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giurgiulescul@yahoo.com

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Contact: Chemistry-Biology Department, North University of Baia Mare, Victoriei Str. 76, Baia Mare,
Tel. 0040 262-276059; Fax: 0040262-275368
giurgiulescul@yahoo.com; mamihai@yahoo.com

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RESEARCH REGARDING CHEMICAL STABILIZATION OF OILS RICH IN LONG CHAIN POLYUNSATURATED FATTY ACIDS DURING STORAGE

Elena Titoc^{1*}

1- North University of Baia Mare, Chemistry Biology Department, 76 Victoriei Street, Baia Mare, Romania
*elenat@yahoo.com*

Abstract

The effects of storage temperature on oil quality of carp oil (*Cyprinus carpio*) were examined. Crude fish oils was stored at +4° C. The differences between the various oils and fats lie in the fatty acid composition of the triglycerides and phosphatides, and in the composition of the unsaponifiable fraction and other minor compounds. Fish species contain oil in different amounts, varying according to species, age, gender, location, species-origin characteristics, such as spawning and migration seasons, and also some environmental conditions, such as temperature. The focus of the study was to investigate deterioration tolerance of fish oil extracted from carp and to research the influence of antioxidants addition (C vitamin and grape extract) on the storage stability of carp oil. Chemical quality of the simple oil, oil with C vitamin and grape extract was evaluated with various parameters, including iodine, acid, peroxide, refractive index at various time intervals for 4 weeks of storage. All quality parameters, except iodine, increased during storage. It was concluded that C vitamin had an effective stabilization upon fish oil from grape extract.

Keywords: *fish oil, oil quality, antioxidants, storage*

1. Introduction

Fish oil is one of the main fishery products, with pharmaceutical importance. The fish oils can be extracted from whole body and from liver [1, 2]. Various processing methods have been adopted for the extraction of fish oil from the whole body and liver. They are Soxhlet method, Bligh and Dyer method [3], direct steaming method, solar extraction method [1, 2], Mc Gill and Moffat method [4], etc. Recent studies have clearly shown the importance of omega 3 and omega 6 fatty acids for human health and nutrition [5, 6, 7]. Fortunately, fish oil is one of the best sources of dietary supply of these fatty acids. Oils and fats used for human consumption are natural components of vegetables and land or marine origin animals [8]. Regardless of sources, principal components of these oils and fats are the same.

They are triglyceride oils in that their principal constituents, amounting, usually to over 90% of their total composition, are the tri-esters of fatty acids

and glycerol. The remainder of their composition consists of partial glycerides, such as mono and di-glycerides, free fatty acids (FFA) and other compounds, in small quantities. The differences between the various oils and fats lie in the fatty acid composition of the triglycerides and phosphatides, and in the composition of the unsaponifiable fraction and other minor compounds [9]. Fish species contain oil in different amounts, varying according to species, age, gender, location, species-origin characteristics, such as spawning and migration seasons, and also some environmental conditions, such as temperature [10, 11].

Many studies have shown that fish oil has important roles in prevention of cardiovascular diseases and some types of cancer, including colon, breast and prostate [7, 12, 13]. In addition, fish oil also helps to prevent brain aging and Alzheimer's disease [14]. Many health experts suggest that two to three servings per week of seafood should be consumed in order to meet the recommended level of essential

fatty acids for pregnant women, children and elderly people [15, 16]. Seafood is the best source of dietary supply of omega-3 fatty acids, including eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA). It has been suggested that consuming EPA and DHA may reduce the risk of mortality from cardiovascular disease in people who have already experienced a cardiac event [7]. Von Schacky and Harris [17] proposed the omega-3 index (percentage of EPA + DHA of total fatty acids in red blood cells) as a risk factor for sudden cardiac death and concluded that this index should be higher than 8 %.

The effect of LC *n*-3 PUFA when treating asthma has been reviewed by Reisman, Schachter, Dales, Tran, Kourad, Barnes, Sampson, Morrison, Gaboury and Blackman [18]. Zulfakar, Edwards and Heard [19] have reviewed the works that have examined the potential use of *n*-3 PUFA in psoriasis. Several recent reviews have studied the influence of omega-3 PUFA on bowel diseases [20, 21, 22]. All this scientific activity regarding long chain omega-3 PUFA usually ends up by advising an intake of this type of PUFA, either through the natural food products that contain them or as a supplement in the diet for beneficial effects on health [23].

The main purpose of this study was to investigate the stability of fish oil during storage. Fish oil has important industrial applications in food, pharmacy, cosmetics and paint products.

Due of these wide areas of applications, quality and stability of fish oil have gained more importance. Fish oil spoils in two major ways, like oils from animal and vegetable sources: oxidative spoilage and hydrolytic spoilage [24]. Due to its high content of polyunsaturated fatty acids, including EPA and DHA, fish oil is highly susceptible to oxidative spoilage [11], and the rate of fish oil oxidation is significantly different from that of other oils. Fish oils also include high concentrations of phospholipids, containing unsaturated fatty acids, which make them even more sensitive than other

oils. The break in the induction curve is less sharp, and the beginning of the increase in the peroxide value occurs sooner [25, 26]. The course of oxidation is often quite different between extracted fish oils and lipids in fish tissues [27-31]. The autoxidation of fish oils is the most important cause of quality deterioration [32-35]. Undesirable flavours and odours develop at very low peroxide values at an early stage of oxidation, even during the induction period [36, 37].

For evaluation of oil stability and monitoring of deterioration during storage, commonly methods include iodine value (IV), peroxide value (PV), acid value (AV), refractive index were used. There are some important limits posed by regulatory agencies for quality and acceptability of oils for human consumption: for example, 8 meq O₂/kg of oil is the limit of acceptability for PV [33]. The focus of the study was to investigate deterioration tolerance of fish oil extracted from carp (*Cyprinus carpio*) and to research the influence of antioxidants addition (C vitamin and grape extract) on the storage stability of carp oil.

2. Materials and methods

2.1. Material

The material used in the research consisted of the following types of oil:

- carp oil obtained by fat melting collected from farmed carp,
- carp oil with C vitamin (1g/100 ml),
- carp oil with grape extract (1g/100 ml).

2.2. Chemical analysis

2.2.1. Acid value (AV)

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 phenolphthalein, as an indicator. Acidity was expressed as oleic acid grams to 100 grams sample [37].

2.2.2. Peroxide value (PV)

Peroxide value was determined using UV-VIS T60U spectrophotometer (Bibby Scientific, London, UK): operating temperature 5 – 45°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 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 [36].

2.2.3. Iodine value (IV)

Iodine value was determined using Hanus method. 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 $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ using starch (1.0 g/100 mL) as an indicator. IV was calculated as g $\text{I}_2/100$ g sample [37].

2.2.4. Refractive index

The values of refractive index are conditioned by the nature and proportion of fatty acids, unsaturated fatty acids increase the value, and saturated fatty acids decrease it. To determine the refractive index we used the PAL-RI (Tokyo, Japan) with the following technical characteristics: field: 1.3306-1.5284; resolution: 0.0001; accuracy: ± 0.0003 ; measuring temperature: 5-45°C (resolution 1°C); measuring time: 3 s; in accordance with the requirements of EMC Directive 93/68/EEC.

3. Results and discussion

During refrigerated storage, acidity presented an upward trend for the three types of fish oil, hydrolysis was carried out in fast-paced, but the acidity did not exceed the maximum permissible value of 1% (g oleic acid) in the fourth week of refrigeration, oil with added vitamin C recording lower values (Figure 1).

Iodine value shows a downward trend over the three types of oil storage, due to unsaturated degree reduction by fatty acids oxidation, a less pronounced decrease was observed for oil with C vitamin (Figure 2).

For simple carp oil, peroxide value shows a relatively slow growth until the second week, which corresponds to the initiation stage of oxidation in which a small amount of peroxides are formed, followed by a surge in the third week, corresponding to the propagation phase, in which the largest amount of peroxides are formed.

In the fourth week the peroxide value decreased due to the formation of secondary oxidation compounds. In the case of oil with C vitamin and grape extract, peroxide index had an upward trend, in these cases the end stage did not installed in the fourth week, so there are no secondary oxidation compounds, we can say that the antioxidants (C vitamin and grape extract) can be used to the chemical stabilization of fish oil, C vitamin showing stronger antioxidant effect. Refractive index presented an irregular trend during the four weeks of refrigerated storage, due to fatty acids variations because of oxidation and hydrolysis processes installation (Figures 3 and 4). Results are expressed as the mean of three determinations.

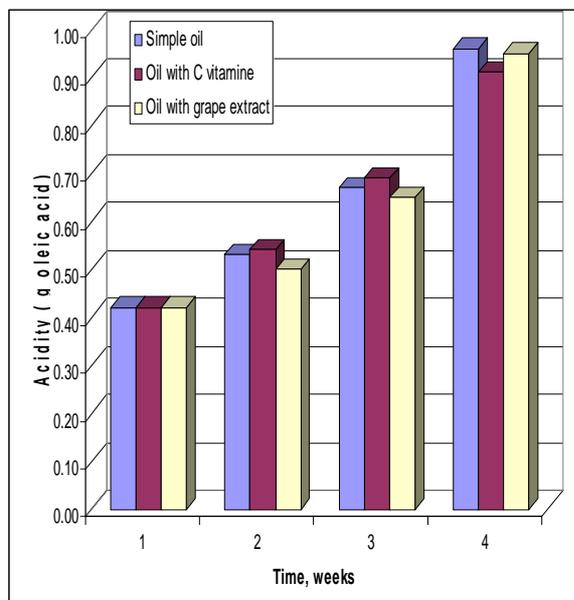


Figure 1. Acidity variation of oil samples

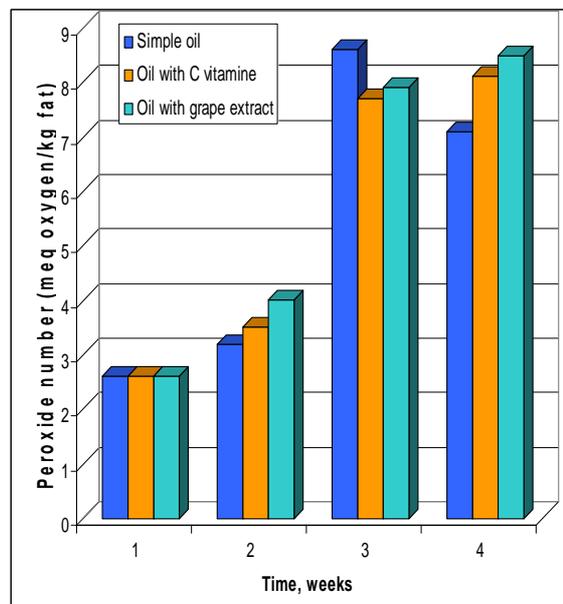


Figure 3. Peroxide index variation for oil samples

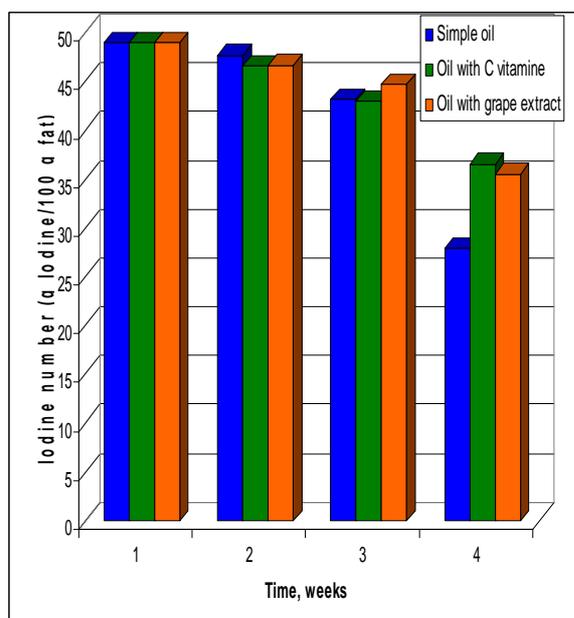


Figure 2. Iodine index variation for oil samples

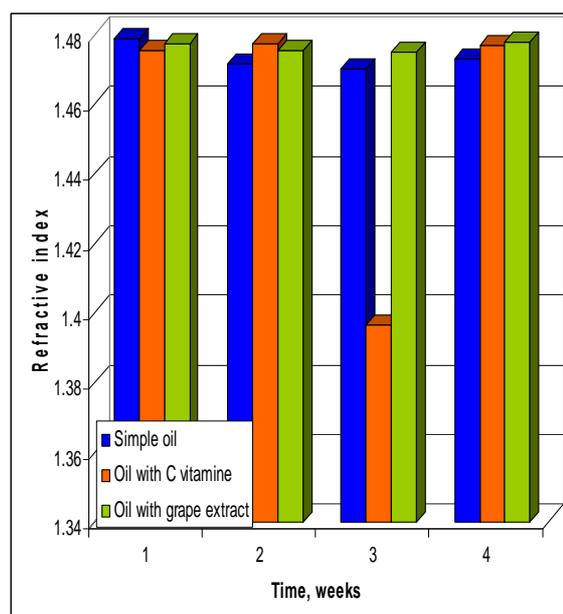


Figure 4. Refractive index variation of oil samples

For simple carp oil, peroxide value shows a relatively slow growth until the second week, which corresponds to the initiation stage of oxidation in which a small amount of peroxides are formed, followed by a surge in the third week, corresponding to the propagation phase, in which the largest amount of peroxides are formed, and in the fourth week the peroxide value decreased due to the formation of secondary oxidation compounds. In the case of oil with C vitamin and grape extract, peroxide index had an upward trend, in these cases the end stage did not installed in the fourth week, so there are no secondary oxidation compounds, we can say that the antioxidants (C vitamin and grape extract) can be used to the chemical stabilization of fish oil, C vitamin showing stronger antioxidant effect. Refractive index presented an irregular trend during the four weeks of refrigerated storage, due to fatty acids variations because of oxidation and hydrolysis processes installation (Figures 3 and 4). Results are expressed as the mean of three determinations.

4. Conclusions

Iodine value shows a downward trend over the three types of oil storage, due to unsaturated degree reduction by fatty acids oxidation, a less pronounced decrease was observed for oil with C vitamin.

It was found that addition of C vitamin and grape extract in fish oil delay the oxidative process, an effective stabilization was observed for oil with C vitamin, and storage temperature had important effect on storage stability of fish oil.

We can say that the antioxidants (C vitamin and grape extract) can be used to the chemical stabilization of fish oil, C vitamin showing stronger antioxidant effect.

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RESEARCH ON THE CONTAMINATION OF VEGETAL PRODUCTS IN MARAMURES AREA

Laura Bretan^{2,3,4*}, **Otto Ketney**^{1,2,3}, **Florin Boltea**^{2,3,5}

1 - "Lucian Blaga" University of Sibiu, Faculty of Agricultural Sciences, Food Industry and Environment Protection, Department of Food Biotechnology – 550024-Sibiu, Dr. Ion Ratiu Street, no. 7-9, Romania

2* - North University of Baia Mare, Faculty of Sciences, Chemistry Biology Department 430122- Baia Mare, Victoriei street, no. 76, Romania, *laura_sya@yahoo.com*

3 - Sanitary veterinary and food safety direction, Vasile Alecsandri street no. 66, Baia Mare, Maramures

4 - University of Agricultural Sciences and Veterinary Medicine Cluj-Napoca, Calea Mănăştur Street, no. 3-5, Cluj-Napoca

5 - University of Agricultural Sciences and Veterinary Medicine of Banat Timișoara, Food Technology Faculty, 119 A, Aradului street, 300645, Timișoara

Abstract

Tackling the ecological thinking and studies proposed by some researchers suggesting the fact that the living systems are in a dynamic equilibrium guide us to the assessment of a polluted environment as an imposed accent. Thus, anthropological activities are responsible for the destroying of the environment equilibrium, through qualitative and quantitative alterations of the animal and vegetable species, which allows them to be the vectors of toxic molecules or to accumulate them in target organs and products. Heavy metals get into the food chain through soil, plants, animals; this is why the thorough knowledge of these aspects is very important. The transfer of these inorganic compounds from the soil to the plants and the food chain of animals and humans has become a research subject since the beginning of the modern agricultural chemistry and the investigations regarding the human and animal health and nutrition.

Keywords: *heavy metals, lead, cadmium, salad, spinach, apples, pears*

1. Introduction

Lead, mercury, cadmium are, together with arsenic, the most common and spread harmful metals in the environment [1, 2]. They are also among the metallic contaminants with the most encountered problems in the industry of food processing. Each of them is responsible for high scale incidents of contamination and, despite the steps that were taken by the governmental authorities; they continue to be possible threats to the safety of consumers, in certain circumstances. They are included especially in the official regulations regarding the food safety in many countries and among them are responsible for much official documentation, more than other food contaminants.

For many consumers, these three metals are elements that come for the first

time in mind when contamination with heavy metals is mentioned [3, 4].

According to some authors [5], there are six major categories of sources of environment contamination with heavy metals: natural sources, products used in agriculture, pollutants resulted from mining activities, industrial emissions, thermal power station emissions and vehicle emission [6, 7, 8]. Because of the connection between soil and the metals in our bodies, through the food products, we have to give a certain attention to the nature of the soil and to the way in which this contributes to the metallic content of the food products.

The soil is not only the main source of all the metals that get accumulated in a natural way in the plants and animals. In a world where the spreading in the environment of the elements could harm as a result of the human activity is a growing

problem, it is important that those that are preoccupied with the food safety to consider the metals in the soil as an integrated part of their processes of planning and management [9]. The absorption of heavy metals from the soil in plants has a major role in the contamination of plants with heavy metals [10].

In this paper we mean to do a comparative study regarding the contamination of salad, spinach, apples and pears with Pb and Cd in two different places: Baia Mare and Sighetu Marmatiei. The first place is well known for its polluting potential due to the industries in the area. As an area considered a witness we have chosen Sighetu Marmatiei, situated at 60 km from the major sources of pollution and which is separated from them by the Gutai Mountains. There have been analyzed 20 samples of salad, 20 samples of spinach, 20 samples of apples and 20 samples of pears from the polluted area of Baia Mare. From the unpolluted area (witness), there have been analyzed 20 samples of salad, 20 samples of spinach, 20 samples of apples and 20 samples of pears.

2. Materials and methods

The mineralization takes place in order to eliminate the organic substances that could interfere through the absorption of wavelength specific to metals. For the mineralization of the samples in order to determine the metals we have used the wet mineralization. For this determination, it was used a spectrophotometer of atomic absorption type SpectrAA 220 VARIAN.

3. Results and discussions

As a result of the determination of the lead and cadmium concentrations in vegetal products: salad, spinach, apples and pears, the following values pointed in the following table and graphics have been

obtained (Table 1, Figure 1 and Figure 2). The major polluters of vegetation are especially Pb, Cd, Cu, Zn, Fe, Mn. Pollution is mainly realized through the polluted soil, but also through the polluted atmosphere, the level of pollution in this way varies between 10.37 – 23.37%. Lead is a ubiquitous element, being an element of the earth shell and the soil, from where it is taken by plants and animals.

4. Conclusions

The major polluters of vegetation are especially Pb, Cd, Cu, Zn, Fe, Mn. Pollution is mainly realized through the polluted soil, but also through the polluted atmosphere, the level of pollution in this way varies between 10.37 – 23.37%. Lead is a ubiquitous element, being an element of the earth shell and the soil, from where it is taken by plants and animals. Its provenience in the ambient environment is directly proportional to the pollution provoked by the use of the metal in industry, vehicles' exhaust, but also by its presence in almost every objects of current use: pipes, canes, cosmetics, paints, etc.

The lead evacuated in the atmosphere by different sources can get directly into the organism with the inspired air, or indirectly, after the depositing on the surfaces or soil, then water, food, etc., as well as after the intake from the soil by plants, some species absorbing more than others, especially in certain parts. Cadmium is a very toxic metal, without any biogenic role, and its compounds don't have any pharmaceutical use, being one of the elements known as cancerous. This represents a source of pollution for the environment, together with lead and zinc, those coming from foundry and mining. Cadmium gets into the environment especially through the industrial emissions, contaminating the air, water, soil and plants.

Table 1
Concentration of heavy metals

Sample no.	Area	The concentration of heavy metals mg/kg $\bar{x} \pm \text{RSD}^*$	
		Pb	Cd
1	Baia Mare	0.69 ± 0.5	0.15 ± 0.1
2		0.78 ± 0.6	0.14 ± 0.3
3		0.8 ± 0.5	0.15 ± 0.2
4		1.3 ± 0.2	0.03 ± 0.2
5		0.95 ± 0.4	0.02 ± 0.5
6	Sighetu Marmatiei	0.05 ± 0.2	0.07 ± 0.1
7		0.15 ± 0.1	0.09 ± 0.1
8		0.2 ± 0.3	0.08 ± 0.3
9		0.13 ± 0.4	0.02 ± 0.4
10		0.08 ± 0.2	0.07 ± 0.3
11	CMA	0.57 ± 0.3	0.14 ± 0.1
12		0.62 ± 0.1	0.16 ± 0.3
13		0.75 ± 0.3	0.12 ± 0.2
14		1.12 ± 0.4	0.13 ± 0.2
15		0.68 ± 0.7	0.17 ± 0.5

* Relative standard deviation for three determinations

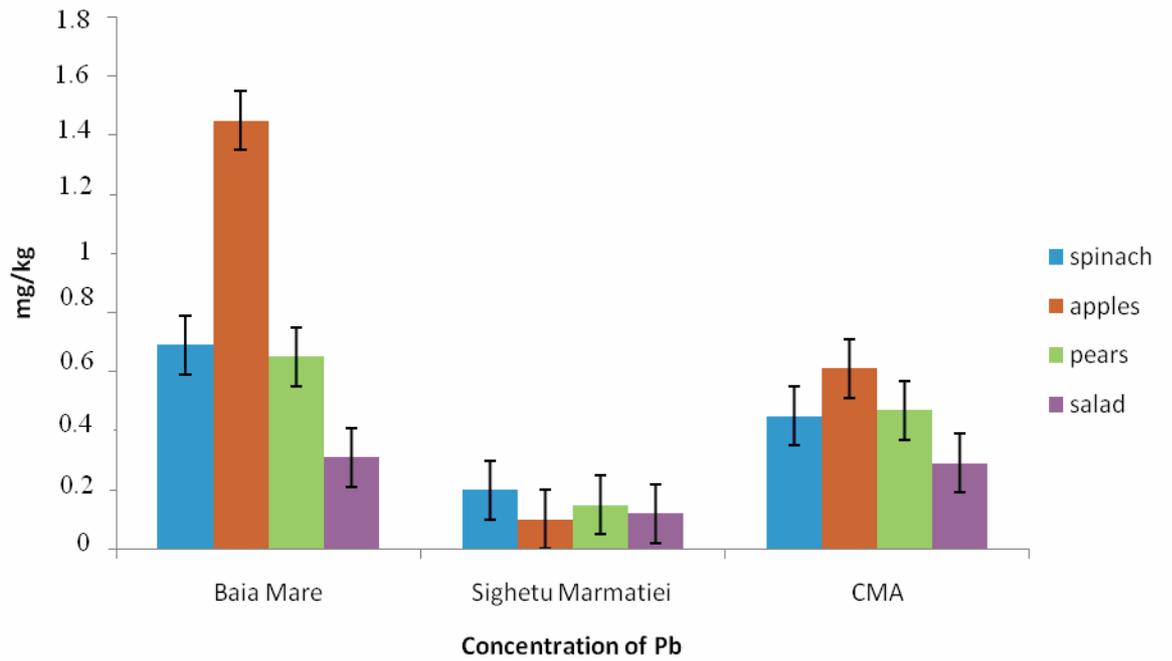


Figure 1. Variation of lead concentration in different areas of Maramures

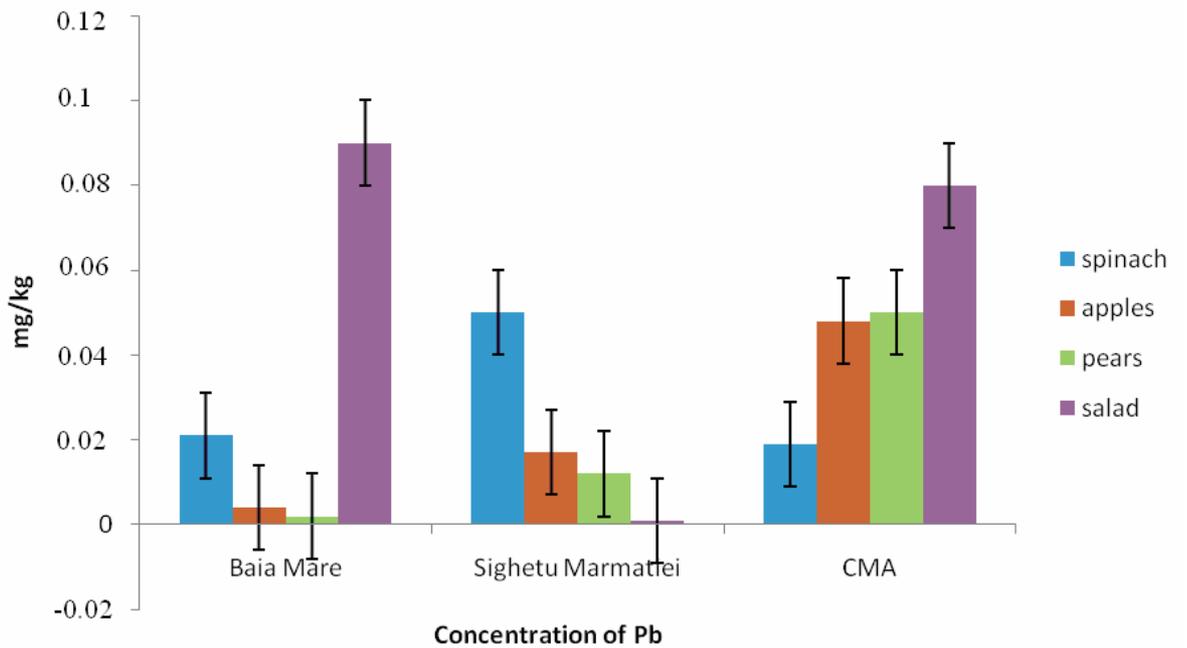


Figure 2. Variation of cadmium concentration in different areas of Maramures

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ASPECTS OF FUNGAL MICROFLORA ON MEAT AND MEAT PRODUCTS

Zorica Voşgan*¹, Daniela Mureşan¹, Corina Dragos², Marian Jelea¹

¹North University of Baia Mare, Str. Victoriei, 76, Romania, 430122

* zori_v13@yahoo.com

²Sanitary Veterinary and food safety direction, Baia Mare, Maramures

ABSTRACT

Consumption foods of animal origin, which occupies an important place in a rational human nutrition, is not without danger. In some cases, molds can cause respiratory problems and allergic reactions, particularly aggressive and dangerous. Moulds developed on meat and meat products can be isolated by cultivation on selective nutrient media. The microbiological analysis of eight samples were identified following types of molds: *Penicillium sp.*; *Rhizopus sp.*; *Cladosporium sp.*; *Mucor sp.*, *Aspergillus sp.*. From Sibiu salami was isolated pure strain of *Penicillium nalgiovense*. In this way, could see the risks to which we expose ourselves when not kept proper safety measures.

Keywords: secondary metabolites, *Aspergillus sp.*, molds, macroscopic and microscopic aspects.

1. Introduction

Progress in recent decades in the technology of processing, preservation and handling do not exclude the possibility of alteration or contamination with pathogenic microorganisms in meat. Because meat composition and its origin, is often involved in the spread of disease or the onset of toxic food infections [1, 2].

The conditions of the environment in the manufacturing rooms, stores, refrigerators and shops are very suitable for the development of moulds inside the products, but more frequently on the surface of various sorts of meat and meat products [3]. Among the most common genera remember: *Penicillium sp.*, *Aspergillus sp.*, *Cladosporium sp.*, *Rhizopus sp.*, *Mucor sp.*, *Sporotrichum sp.*, *Thamnidium sp. etc.*

Fungus infections amended sensory qualities of the products: appearance, color, smell, taste. The reason for an increasing interest is the ability of moulds to produce secondary metabolites

– mycotoxins – that have unfavourable effects, such as carcinogenesis, mutagenicity, and high thermostability [4]. When the temperature and relative humidity are optimal after contamination,

there is also a risk of mycotoxin production [5]. According to Ostrý [6], the production of mycotoxins in meat and meat products is enabled by the following factors:

- the presence of oxygen
- temperature in range 4 ± 40 °C
- pH-value between 2.5 and 8 (with an optimum between 5 and 8),
- minimum water activity of 0.80
- maximum salt concentration of 14%.

Number of fungi in the flesh is quite small but can grow or not, depending on the pre-processing, when several factors can intervene.

In meat products the water activity is low, they are subjected to drying and curing process of long duration (type salami Sibiu) and seeded with noble spores of mold.

Fungal species of the genus *Penicillium* are a frequently used as commercial starter cultures for mold ripened foods. *Penicillium notatum*, *Penicillium nalgiovense* and *Penicillium chrysogenum* have been isolated from dry-cured salami. These molds help in flavor development by decomposing excess lactic acid and

inhibiting the growth of other undesirable molds.

On the other hand, it is ascertained that in nature about 70-80 per cent of *Penicillium* species are capable of producing mycotoxins, many of which are effectively detected in the cultural media of the isolates belonging to many variants species that colonize salami, as well as on the salami [7÷ 10].

2. Materials and methods

8 samples of meat and meat products (traditional sausage, pate, kaiser, salami ham, thawed meat, Transylvanian ham) were selected for isolation of pure strains of fungi, plus Sibiu salami seeded with a starter culture *Penicillium nalgiovense*.

To isolate pure moulds strains were used the following nutritional media: water-peptone- glucose and Sabouroud medium.

Fungal spores collected from meat and meat products were inoculated in liquid medium for 24 - 48 hours, after which made the inoculation in dishes on solid medium Sabouroud. Petri dishes seeded were incubated for 72 - 120 hours at room temperature, until the appearance of fungal colony. The procedure was repeated to obtain pure strains of colonies and to take pictures at macroscopic and microscopic level, *in vivo* between the slide and the lamella, for identify the type of mould on the product.

To take the pictures at macroscopic and microscopic level we used the following equipments: MLB 2100 A. Kruss Optronic biological microscope with 3 field glasses, Nikon Coolpix P5000 digital camera, Fujifilm FinePix S 9600 digital camera.

3. Results and discussion

Of the eight samples analyzed were isolated and identified the following types of: *Penicillium sp.*; *Rhizopus sp.*; *Mucor sp.*; *Cladosporium sp.*; *Aspergillus sp* (Table 1).

Contaminated samples, fungal colonies developed on solid medium, macroscopic aspects of fungi isolated in pure strains and microscopic aspects are presented in the Figures 1 and 2.

Table 1. Genus of molds developed on meat and meat products

Product origin	No.	Products	Genus
Household products	1	Traditional sausage	<i>Penicillium.sp.</i>
	2	Thawed meat	<i>Penicillium.sp.</i>
Marketed products	3	Pate pork	<i>Penicillium.sp.</i>
	4	Kaiser	<i>Penicillium.sp.</i>
			<i>Rhizopus.sp.</i>
	5	Salami	<i>Aspergillus.sp.</i>
			<i>Penicillium.sp.</i>
	6	Ham	<i>Mucor.sp.</i>
7	Transylvanian ham	<i>Cladosporium.sp.</i>	
8	Sibiu salami	<i>Penicillium nalgiovense</i>	

On the products home made, with no food additives, have identified only species of the genus *Penicillium sp.*

The products produced industrially and sold in shops presented the following types: *Penicillium sp.* the four samples (pate, kaiser, salami, Sibiu salami), *Rhizopus sp.* the kaiser, *Aspergillus sp.* the salami, *Mucor sp.* the ham, *Cladosporium sp.* the Transylvanian ham.

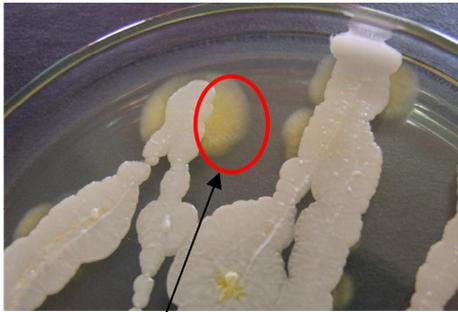
The presence of the genus *Aspergillus sp.* aflatoxicogenic is the most worrying.

Cvetni and Pepelnjak [11] reported a 20% average occurrence of *Aspergillus flavus* and *Aspergillus parasiticus* in smoked meat products, pork salami and sausage, bacon and ham.

Household products	<p>Sausage home</p> 	<p>Thawed meat</p> 
Petri dish of fungal colonies	<p><i>Penicillium sp.</i></p> 	<p><i>Penicillium sp.</i></p> 
Microscopic aspects in vivo	<p><i>Penicillium sp.</i></p> 	<p><i>Penicillium sp.</i></p> 

Figure 1. Macroscopic and microscopic aspects of fungi developed on the home made meat and meat products

<p>Pate pork</p>		
<p>Petri dish of fungal colonies</p>	<p><i>Penicillium sp</i></p> 	<p><i>Penicillium sp .</i></p> 
<p>Microscopic aspects <i>in vivo</i></p>	<p><i>Penicillium sp.</i></p> 	<p><i>Penicillium sp.</i></p> 
<p>Kaiser</p>		

<p>Petri dish of fungal colonies</p>	<p><i>Penicillium sp.</i></p> 	<p><i>Rhizopus sp.</i></p> 
<p>Microscopic aspects in vivo</p>	<p><i>Penicillium sp.</i></p> 	<p><i>Rhizopus sp.</i></p> 
<p>Salami</p>		
<p>Petri dish of fungal colonies</p>	<p><i>Aspergillus sp.</i></p> 	<p><i>Penicillium sp.</i></p> 
<p>Microscopic aspects in vivo</p>	<p><i>Aspergillus sp.</i></p> 	<p><i>Penicillium sp.</i></p> 

<p>Ham</p> <hr/> <p>Transylvanian ham</p>		
<p>Petri dish of fungal colonies</p>	<p><i>Mucor sp.</i></p> 	<p><i>Cladosporium sp.</i></p> 
<p>Microscopic aspects in vivo</p>	<p><i>Mucor sp.</i></p> 	<p><i>Cladosporium sp.</i></p> 

Figure 2. Macroscopic and microscopic aspects of fungi developed on the studied meat and meat products

4. Conclusions

The results from this study indicate the presence of the genus *Penicillium sp.* with the highest incidence of meat and meat products, obtained in their own households as well as those of trade.

The products marketed were isolated genera: *Rizopus sp.*, *Cladosporium sp.*, *Aspergillus sp.*, *Mucor sp.*

Flavourings, especially spices, added to meat can also considerably contribute to the total mould contamination of final products. An essential role is the storage condition, because the factors that lead to the emergence of molds are: high temperature, high humidity, reduced air circulation and changes in physico-chemical properties.

By respecting of minimum safety conditions to avoid spore germination and vegetative forms which can produce toxic metabolites dangerous to consumer health.

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EFFECT OF DIFFERENT PARTICLE SIZES OF JAGGERY POWDER ON STORABILITY

Adagale P. V.¹, Unde P. A.¹, Syed Imran Hashmi^{1*}, Abdul Raheem¹

1- Department of Food Trade and Business Management, College of Food Technology, M.A.U., Parbhani (MS) India,

** imran.foodresearch@gmail.com*

Abstract

The investigations were carried out to study the effect of different particle size and packaging materials on storability of jaggery powder on the basis of changes in chemical composition and organoleptic characteristics. Jaggery powder of three different grades viz., coarse (0.500 -0.708 mm), medium (0.351 - 0.420 mm) and fine (0.211 - 0.296 mm) were prepared and packed in 100 gauge polyethylene bag. The samples were stored at room temperature for the period of 6 months. In order to optimize the particle size of jaggery powder, the changes in chemical composition and organoleptic properties were evaluated during storage. The results revealed that change in chemical composition was lower in case of coarse jaggery powder. The coarse jaggery powder having particle size in the range 0.500 – 0.078 mm) was found more acceptable among all other powder sizes after storage period of six months in terms of its chemical properties and organoleptic characteristics.

Key words: *Jaggery, powder, storability, particle size*

1. Introduction

Jaggery is one of the most important sweeteners in India. Jaggery (also known as *gur*) is a traditional unrefined non-centrifugal sugar consumed in Asia, Africa, Latin America, and the Caribbean [1]. It is a concentrated product of cane juice without separation of the molasses and crystals, and can vary from golden brown to dark brown in color. It contains up to 50% sucrose, up to 20% invert sugars with some other insoluble matter such as ash, proteins and bagasse fibers [2]. It is directly consumed by human and used in animal feed mixtures. Jaggery is a natural sweetener made by the concentration of sugarcane juice prepared without the use of any chemicals [3]. Jaggery has great nutritive and medicinal value. Jaggery purifies the blood, prevents the rheumatic afflictions and disorders of bile and process properties of higher order [4, 5]. Jaggery contains proteins, vitamins and minerals, which are essential constituents for the body. It is also a potent source of iron and copper [6]. Jaggery is an energy food that is said to purify blood,

regulate liver function and keep the body healthy. It has also been prescribed in various diseases like jaundice, breathlessness and kidney problems [7]. Jaggery industry is the dominant decentralized cottage industry of India, which meets about 40 to 50% sweetener requirement of Indian population. About one third to one half jaggery produced needs to be stored every year [8]. Jaggery is usually available in the market in the form of one or half kg block. However, these blocks are not feasible for transportation and retailing. The loss of jaggery during storage ranged from 7 to 25% depending upon storage conditions [9]. Hence, in present investigation, efforts were made to convert jaggery into different particle sizes and further prepared jaggery powder is analyzed for its physicochemical and organoleptic characteristics during storage for the period of 6 months.

2. Materials and methods

The present research work entitled “effect of different particle size of jaggery powder on storability” was carried out in Department of Agricultural Process Engineering, M.P.K.V, Rahuri during the year 2005-06.

2.1. Experimental analysis

The Jaggery (var. CO92005) was procured from Regional sugarcane and Jaggery Research Station, Kolhapur. It was free from impurities, light brown in color, grainy structure and uniform size. The pieces of solid jaggery were shed dried under controlled temperature of 27°C till external surface becomes slightly rough and losses its smoothness. Size reduction was done by hammer (the beater of hammers mill that rotates in closed steel chamber at 3000 rpm and forces the ground through a screen of 18 meshes). The jaggery powder obtained was analyzed for its properties and quality. Three sizes of powder jaggery (i.e. course, medium and fine) were obtained on the basis of sieve analysis of jaggery powder. The powder jaggery properties viz., sieve analysis [10], fineness modules [11] and uniformity index [12] were determined. Average particle size was calculated using equation [12].

$$\text{Average particle size} = 0.135 \times (1.336)^{\text{FM}}$$

2.2. Packaging and storage of jaggery powder

Jaggery powder (course, medium and fine) were further packed in 100 gauge polyethylene bags. These samples were further stored at room temperature (27°C) in M.P.K.V., Rahuri.

2.3. Physicochemical properties

The physicochemical properties viz. reducing sugar, non reducing sugar, pH, colour and moisture content of jaggery powder were determined by using standard methods [13]. Colour of jaggery solution was measured on Klet summerson photoelectric colourimeter using green filter.

2.4. Organoleptic Evaluation

Organoleptic evaluation was carried out every month during storage. A panel of 10 judges using Nine-point hedonic scale judges the colour, taste, texture, flavor and overall acceptability of samples [14].

3. Results and discussion

3.1. Chemical composition of Jaggery powder

The chemical composition of jaggery powder (var. Co 92005) was determined in Table 1. The moisture content, pH and colour found were 1.2 per cent, 6.6 and 0.18 per cent, respectively. The reducing sugar and non-reducing sugar obtained were 8.51 per cent and 78.56 per cent, respectively.

3.2. Grading of jaggery powder

The sieve analysis of jaggery powder for fineness modulus, size (average particle diameter) and uniformity index was done and data is tabulated in Table 2.

Based on sieve analysis the jaggery powder was graded into three grades viz., coarse, medium and fine. The particle sizes of 0.500 – 0.708, 0.351 – 0.420 and 0.211 – 0.296 mm were considered coarse, medium and fine, respectively. The fineness modulus, average particle diameter and uniformity index found were 4.14, 0.45, and 5:2:3, respectively. Similar studies were done for rice products [10].

3.2. Effect of particle size on chemical properties of jaggery powder

The data of effect of particle size of jaggery powder during storage on moisture content, pH, reducing sugar, non reducing sugar and colour of jaggery powder is depicted in Table 3. The results revealed that moisture content of jaggery powder significantly increased during the storage period. This may be due to the high water vapour transmission rate of 100 gauge polyethylene bags due to which there was

increase in moisture content of jaggery powder. The moisture content of jaggery powder ranged from 1.20 to 1.74 per cent. Maximum moisture was observed in case of fine jaggery powder stored for 6 months while minimum water absorption after 6 months of storage was observed in coarse particle sized jaggery powder. On the basis of moisture, it could be concluded that particle size is inversely related to moisture absorption in jaggery powder. The pH of samples varied from 6.60 to 6.48. pH of sample found to be reversibly related to storage period. pH decreased with increase in storage period and increased in particle size. The decrease in pH during storage may be due to change in chemical properties which are affected by storage conditions.

The reducing sugar content of jaggery powder found to increase with increase in storage period and particle size. While, non reducing sugar decreased with increase in storage period and particle size. The colour of sample found to be maximum in case of coarse particle size which increased with increase in storage period.

3.3. Effect of particle size and storage on organoleptic properties of jaggery powder

The data on sensory attributes viz., colour, taste, texture, flavor and overall acceptability of jaggery powder is plotted and shown in Figure 1. The results revealed that there were no changes on taste with different particle sizes. However, colour and textural properties of jaggery powder changed drastically with increase in size of particles.

The overall acceptability was ranged from 7.6 to 8.4 for all the particle sizes (Figure 1). The overall acceptability was found to be maximum in case of coarse powder (8.4), followed by medium (8.1) and minimum overall acceptability was seen in case of fine powder (7.6). On the basis of data generated on organoleptic evaluation, it could be predicted that coarse powder was found more acceptable compared to medium and

fine powder with respect to organoleptic characteristics.

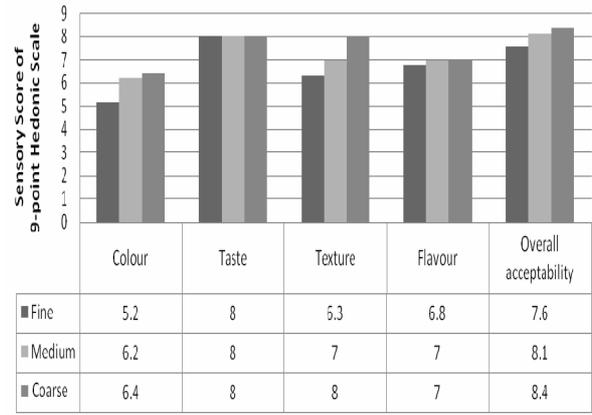


Figure 1. Organoleptic evaluation of Jaggery Powder

4. Conclusions

Jaggery powder contained 1.20% moisture, 6.60 pH, 8.51% reducing sugar and 78.56% non reducing sugar. The colour of prepared jaggery powder found to be 0.18. The particle size significantly affected on the chemical properties of jaggery. With increase in particle size, moisture, reducing sugar and colour of jaggery increased while pH and non reducing sugar content was found to decrease.

Organoleptic evaluation of samples after 6 months of storage reported best quality of coarse particle size followed by medium and fine jaggery powder. In all, it could be concluded that jaggery powder with coarse particle size (0.500 – 0.708 mm) found to desirable amongst all the samples in terms of its physico-chemical properties and organoleptic evaluation.

Table 1.
Chemical composition of jaggery powder

Parameter	Value
Moisture content, %	1.20
pH	6.60
Reducing sugar, %	8.51
Non-reducing sugar, %	78.56
Colour	0.18

Table 2.
Sieve analysis of jaggery powder

Grading	Size (mm)	Fineness Modulus	Average Particle Size (mm)	Uniformity Index
Coarse	0.500 – 0.708	4.14	0.45	5:2:3
Medium	0.351 – 0.420			
Fine	0.211 – 0.296			

Table 3.
Effect of particle size and storage on physic-chemical properties of jaggery powder

Sr. No.	Particular	Particle size	Storage period (methods)						
			0	1	2	3	4	5	6
1	Moisture	Fine	1.20	1.23	1.28	1.35	1.51	1.65	1.74
		Medium	1.20	1.20	1.26	1.32	1.47	1.60	1.68
		Coarse	1.20	1.20	1.24	1.29	1.43	1.55	1.62
2	pH	Fine	6.60	6.60	6.59	6.59	6.58	6.54	6.50
		Medium	6.60	6.60	6.58	6.58	6.57	6.53	6.49
		Coarse	6.60	6.60	6.57	6.57	6.56	6.52	6.48
3	Reducing sugar	Fine	8.51	8.51	8.51	8.52	8.53	8.54	8.56
		Medium	8.51	8.51	8.51	8.52	8.53	8.55	8.57
		Coarse	8.51	8.51	8.51	8.53	8.54	8.56	8.58
4	Non reducing sugar	Fine	78.56	78.56	78.56	78.55	78.54	78.53	78.53
		Medium	78.56	78.56	78.56	78.55	78.53	78.52	78.52
		Coarse	78.56	78.56	78.55	78.54	78.52	78.51	78.50
5	Colour	Fine	0.18	0.19	0.21	0.22	0.24	0.26	0.27
		Medium	0.18	0.20	0.21	0.23	0.25	0.27	0.28
		Coarse	0.18	0.21	0.22	0.24	0.26	0.28	0.29

* Each value is average of 5 determinations

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FRESHNESS DETERMINATION OF POULTRY MEAT

Florin Boltea^{2,3,5*}, Laura Bretan^{2,3,4}, Otto Ketney^{1,2,3}

1 - "Lucian Blaga" University of Sibiu, Faculty of Agricultural Sciences, Food Industry and Environment Protection, Department of Food Biotechnology – 550024-Sibiu, Dr. Ion Ratiu Street, no. 7-9, Romania

2* - North University of Baia Mare, Faculty of Sciences, Chemistry Biology Department 430122- Baia Mare, Victoriei street, no. 76, Romania, laura_sya@yahoo.com

3 - Sanitary veterinary and food safety direction, Vasile Alecsandri street no. 66, Baia Mare, Maramures

4 - University of Agricultural Sciences and Veterinary Medicine Cluj-Napoca, Calea Mănăştur Street, no. 3-5, Cluj-Napoca

5* - University of Agricultural Sciences and Veterinary Medicine of Banat Timișoara, Food Technology Faculty, 119 A, Aradului street, 300645, Timișoara, boltea_florin@yahoo.com

Abstract

In this study was assessed the quality of poultry meat during the refrigeration by content analysis easily hydrolyzable nitrogen with colorimetric method using Nessler reagent, measuring the variation in color intensity of ammonia formed in poultry meat with Nessler reagent, study results were compared with the legal limit allowed by Romanian legislation. In meat and meat products during storage at refrigerated temperatures can develop a highly toxic substances such as ammonia, hydrogen sulphide, peroxidase, putrescine, and cadaverine formed by decarboxylation of amino acids in meat as a consequence of the process autolytic decomposition process in meat. The determination of ammonia (easily hydrolysable nitrogen) in poultry meat is critical for daily quality control of production and for specification in contracts.

Keywords: *easily hydrolysable nitrogen, refrigerated meat, ammonia*

1. Introduction

The meat composition includes a host of chemical compounds in muscles, they include free fatty acids, glycerol, triglycerides, phospholipids, and non-protein nitrogenous components such as DNA, RNA, ammonia, amine groups, and vitamins, also there are glycogen granules and ATP.

In Europe, the average loss in quantity of fresh poultry meat is estimated to be 1-3%, although some processing plants report up to 6% trimming loss in breast meat alone. At 1% loss, it can be estimated that 54.600 tonnes of prime broiler meat was lost during the year 1996. In December 1996, the producer price (pound sterling (£) equivalent) was 56 kg⁻¹

in the Netherlands, the lowest cost producer in Europe, and at this producer price, the actual loss for the year 1996 can be estimated at over £30 million. If the retail price of fresh turkey meat is considered to be £3 kg⁻¹ and the minimal loss due to trimming happens to be 1%, then it is estimated that the European turkey industry had lost about £50 million worth of prime meat in the year 1996. Although the trimmings and downgraded portions or carcasses would have been sold at a reduced price, this certainly shows the importance of carcass and meat quality.

To address the concerns of poultry processors and retailers, scientists around the world are relentlessly pursuing research and development [1, 2].

In meat and meat products during storage at refrigerated temperatures can develop a highly toxic substances such as ammonia, hydrogen sulphide, peroxidase, putrescine, and cadaverine formed by decarboxylation of amino acids in meat as a consequence of the process autolytic decomposition process in meat .

Qualities measurements of chemical or physical-chemical properties, which are directly relevant to food quality, are found less frequently for process control in the meat industry [3].

To assess the quality and freshness of meat during storage was determined ammonia (easily hydrolysable nitrogen) with Nessler reagent. Method consists in measure the quantity of nitrogen present in the form of dissolved ammonia and ammonium ions. The test involves dosing in the reaction medium with Nessler's reagent (solution of potassium tetraiodomercurate) photolorimetric method [4-6]. The products were purchased from the slaughter house immediately after slaughter and stored for 10 days at 4 °C.

2. Materials and methods

2.1. Sample preparation

Meat for analysis was minced twice after that was weighed 10 grams (10.000 mg) of the analytical balance and placed in a 100 volumetric flask and brought to volume with bidistilate water closed and with a lid to shake and leave to rest for 10 minutes.

2.2. Reagents

Bidistilate water without ammonia

Nessler reagent (tetraiodomercurate bipotasic solution in potassium hydroxide): 5 g potassium iodide dissolved in 5 cm³ of hot water in an Erlenmeyer flask. Add hot saturated solution of mercuric chloride until the precipitate formed is no longer dissolved. After cooling the solution

separate, decant a 100 cm³ volumetric flask. Add 15 g potassium hydroxide dissolved in 30 cm³ water and bring to volume with water. Add 0.5 cm³ saturated solution of mercuric chloride, allow to make the solution above the precipitate and separated by decantation, pass in a clean and kept in the dark.

Alkaline mixture: 10 g sodium carbonate and 10 g sodium hydroxide dissolved in few ml of bidistilate water in a 100 ml volumetric flask and completed to volume with bidistilate water.

Standard stocks solution of ammonia is obtained by weighing the analytical balance 1000 mg ammonium chloride dissolved in a 100 volumetric flask, bring to volume with bidistilate water after a dilution is made by taking 1 ml of this solution and introducing it into a 1000 ml volumetric flask and bringing to volume with bidistilate water so that the solution finally have a concentration of 0.01 mg / l ammonium chloride.

Seignette salt (potassium sodium tartrate): 392 g Seignette salt NaKC4H4O6·4H2O dissolve in 784 ml 20% NaOH solution. Mix well and after two hours may be used without shaking the bottle.

2.3. Description of working procedures

Ammonia in aqueous extract of meat sample form with solution of potassium tetraiodomercurate (II) (K₂[HgI₄]) (Nessler reagent) complex colored, yellow-orange oximercury ammonium iodide, colour intensity was read photolorimeter to the wavelengths of 425 nm.

100 ml aqueous meat extract were placed in a cylinder with a stopper and add 1 ml alkaline mixture and shaken. It was the supernatant 10 ml, was added 2 ml Seignette salt and 2 ml Nessler reagent was shaken and left to stand 10 minutes later which was centrifuged at 3500 rpm for 10 min with a rotating centrifuge ROTANTA model 460 then read colour intensity in

WTW photocolorimeter SpectroFlex model 6100 to 425 nm in 1 mm cuvette. Extinction values of the sample were interpolated from a standard curve which was performed after the scheme in Table 1.

3. Results and discussions

During the 10 days in which meat was kept at 4 °C was taken each day 10 g sample which was analyzed for ammonia content by reaction of ammonia with Nessler reagent.

The intensity of color formed following reaction similarly to the standard solution of ammonia reading. Extinction values of the sample were interpolated on the calibration curve.

In the Table 1 are presented the results content increases of the ammonia in meat during the 10 days. From Table 3 we can see that the meat from of the 9th day the value exceeds the limit from 35 mg, g NH₃/100 permitted by Romanian legislation, limit stipulated in article 12 of Order 975/1998 which provides that in the pork meat the easily hydrolysable nitrogen over 35 mg NH₃/100 g limit is unfit for human consumption through clear signs of deterioration (Order 975/1998). Variation of hydrolysable nitrogen (mg NH₃ /100g meat) during storage to de 4°C can be seen in Figure 2.

4. Conclusions

The determination of ammonia (easily hydrolysable nitrogen) in poultry meat is critical for daily quality control of production and for specification in contracts.

The traditional ammonia methods (easily hydrolysable nitrogen) see. SR 9065-7:2007 is relatively accurate, but it is time-consuming; exposes the analyst to toxic fumes, concentrated acid, and alkali; and produces chemical wastes that must be disposed compared with photocolorimetric method of this paper which is somewhat quicker than the traditional method SR 9065-7:2007.

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Table 1
Standard solution realization for calibration

Concentration of ammonia solution	mg/L	0	0.02	0.04	0.06	0.08	0.1
Standard solution	mL	0	2	4	6	8	0
Bidistilate water	mL	0	8	6	4	2	1
Seignette salt	mL				2		
Nessler reagent	mL				2		

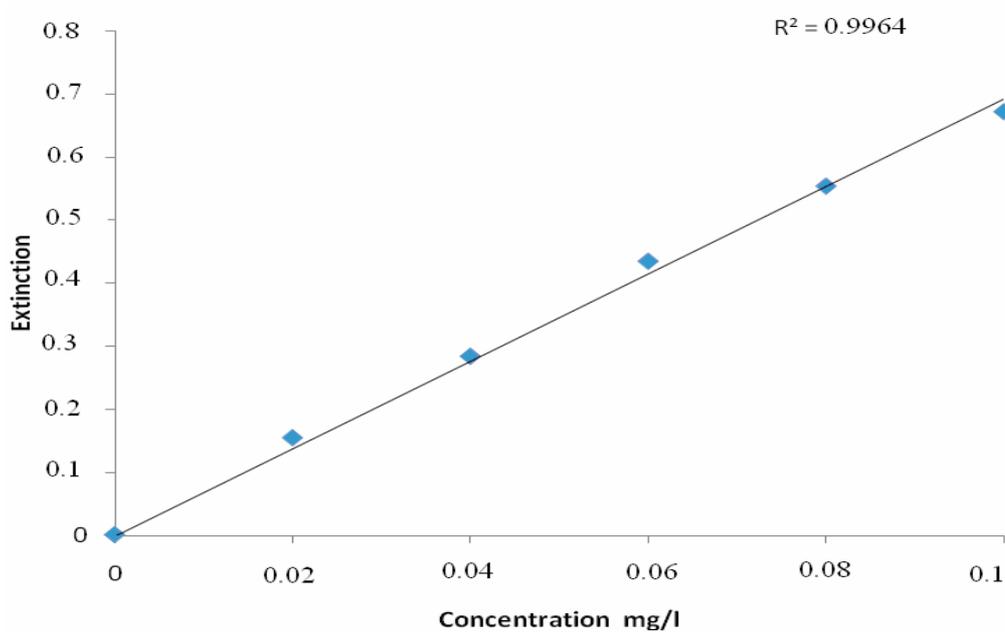


Figure 1. Calibration curve

Table 2
Results of standard solution readings at photocolorimeter

Standard solution concentrations (mg)	Abs.1*	Abs.2*	Media	RSD**%
0.00	0.002	0.002	0.002	0.000
0.02	0.156	0.155	0.155	0.454
0.04	0.287	0.282	0.284	1.242
0.06	0.436	0.434	0.435	0.325
0.08	0.553	0.555	0.554	0.255
0.10	0.671	0.673	0.672	0.210

* Absorbance; ** Relative standard deviation

Table 3
Results of experimental data

Day	Sample masses g	Abs.1*	Abs.2*	Media	RDS** %	Concentration mg NH ₃ / 100 g
1	10.018	0.105	0.104	0.105	0.676	23.484
2	10.022	0.141	0.142	0.142	0.499	24.736
3	10.025	0.172	0.174	0.173	0.817	26.509
4	10.002	0.185	0.186	0.186	0.381	26.942
5	10.008	0.201	0.202	0.202	0.350	29.229
6	10.003	0.215	0.216	0.216	0.328	31.399
7	10.013	0.224	0.227	0.226	0.940	35.824
8	10.016	0.243	0.247	0.245	1.154	37.224
9	10.011	0.268	0.262	0.265	1.600	37.242
10	10.010	0.275	0.273	0.274	0.516	38.212

* Absorbance; ** Relative standard deviation

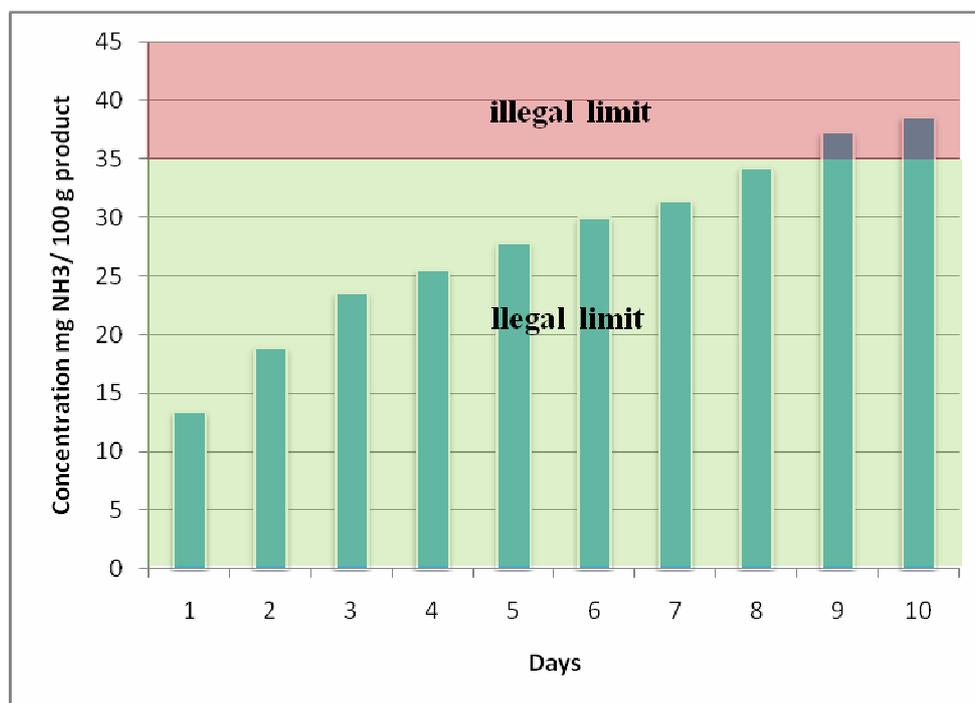


Figure 2. Variation of hydrolysable nitrogen (mg NH₃/100g meat) during storage at 4 °C

STUDIES ON EXTRACTION OF GINGER OIL AND ITS MICROENCAPSULATION

Kadam M.L.¹, Syed Imran Hashmi^{2*}, Kale R.V.³

1- Department of Oil Technology, Laxminarayan Institute of Technology (LIT), R.T.M.U., Nagpur

2*- Department of Food Trade and Business Management, College of Food Technology, M.A.U., Parbhani – 431401 (MS) India

3- Department of Food Science and Technology, Shivaji University, Kolhapur (MS) India

* imran.foodresearch@gmail.com

Abstract

Ginger (*Zingiber officinale* Roscoe, *Zingiberaceae*) is one of most important spices in India. The flavors of ginger are typically due to its essential oils characterized by warm, spicy, and woody notes, with slight lemon notes. Ginger essential oil finds in application in food and fragrance industries. However, processing conditions can cause degradation of ginger essential oil reducing its functional properties which could be prevented by microencapsulation. In present investigation, efforts were made to prepare ginger oil from dry ginger by hydro distillation method and its physical-chemical properties were observed. Further, systematic efforts were taken to standardize the spray drying condition for preparation of microencapsulated ginger oil powder by using acacia gum as wall material. The observations related to physical-chemical and organoleptic characteristics prepared powders revealed that the inlet temperature of 160°C is optimum in microencapsulation of ginger oil.

Keywords: *Zingiber officinale*, spray drying, physical-chemical properties, organoleptic characteristics

1. Introduction

The use of herbs and spices in the flavoring of foods is almost as old as man himself [1]. The spice ginger (*Zingiber officinale* Rosc) is obtained from the underground stems or rhizomes of a herbaceous tropical perennial belonging to the family *Zingiberaceae*. Its medical value is increasingly being recognized [2]. Ginger originated in South-East Asia, probably in India [3]. The name itself supports this view. The Sanskrit name 'Singabera' gave rise to Greek 'Zingiberi' and later the generic name *Zingiber* [4]. India and China are the dominant suppliers to the world market. The export of ginger during 2008-09 was 322970 tons valued at Rs.1, 581.75 Lakh. Indian ginger is popular in importing countries. The export potential of gingers produced in other states is very limited because of their higher fiber and moisture content.

Ginger essential oils or sometimes called volatile oils are believed to be that small portion of ginger, which imparts the characteristics odors and flavor most

closely associated with the vegetative matter of ginger. Ginger essential oils have a very high commercial value due to its therapeutic properties [5]. It is widely used in aromatherapy, medicine and as well as flavoring food and drink industries [6]. There are two ways of extraction, that is using steam distillation and solvent extraction. Steam distillation method is used for temperature sensitive material like natural aromatic compounds. For this method, there is no solvent is used to extract the material but pure water is the main component to do it [7].

Preventing illness by diet is a unique opportunity for innovations in development of functional foods [8, 9]. Ginger essential oil can be incorporated in various foods as a nutraceutical ingredient and for its flavor characteristics. Processing conditions of foods can cause degradation of ginger essential oil reducing its functional properties. It can also react with components present in the food system, which may limit bioavailability, or change the color or taste of a product. In

many cases, microencapsulation can be used to overcome these challenges.

Microencapsulation can provide a physical barrier between the core compound and the other components of the product. More especially, in the food field, microencapsulation is a technique by which liquid droplets, solid particles or gas compounds are entrapped into thin films of a food grade microencapsulating agent. Some researchers proposed six reasons for applying microencapsulation in food industry: to reduce the core reactivity with environmental factors; to decrease the transfer rate of the core material to the outside environment; to promote easier handling; to control the release of the core material; to mask the core taste; and finally to dilute the core material when it should be used in only very small amounts [10].

The different types of microcapsules and micro spheres are produced from a wide range of wall materials (monomers and/or polymers) and by a large number of different microencapsulation processes such as: spray-drying, spray-cooling, spray-chilling, air suspension coating, extrusion, centrifugal extrusion, freeze-drying, conservation, rotational suspension separation, co-crystallization, liposome entrapment, interfacial polymerization, molecular inclusion, etc. [10, 11, 12, 13, 14]. Microencapsulation by spray-drying has been successfully used in the food industry for several decades [12], and this process is one of the oldest encapsulation methods used since the 1930s to prepare the first encapsulated flavors using gum acacia as wall material [10]. Important applications are to coat colorants, flavors, vitamins, and other sensitive food ingredients in order to increase their shelf life [10, 15]. Microcapsules are also used in applications such as carbonless copy paper, pharmaceuticals, pesticides and scented strips. In the food industry the use of microencapsulation to protect, isolate or control the release of a given substance is of growing interest. Converting a liquid into a powder allows alternative use of

ingredients. One of the largest food applications is the encapsulation of flavors like that of ginger.

Numerous wall materials or encapsulating agents are available for food application. Gums Arabic, hydrolyzed starches, and emulsifying starches are most commonly used as wall materials [10, 16, 17]. Typically, the effective wall materials for spray drying should have functional properties, including good emulsification, film forming, high solubility, low viscosity at high concentrations and low cost properties [17, 18].

Recently, there is an increasing trend in food and pharmaceutical industries are towards replacing synthetic additive with natural products. With the increasing knowledge of the positive functions of ginger essential oils, more and more people take interest in some foods or pharmaceuticals containing ginger essential oil as ingredient, such as beverages, baked goods, oils, capsules and tablets. The reasons for encapsulating a ginger essential oil includes: reduce reactivity with the environment (water, oxygen, light), decrease evaporation or transfer rate to the outside environment, promote handling ability, release control, mask taste, and dilute to achieve a uniform distribution in the final product when used in very small amounts [19]. In present investigation, the efforts were made to extract essential oil from ginger. The physical-chemical properties of extracted essential oil were studied. Further it was microencapsulated by spray drying and physicochemical properties of microencapsulated ginger essential oil powder were measured.

2. Materials and methods

2.1. Raw material selection and analysis

Selection of ginger of Essential Oil Extraction: The technological feasibility of both fresh and dry ginger was assessed to obtain essential oil. During initial experiments, it was observed that dried

ginger yields more ginger oil as compared to the fresh ginger. Henceforth dried ginger is used for the experimental purpose. The ginger used in this research was purchased from the local market.

Analysis of Ginger: Moisture content, crude protein and crude fat were analyzed by the standard method [20]. The carbohydrate content was calculated by differential method.

2.2. Ginger oil extraction & its physicochemical properties

Flow sheet for extraction of ginger oil was summarized in Figure 1.

Operational Conditions: Ginger oil was extracted by hydro distillation method with optimized operational condition. The operational conditions include optimized sample, temperature, extraction time, and ratio of ginger to water. *Temperature:* The operating temperature for experiments carried out was varied from 100°C to 150°C. The experiments using distillation were carried out only at atmospheric pressure (i.e. 760mm of Hg). *Extraction time:* the term extraction time is used for the duration of time it took for experiment to run. In this research, the experiments were carried out from 2.30 to 4 hrs of extraction time.

Ratio of ginger to water: the experiments being carried out using the equipment set up. The ratio of ginger to water was 1 kg of sample to 10 liter of distilled water was used. *Refractive index:* the refractometer is the fastest and reliable technique in quality control assessments and conformation in this research. The literature value for the refractive index of ginger oil is in the range of 1.4800 to 1.4950. The ginger oil with the RI value of 1.4888 to 1.4895 were collected from the hydro distillation of ground ginger at atmospheric pressure was used for the vapor pressure measurement.

Experimental procedure: the experiment was carried out at atmospheric

pressure. 1kg of sample is weighed and put into the still proper without using a basket. Then, 10 liters of distilled water was added into the vessel. The cover was then closed and heated for 4 hours. During the whole experiment the condensation was going on, condensate content water and ginger oil. The condensate was channeled out in a separating funnel. The condensate was then allowed to settle for a few minutes before the ginger oil was collected. Next, the ginger oil collected and analyzed for its refractive index, weighed and stored in air tight glass container.

2.3. Analysis of ginger oil

Color of Ginger oil was determined by visual observation. *Moisture content, flash Point, acid value, specific gravity and refractive index* of oil was measured by standard method [20].

Non volatile matter of oils: Non-volatiles were analyzed as per the reported method. 0.2 g of the ginger oil was weighed accurately in an evaporating dish and heated in a hot air oven at 100°C for 45 min. The evaporating dish was cooled and weighed again. The loss in the weight corresponds to volatile oil, and the mass left behind to that of the non-volatiles or fixed oils. All the results were expressed as % w/w of the sample.

Determination of optical rotation: In case of optical rotation determination, 10 ml polarimeter tube containing oil was placed in the trough of the instrument between polarizer and analyzer. Care was taken in filling the tube to avoid the entrance of air bubble which could disturb the rotation of light. Analyzer was slowly turned until both the halves of the field were viewed through the telescope. The direction of rotation was determined, if the analyzer was turned counter clock wise from the zero position to obtain the final reading, the rotation is levo (-) if clock wise and dextro (+) if anti clockwise.

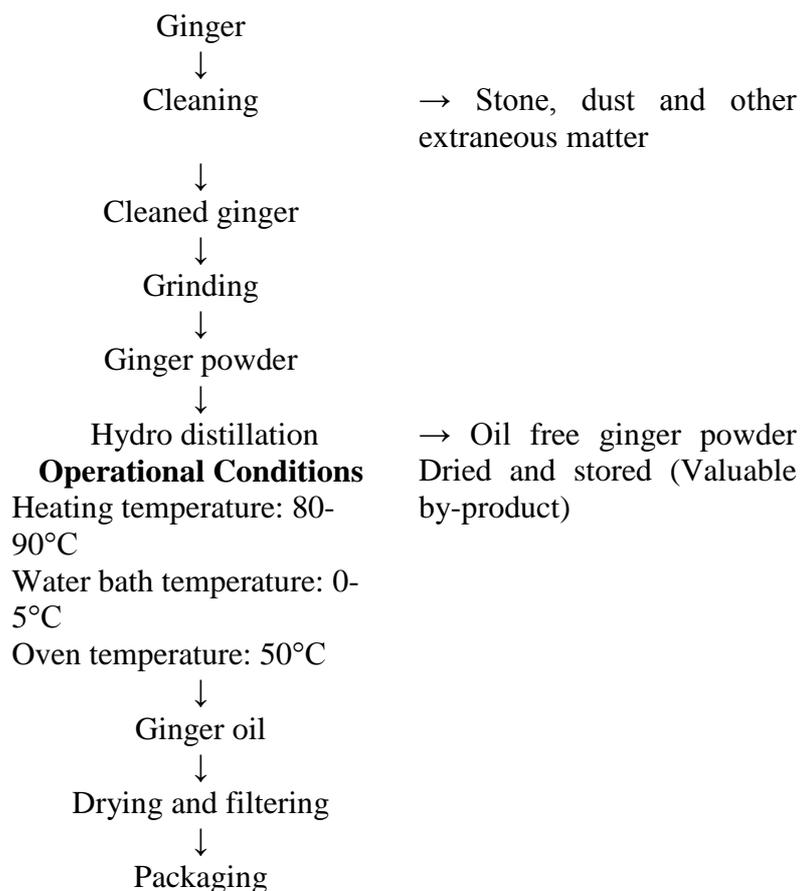


Figure 1. Process flow chat for Extraction of ginger oil

2.4. Microencapsulation of ginger oil and analysis of encapsulated powder

Preparation of Feed Mixture and Microencapsulation by Spray Drying

Gum acacia is used as carried material for microencapsulation of ginger oil due to its high water solubility, low viscosity and ability to act as an oil-in-water emulsifier. Wall material (feed mixture) was prepared by dissolving the desired amount in deionized water at 50°C stirring overnight to enhance hydration. In a beaker 80 gm of gum acacia was dissolved with 200 ml of distilled water, solution was kept on stirring to dissolve gum acacia. Similar type of formulations of feed mixture was prepared by others [21]. Further 20 gm of ginger oil was added and homogenized vigorously (10000rpm/5min) with an Ultra Homogenizer at ambient temperature (22°C) for 15 min. Maintain the pH of the solution around 4.5-5 and viscosity at 25°C measured by Brookfield viscometer was 35cP. The prepared feed mixture was slowly agitated till spray drying and the feed mixture was spray dried at the specified conditions.

Physical-chemical Analysis of Microencapsulated Ginger oil powder

Moisture content: moisture content of microencapsulated powder was determined by standard method [20].

Microencapsulation efficiency (M.E.): Microencapsulation efficiency was calculated as follows [22]. The total oil content of the powder was determined by Soxhlet extraction. The extractable oil was determined by gently shaking capsules in petroleum ether for 10 min. The solvent was filtered and the extractable fat was determined gravimetrically. The encapsulation efficiency (EE) was calculated as follows:

$$\text{Encapsulation efficiency} = (\text{total oil} - \text{extractable oil}) \times 100 / \text{total oil}$$

Particle size distribution: the particle size distribution of microencapsulated powder was determined using Laboratory Test Sieve (AS 200 digit, ASTM E 11, RETSch, Germany).

Bulk density of powder: the Bulk Density of microencapsulated ginger essential oil powder was measured by method given other authors [23].

Organoleptic Evaluation

The sensory evaluation of microencapsulated ginger oil powder was carried out by a 25 member trained panel comprising of postgraduate students and academic staff members of the faculty who had some previous experience in sensory evaluation of herbs and spices products. The panel members were requested in measuring the terms identifying sensory characteristics and in use of the score. Judgments were made through rating products on a 9 point Hedonic Scale [24] with corresponding descriptive terms ranging from 9 'like extremely' to 1 'dislike extremely'.

Statistical analysis

The analysis of variance of the data obtained was done by using Completely Randomized Design (CRD) for different treatments as per the methods given by others [25]. The analysis of variance revealed at significance of $P < 0.05$ level, S.E. and C.D. at 5 % level is mentioned wherever required.

3. Results and discussion

In present investigation efforts were made to standardize the process for microencapsulation of ginger essential oils using spray drying. The data was generated during present investigation is summarized as follows.

Proximate Composition of Ginger

The sample was analyzed for their proximate compositions and depicted in Table 1. Oleoresin content of ginger was found to be 6.79% which is use as a

flavoring compound also for studies on extraction of essential oil and its encapsulation. Moisture content of ginger was found to be 12.13 %, which is an important parameter for condition during oil extraction. While Ash, protein and fiber content of ginger was found to be 8.08%, 16.27% and 6.9%, respectively.

The color of obtained ginger oil varied from pale yellow to brown with a warm, citrus & woody Characteristic odor in liquid state. The flash point of obtained ginger oil was observed to be 56°C, while the specific gravity 20°C varied in the range of 0.8765 to 0.8892. The Refractometer is the fastest and reliable technique in quality control assessments and conformation in this research. The literature value for refractive index (RI) of ginger oil is in the range of 1.488 to 1.494 [26]. In present investigation, the refractive index at 20°C observed to be in the range of 1.478 - 1.4913 which is at par with literature value. The standard values for optical rotation of ginger oil are in the range -26 to -50 while the observed values were in the range of -29 to -48°C. Non volatile matter present in oil was 4.17% and easily soluble in ethanol having acid value 5.28. Moisture content of essential oil was surprisingly high with an average of 8.76 per cent.

Standardization of process for microencapsulation of ginger oil by Spray Drying

In the present investigation efforts were made to standardize the process for microencapsulation of ginger oil by spray drying under different drying conditions. The prepared ginger oil and gum acacia feed mixture was encapsulated by spray drying by using LSD – 48 MINI SPRAY DRIER JISL. The microencapsulation of ginger oil was standardized by monitoring effect of different Inlet temperature combinations while other parameters such as feed flow rate, rotational speed of atomizer and flow rate of air are kept

constant at the values of 500ml/hr, 30.000 rpm and 110kg/hr, respectively.

Preliminary experimental work was done with different inlet temperatures for spray drying of feed mixture at the feed flow rate of 500ml/hr so as to select a range of temperature could be used in ginger oil encapsulation. In order to become better acquainted with spray drying equipment, numerous practice attempts were made and through trial and error, coupled with continued informal sensory evaluation by a panel of food scientists. As result of these preliminary trials, it was observed that at the lower inlet temperatures (between 110 to 140°C), the powder so prepared was of inferior quality with larger particle sizes having lower flow ability while at the inlet temperatures above 180°C were resulting in very minute particle size causing dusting of prepared powder with lower flavors profile. Hence on the basis of these results, it was decided to systematically study the spray drying of ginger oil encapsulation feed mixture in the range of 150 to 180°C. Further systematic studies have been carried out by observing the different physical-chemical properties of encapsulated ginger oil powder so as to optimize spray drying inlet temperature. All the trials were done in triplicate and the averages of these triplicate measurements recorded. Additional determinations were carried out if the single values from the triplicates deviated by more than $\pm 0.6\%$ from the triplicate mean. Results from microencapsulation of ginger oil by using a laboratory scale spray dryer are shows in Table 2.

The physical-chemical properties of powder are important in judging its suitability for microencapsulation. Various physical-chemical properties of encapsulated ginger oil powder prepared with varying inlet temperature of from 150°C to 180°C are given in Table 3.

Moisture content of powder is one of the most crucial factors in justifying the suitability of powder for its end use.

Table 1
Proximate composition of ginger

Sr. No.	Parameters	Value (%)
1	Moisture content	12.13
2	Ash content	8.08
3	Protein	16.27
4	Fiber	6.87
5	Oleoresin	6.79
6	Starch	50.86

* Each value is average of 5 determinations

Table 2
Physical and Chemical Properties of ginger oil

Sr. No.	Parameter	Results
1	Color	Pale yellow to brown
2	Odor	A warm, citrus & woody Characteristic of Ginger
3	Appearance	Mobile liquid
4	Flash Point	56°C
5	Specific Gravity@ 20° C	0.8765 - 0.8892
6	Refractive Index@ 20°C	1.4800 - 1.4940
7	Non Volatiles Matter	4.17 %
8	Optical Rotation	-29 to -48°
9	Solubility	Soluble in alcohol (95%)
10	Acid Value	5.28
11	Moisture content	8.76%.

* The values/characteristics presented in Table 2 represent the range or average of 5 determinations.

Table 3
Effect of different Inlet temperature on physical-chemical properties of microencapsulated ginger oil powder

Inlet Temperature (°C)	150	160	170	180
Outlet Temperature (°C)	99	108	112	126
Moisture Content (%)	5.90	4.01	3.67	2.21
Bulk Density (g/ml)	0.79	0.74	0.71	0.69
Microencapsulation efficiency	92%	91%	87	82
Average particle size (µm)	55.9	41.2	37.8	32.1
Overall Acceptability	7.9	8.5	8.1	7.8

During present investigation, moisture content of spray dried powder found to be inversely related to inlet temperature of spray drying.

Moisture content of the spray dried encapsulated ginger oil powder at varying temperatures found to be in the range of 5.90 to 2.21 per cent. It was observed that if moisture content of powder will be higher (as in case of sample dried at 150°C), the powder have susceptibility for microbial spoilage while too much lower moisture (as in case of sample dried at 180°C), dustiness of powder is observed which is undesirable. The moisture content of sample dried at 160°C is observed to be 4.01 per cent which is sufficient enough for the long term of storage of powder without any microbial deterioration under specific conditions while at this moisture, no dusting of powder was observed. With respect to bulk density, it was observed that bulk density of spray dried encapsulated ginger oil powder decreases with increase in inlet temperature and vice versa. The encapsulation efficiency is defined as the proportion of ginger oil that was not available to the extracting solvent and, consequently, less exposed to the environment. It is related to the commercial powder quality so it has an effect on volatility and flow powder properties. This reflects the amount of oil recovered from powder particles during the solvent-extraction process, which is composed of both surface and internal oil entrapped within the wall matrix. In this study the encapsulation efficiency was highest at lower temperature and found to decrease with increase in temperature. Finally, overall quality of prepared microencapsulated ginger oil powder was assessed on the basis of sensory evaluation. During sensorial evaluation, overall acceptability was decided on the basis of major quality characteristics including overall appearance and flavor profile of the powder. It was found that maximum sensorial score was reported in case of powder at 150°C, slight clamminess was observed due to higher moisture content

while at the temperature of 180°C flavor properties has shown minimum values. Maximum overall acceptability of powder was observed at the inlet temperature of 160°C, which may be due to higher flavor characteristics and better flow ability of powder with acceptable particle size.

4. Conclusion

For a developing country like India, ginger oil production by hydro distillation could be considered to be much more economical compared to other methods of essential oil production. High quality industrial grade ginger oil was prepared by hydro distillation method under laboratory conditions. It was observed that Gum Arabic is successful in encapsulating ginger oil. The degradation of ginger oil is higher at the elevated temperature. When compared to previous methods, the current approach showed comparable or improved encapsulation efficiency.

In this experiment, it could be concluded that spray drying at the inlet temperature of 160°C is ideal for microencapsulation of ginger oil powder.

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THE MICROWAVES EFFECTS AGAINST THE PATHOGENS *ESCHERICHIA COLI* AND *STAPHYLOCOCCUS AUREUS* PRESENT IN RAW MILK

Anca Dumuta^{1*}

*1** - North University of Baia Mare, Faculty of Sciences, Chemistry Biology Department 430122- Baia Mare, Victoriei street, no. 76, Romania, codre_anca@yahoo.com

Abstract

In this study, the effects that microwaves have on the pathogens *Escherichia coli* and *Staphylococcus aureus* were investigated. These microorganisms can contaminate the raw milk during the milking procedure, from 3 main sources: the milking equipment, the teat surface and from within the udder or after the pasteurization of the milk, having as sources of contamination, the equipment and the human factor, all this sources generating microbiological risks.

Keywords: *microbiological risks, pathogens, microorganisms' destruction*

1. Introduction

Already for decades the processing of milk using microwaves action is an alternative for the conventional processes of pasteurization or sterilization of milk. Microwaves heating generates a volumetric heating in the material, resulting in a high energy efficiency and a reduction of the heating time [1]. Heating of food due to the microwaves action results from the conversion of the microwaves energy into heat by friction of water molecules vibrating because of the rapid fluctuation in the electromagnetic field [2, 3]. The microwaves heating is influenced by many factors, including dielectric properties, volume and shape of the material, but also the design and the geometric parameters of the microwave equipment [1]. The dielectric properties are dependent on the composition of the treated material and its humidity and salt content are two determining factors [4, 5].

The fact that the microwaves radiation helps preserve food by reducing the presence of active microbiological cells has been demonstrated by a series of studies undertaken on foods such as turkeys, meats, milk, corn on the cob, chickens, frozen foods and potatoes [6, 7, 8, 9, 10, 11, 12].

Whilst the microwaves ovens are widely used for food preparation, insufficient information is available on the consequence of microwave heating on the composition and the nutritional quality of food [13].

Considering that relatively few studies investigated the influence that microwaves have on the microorganisms possibly present in milk as shown in the above paragraph, this study aims to inquire the effects that microwaves have on the pathogens *Escherichia coli* and *Staphylococcus aureus* inoculated in raw milk.

2. Materials and methods

In order to study the effects that microwaves have on these pathogens, we inoculated a quantity of 500 ml of raw milk with a reference strain for each pathogen and then we incubated the 2 types of milk for 24 hours at 37 °C in the *Staphylococcus aureus* case and at 44 °C in the *Escherichia coli* case. In each situation, we analyzed 6 samples of 50 ml milk /sample using the specifically colony counting techniques, after the incubation time and after exposing them to the microwaves action of a home-type oven during different periods of time: T0 – milk

sample not treated with microwaves, T15 – milk sample treated for 15 seconds with microwaves, T30 - milk sample treated for 30 seconds with microwaves, T45 - milk sample treated for 45 seconds with microwaves, T60 - milk sample treated for 60 seconds with microwaves and T120 - milk sample treated for 120 seconds with microwaves.

The samples were exposed to the microwaves action of a Hansa AMM 21 E80GH microwave oven at electrical power of 800 W during the different periods of time mentioned before.

To investigate the microwaves influence against the *Staphylococcus aureus* pathogen we applied the Technique using Baird-Parker agar medium and against the *Escherichia coli* pathogen the Colony-count technique at 44 °C using 5-bromo-4-chloro-3-indolyl β-Dglucuronide.

3. Results and discussions

The values obtained for the number of pathogen germs present in 1 ml of milk sample after using the specifically colony counting techniques are present in Tables 1 and 2.

Table 1. The number of *Staphylococcus aureus* germs present in 1 ml of milk sample inoculated and microwave treated for different periods of time

Microwaves time exposure of the sample, s	0	15	30	45	60	120
The number of <i>Staphylococcus aureus</i> germs present in 1 ml of milk sample	Massive infestation	8x10	0	0	0	0

Table 2. The number of *Escherichia coli* germs present in 1 ml of milk sample inoculated and microwave treated for different periods of time

Microwaves time exposure of the sample, s	0	15	30	45	60	120
The number of <i>Escherichia coli</i> germs present in 1 ml of milk sample	Massive infestation	0	0	0	0	0

The images of the Petri dishes containing some of the samples after the incubation time are presented in the

Figures 1-5. In Figures 1 and 4, corresponding to the samples of the two pathogens not treated at microwaves, one can observe that the number of microorganism's germs is very high and can not be counted due to the situation of massive infestation.

After exposing the milk samples to the microwaves action, there can be seen a significant reduction of the microorganisms number as the microwave time exposure is increased.

The number of *Staphylococcus aureus* germs decrease from a massive infestation to the value of 8 x10 germs / ml after 15 s of microwave exposure and then to the noteworthy value of 0 germs/ml after 30 s of exposure. This last value is maintained for all the rest of the microwave exposure intervals. The *Escherichia coli* case study is similar to the *Staphylococcus aureus* case, except that this pathogen is reduced to the 0 value even after the first 15 s of microwave exposure.

It seems that the microwaves action against the microorganisms present in foods is influenced by the intrinsic characteristics of the products being processed (pH, humidity, Ox reduction potential, antibodies present, biological structures, chemical composition, amount and geometry of the food) and extrinsic factors (temperature, humidity, ambient gases, frequency and intensity of the radiation, time of exposure, position of the foods in relation to the effective radiation field, among others). Also, very important are: the chemical and physical composition of the microorganisms being irradiated, their stage of development (vegetative cell, spore or development phase, wet or dry, etc.) and their initial amount. [14].



Figure 1. The milk sample inoculated with Staphylococcus aureus and not treated at microwaves

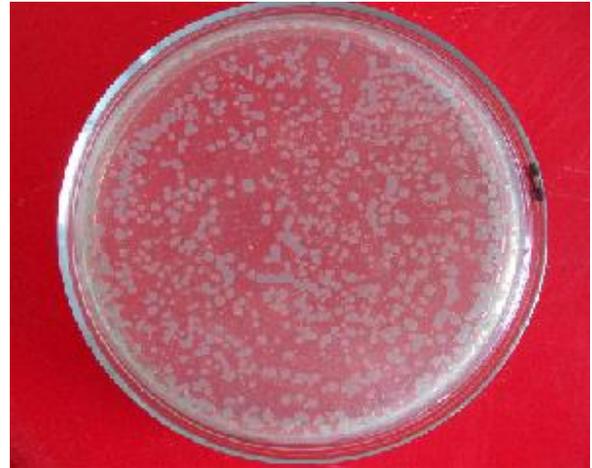


Figure 4. The milk sample inoculated with Escherichia coli and not treated at microwaves



Figure 2. The milk sample inoculated with Staphylococcus aureus and treated for 15 seconds with microwaves

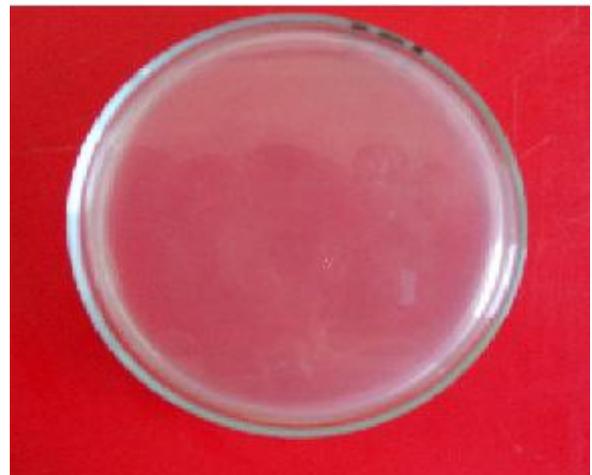


Figure 5. The milk sample inoculated with Escherichia coli and treated for 15 seconds with microwaves



Figure 3. The milk sample inoculated with Staphylococcus aureus and treated for 30 seconds with microwaves

The mechanism of destruction of microorganisms through microwaves is debatable. Although some studies have shown that the thermal effect of the microwaves heating is essential in destroying microorganisms due to the fact that the absorption of microwave energy can increase the temperature of foods rapidly, deactivating microorganisms and performing pasteurization or sterilization [14], others have explained this destruction by non-thermal effects, because a lower final temperature may be needed to kill microorganisms. The destruction by non-thermal effects or "cold pasteurization" is sustained by four predominant theories:

selective heating, electroporation, cell membrane rupture, and magnetic field coupling [15].

Albeit there is a debate on the mechanisms of microwave-induced death of microorganisms, there is no doubt about its destructive effect as microwave destruction of many microorganisms has been reported, including: *Bacillus cereus*, *Campylobacter jejuni*, *Clostridium perfringens*, *E. coli*, *Enterococcus*, *Listeria monocytogenes*, *Staphylococcus aureus*, *Salmonella enteridis*, *Salmonella sofia*, *Proteus mirabilis* and *Pseudomonas aeruginosa*, *Aspergillus niger*, *Penicillium* and *Rhizopus nigricans* [16]. No pathogen has been reported to be resistant at the microwaves action [17].

4. Conclusions

The present study examined the effects of microwave treatment on the pathogens *Escherichia coli* and *Staphylococcus aureus* inoculated in raw milk.

After exposing the inoculated milk samples to the microwaves action for 0s, 15s, 30 s, 45 s, 60 s and 120 s, there can be seen a significant reduction of the microorganisms number as the microwave time exposure increased. Thus, the samples of the two pathogens not treated at microwaves have a very high number of germs that can not be counted due to the situation of massive infestation. But only after 15 s of microwave exposure the number of *Staphylococcus aureus* germs decreased to the value of 8×10 germs / ml and the number of *Escherichia coli* is reduced to 0. From this point forward, during the rest of the microwave exposure intervals, the two pathogens are reduces to the noteworthy value of 0 germs / ml.

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THE PHYSIC-CHEMICAL QUALITY OF CHEESE IN MARAMUREŞ COUNTY

Bretan Laura^{*1,3,4}, **Pop Vasile**^{1,3}, **Ketney Otto**^{1,2,3}, **Boltea Florin**^{1,3,5}

1* - North University of Baia Mare, Faculty of Sciences, Chemistry Biology Department 430122- Baia Mare, Victoriei Street, no. 76, Romania (* Corresponding author: e-mail: laura_sya@yahoo.com)

2 - "Lucian Blaga" University of Sibiu, Faculty of Agricultural Sciences, Food Industry and Environment Protection, Department of Food Biotechnology – 550024-Sibiu, Dr. Ion Ratiu Street, no. 7-9, Romania

3 - Sanitary Veterinary and Food Safety Direction, Vasile Alecsandri Street no. 66, Baia Mare, Maramures

4 - University of Agricultural Sciences and Veterinary Medicine Cluj-Napoca, Calea Mănăştur Street, no. 3-5, Cluj-Napoca

5 - University of Agricultural Sciences and Veterinary Medicine of Banat Timişoara, Food Technology Faculty, 119 A, Aradului street, 300645, Timişoara

Abstract

The quality of the finite product is the sum of the qualities of all the steps taken in the process through which the raw material goes, possibly being made through a collective effort and common acceptance of the competition qualitative criteria. The concept of "the quality of a food product" includes the requirements of nutritional, hygienic, sanitarian, technological and commercial order. The nutritive criterion refers to the food content in proteins, carbohydrates, lipids, mineral substances and vitamins.

Key words: *quality, cheese, water, fat/ S.U.*

1. Introduction

The producers are tempted to obtain a substantial profit, in the detriment of the quality of the food product due to the high sum of conditions influencing the last one [1,2,3]. The responsible authorities supervise the observance of all requirements imposed by European requirements by producers. In order to appreciate the quality of cheese, a series of determinations which allow establishing whether those products are made according to the intern standards and requirements are made. Cheese plays an important part in nutrition. They represent an important source of nutritive factors, with high biological value, concentrated in a small volume and with a high digestion [4]. The nutritive value of cheese is given by the high content in protein substances and fat easy to assimilate, mineral salts of calcium, phosphor, magnesium, sodium and chlorine, as well as vitamins [5]. Trough the concentration of fat in the obtained coagulant by the precipitation of casein, cheese becomes a source of A, D, E, K liposoluble vitamins more important than milk. The energetic value of cheese is

conditioned by the content of fat of the product.

2. Materials and methods

115 samples of Dalia cheese were examined throughout the year 2010. The samples of Dalia cheese were taken from different private enterprises from the Maramures county. According to STAS 10008-81, the maximum admitted quantity of water (%) in Dalia cheese is of 44%, and the fat according to S.U. must be of minimum 40%.

The determination of water in the cheese was made by using the method of drying in stove, and the ratio fat /S.U. was made by using the butirometric method Van Gulik.

3. Results and discussions

From the 115 samples of Dalia cheese, 102 analysed samples were situated above the reference value of maximum 44% water. At the G/SU parameter, 14 samples were situated under the value of 40%. The obtained results are emphasised in Table 1 and Figures 1 and 2:

Table 1.
The values of the physical-chemical parameters of Dalia cheese

No.	WATER (%)	G/SU	No.	WATER (%)	G/SU	No.	WATER (%)	G/SU
1	46.84	40.4	40	50.7	32.4	79	48.35	41.6
2	43.67	35.5	41	48	40.3	80	47.35	40.8
3	47.62	40	42	50.7	33.4	81	48.35	40.6
4	42.3	43.3	43	47.8	40.2	82	48.16	40.5
5	49.5	31.6	44	47.3	41.7	83	44	41.1
6	44	40.6	45	48.32	40.6	84	44	41.1
7	43.72	40.8	46	48.63	40.8	85	47.35	40.8
8	39	37.7	47	47.3	41.7	86	47.83	41.2
9	41.1	37.3	48	46.31	40.9	87	46.5	39.1
10	47.62	42	49	47.3	40.7	88	48.34	41.6
11	49.5	44.5	50	47.34	40.8	89	47.32	41.7
12	46.38	40	51	47.3	41.7	90	48.46	41.6
13	48.8	31.2	52	47.35	40.8	91	47.35	41.7
14	48.1	30.8	53	47.24	40.7	92	47.54	40
15	48.65	41.3	54	46.32	40.9	93	48.05	40.4
16	48.7	42.8	55	47.3	41.7	94	48.35	41.6
17	47.2	41.6	56	54.3	43.7	95	47.3	41.7
18	48.3	40.6	57	48.35	39.6	96	48.3	40.6
19	53.1	41.5	58	47.4	40.7	97	48.16	40.5
20	47.32	41.7	59	46.35	40	98	48.6	40.8
21	48.03	41.3	60	47.35	41.7	99	47.5	41.9
22	48.32	40.6	61	48.8	40.9	100	48.62	40.8
23	48.3	40.6	62	44	38.4	101	47.3	41.7

24	47.62	41	63	44	38.4	102	48.7	41.9
25	47.19	41.6	64	48.36	40.6	103	47.3	41.7
26	47.3	41	65	48.64	40.8	104	47.38	40.8
27	47.35	40.8	66	46.94	40.5	105	48.43	40.7
28	47.03	40.5	67	48.64	38.9	106	48.3	40.6
29	46.92	40.5	68	47.3	41.7	107	48.35	40.6
30	49.62	41.6	69	48.64	40.8	108	48.35	41.6
31	47.05	41.5	70	48.37	42.6	109	48.7	41.9
32	47.35	41.7	71	47.3	41.7	110	47.35	41.7
33	48.62	41.8	72	47.19	41.6	111	48.7	40.9
34	46.7	45	73	46.85	41.3	112	48.7	41.9
35	47.35	40.8	74	42.8	48	113	48.5	41.7
36	47.9	36.6	75	47.6	42.9	114	48.64	42.8
37	44	40	76	43.2	41.3	115	48.75	41.9
38	48.73	42.9	77	47.35	41.7	x	47.456	40.6721
39	48.18	40.5	78	48.36	40.6	d.s	1.9455	2.37085

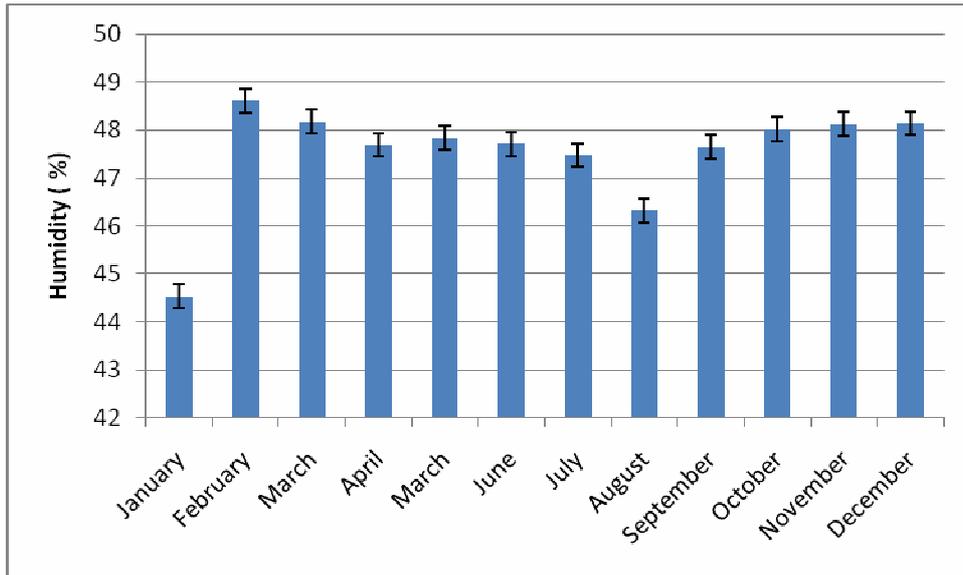


Figure 1. The quality of cheese according to humidity in 2010

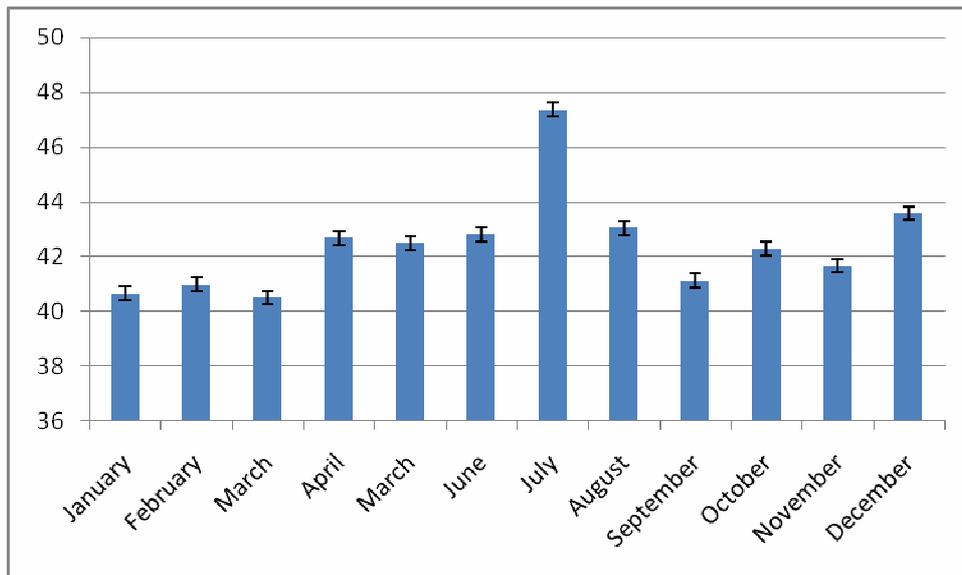


Figure 2. The quality of cheese according to G/SU ration in 2010

4. Conclusions

The ratio of humidity at the analysed samples was $47.45 \pm 1.9\%$, with a variation coefficient of 4.09%.

The G/SU ratio was $40.67 \pm 2.37\%$ with a variation coefficient of 5.82%.

From the total of analysed samples for water, 88.69% did not correspond.

From the total of analysed samples for G/SU parameter 12.17 % did not correspond.

A too high quantity of water in cheese denotes the fact that heating, pressing, cutting of the coagulant, sweeping were not made properly.

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USE OF TiO₂ PHOTOCATALYST AS ALTERNATIVE MEANS FOR THE COTTAGE CHEESE PRESERVATION

Peter Anca^{1*}, Bregnya Denisa¹, Mihaly-Cozmuta Leonard¹,
Mihaly-Cozmuta Anca¹, Nicula Camelia¹

¹ North University of Baia Mare, Chemistry-Biology Department, Victoriei 62 A, 430083, Baia Mare, Romania
* peteranca@yahoo.com

Abstract

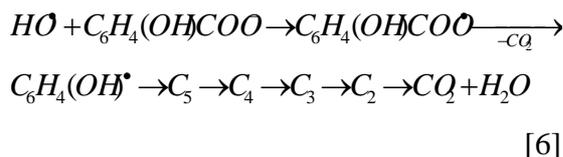
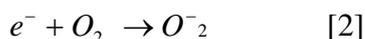
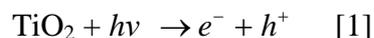
Various types of packages containing TiO₂ for the cottage cheese preservation, in air, at the room temperature, were tested. A nano-sized film of amorphous and crystallized TiO₂ respectively, was coated onto a polypropylene film. Thus, four types of packages: polypropylene-amorphous TiO₂, polypropylene-amorphous TiO₂-polypropylene, polypropylene-crystallized TiO₂ and polypropylene-crystallized TiO₂-polypropylene were obtained. The cottage cheese preservation was monitored by acidity, chlorine and fat contents, during 3, 8 and 11 days. FTIR analyses were performed, to observe if TiO₂ penetrated cottage cheese, especially when the TiO₂ is not in contact with cheese. Acidity, chlorine content and fat of the cottage cheese packed in bags containing TiO₂ decrease in time. Moreover, the acidity decrease is more significant when polypropylene-TiO₂ bag was used, in comparison with the case when polypropylene-TiO₂-polypropylene was used. Those three monitored parameters decrease when, in the same conditions, crystallized TiO₂ was used, demonstrating the higher photoactivity of the crystallized TiO₂. The FTIR spectra of the samples packed in polypropylene-amorphous TiO₂ and polypropylene-amorphous TiO₂-polypropylene showed the presence of the TiO₂ in cheese only after 8 and 11 days. In the case of using crystallized TiO₂, it's very interesting to observe that only after 3 days, the cheese contain TiO₂ and after 8 days of storage, no peaks assigned to the Ti-O vibration in the FTIR spectra of cheese packed in polypropylene-TiO₂ and polypropylene-TiO₂-polypropylene were observed.

Keywords: FTIR analyses, acidity, chlorine content, amorphous, crystalline

1. Introduction

Given the worldwide growth of the population and, subsequently, of the demand for food, producing healthy food, properly preserved and safe for human use has become a high - priority research topic. Nanotechnologies represent an alternative to the classical methods of food disinfection, preservation and protection (featuring their own several disadvantages). Nanomaterials have diverse applications in food safety, in the detection and destruction of saprophytes, in food packaging, because they exhibit properties not found at the macro-scale. [1]. Application of nanomaterials in the field of food security is due to their photocatalytic activity. One of the most performing photocatalysts is TiO₂, due to its chemical stability, to its resistance to photo-corrosion, lack of toxicity and low cost [2-9].

The mechanism of photocatalysis consists in the attack of the photons with the energy $h\nu$ equal or greater than the band gap (E_g) of TiO₂ on the semiconductor particle, which induces the generation of charge carriers (electrons and holes) (reaction 1) and the transfer of electrons (e^-_{CB}) from the valence band (VB) into the conduction band (CB), leaving in VB the holes (h^+_{VB}) [10]. The electrons in CB reduce the dissolved oxygen (reaction 2), by generating oxygen radicals and the holes from VB oxidize the chlorine ions (reaction 3) and hydroxyl groups on the TiO₂ surface (reaction 4), forming chlorine radicals, that will transform in chlor molecules (reaction 5) and hydroxyl radicals, which will initiate the oxidation of the organic and inorganic compounds, including lipids, glucides, ...(reaction 6).



Many factors influence the photocatalytic activity of TiO_2 : TiO_2 polymorphism (type of the crystalline structure) and the dimension of the crystalline particles [17], the specific surface area [17] and the photocatalyst mass [11].

Research studies [11, 18] have demonstrated that the antimicrobial activity goes up as the TiO_2 mass grows, because the number of active centres that generate charge carriers (electrons and holes) goes up, thus initiating the decomposition of cellular organic compounds. Even more, it is important to bear in mind that the antimicrobial efficiency of TiO_2 is the same in the case of unsupported powders, as well as for the supported powders, generally on polypropylene [11, 12].

In terms of the preservative activity of TiO_2 , research has demonstrated that the presence of TiO_2 in the packaging for pasteurized milk, stored for over 7 days, reduces the degradation of vitamin A and the generation of riboflavin, responsible for the unpleasant smell [14-16]. Also, by packaging jujube fruits [13] for over 12 days at room temperature, with a material containing TiO_2 , there will be no impact on the fruits neither in terms of freshness, nor as regards their physical-chemical and physiological properties. Other advantages of the TiO_2 - based nanomaterials used to produce smart packaging systems include: the simple preparation technology, industrial feasibility and the low cost [13].

The aim of this research is to test the preservation activity of amorphous and

crystallized TiO_2 contained in polypropylene packages on the cottage cheese. Moreover, it has been of interest to establish if the TiO_2 penetrate the cheese in packages in which the photocatalyst is not in the contact with the aliment.

2. Materials and methods

2.1. Cottage cheese

The cottage cheese was homemade prepared from fresh cow milk, according to the traditional brewing method.

2.2. Packaging materials

The crystallized TiO_2 was obtained by heat treatment of the commercial amorphous TiO_2 at 500°C , 2 hour, the heating/cooling speed being $4^\circ\text{C}/\text{min}$, in a Caloris furnace (2006).

A suspension of amorphous and crystallized TiO_2 (0.1 g) in isopropyl alcohol (2 ml) was prepared and coated on 200 cm^2 polypropylene film, using a knife, and dried in air for 10 minutes. Packages, in which, a second polypropylene film was deposited onto amorphous or crystallized TiO_2 were also prepared. In the as obtained packages, 55 g cottage cheese was measured and packed. In the packages with the structure polypropylene-amorphous TiO_2 and polypropylene-crystallized TiO_2 the cheese is in contact with TiO_2 and in the packages with the structure polypropylene-amorphous TiO_2 -polypropylene and polypropylene-crystallized TiO_2 -polypropylene the cheese is not in contact with TiO_2 .

The bags were maintained in laboratory, at 22.5°C , for allowing the visible light to excite the TiO_2 particles.

At 3, 8 and 11 days acidity, chlorine content, fat and FTIR analyses were made.

2.3. Acidity measurements

5 g of cottage cheese was mixed with 5 ml ultrapure water and than 40 ml water at $35\text{-}40^\circ\text{C}$ and 2 drops of phenolphthalein 1% (S.C. Chemical Company S.A. Iași, România) were added.

The mixture was titrated with 0.1N solution of NaOH (S.C. Chemical Company S.A. Iasi, România), until the pink color appearance that will persist as least 1 minute. The acidity was calculated using the formula [19]:

$$\text{Acidity (Thörner degrees)} = 10 \cdot V \quad (1)$$

where:

V – volume of NaOH 0.1 N solution used to titration 9 (ml)

Every experiment was repeated two times and the average value was noted.

2.4. Chlorine content determination

10 g cottage cheese was mixed with 100 ml ultrapure water at 60-65°C. The mixture was stirred for 20 minutes in the Heidolf stirrer (2009) and than was filtered. 10 ml of the obtained extract were introduced in an Erlenmeyer vessel and, subsequently, 3-4 drops of potassium chromate (S.C. Chemical Company S.A. Iași, România) were added. The mixture was titrated with 0.1 N solution of silver nitrate (S.C. Chemical Company S.A. Iasi, România) until the color changed in red-brown.

The chlorine content was calculated using the formula [19]:

$$Cl \text{ (g/100 g sample)} = \frac{V \cdot 0.00585 \cdot 10}{m} 100 \quad (2)$$

where:

V – volume of AgNO₃ 0.1 N solution used to titration (ml).

m – cheese mass (g).

0.00585 – mass of sodium chloride corresponding to 1 ml 0.1 N solution silver nitrate.

10 – ratio between the total volume of the aqueous extract and the analyzed extract volume

Every experiment was repeated two times and the average value was noted.

2.5. Fat content

The fat content was determinate by solvent extraction, using a VELP extraction system (2009), followed by solvent evaporation. The used solvent was ethanol. 5 g of cottage cheese was introduced in the cotton bag and in the solvent vessel, 75 ml ethanol 96% (S.C. Chemical Company S.A. Iași, România) were added. The work temperature was 210°C. The extraction process presents three stages: 1) immersion (90 minutes), when the sample was imersed in solvent, 2) washing (20 minutes), when the sample was allow to eliminate the solvent from the cotton bag and 3) recovery (10 minutes), when the remained solvent was recovered. After the extraction, the mixture ethanol – lipids was transfered into a porcelain vessel and heated slowly to 60°C, when the solvent was evaporated.

The fat content was estimated in g/100 g cheese.

2.6. Structure analyses

The structure analyses were performed on a Perkin Elmer FTIR 2000 instrument (2010). Sample wafers consisted of 100 mg spectral quality KBr and ca. 1 mg cheese sample.

3. Results and discussion

3.1. Organoleptic characteristics of cheese

The macroscopic images of the cheese maintained for 3 days, in packages containing crystallized TiO₂ are presented in Figure 1. The aspect of the cheese packed in polypropylene-crystallized TiO₂-polypropylene is quite different from that of the sample maintained in polypropylene-crystallized TiO₂ and is similar with that of the fresh prepared cottage cheese (white color, nice flavor). The color of the cheese maintained in polypropylene-crystallized TiO₂ package is also white, but the flavor is altered. In both samples, white-yellowish microorganism colonies are observed, but in the sample packed in polypropylene-crystallized TiO₂, the microorganisms are homogeneously

dispersed in all cheese samples, in comparison with the cheese maintained in polypropylene-crystallized TiO_2 -polypropylene, which is covered only with a thin layer of yellowish film of microorganisms.

The same color and flavor changes were observed also for the samples maintained in the packages containing amorphous TiO_2 (not show), after 3 days of storage.

The cheese kept in air became yellow and dried.

After 8 days, the cheese maintained in the polypropylene film became yellow (Figure 2), having an altered flavor and consisting in homogeneous dispersed mucilaginous areas, as result of the microorganisms activity. The cheese packed in polypropylene-amorphous TiO_2 bag (Figure 2) presents an aspect less altered than the above discussed sample, the mucilaginous areas are reduced and the smell is relative fragrant. The color, aspect and flavor of the cheese maintained in polypropylene-amorphous TiO_2 -polypropylene package are similar with those of the fresh prepared cottage cheese (Figure 2). The aspect, color and flavor of the cheese packed in bags containing crystallized TiO_2 (not show) are similar with those observed when the amorphous TiO_2 was used.

The cheese packed in the polypropylene film became completely mucilaginous after 11 days, having a yellow color and advanced altered flavor (Figure 3). The cheese packed in polypropylene-amorphous TiO_2 bag contains isolated cheese-like lumps. In the cheese maintained in polypropylene-amorphous TiO_2 -polypropylene, these lumps are predominant (Figure 3).

The conclusion is that the less altered cheese, after maintaining it, at room temperature, for 8 and 11 days, is that packed in the polypropylene- TiO_2 -polypropylene package. No macroscopic changes by using amorphous or crystallized TiO_2 were observed.

3.2. Physical-chemical characteristics of cheese

The photoactivity of TiO_2 , that confer it the preservation ability, was monitored by the acidity level. The increase of acidity, in time, demonstrates the cheese alteration. By analyzing Figure 4, the acidity of the cheese samples from all the investigated packages decreases in time, or has remained almost unchanged (samples maintained in polypropylene). The acidity decrease of the samples packed in bags containing TiO_2 could be explained by the fact that titania decomposes, through photocatalysis, the organic compounds from cheese, including those resulted from a slight lactic fermentation process.

The most intense decrease in acidity, after 8 days, was observed when packages containing crystallized TiO_2 were used (65.2 % for polypropylene-crystallized TiO_2 and 56.2 % for polypropylene - crystallized TiO_2 -polypropylene). The acidity decrease of samples packed in bags containing amorphous TiO_2 , after 11 days, was 39.13 % for polypropylene, 50.86 % for polypropylene-amorphous TiO_2 and 40% for polypropylene-amorphous TiO_2 -polypropylene. The decrease of acidity observed in the sample packed only in polypropylene is explained by the photolysis process which occurs during storage.

The chlorine content of the cheese samples from all types of packages decreases in time (Figure 5). By comparing the packages containing amorphous TiO_2 , one observes that the significant decrease appears when the polypropylene- TiO_2 -polypropylene package was used, being followed by polypropylene- TiO_2 structure. The significant reduce of the chlorine content achieved in the case of polypropylene bag using, can be explained by the photolysis process that occurs during the storage. Regarding to the values corresponding to the packages containing crystallized TiO_2 , one observes that the higher reduce was obtained when

polypropylene-TiO₂-polypropylene was used and only after 8 days of storage.

The fat content of the cheese samples from all types of packages decreases in time (Figure 6). By comparing the packages containing amorphous TiO₂, one observes that the significant decrease appears when the polypropylene-TiO₂-polypropylene package was used, being followed by polypropylene-TiO₂ structure. The lower reduce of the fat content was achieved in the case of polypropylene bag, due to the fact that the photolysis process occurs during the storage. Regarding to the values corresponding to the packages containing crystallized TiO₂, one observes that the most significant decrease was obtained when polypropylene-TiO₂-polypropylene was used and only after 8 days of storage (96.1 %).

The higher decrease of the investigated parameters, when the crystallized TiO₂ was used, even after a short period of storage, is explained by the fact that the crystallized TiO₂ presents higher photocatalytic activity than the amorphous titania [20].

3.3. Structural characteristics of cheese

An important aim of the study is to establish qualitatively and quantitatively if the TiO₂ penetrate the cheese. It is interesting to observe that the cheese packed, for 3 days, in polypropylene-amorphous TiO₂ bags contains not TiO₂ (Figure 7, spectrum black). According to Choi [21], the peaks at 750-1080 cm⁻¹ are assigned to Ti-O vibration. The peaks observed between 1243 and 1639 cm⁻¹ are typical IR band of CH₃ or CH₂ [21]. Small peaks observed between 3200 and 3400 cm⁻¹ correspond to O-H vibration [21]. In comparison with the sample maintained 3 days, that packed for 0 period higher than 3 days (8 and 11 days, respectively) in polypropylene-amorphous TiO₂ bags contains TiO₂, because in these spectra, peaks assigned to Ti-O vibration (at 1074.35 cm⁻¹ in red spectrum and at 1078.26 cm⁻¹ in blue spectrum) were detected.

Taking in consideration the fact that the peak height is proportional with the quantity of TiO₂ from cheese, we have calculated the height of the peak at 1074 cm⁻¹, corresponding to the Ti-O vibration (Table 1). So, we have presumed that an increase of the peak height suggest an increase of the TiO₂ quantity in cheese. Regarding to the results corresponding to the polypropylene – amorphous TiO₂ packages, one observes that the peak height decrease in time, meaning that the quantity of TiO₂ increase after 3 days of maintaining until 8 days, and then decrease.

FTIR spectra of the cheese samples maintained in polypropylene-amorphous TiO₂-polypropylene bags are presented in Figure 8. No peaks assigned to Ti-O vibration are observed in the black spectrum, corresponding to 3 days of storage. At 8 and more days, peaks at 1074 cm⁻¹ are observed, that are assigned to the Ti-O vibration. Also, in the case of polypropylene - amorphous TiO₂-polypropylene packages, one observes that the peak height decreases from 8 to 11 days (Table 1), suggesting that the TiO₂ quantity from cheese decreases in time. The peaks observed between 1200 and 1650 cm⁻¹ are typical IR band of CH₃ or CH₂ [21]. Small peaks observed between 3200 and 3400 cm⁻¹ correspond to O-H vibration [21].

The cheese samples packed in bags containing crystallized TiO₂ accumulate titania only after 3 days of storage (Figure 9, black and red spectra), while after 8 days of storage, no peaks corresponding to Ti-O vibration appear. As we expected, the height of the peak at 1074 cm⁻¹ is higher for polypropylene-crystallized TiO₂ package (2.77 %T) (Table 1), in comparison with polypropylene-crystallized TiO₂-polypropylene package (2.59 %T). Additionally, peaks at 1200 - 1650 cm⁻¹ typical IR band of CH₃ or CH₂ and small peaks between 3200 and 3400 cm⁻¹ corresponding to O-H vibration are observed.

Table 1. Values of the height (T %) corresponding to the peak appeared at 1078-1071 cm^{-1} from FTIR spectra, assigned to the Ti-O vibration (P-polypropylene, am-amorphous, cr-crystallized) [21]

	P-am TiO ₂	P-am TiO ₂ -P	P-cr TiO ₂	P-cr TiO ₂ -P
3 days	no peak	no peak	2.7	2.9
8 days	2.47	2.9	no peak	no peak
11 days	1.3	2.2	-	-

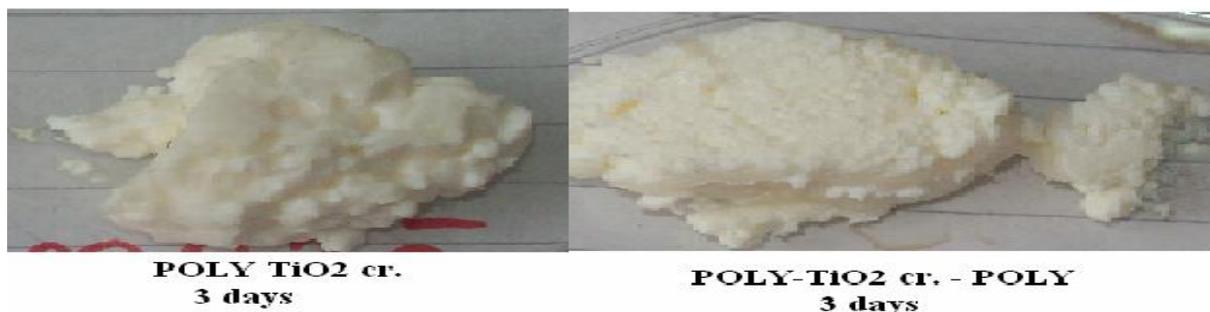


Figure 1. Macroscopic view of the cheese maintained for 3 days in the polypropylene-crystallized TiO₂ and polypropylene-crystallized TiO₂-polypropylene packages (poly-polypropylene film, cr.-crystallized)

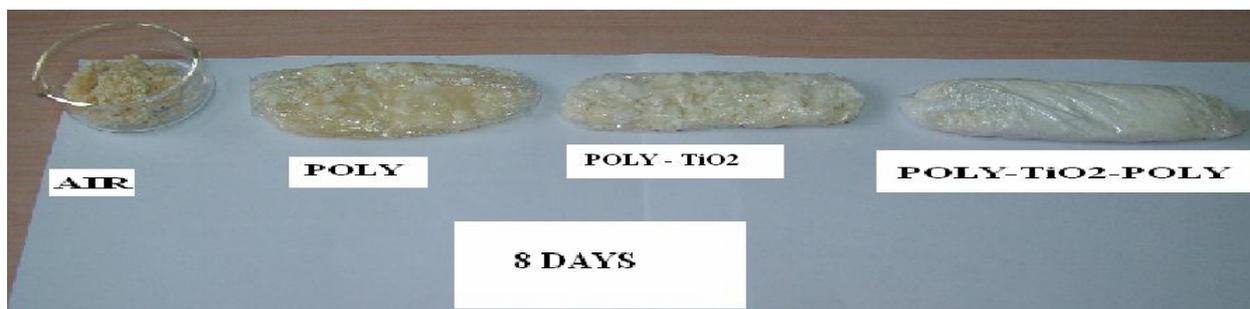


Figure 2. Macroscopic view of the cheese maintained for 8 days in the prepared packages containing amorphous TiO₂, in comparison with the same cheese kept in a glass vessel in air (poly-polypropylene film)



Figure 3. Macroscopic view of the cheese maintained for 11 days in the prepared packages containing amorphous TiO₂, in comparison with the same cheese kept in a glass vessel in air (poly-polypropylene film)

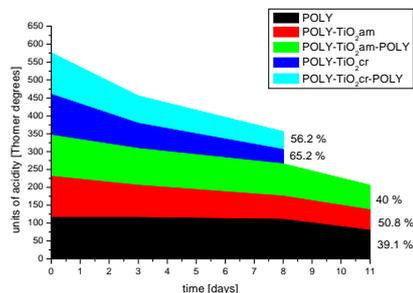


Figure 4. Variation of the acidity of the cheese samples packed in different packaging, in time (poly-polypropylene film, am-amorphous, cr-crystallized). The percents represent the acidity decrease, at the final of the experiment

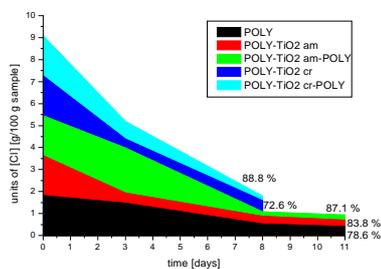


Figure 5. Variation of chlorine content of cheese samples packed in different packaging, in time (poly-polypropylene film, am-amorphous, cr-crystallized). The percents represent the chlorine content decrease, at the final of the experiment

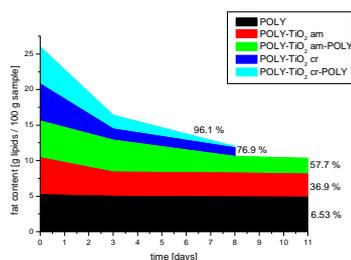


Figure 6. Variation of fat content of cheese samples packed in different packaging, in time (poly-polypropylene film, am-amorphous, cr-crystallized). The percents represent the fat decrease, at the final of the experiment

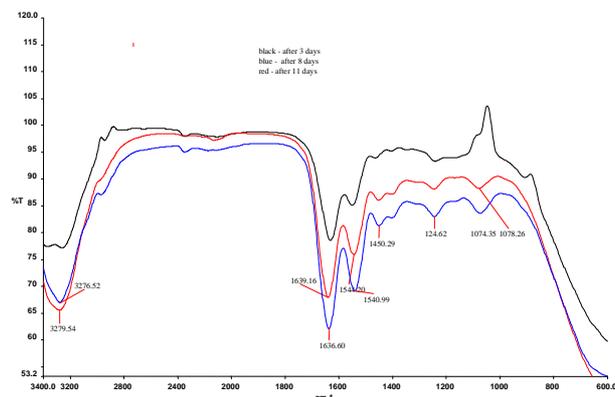


Figure 7. FTIR spectra of cheese packaged in polypropylene-amorphous TiO₂ bags, for different periods of time

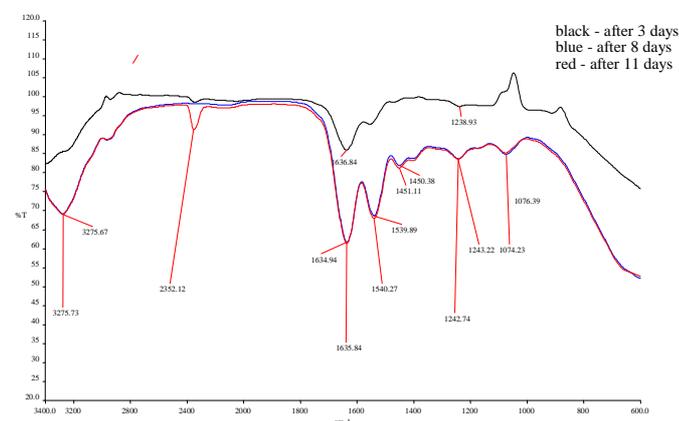


Figure 8. FTIR spectra of cheese packaged in polypropylene-amorphous TiO₂-polypropylene bags, for different periods of time

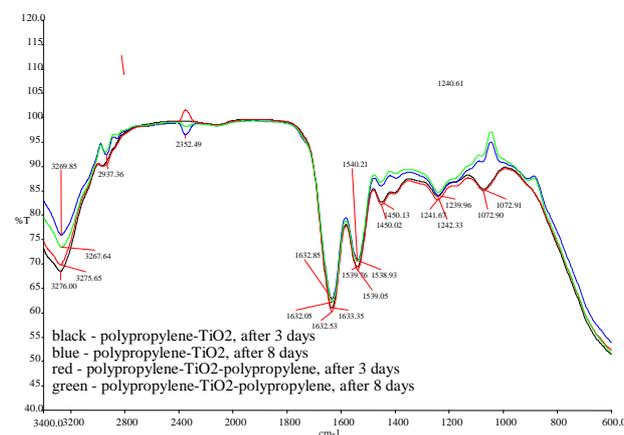


Figure 9. FTIR spectra of cheese packaged in bags containing crystallized TiO₂ and maintained 3 and 8 days, respectively

From those two packages containing crystallized TiO₂, the lowest quantity of titania was accumulated by the cheese packed in the polypropylene-TiO₂-polypropylene package, after 3 days.

After 11 days of storage, the lowest quantity of amorphous titania was accumulated by the cheese packed in the polypropylene-TiO₂ package.

4. Conclusions

- Keeping the cottage cheese in packages like polypropylene-crystallized TiO₂ and polypropylene-crystallized TiO₂-polypropylene, preserve its aspect, color and flavor.
- The highest acidity decrease was obtained when the cheese was packed in polypropylene-crystallized TiO₂ for 11 days.
- The highest chlorine and fat decrease were observed when the cheese was packed in polypropylene-crystallized TiO₂-polypropylene, for 11 days.
- The quantity of titania accumulated in the cheese decrease in time.
- Packages containing crystallized TiO₂ and moreover those having the structure polypropylene-TiO₂-polypropylene are the most efficient for cottage cheese preserving, from all the studied package types.

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STUDY ON IMPROVING PERFORMANCE PISTON PUMPS USED IN FOOD INDUSTRY

Ioan Radu Şugar^{1*}, Ana Maria Şugar²

1 - North University of Baia Mare, Faculty of Engineering, Department of Engineering and Technology Management, 430083- Baia Mare, Dr. Victor Babeş str., no. 62A, Romania, radusugar@yahoo.com
2-High School sports program Baia Mare, 430122- Baia Mare, B-dul Republicii str, no 3. Romania, anasugar@yahoo.com*

Abstract

Fluids are very important in achieving the technology, either through direct participation in various operations or to provide transportation between the machines. Many operations process of the food industry, handling products in a fluid state. Products are composed of liquid state and liquid state of solid state. Fluid transport by pipeline is done with pumps. Pumps are machines made of static pressure rise to movement of fluid from one place to another. Principle of operation is their mechanism of movement of the pumping element that the fate of the energy available at the drive shaft of the pump fluid pressure energy. By pumping means you are lifting the total energy of a fluid through a machine (pump), the purpose of transporting it. To this end, the pump energy is converted into mechanical energy for driving the hydraulic pump. The energy absorbed pump provides a flow and a certain lift. Piston pump pumping the liquid carried by the movement of the piston in the cylinder reciprocating pump, while successive synchronized, close and open valves on suction and discharge that fluid access to the pump, that pump out the liquid. Involvement of the piston can be achieved with an electric motor, internal combustion engine, steam engine, manual, etc. Determine shaft speed directly proportional flow pump, but does not affect the general discharge pressure. At the pump discharge pressure is determined by the pressure of the pumping, which determines the required pump power.

Keywords: *piston, pumps, valves, speed, liquid.*

1. Introduction

Simple effect piston pumps are piston pumps that have one active front in contact with liquid, the opposite side being in contact with air.

Pumping action of piston displacement occurs only in one direction, the reverse movement of the plunger is empty.

The Figure 1 is present in single-piston pump effect.

The movement of the piston to the right, the volume between cylinder and piston in front piston is increased, achieving depression. Due to the depression created by the valve 9 is closed while the valve 7 opens, allowing fluid into the cylinder inlet. The liquid will occupy a volume issued by moving its plunger.

The left piston displacement volume of liquid in a cylinder in front of the piston is subjected to pressure. The pressure created causes the suction valve closing and opening valve discharge July 9, allowing the liquid discharge pipe 8. Upon further displacement piston to the right will be the new liquid suction pump cylinder and the movement of his left discharge.

Piston pumps can have different shapes (Figure 1), depending on the nature of the liquid pumped, its temperature, viscosity, suction or discharge pressure, etc.

The movement of the piston to the right, the volume between cylinder and piston in front piston is increased, achieving depression. Due to the depression created by the valve 9 is closed

piston engine drive. Transmission movement is driven directly; the piston engine is driven by steam or compressed air. Direct-acting piston pumps can be single cylinder - Simplex - or by two parallel cylinders - duplex.

Flow pump. If we consider the operating principle of the pump, then a full rotation of the crank ϕ is carried in C by B aspiration, and the rotation of C in A through D is carried out repression, Figure 2.

In one full rotation, the piston makes a stroke and aspiring to the right flow of fluid Q .

The left piston stroke displacement and repression carried out the same flow of fluid intake. For speed of crank in the crank-slider mechanism:

$$Q = 60s'n A \text{ [m}^3\text{/h]} \quad (4)$$

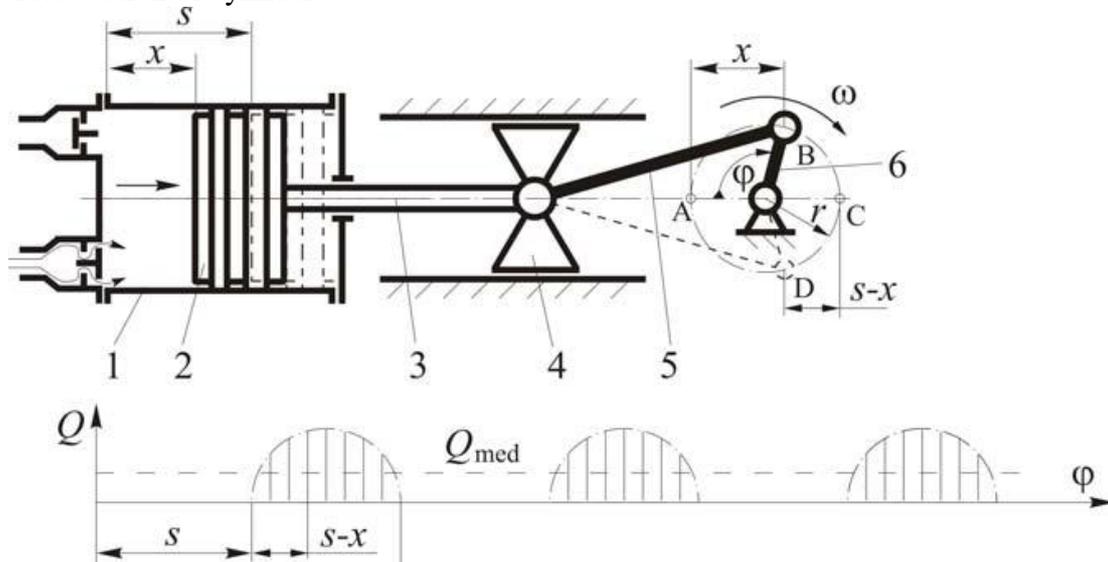
This flow rate is theoretically ideal to function properly. Actual flow piston pump is simple effect:

$$Q_r = \eta Q \quad (5)$$

where: η - pump volumetric efficiency.

$$Q = s.A \quad (3)$$

where: s - stroke, $s = 2r$
 A - cross section of cylinder



*Figure 2. Crank-sliding mechanism that drives the piston pump piston flow the simple effect [7]:
 1-cylinder, 2-piston-rod 3, 4-runners, five-rod, 6-lug*

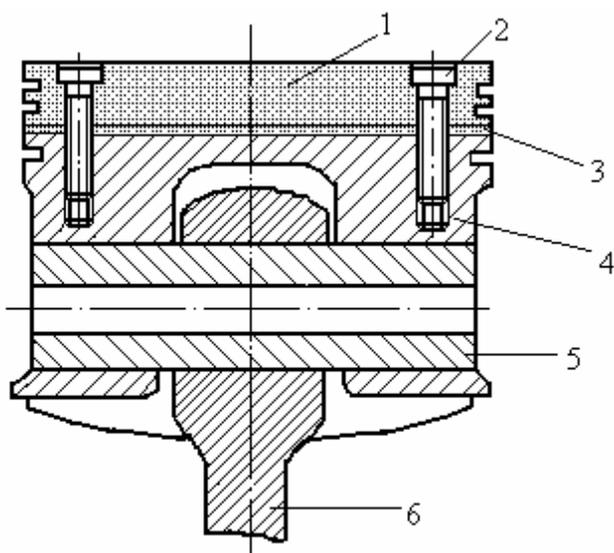
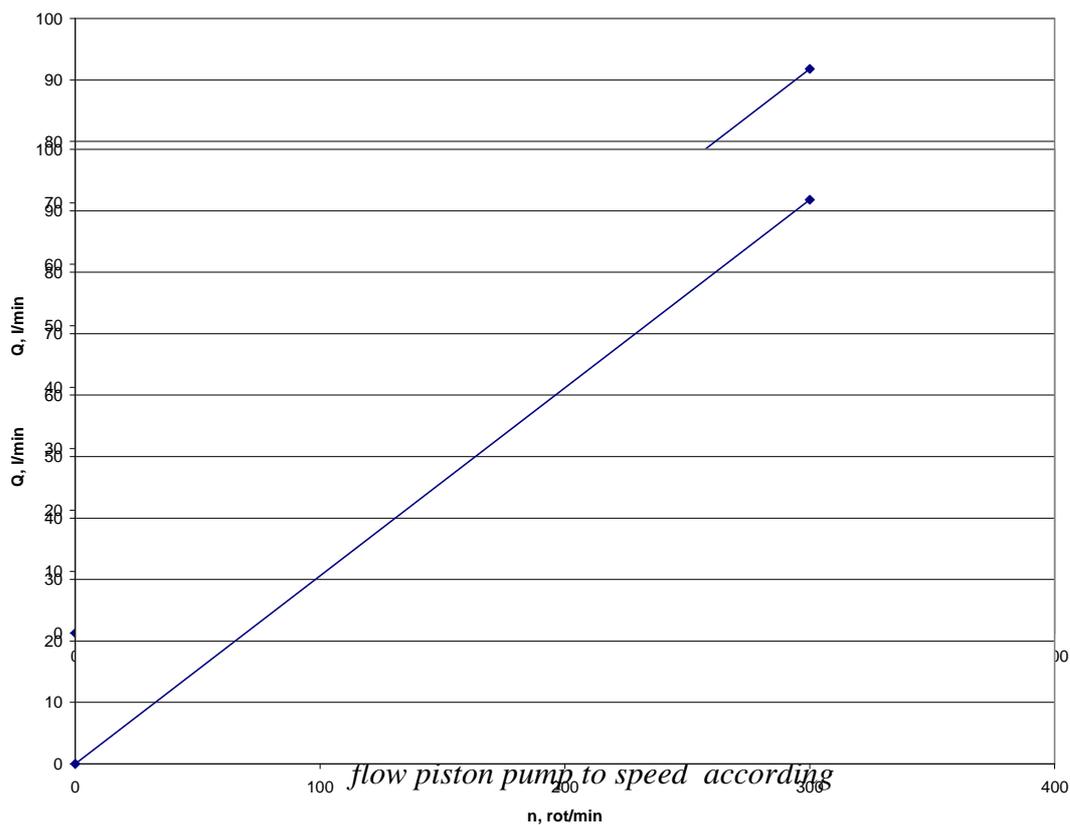


Figure 3. Structure of the piston head isolation laminated plate [5]:

- 1 – sintered plate of silicon nitride, 2-screws;
- 3 - aluminum titanate sintered plate;
- 4-piston, 5-pin, 6-rod



3. Results and discussions

The biggest drawback in the operation of simple effect piston pump is operating pulsed regime.

Unevenness of the flow pump is the ratio of maximum flow rate, Q_{max} and average flow, Q_{med} . For the simple effect piston pump $\delta = \pi = 3.14$. Drive speed can not exceed certain limits determined by the effects of hydraulic and mechanical proper functioning of the pump.

Pump drive speed is a key feature, directly influences pump parameters: flow, pressure, power, efficiency.

To isolate the head piston can use several constructive solutions. One of them is shown in Figure 3. In Figure 4, submitted to flow achieved if a piston pump has a diameter $D = 73$ and $s = 77$ race.

4. Conclusions

- Speed drive, which determines the speed of the piston and consequently the liquid, must take account of its characteristics: viscosity, density, temperature, pressure.
- Usually this type of speed pump system is located around 300 rpm.
- Volumetric efficiency takes into account primarily the inertia of the closing - opening the valve, then the leakage around the piston rings because they wear a shirt or cylinder, or even escape.
- Piston pump flow is directly proportional to crankshaft speed.
- To obtain high throughput can use several cylinders.
- By using composite materials in construction increases reliability and reduces piston its mass, as a beginning to increase efficiency.

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8. TISAN, V (2007). *Utilaje în Industria Alimentară*. Editura Risoprint, ISBN 978-973-751-1, Cluj-Napoca.

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