of this, the assimilation of food during the time of recovery is much assisted.

In Germany there is a widespread use of the red beetroot juice, which is available in pasteurized form in bottles, as a powerful restorative during convalescence. It follows that beets are also good for general weakness and debility of all sorts. In combination with other juices, especially carrot and cucumber, red beetroot juice is not only a splendid blood builder but also one of the finest therapies for sexual

weakness, kidney stones, gall bladder, kidney, liver and prostate troubles.

Nevertheless, the popularity of yoghurt has been propelled by the availability of the sweetened fruit or vegetables flavored products. The addition of these new flavors enhances the versatility of taste, color and texture for the consumers.

2. Materials and methods

2.1 The determination of the acidity

In a 100 ml Erlenmeyer glass we put 10 ml of yoghurt with red beetroot juice and 3 drops of phenolphthalein. We titrated the mixture with sodium hydroxide (NaOH) N/10 until the pink – pal color obtained persisted for 30 seconds.

Acidity $^oT = V \times 10$

$^oT = 15 \times 10 = 150$

Where: V - the NaOH N/10 volume used for the titration, ml;

V = 15;

10 - Yoghurt volume used for the determination.

2.2 The determination of the yoghurt fat by the acid-butirometric method (Gerber)

The fat content of the yoghurt with beetroot juice can be read in percentage directly on the graded scale of the butirometer after a previous dissolution of the proteic substances from yoghurt under the sulphuric acid action and after the separation of the fat by the centrifugation in the presence of a small amount of isoamyl alcohol .

The yoghurt with red beetroot juice has a fat content of 4,1 %.

2.3 The determination of the dry substance content of the yoghurt with beetroot juice

The dry substance content in the yoghurt with beetroot juice was determined through the evaporation of the water existing in the yoghurt sample, using the drying oven with a 102°C temperature, until the sample had a constant mass.

$$\text{d. s. \%} = \frac{m_2 - m_0}{m_1 - m_0} \bullet 100$$

Where:

m_0 – the capsule's quantity, in grams (g);

m_1 – the capsule's quantity and the product's quantity before drying, in g;

m_2 – the capsule's quantity and the product's quantity after drying, in g .

$$\text{\% DS} = \frac{6{,}523 - 4{,}326}{14{,}326 - 4{,}326} \times 100 = 21\ \%$$

3. Results

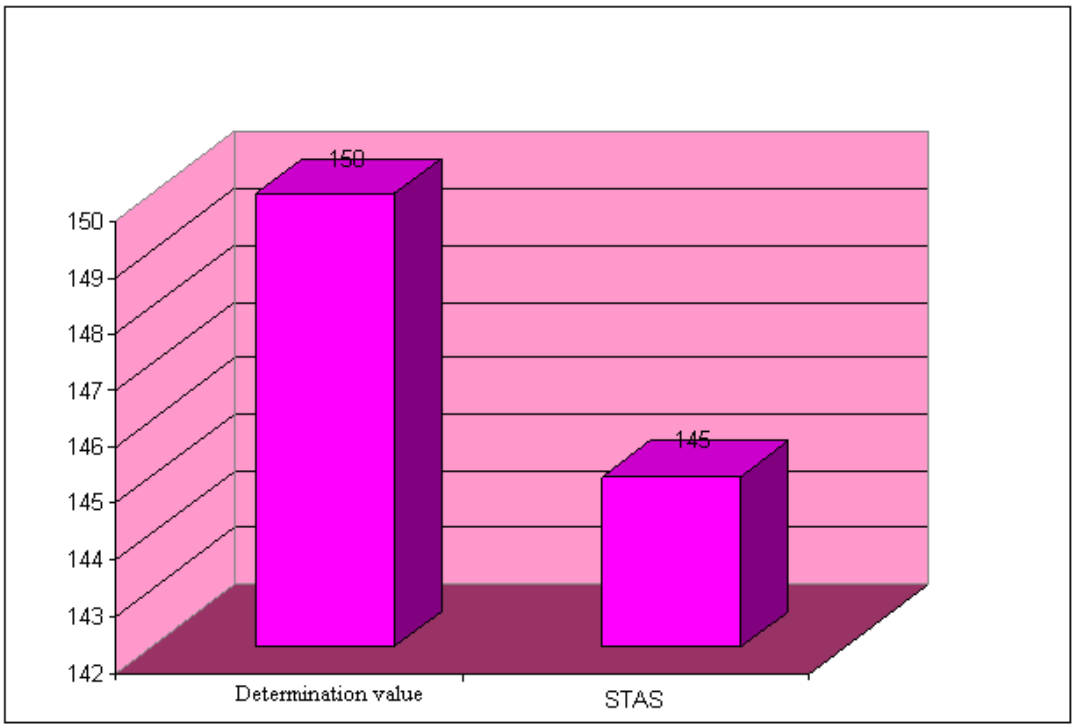

Fig. 1. Comparison between our determination value and the acidity value of the normal yoghurt as it is given by the STAS

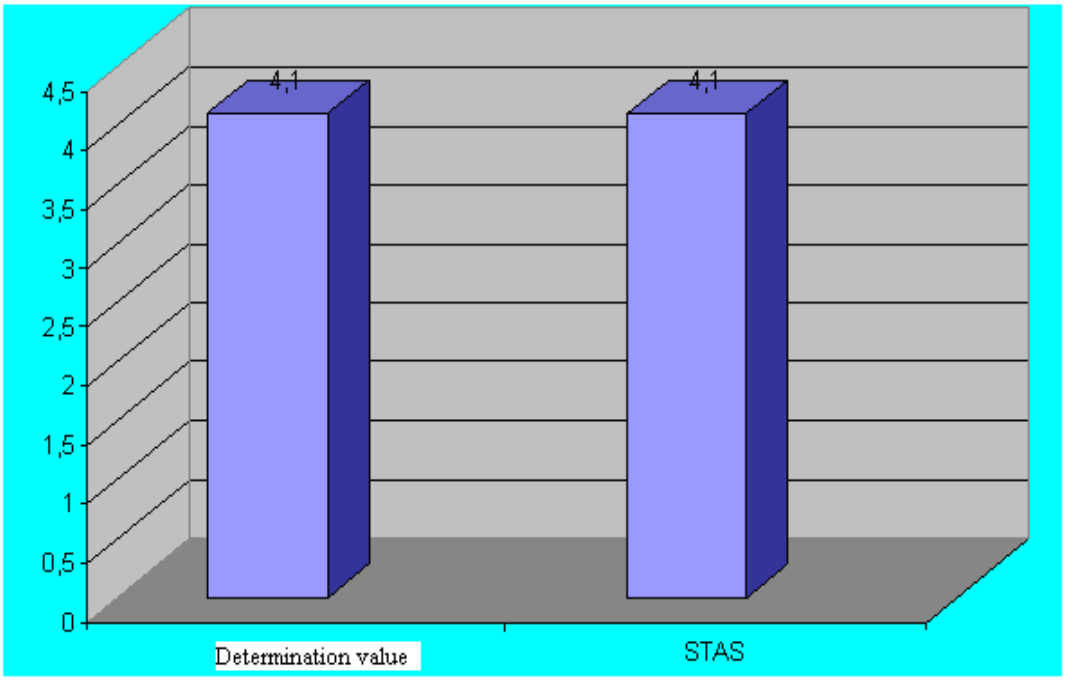

Fig.2 The determination value, 4,1 %, corespondes with the value given by the STAS for the yoghurt with fruits or vegetables juice addition

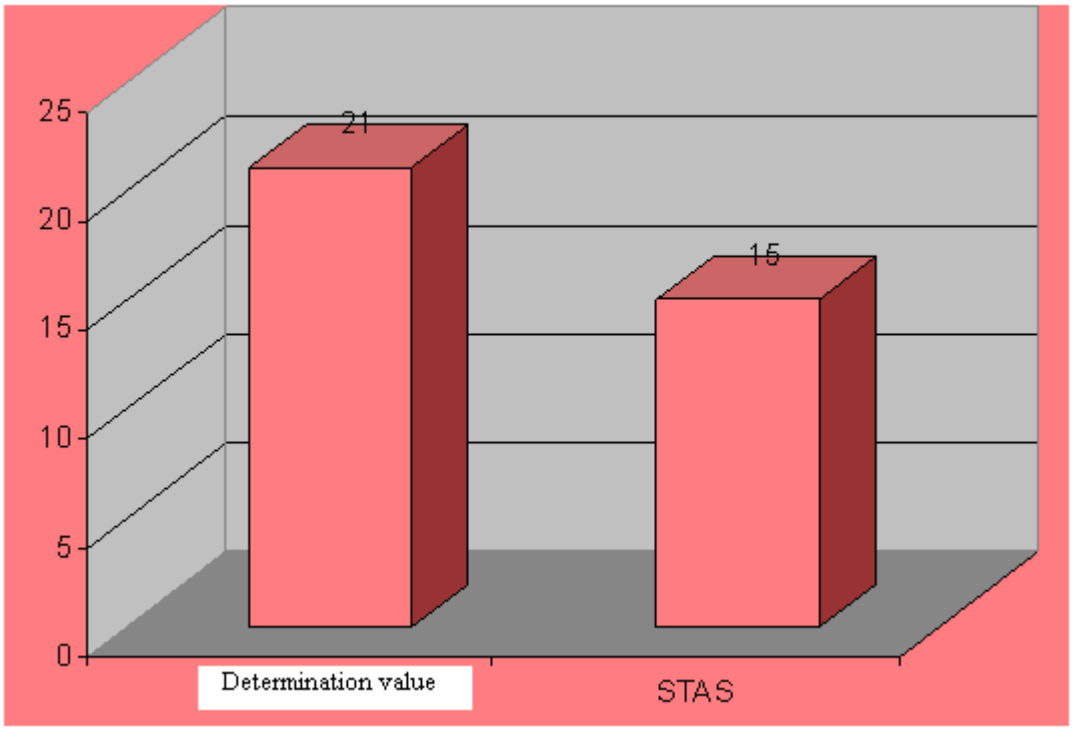

Fig. 3. . Comparison between our determination value and the dry substance value of the normal yoghurt as it is given by the STAS

4. Conclusions

The fat determination showed that the beetroot juice yoghurt is a very fat type of yoghurt, the extra type, because it has a fat content of 4,1 %, whereas the normal type of yoghurt has a fat content of 2,8 %.

The acidity determination indicated that the beetroot juice yoghurt has a higher acidity level, 150 ºT, than the normal yoghurt which has an acidity level of 145 ºT. This fact is due to the addition of the beetroot juice which contains acids extracted from the beetroot.

Also, as the determination showed, the dry substance content of the beetroot juice yoghurt, 21 %, is higher than that of the normal yoghurt, 15 %. This fact is also generated by the beetroot juice added to our type of yoghurt and also by the concentration technological operation which appears in the beetroot juice yoghurt technological flux.

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EVALUATION OF ALTERATIVE PROCESSES IN BEEF TALLOW UNDER REFRIGERATION AND FREEZING STORAGE

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Abstract

Physicochemical characteristics and freshness indicators of beef tallow during refrigeration (2 ... 4^{o}C) and freezing (-15 ...- 18^{o}C) storage were studied. Alteration (hydrolysis and oxidation) of food is responsible for the degradation of sensory quality, nutritional value and even the formation of toxic substances such as peroxides, which requires intimate knowledge of these processes and taking appropriate measures to avoid losses that can be registered. Changes in freshness parameters and the installation of alterative processes when beef tallow becomes improperly for consumption were studied, inducing fatty acids content, acidity, peroxide value (PV), iodine value (IV) and the presence of epyhidrinic aldehyde. There was an increase of titrabile acidity during storage, beef tallow advanced hydrolysis was installed after 3 months and 20 days, under refrigeration, and after 6 month under freezing conditions. Hydrolysis processes were installed more quickly in terms of refrigeration and freezing than oxidative processes, being intensified by lipases presence. Results showed that beef tallow was more resistant to oxidation than to hydrolysis, due to low water content (0,15%) and to low polyunsaturated fatty acids content (8.4%), advanced oxidation was installed after 5 months in chilled beef tallow and after 8 months in frozen beef tallow.

Keywords: *beef tallow, hydrolysis, oxidation, refrigeration, freezing*

1. Introduction

Beef tallow is one of the most complex fats found in nature. This complexity stems from the extreme diversity of its fatty acids (FA) (e.g., chain length, degree of unsaturation and branching). It also contains thousands of triacylglycerol (TAG) species, its nutritive value is based on fat content (Gus, 2003).

Hydrolysis and oxidation occurring in animal fats during their storage have resulted in the depreciation of their quality and their exclusion from the diet. Hydrolysis is the type of alteration which is finalized with the release of the two primary components: fatty acids and glycerine. The first factor which requires hydrolysis is the water content of fat, the other factor being hydrolitic specific enzymes (Ciobanu, D., & Ciobanu, R., 2001; Banu et al., 2002).

Lipid oxidation includes fatty acid oxidation and generates compounds that affect food quality and even nutrition and food safety (Gertz, Klostermann, & Kochhar, 2000; Tofana Maria, 2006). Off-flavorings, nutritional losses and other deteriorative changes in fats are concerned with the changes that result from reaction with atmospheric oxygen, oxidative rancidity, or by hydrolytic reactions catalyzed by lipases from food or from microorganisms. Oxidative rancidity or autooxidation cannot be stopped by lowering the temperature of storage since it is a chemical reaction with low activation energy.

Research into the problems, concerning oxidative deterioration has been pursued for many years but is has been given a boost by the recognition that such oxidations can cause damage to cell membranes and DNA (Moller & Wallin, 1998) that may be involved in aging process (Liu et al., 1998), hypertension (Russo et al., 1998) and cancer growth (Navarro et al., 1999).

Autooxidation is the reaction of atmospheric oxygen and lipids, because unsaturated ties, after this process being irreversibly compromised the quality of fatty substances, not only in terms of organoleptic (taste and aroma) but also in terms of toxicology. Lipid oxidation involves radicals reactions which unfolds in three steps (Tofana, 2006).

Lipid oxidation proceeds through a typical self-propagating free radical mechanism where oxygen attack occurs mainly at the allylic positions adjacent to double boubds (Nawar, 1998). The photosensitised route is an alternative oxidative pathway that involves the direct reaction of excited singlet oxygen ($^1O_2$) to unsaturated lipids in the presence of sensitisers (Frankel, 1980; Hamilton, 1989; Rawls & Van Santen, 1970). In the peroxidation of unsaturated fatty acids, lipid hydroperoxides form during the propagation phase.

These compounds are unstable and decompose rapidly, giving rise to a range of new free radicals and other non-radical compounds, including alkoxyl and alkyls radicals, aldehydes, ketones, as well as variety of carboxyl compounds that form a complex mixture of secondary lipid oxidation products.

Volatiles such as hexanal and pentanal have been associated with the development of undesirable flavours and have been proposed as potential markers of fresh product quality (Contarini & Pavolo, 2002; Karatapanis, Badeka, Riganakos, Savvaidis, & Kontominas, 2006; Kim & Morr, 1996; Marsili &

Miller, 2003; Toso, Procida, & Stefano, 2002).

Reached at this stage, fat becomes unfit for consumption. Of the chemical, specific reaction for aldehydes identification (Kreis) will be positive and regardless of the intensity of the reaction (weak positive, positive or mostly positive), fat should be excluded from the food circuit. In this stage of oxidation, organoleptic changes are installed, easily discernible using the senses: yellow color, smell and taste of rancidity (Popescu & Meica, 1995).

Peroxide index provides us information on the incipient oxidation, and Kreis reaction illustrates advanced oxidation. In table 1 are presented the compounds formed in fat autooxidation processes.

Research motivation is the determination of physicochemical indicators in fresh beef tallow, and the moment when occur changes in the organoleptic and physicochemical parametres of beef tallow stored under refrigeration and freezing, following hydrolysis and oxidation, making it unsuitable for human consumption.

2. Materials and methods

2.1 Samples

Beef tallow was obtained by raw tallow melting, collected from „Baltata Romaneasca" race, female, age of 8 years, and stored under refrigeration (2 ... 4^{o}C) and freezing (-15 ...- 18^{o}C), following the installation of altering processes (hydrolysis and oxidation).

2.2 GC analysis

Fatty acid composition was determined using gas chromatography (GC-FID) Shimadzu GC-17 A (England) coupled with flame ionisation detector . Gas chromatography column is Alltech AT-Wax, 0.25 mm I.D., 0.25 μm thick stationary phase (polyethylene), used helium as carrier gas at a pressure of 147 kPa, temperature of the injector and detector was set to 260^{o}C, the oven programm was the following: 70^{o}C for 2 min., then the temperature was raised up to 150^{o}C with a gradient of 10^{o}C/min., a level of 3 min. and the temperature was raised up to 235^{o}C with a gradient of 4^{o}C/min (SR EN 14082, 1998; ISO 3976, 2006).

Samples preparing to GC analysis: were weight 50 mg sample, was add 1 ml benzene, from dilution were taking 100 μl and mixed with 200 μl internal standard (nonadecanoic acid 19:0), 1 ml benzene, 2 ml methanol, 4 drops H_2SO_4 conc., was heated at 80^{o}C for 2 hours. Then we passed to esters extraction: esterificated sample was passed into a separating funnel, were add 10 ml hexane and 2 ml

distilled water, the upper was retained, filtered on cellulose filter, anhidrificated with anhydrous Na_2SO_4, dry with a rotary evaporator, then was resume in 1 ml hexane and 1 μl sample was injected into gas chromatograph.

The method consists in transforming of fatty acids in methyl esters in the sample under analysis, followed by separation of components on a chromatography column, their identification by comparison with standard chromatograms and quantitative determination of fatty acids. By chromatography separation, the sample chromatogram is obtained, in which fatty acids are recorded in the form of peaks separated from each other by increasing the length chain, and at the same length chain by increasing of unsaturated degree. By comparing the distances of each peak from analyzed sample chromatogram with peaks distances from standard chromatograms, we identify each fatty acid present in the analyzed sample. Results were expressed as w/w (%) total fatty acids (SR EN 14082, 2003; ISO 3976, 2006).

2.3 Titrable acidity

Determination of acidity is the basic criterion for assessing the installation and intensity of hydrolysis. The method consists in neutralizing acidity with sodium hydroxide 0.1 N, using phenophtaleine, as an indicator. Acidity was expressed in oleic acid grams to 100 grams sample (SR EN 14082, 1998, 2003).

2.4 Iodine value

Iodine value was determined using Hanus method (SR EN 14082, 2003). Approximately, 0.5 g sample (dissolved in 15 ml CCl_4) was mixed with 25 ml Hanus solution (IBr) to halogenate the double bonds. After storing the mixture in dark for 30 min., excess IBr was reduced to free I_2 in the presence of 20 ml of KI (100 g/l) and 100 ml distilled water. Free I_2 was measured by titration with 24.9 g/l $Na_2S_2O_3 \cdot 5H_2O$ using starch (1.0 g/100 ml) as an indicator. IV was calculated as g I_2/100 g sample.

2.5 Spectrophotometric determination of peroxide value (PV)

Peroxide value was determined using UV - VIS T60U spectrophotometer (England): operating temperature 5 – 45^oC; field wavelength 190 - 1100 nm; wave length accuracy 0.1 nm (ISO 3976, 2006).

This protocol was based on the spectrophotometric determination of ferric ions (Fe^{3+}) derived from the oxidation of ferrous ions (Fe^{2+}) by hydroperoxides, in the presence of ammonium thiocyanate (NH_4SCN). Thiocyanate ions (SCN^-) react with Fe^{3+} ions to give a red-violet chromogen that can be determined

spectrophotometrically, the absorbance of each solution was read at 500 nm.To quantify PV, a calibration curve (absorbance at 500 nm vs. Fe^{3+} expressed in µg) was constructed and peroxide value was expressed as meq O_2/kg sample.

2.6 Kreis reaction

By Kreis reaction we identify aldehydes results in advanced stages of fat oxidation.Epyhidrinic aldehyde, formed during advanced oxidation of fats, released in an acid environment, reacts with phluoroglucine, giving a colored compound. Color intensity is proportional to the quantity of epyhidrinic aldehyde, and so with the oxidation process (SR EN 14082, 1998, 2003).

3. Results and discussion

3.1 Physicochemical examination of chilled beef tallow

The content of saturated fatty acids in bef tallow was higher (57.13%) than that of monounsaturated fatty acids (34.47%) and polyunsaturated fatty acids (8,4%), major fatty acids presented were palmitic (26.72), stearic (26.21) and oleic acids (figure 1).

Oleic acid was determined in the largest proportion (30.14%) these results are in agreement with previous studies on this type of fat (Glew, Okolo, Chuang & Vanderjagt, 1999; Samet-Bali, Ayadi & Attia, 2008).

To watch the acid hydrolysis were determined following values for titrable acidity of beef tallow stored under refrigeration (2 ... 4^{o}C), determinations were made at 5 days intervals: for fresh beef tallow acidity was 0.16% (g oleic acid); for beef tallow to 5 days refrigeration 0.17%; to 10 days refrigeration 0.18%; to 15 days refrigeration 0.19%; to 20 days refrigeration 0.20%; to 25 days refrigeration 0.21%; to 30 days refrigeration 0.23%; to 35 days refrigeration 0.25%; to 40 days refrigeration 0.27%; to 45 days refrigeration 0.29%; to 50 days refrigeration 0.32%; to 55 days refrigeration 0.35 %; to 60 days refrigeration 0.38%, to 65 days refrigeration 0.41%, to 70 days refrigeration 0.45%, to 75 days refrigeration 0.49%, to 80 days refrigeration 0.53%, to 85 days refrigeration 0.59%, to 90 days refrigeration 0.65%, to 95 days refrigeration 0.71%, to 100 days refrigeration 0.77%, to 105 days refrigeration 0.84%, to 110 days refrigeration 0.93%, and for beef tallow to 115 days refrigeration 1.08%.

Results showed that for beef tallow, hydrolysis did not developed very rapidly, to 45 days of refirgeration was registered a moderate increase of acidity, and this enhanced during storage. It was found that advanced hydrolysis process appeared after 110 days under refrigeration, acidity exceeded 1% (g oleic acid), the maximum permitted value, because were released fatty acids from triglycerides, there were changes in taste and odor, beef tallow become improper for consumption.

In assessing the degree of freshness and intensity of oxidation process for chilled beef tallow were determined iodine index, peroxide index as an indicator of incipient oxidation (Naz, Siddiqi, Sheikh, & Asad Sayeed, 2005) and the presence of epyhidrinic aldehyde as an indicator of advanced oxidation (Popescu & Meica, 1995), determinations were performed at one month intervals until it was pointed Kreis positive feedback, when was determined the installation of advanced oxidation process.

Were determined the following values for iodine index: for fresh beef tallow 45.6 g I_2/100 g sample, beef tallow to 1 month refrigeration 44.7, to 2 months refrigeration 43.5, to 3 months refrigeration 41.8, to 4 months refrigeration 37.1, and beef tallow to 5 months refrigeration 36.3.

In the first 3 months, iodine index values felt slightly, in month 4 the decrease was more pronounced, in line with the propagation phase of lipid oxidation that formed the largest quantity of hydroperoxides, then the decrease presented a slow slope as indicated in figure 2.

During the refrigeration storage there was a fall of iodine index values, because with the beginning of oxidation processes decreased the degree of unsaturation due to unsaturated fatty acids oxidation (Vito, Ferioli, Riciputi, Iafelice, Marconi, & Caboni, 2008).

For fresh beef tallow, the peroxide value was determined to be 0.7 meq O_2/kg and followed an upward slope. In the first 3 months of storage under refrigeration there was a slow increase of the peroxide index, which corresponded to the initiation phase of oxidation (Naz et al., 2005), followed by a sharp increase corresponding to propagation phase in which were formed the largest amount of hydroperoxides as primary compounds of oxidation, value reached at 4.8 meq O_2/kg in month 5, after which the peroxide value decreased as a result of the split of hydroperoxides in secondary compounds, at this moment Kreis reaction was positive indicating epyhidrinic aldehyde presence (figure 3).

3.2 Physicochemical examination of frozen beef tallow

To follow the acid hydrolysis of beef tallow stored under freezing conditions (-15 ...- 18^{o}C) were determined the following values of titrable acidity, determinations were made at one month intervals: fresh beef tallow had 0.16% (g oleic acid) acidity; beef tallow to 1 month freezing 0.21%; to 2 months freezing 0.29%; to 3 months freezing 0.4%; to 5 months freezing 0.68%; to 6 months freezing 0.85% and to 7 months freezing 1.07% .

The results showed that beef tallow acidity reached 1.07% (g oleic acid) to 7 months under freezing, exceeding the maximum permitted limit, advanced hydrolysis was installed and beef tallow become unsuitable for consuption.

To watch the installation of oxidation process were determined the following values of iodine index for beef tallow store under freezing: for fresh beef tallow 45.6 g I_2/100 g sample; beef tallow to 1 month freezing 45.4; to 2 months freezing 45.1; to 3 months freezing 44.6; to 4 months freezing 43.7; to 5 months freezing 42.6; to 6 months freezing 41.2; to 7 months freezing 39.4 and to 8 months freezing 33.9. Results showed a fall of iodine index values, because with the beginning of oxidation processes decreased the degree of unsaturation due to unsaturated fatty acids oxidation (Vito et al., 2008), (figure 4).

Figure 5 illustrates that in the first 6 months of storage under freezing, there was a slow increase of the peroxide value, which corresponded to the initiation phase of oxidation, followed by a sharp increase corresponding to propagation phase in which were formed the largest amount of hydroperoxides as primary compounds of oxidation (Naz et al., 2005), value reached at 4.7 meq O_2/kg, in the next month the growth was relatively constant because the balance formed between peroxides and secondary compounds, and after 9 months the peroxide value decreased as a result of the split of hydroperoxides in secondary compounds, in this moment Kreis reaction was positive indicating epyhidrinic aldehyde presence.

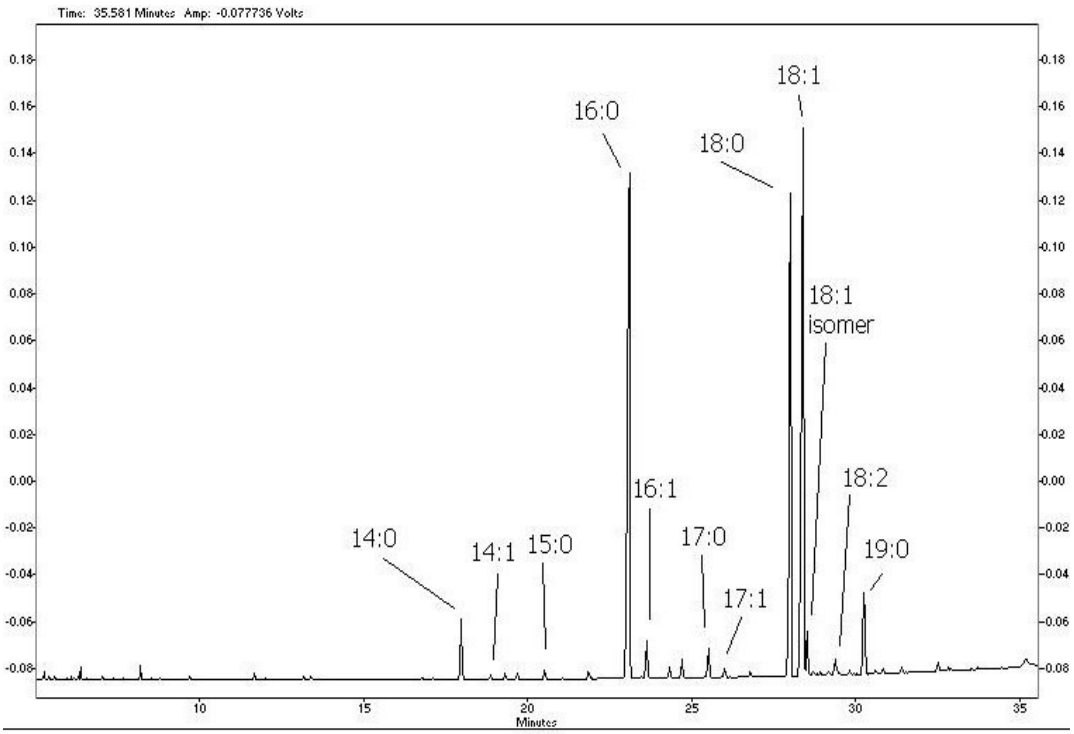

Fig. 1. Fatty acids composition of beef tallow

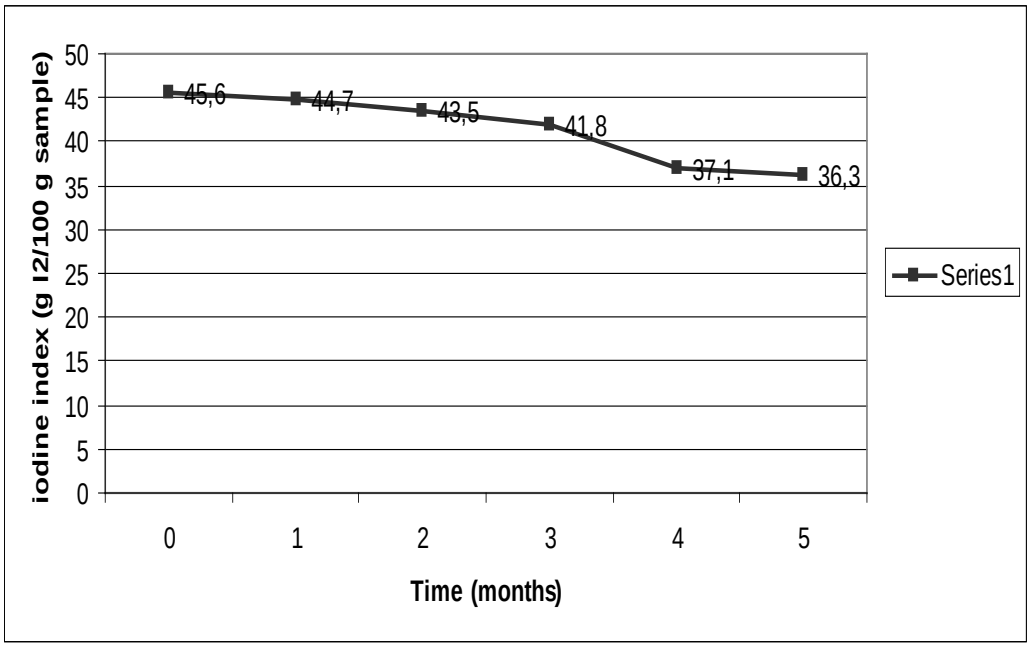

Fig. 2. Iodine index variation of chilled beef tallow

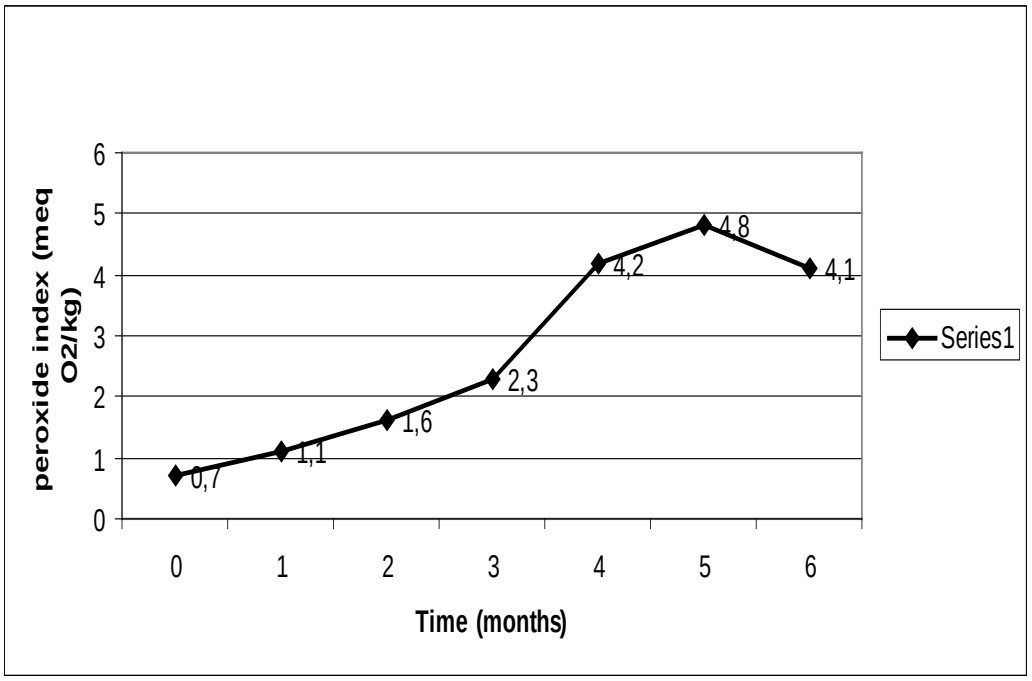

Fig. 3. Peroxide index variation of chilled beef tallow

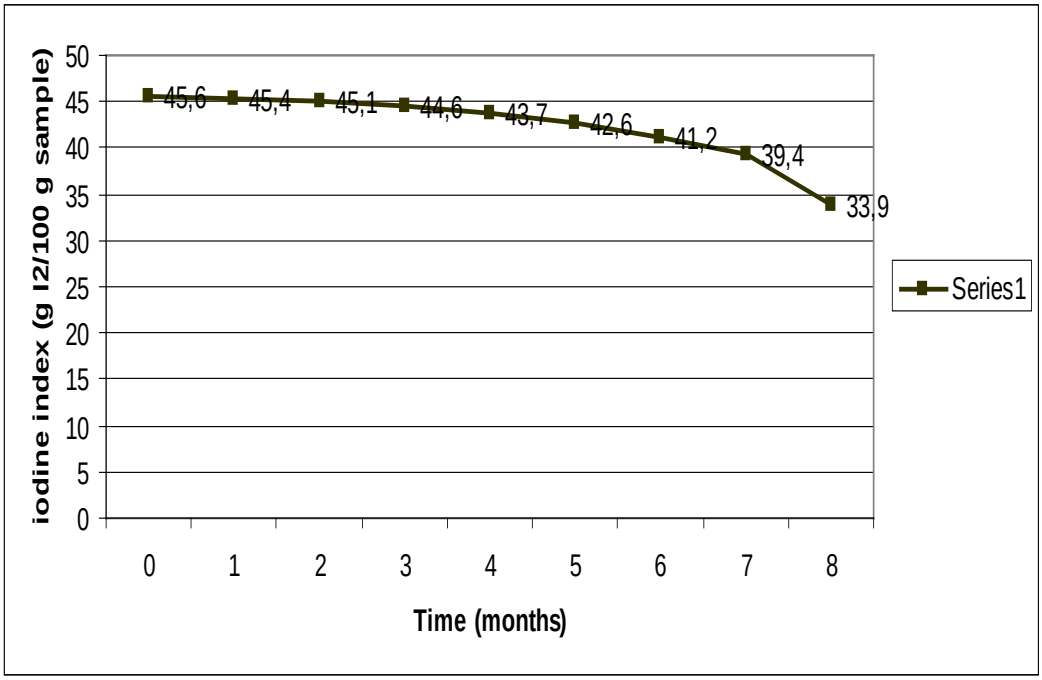

Fig. 4. Iodine index variation of frozen beef tallow

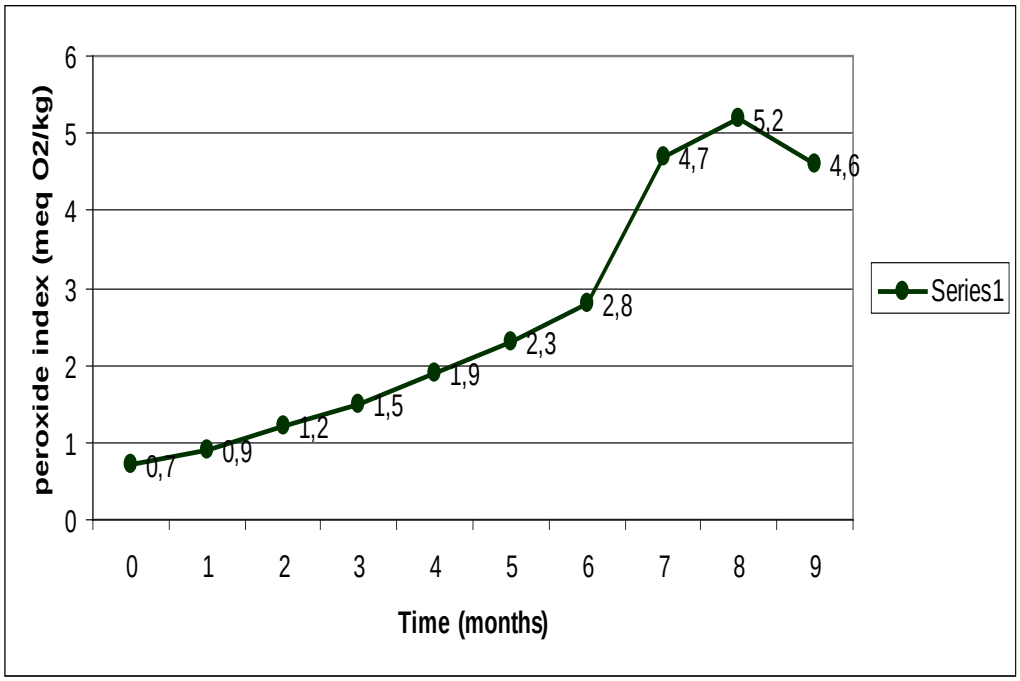

Fig. 5. Peroxide index variation of frozen beef tallow

4. Conclusions

Determination of chemical composition of animal fats is important in establishing organoleptic and physico-chemical parameters, their variation in time, being an indicator of their stability compared to alterative processes.

The timing of changes occurring in hydrolysis and oxidation processes of beef tallow has particular importance in assessing the quality and its validity.

In frozen beef tallow, altering processes took place more slowly than in that stored under refrigeration. Hydrolysis process was installed more quickly in terms of refrigeration and freezing than oxidative process, the latter being reduced by low temperature storage and the low content of unsaturated fatty acids.

Hydrolysis was translated by increasing of titrable acidity until it exceeds 1%, advanced hydrolysis was installed after 3 months and 20 days in chilled beef tallow and after 6 months in frozen beef tallow, advanced oxidation was installed after 5 months in chilled beef tallow and after 8 months in frozen beef tallow.

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COMPARISON BETWEEN SOME PHYSICAL - CHEMICAL CHARACTERISTICS OF CACAO MILK AND RAW MILK

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Abstract

The paper presents a comparison between some physical - chemical characteristics of the cacao milk and of the raw milk. For this comparison we made the following determinations for both types of milk: the determination of the dry substance using the drying oven with a 102 °C temperature, the determination of the proteic substance by titration with sodium hydroxide (NaOH) N/10, the milk pasteurization control by the starch and potassium iodide test and the pH determination using the indicator paper.

Keywords: *cacao milk, raw milk, dry substance, proteic substance, lactoperoxidase, pH*

1. Introduction

Cacao milk is produced respecting the technological instructions given by the standards. The ingredients used for this technology are: raw milk with 3,2 % fats, sugar, cream, 0,8 % cacao powder and carrageenan as a stabilizer.

The characteristics of the raw milk must correspond to the standard STAS 9535 / 2-87. Also, in this technology is not admitted the use of preserving substances, alkalinizing substances, neutralizing substances or other substances which are not naturally present in the normally composition of the milk.

2. Materials and methods

2.1 The determination of the dry substance

The dry substance content in milk was determined through the evaporation of the water existing in the milk sample, using the drying oven with a 102 °C temperature, until the sample had a constant mass.

$$\text{S.U.\%} = \frac{m_2 - m_0}{m_1 - m_0} \bullet 100$$

Where:

m_0 – the capsule's quantity, in grams (g);

m_1 – the capsule's quantity and the product's quantity before drying, in g;

m_2 – the capsule's quantity and the product's quantity after drying, in g.

2.2 *The determination of the proteic substance*

In a 100 ml Erlenmeyer glass we poured out 10 ml of milk and 10 drops of phenolphthalein. We titrated the mixture with sodium hydroxide (NaOH) N/10 until the pink – pal colour obtained persisted for 30 seconds. Then, we added 2 ml of formaldehyde solution which decoloured the mixture and titrated again with sodium hydroxide (NaOH) N/10 until the pink – pal colour.

The protein concentration in milk can be calculated with the formula:

The total quantity of protein: P = V • 1, 95, %

The total quantity of casein: C = V • 1, 51, %

Where: V = the NaOH N/10 volume used for the second titration.

2.3 The milk pasteurization control

We controlled the milk pasteurization by the starch and potassium iodide test. The lactoperoxidase is a mammary enzyme and it is destroyed at 75 °C in 30 minutes or at 80 °C in 10 minutes.

In 2 test tubes, we put 5 ml of raw milk and 5 ml of cacao milk. In both tubes we put 5 drops of starch and potassium iodide solution and 5 drops of oxygenated water. If the color of the sample in the tube becomes dark-blue this signifies that in the sample exists lactoperoxidase, which means that the milk is raw or that the pasteurization took place at temperatures below 80 °C.

2.4 The pH determination

We determined the pH values for both cacao milk and raw milk using indicator paper.

3. Results and discussion

2.1 Results:

- *Cacao milk:*

$$DS\% = \frac{135,6830 - 133,7381}{143,8991 - 133,7381} \times 100 = 19,14\ \%$$

- *Raw milk :*

$$DS\% = \frac{46,5331 - 44,6668}{54,7912 - 44,668} \times 100 = 18,43\ \%$$

2.2 Results:

- *Cacao milk*

V = 2, 6 ml NaOH.

Protein: P = V •1, 95 = 2, 6 • 1, 95 = 5, 07 %

Casein: C = V •1, 51 = 2, 6 • 1, 51 = 3, 92 %

- *Raw milk*

V = 1, 8 ml NaOH.

Protein: P = V •1,95 = 1,8 • 1,95 = 3,51 %

Casein: C = V •1, 51 = 1, 8 • 1, 51 = 2, 71 %

The results showed that the cacao milk was pasteurized at temperatures above 80 °C and that the sample in the other test tube was a sample of raw milk which was not pasteurized.

The results showed that the pH values were: 6 for the cacao milk and 7 for the raw milk.

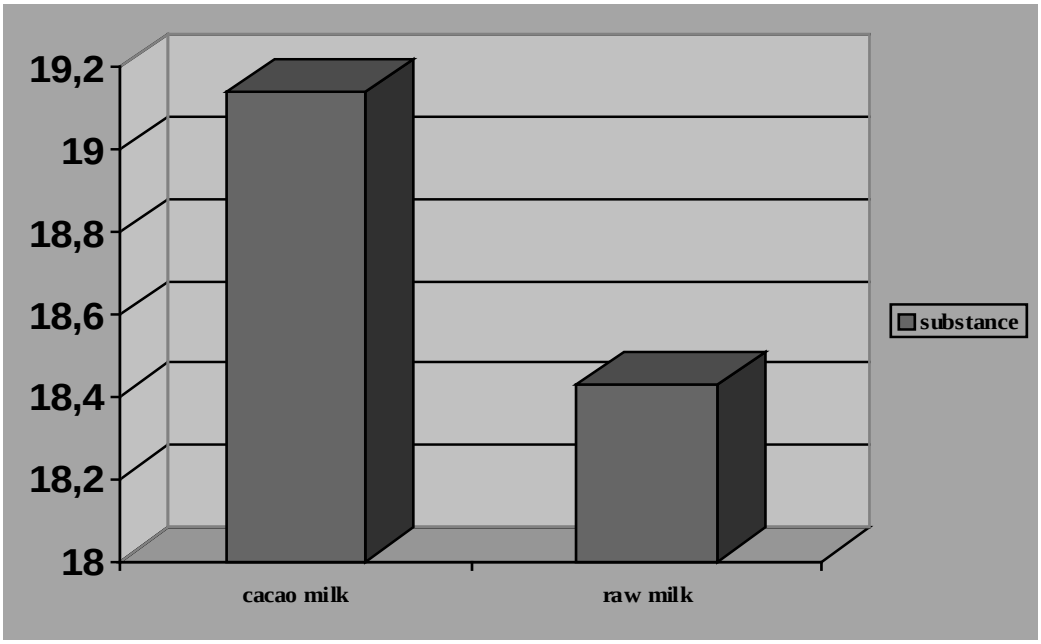

Fig. 1. Dry subtance content for the cacao milk and for the raw milk

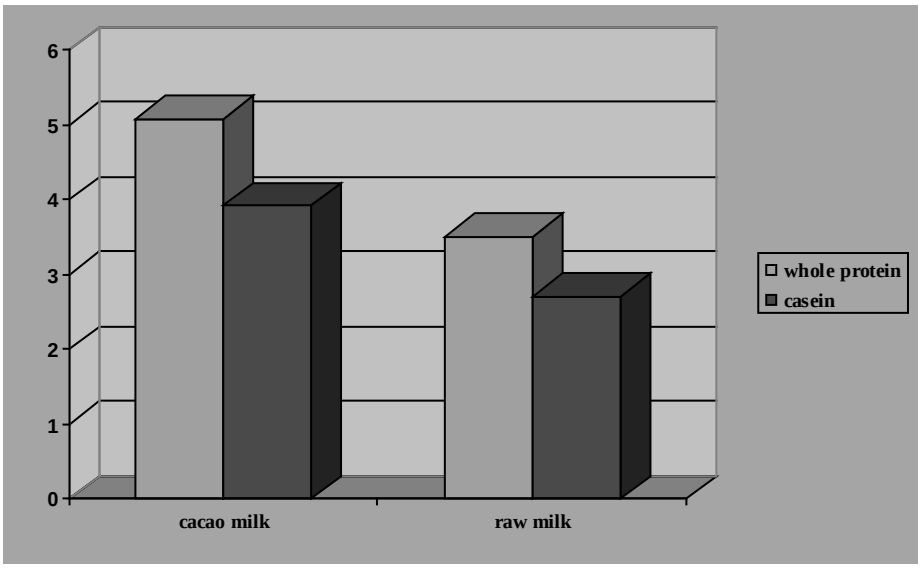

Fig. 2. Protein and casein content for thecacao milk and for the raw milk

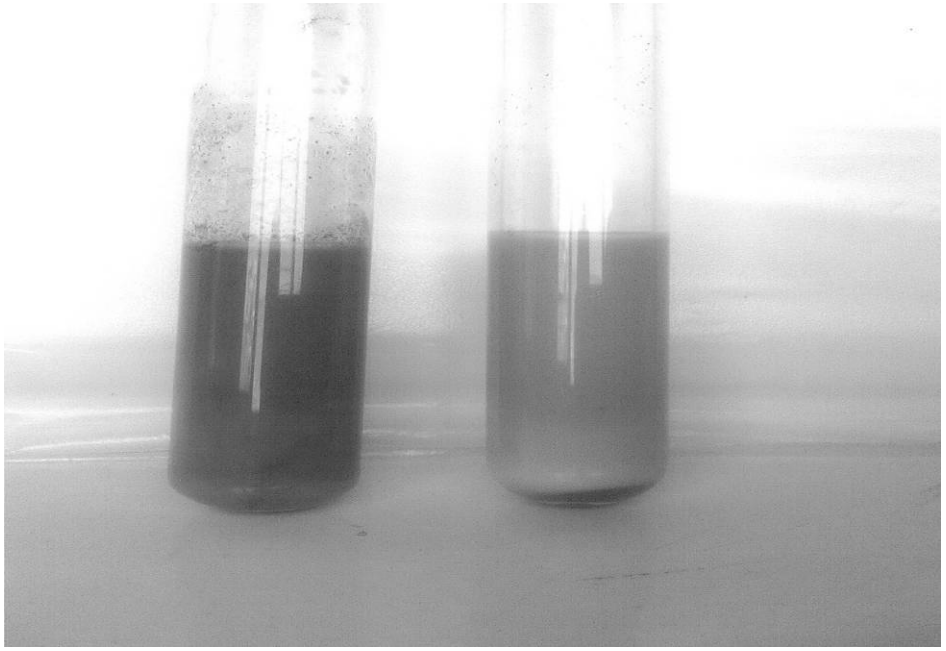

Fig. 3. Cacao milk sample (in the left) and raw milk sample (in the right)

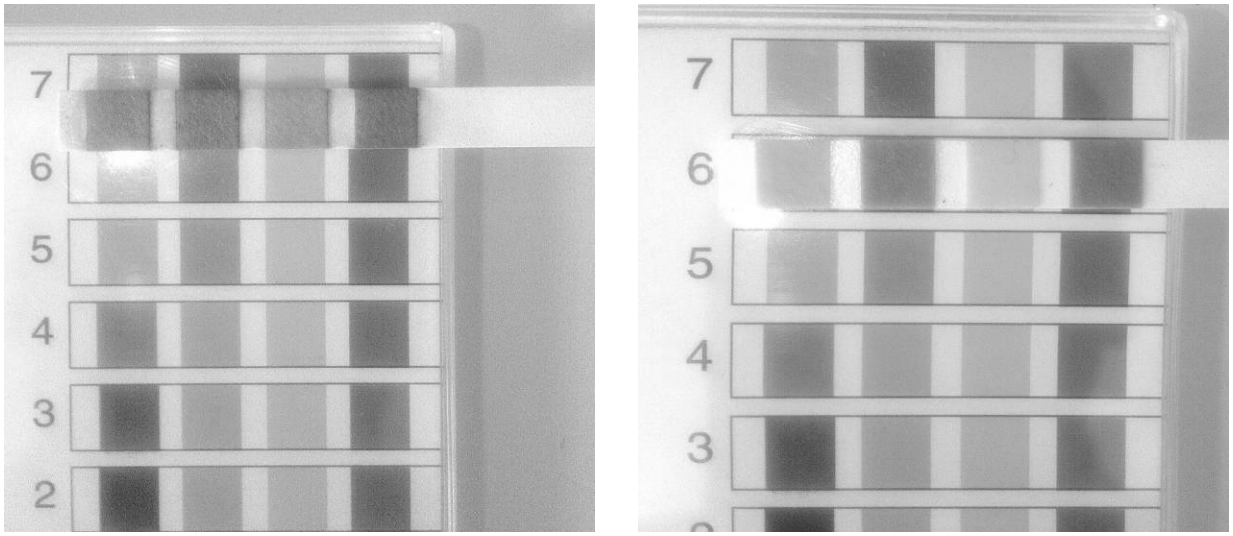

Fig. 4. pH value for the cacao milk (left) and pH value for the raw milk (right)

4. Conclusions

The dry substance of the milk is constituit of fat, proteic substance, lactose and mineral salts. By the determination of this characteristic we can estimated the quality of the milk and also can establish milk falsifications. As we can see the content of the dry substance which remains after the water evaporation from our cacao milk is 19,14 % and is higher than the content of the dry substance in the raw milk which is 18,43 %. This fact shows that the dry substance of the raw milk increases with the addition of the cacao necesary for the obtaining of the cacao milk.

The casein is an amorphous substance, therefore it has acid groups (- COOH) and also alkaline groups (-NH_2). In the method that we used the alkaline groups are blocked by formaldehyde and the acid groups are titrated with sodium hydroxide (NaOH). The determination showed that the cacao milk has a higher content of proteic substance than the raw milk with a difference of more than 1 % between the 2 types of milk.

The milk pasteurization control showed that the cacao milk was pasteurized at temperatures above 80 °C and that the sample in the other test tube was a sample of raw milk which was not pasteurized. This can be explained by the fact that the lactoperoxidase decompose the oxygenated water (H_2O_2) added in the milk samples into water (H_2O) and atomic oxigen (O_2). The oxigen, at its turn

oxidates the potassium iodide, freeing the iod, which in the presents of starch forms a blue coloure.

In the determination of the pH, the results showed that the cacao milk has an acid pH different from the raw milk pH which is neutral.

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RESEARCH ON QUALITY OF POULTRY MEAT RELATED TO GROWTH SYSTEM

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Abstract

The paper studies the influence of extensive and intensive growth systems on some physically-chemically characteristics of poultry meat. Humidity, protein and fat contents are higher in case of meat coming from broilers growth in intensive system, and the alteration rate also. High content of minerals was determined in the meat coming from broilers growth in extensive system.

Keywords: *poultry meat, growt system, quality of meat*

1. Introduction

The poultry meat has an important role in human diet due theirs nutritional qualityies. Comparing with others domestic animals, meat producing, the poultry has the advantage to supply fresh meat. Untill the end of the second war, the poultry meat were produced in individual households considered as extensive growth system. The requirements related to poultry growth's organizing as industrial activity appeared and developed once people migration from villages to towns in the same time with increasing the consumption of poultry products. In present days, the extensive poultry growing system has low proportion on meat market, it been replaced by intensive industrial system. As consequence, the consumers are splitted in two parts, each one considering that the favorite system produces the highest quality and nutritional meat.

The purpose of the paper is to compare some nutritional characteristics of poultry meat coming both from extensive and intensive growth system.

2. Materials and methods

The research were made on broiler growth up in intensive and extensive systems. *The intensive growth system* was realized in paddocks, feeding with combined feeds, in respect of nutritional balance and age ranges. Watering was at discretion. *The extensive growth* system (peasant) was made in the yard, on a larger surface, without any vegetation, housing in cages, feeding with corn grains. Watering was at discretion. After sacrificing of broilers, samples of breast and thigh were collected for analysis of:

a) The humidity analysis was done by drying of samples at 105^{o}C until the constant weight.

b) The content of total minerals were analyzed based on mineralization of meat samples to ash.

c) Kjeldahl method was used for measuring the *protein content*. The nitrogen content was multiplied by 6.25 for obtained the protein content in meat samples.

d) The fat content was determined using Soxhlet method.

e) The freshness level was assessed during 5 days, considering ammonium content.

3. Results and discussion

a) The levels of humidity in meat samples are presented in Figure 1.

As the Figure 1 indicates the humidity levels are higher in samples coming from extensive growth system as follow:

- the humidity content in thigh sample coming from extensive system is higher with 2.84% than that coming from intensive system
- the humidity content in breast sample coming from extensive system is higher with 1.14% than that coming from intensive system

As the Figure 2 presents the analyzed meat samples are framed between lean meat and average fat meat (according to STAS*****).

b) The contents of total minerals in meat samples are indicated in Figure 3.

The contents of minerals in breast and thigh of broilers growed up in intensive system are significantly increased comparing with those in samples coming from extensive system:

- the mineral content in thigh is with 13.68% higher
- the mineral content in breast is with 13.40% higher

According to minerals content the meat samples coming from intensive system are framed between lean meat and average fat

meat while the meat samples coming from extensive system are framed between average fat and fat meat (Figure 4).

c) The protein contents in meat samples are indicated in Figure 5.

According to protein content the meat samples coming from both systems are framed between lean and average fat meat (Figure 6).

As the Figure 5 indicates, the content of protein in samples coming from broilers coming from extensive system was higher comparing with that of samples coming from intensive system as follows:

- with 5,8% in thigh
- with 0,05% in breast, which is an insignificant value.

High concentration of proteins presents in meat samples coming from broilers growth up in extensive system can explain the difference of humidity of two systems due the higher quantity of sarcoplasmatic proteins, which deposits a large amount of water.

d) Fat content in meat samples coming from studied growth systems is illustrated in Figure 7.

The content of fat was significantly increased in breast meat samples coming from broilers growt in extensive system (by 2,82 times higher than in case of intensive system) while the difference between fat in thigh meat samples was insignificantly (Figure 8).

e) The freshness level

The content of ammonium during 5 days of storage is presented in Figure 9.

Analysis of Figure 9 indicates that the ammonium level is higher in the meat samples of broilers growth in extensive system, due the higher level of the proteins. Thus, the alteration rate is higher in case of extensive growth system.

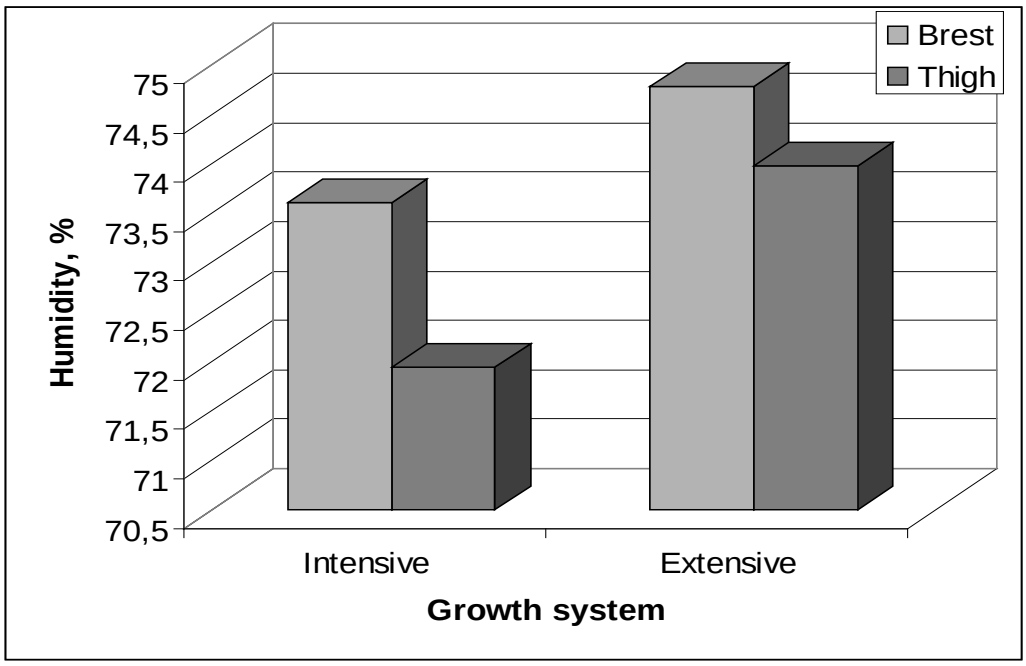

Figure 1. The comparative levels of humidity in poultry meat related to growth systems

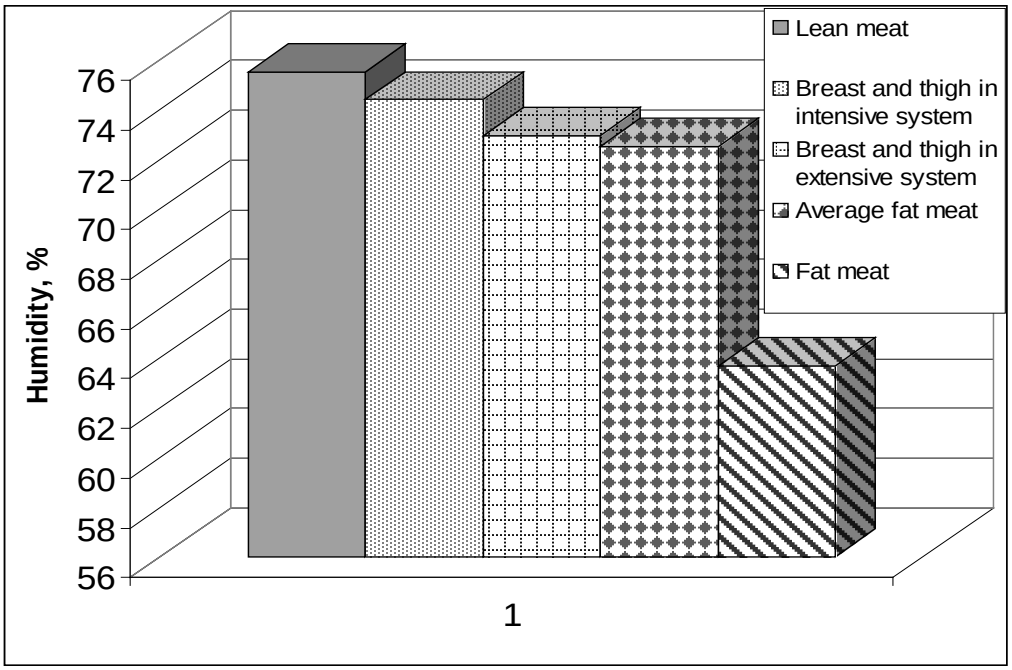

Figure 2. The frame of poultry meat samples in meat standards according to humidity level

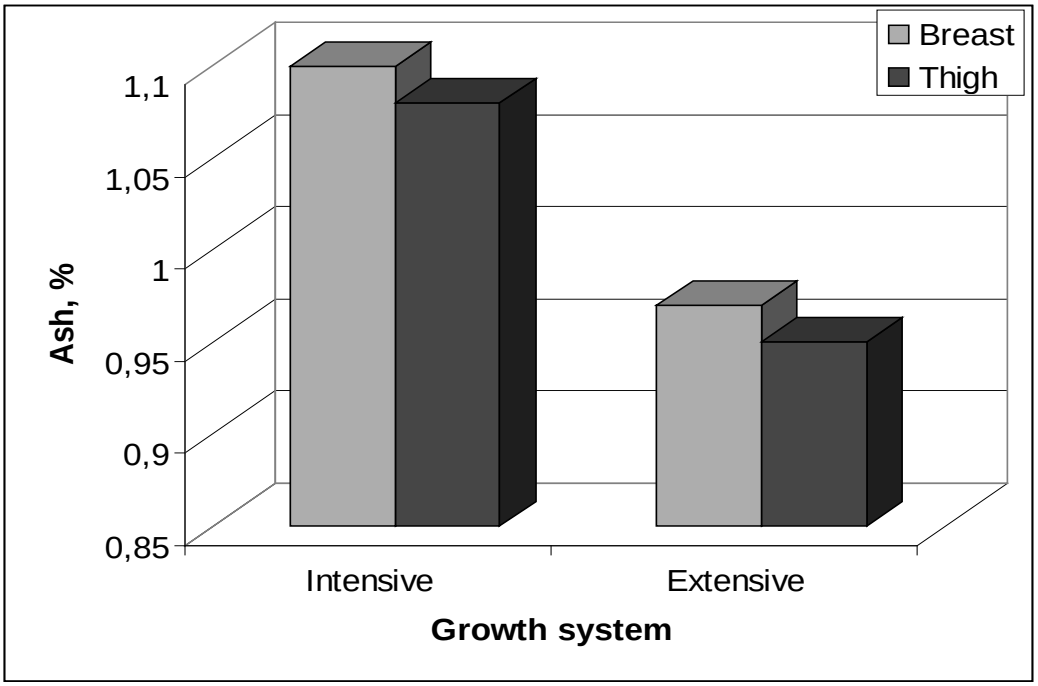

Figure 3. The levels of minerals in meat samples according to growth systems

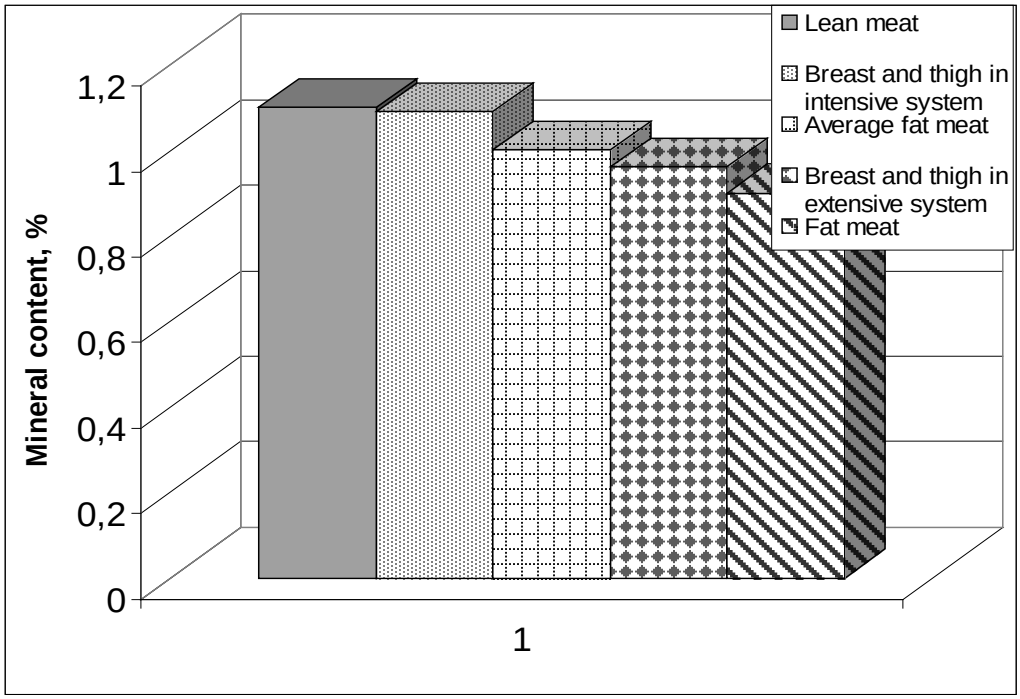

Figure 4. The frame of poultry meat samples in meat standards according to total mineral content

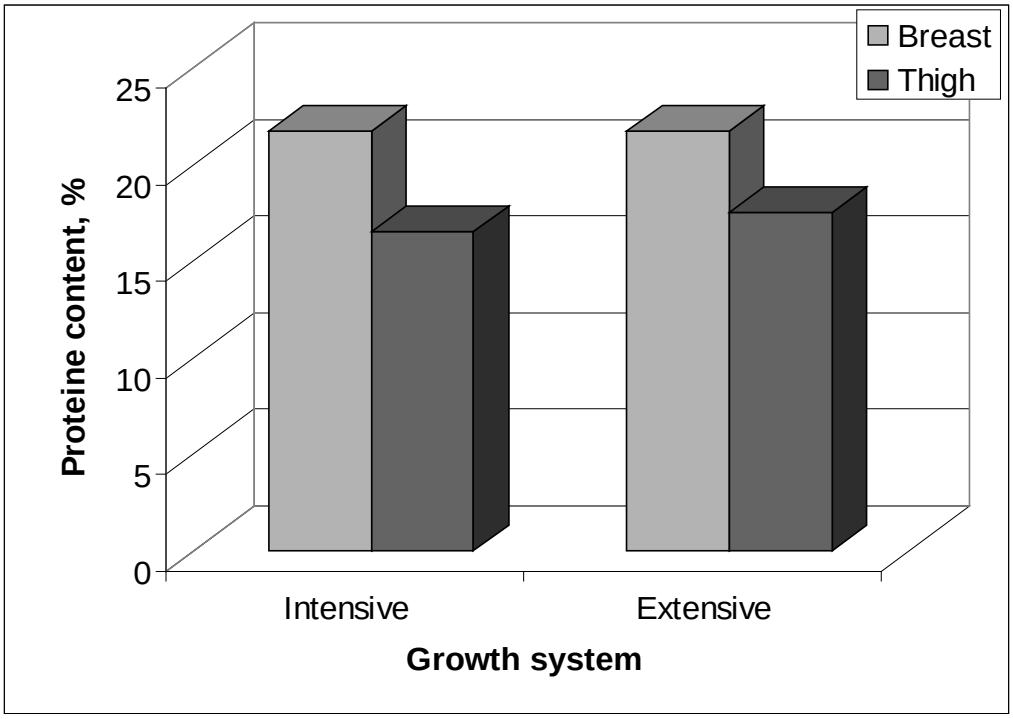

Figure 5. The levels of proteins in meat samples according to growth systems

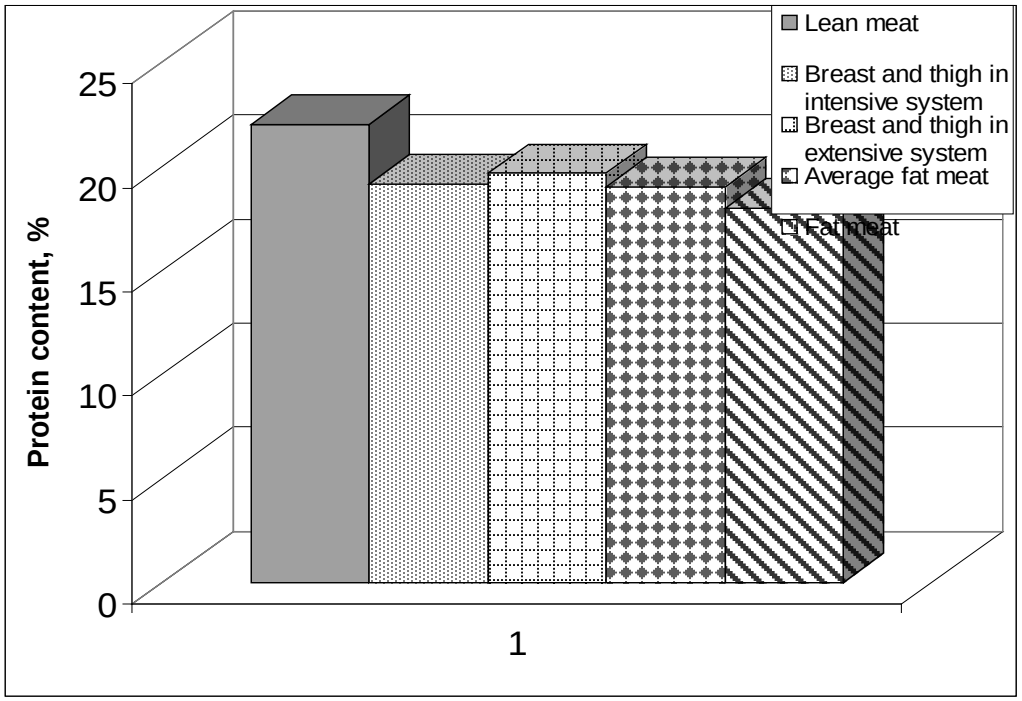

Figure 6. The frame of poultry meat samples in meat standards according to protein content

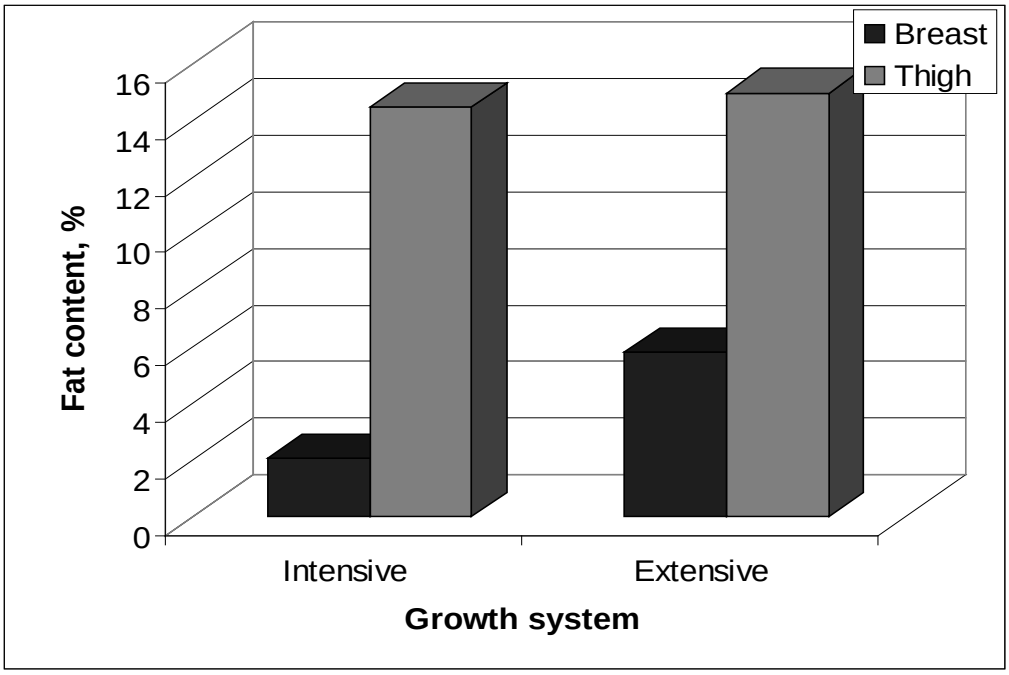

Figure 7. The levels of fat in meat samples according to growth systems

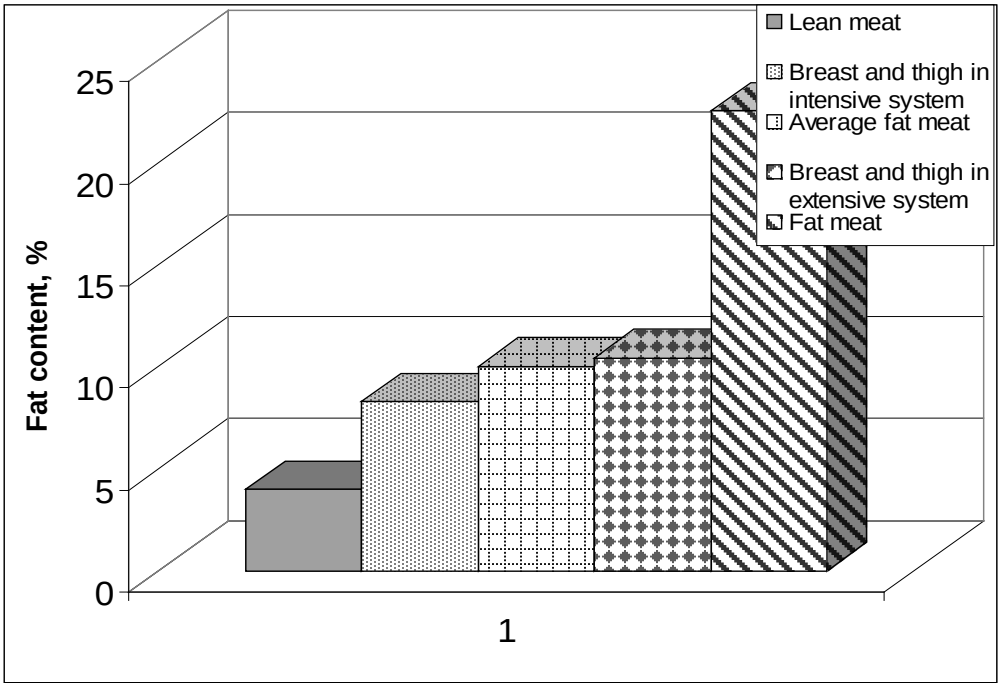

Figure 8. The frame of poultry meat samples in meat standards according to fat content

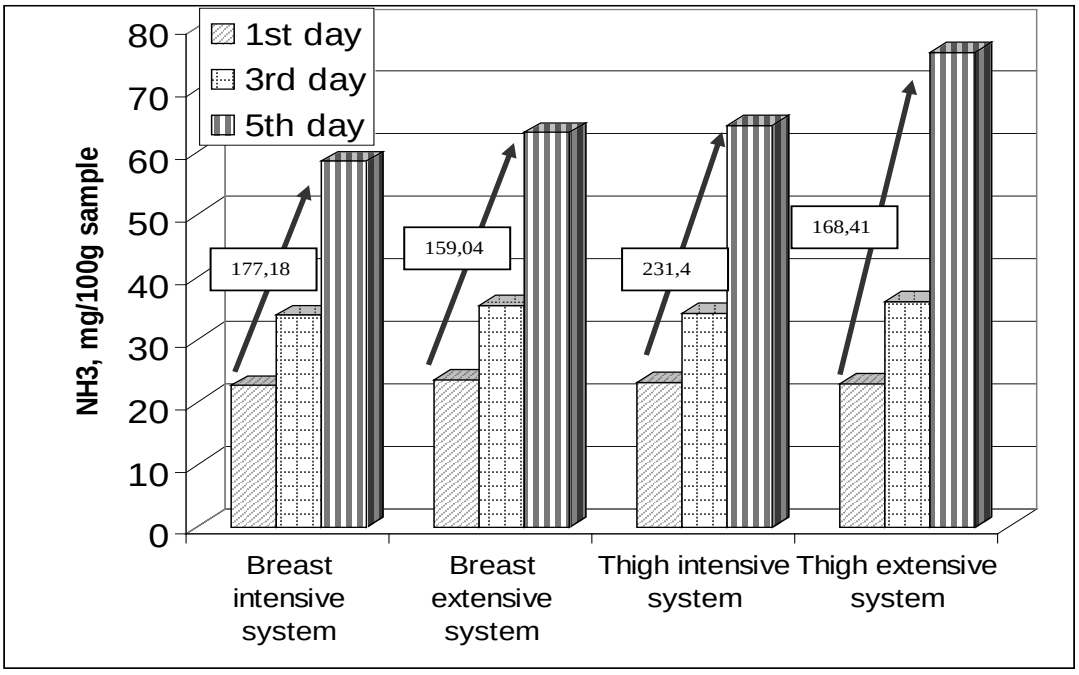

Figure 9. The dynamic of ammonium increase in the meat samples

4. Conclusions

Based on experimental data above presented, we can conclude that the meat coming from broilers growth in extensive system presents the next characteristics comparing with that coming from intensive growth system:

- higher level of humidity, proteins and lipids
- low level of minerals
- higher rate of alteration

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