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APPLICATION OF TITRIMETRIC TECHNIQUE TO DETERMINE PHOSPHATES IN CHEESES

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Abstract: *A method of the titrimetric determination of phosphate (food additives E338 - phosphate acid, E339 - sodium phosphate, E340 - potassium phosphate, E341 - calcium phosphate, E450 - sodium and calcium pyrophosphate,) in cheeses has been developed. The research experimental results regarding the phosphate ion content in hard and spread cheeses (in terms of phosphate acid, %) obtained by means of the developed methodology are described. The correctness and accuracy of the results obtained by developed technique are confirmed by the definition of phosphates in the studied samples of cheeses by photometric method (by the method according to DSTU ISO 3360: 2008). A comparative analysis of the two methods has been performed.*

Keywords: *food additives, phosphates, hard and spread cheeses, titrimetric method, photometric method*

1. Introduction

Today, the use of biological and food additives in the food industry has become an integral component technology [1]. To improvement of the quality of many products and for perfection of the process in the milk industry the phosphates are used. Phosphorus-containing food additives are also used in the production of soft drinks, bakery, confectionery and meat products as stabilizers, acidity regulators, baking powder and a latches color. But most of them are used in the production of hard and spread cheeses. Food additives E338 (phosphate acid H_3PO_4), E339 (sodium phosphate NaH_2PO_4; Na_2HPO_4; Na_3PO_4), E340 (potassium phosphate KH_2PO_4; K_2HPO_4; K_3PO_4), E341 (calcium phosphate $Ca(H_2PO_4)_2$; $CaHPO_4$; $Ca_3(PO_4)_2$) E450 (pyrophosphates $Na_2H_2P_2O_7$; $Na_3HP_2O_7$; $Na_4P_2O_7$; $Ca_2P_2O_7$; $CaH_2P_2O_7$) have a variety of technological properties: soluble alkali phosphate have a pronounced buffer potential to regulate acidity; polyphosphates inhibit growth of gram-positive bacteria, which can significantly increase the shelf life of products; tricalcium phosphate used in the production of bakery products, special drinks, powdered and condensed milk, ice cream, fish and minced meat, alcoholic beverages, convenience foods, breakfast cereals, instant food, confectionery, baking powder, spread cheeses [2].

Phosphates are used as acidity regulator, emulsifier and stabilizer. These salts perform essential function in the production of cheese, because without salt-fin its production is impossible [2, 3].

However, phosphates are found in many foods and can cause adverse reactions (allergy), especially in children. The result of allergy is an altered mental reaction, impulsivity, motor restlessness, impaired concentration, increased aggressiveness

sometimes. Frequent use of food additives E338, E339, E340, E341 in food leads to disturbances in the gastrointestinal tract, vomiting, diarrhea, nausea and aversion to food, weight loss [3, 4]. If a healthy person with food gets a lot of phosphates in her body disturbed calcium metabolism, begins osteoporosis (bones become brittle and relatively easily broken). The products with the addition of phosphate are very dangerous for pregnant women because it is likely to have a child with a variety of disorders in the brain and respiratory system [5, 6].

According to the Committee on the rights of consumers over 80 % of food products, which are sold in the markets of Ukraine, falsified. Therefore, the development and improvement of methods of analysis, including analysis of the food additives content in the food products is an actual problem nowadays. For researches phosphates (food additives E338, E339, E340, E450, E452, etc.) traditionally are using the photometric method [7]. However, this method requires expensive reagents and instruments as well as highly qualified specialists. Not all laboratories can to have such complex providing. Volumetric analysis is a simple and affordable method, which is often used for studies of liquids. The aim of our investigation was to develop (or to adapt) known the titrimetric method of determination of phosphates in milk for research phosphates in hard and spread cheeses.

2. Materials and methods

The determination of phosphates in hard and spread cheeses by photometric method includes the next points: complete destruction of organic compounds under the action of sulfuric acid and hydrogen peroxide; addition of an ammonium molybdate solution in ascorbic acid; measurement of the optical density of blue phosphorus molybdenum complex [8]. The sample was prepared immediately before the definition. Sample of cheese 50±1 g was crushed and stirring until a pasty mass. The sample we put in a Kjeldahl Flask, added concentrated sulfuric acid and were carried out a mineralization: were heated to form the minimum foam by avoiding local overheating and the release of foam from flask; cooled to room temperature; added 3÷4 drops of solution of hydrogen peroxide and heated again. This procedure repeated for as long as the content of the flask will become uncolored and transparent.

To remove traces of hydrogen peroxide continued to heat the flask for 30 min, were cooled. The contents of the flask were transferred to a measuring flask of 100 cm^3 and volume was adjusted to the mark with distilled water. Using a pipette selected 1 cm^3 of obtained mineralize substance and put into measuring flask of 50 cm^3 volume; added a mixed solution of sodium molybdate and ascorbic acid. The volume of solution was adjusted to the mark with distilled water. The flask was put in a boiling water bath for 15 min.

After cooling, the optical density was measured at the wavelength 820 nm in cuvettes with optical path length of 10 mm.

The titrimetric method is simple, cheap and affordable. For develop a methodology of determining phosphates in cheeses by titrimetric method chosen method-prototype for milk.

To find the mass of samples cheeses were taken into account: 1) for determination of phosphates in milk for analysis selected 20 cm^3 of milk (known [9] that in 100 cm^3 of milk is 90÷100 mg of phosphorus, i.e. in 20 cm^3 of milk can be determined ≈20 mg of phosphorus); 2) based on acceptable standards: 100 g of spread cheese includes 2074 mg of phosphorus additives. The mass of cheese sample was found by calculation method:

$$x = \frac{100 \cdot 0{,}02}{2{,}074} \approx 1(g).$$

The sample preparation was carried out similarly as in the photometric definition. From the prepared samples were taken 1 g of cheese, added 20 cm^3 of 0.1 M solution of sodium carbonate.

Ashing in a muffle furnace is an integral part of the preparation of samples. The methodology for milk about ashing of samples says nothing. Based on experimental studies, we made adjustments. We propose before ashing samples in a muffle furnace to make the melting of the samples during slow heating (sand bath or electric stove), preventing the release of foam to full drying of cheese mass. Then, make the ashing in a muffle furnace at a temperature 600°C for 1÷2 min. Further analysis was performed according to the method for milk: an ash quantitatively transferred to a conical flask, was added 30 cm^3 of 0.1 M solution of hydrochloric acid. The solution were evaporated on a sand bath to a volume of ≈15÷20 cm^3 (all ash dissolves). According to the method for milk to solution add 2 drops of 0.1 % solution of methyl orange indicator. The contents of the flask was cooled and titrated by 0.1 M solution of sodium hydroxide to change color to yellow and then by drops add of 0.1 M solution of hydrochloric acid to a neutral reaction (orange color). Our correction: instead of indicator methyl orange to use indicator phenolphthalein. *It is necessary to pay special attention to bring the reaction to neutral.*

In liquid was added 20 cm^3 neutral with respect to phenolphthalein and methyl orange 40 % solution of calcium chloride. The liquid was boiled for 2 min, adding 1÷2 drops of phenolphthalein and titrated by 0.1 M solution of sodium hydroxide to change color from yellow to pink. Again were boiled for 2 min, titrated by 0.1 M solution of sodium hydroxide. This process repeats for as long as after heating, the solution remains pink. Then added 2 drops of phenolphthalein and titrated by 0.1 M solution of sodium hydroxide to change the color of the contents of the flask with pink to red. If color for 10 min weakens to add a few drops of titrant.

The content of phosphate acid (w, %) were calculated by the formula:

$$w_{H_3PO_4} = \frac{V_{NaOH} \cdot N_{NaOH} \cdot E_{H_3PO_4}}{m \cdot 1000} \cdot 100\%,$$

where V_{NaOH} - the amount solution of alkali (mean value), which went on titration, cm^3; N_{NaOH} - concentration of alkali, mol/dm^3; $E_{H_3PO_4}$ - molar equivalent weight of phosphoric acid (32.67 g/mol); m - mass of sample product, g.

3. Results and discussion

We carried the experimental determination of phosphate in the samples of hard and spread cheeses of known domestic manufacturers by the developed methodology. Were studied three samples of hard cheeses and three samples of spread cheeses. To confirm the correctness of the methodology and the reliability of the results, we conducted determination the content of phosphates in the same samples by photometric method according to [7]. The results are shown in Table 1.

Table 1

Phosphates in cheeses

Number of the sample	Phosphates (in terms of phosphoric acid). %	
	Titrimetric method	Photometric method
Hard cheeses		
1.	1.16±0.05	1.19±0.04
2.	1.58±0.02	1.60±0.05
3.	1.09±0.01	1.10±0.015
Spread cheeses		
4.	1.80±0.03	1.85±0.02
5.	1.40±0.025	1.37±0.03
6.	1.60±0.03	1.59±0.035

Comparative analysis of the results obtained by the developed method (titrimetric) and by the method according to [7] (photometric) show that the value of phosphates correlate with each other and differ only in the limits of errors. This clearly comparison chart demonstrates in Fig. 1.

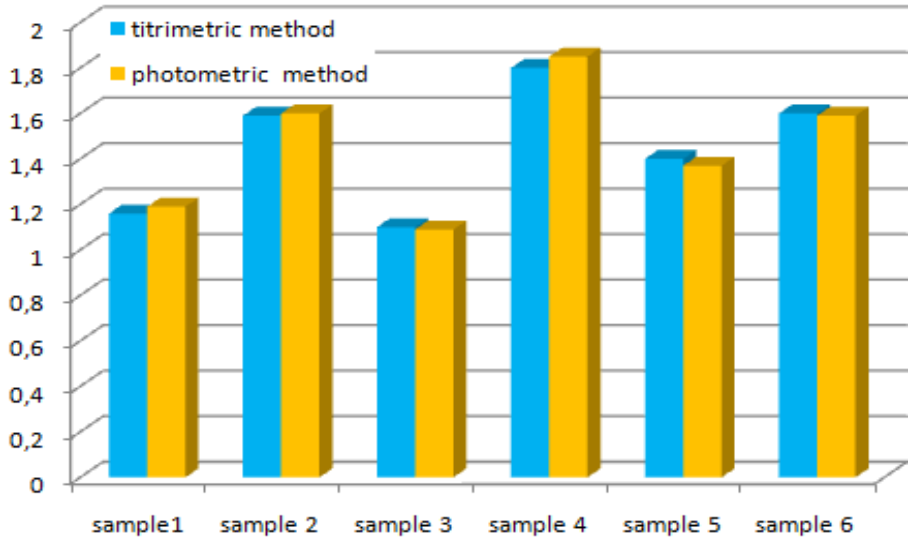

Fig. 1. The comparison chart of content of phosphates in the samples of cheeses

4. Conclusions

Based on the methodology of the titrimetric determination of phosphates in milk developed a method for the titrimetric determination of phosphates in cheeses. By calculation method found mass of the samples (1 g) made adjustments for sample preparation and analysis.

The correctness of the methodology and reliability of the results confirmed by determining the content of phosphate in the studied samples photometric method in according to DSTU ISO 3360: 2008

The method of titrimetric determination of phosphates can be used to determine their as well as in hard and in spread cheeses.

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