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EXPERTISE OF POTATO SNACKS BY OPTICAL MICROSCOPY, FTIR - SPECTROSCOPY, SPECTROFLUORIMETRY AND THIN LAYER CHROMATOGRAPHY

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Abstract. Expertise of four samples of potato snacks with the flavours *Barbecue, Ketchup, Cheese, Sour Cream and Onion* has been carried out with the use of analytical physicochemical methods. It has been found that the mass fraction of moisture in the samples under study ranges 4.4 to 4.7%, and the mass fraction of chlorides does not exceed 2.5%. The mass fraction of starch, which was determined polarimetrically, varies from 66.1% (Sample 2) to 66.7% (Sample 3). The mass fraction of sugars in the potato snack samples does not exceed 1.9%. None of the samples contains foreign material and mineral impurities. The product's physicochemical quality parameters established by the tests conform to the manufacturer's indication that the product contains such ingredients as starch and potatoes (starch and proteins are part of potatoes), oil, maltodextrin, sugars, salt, proteins. Based on characteristic absorption bands in the composition of snacks, FTIR has allowed detecting the presence of the two main components: triglycerides of fatty acids (oils) and polysaccharides (the latter include starches and maltodextrins). Optical microscopy has revealed that the samples have a porous (cellular) structure, with pores distributed throughout the whole mass, which is typical of snacks. Heat treatment of the snacks in the course of extrusion and frying changes the structure of starch to the glassy state: only single whole starch grains are found in the samples. The dietary supplement monosodium glutamate E621 has been identified by thin-layer chromatography on chromatographic plates Sorbfil AF-A-UV. The mobile phases were mixtures of ethanol and distilled water in the ratio 7:3, and n-butanol, acetic acid, and water mixed in the ratio 3:1:1. It has been shown that the turmeric extract E100(i), a natural yellow food colourant, being adsorbed on potato snacks, can be identified in them by its own spectra of diffuse reflection and luminescence spectra, without destroying the samples.

Keywords: expertise, potato snacks, optical microscopy, FTIR spectroscopy.

Introduction. Formulation of the problem

The country's environmental and food safety is a primary national concern. Unfortunately, nowadays, the food industry throughout the world is focused not only on manufacturing high-quality foodstuffs, but also on production of cheap ersatz food containing concentrates, preservatives, chemical colourants, nature-identical components, and chemical additives. These aspects of food security pose problems that should be solved. Today, the quality and safety of food is controlled mainly by classical analytical methods, which are laborious and time-consuming. So, development of simple, rapid, highly sensitive methods of determining the quality of food is a topical and promising task. The modern analytical chemistry techniques characterised by simplicity, expressiveness, high sensitivity, and in some cases selectivity, include the methods of ultraviolet-visible spectroscopy.

Analysis of recent research and publications

The English term *snack*, which has become an international word, denotes food the main purpose of which is helping to overcome quickly the feeling of hunger between meals. Depending on the recipe, snacks can have different merchandising names and be of different types: croutons, crackers, nuts, sweet bread straws, maizepuffs, crisps, biscuits, sunflower seeds, etc. A special place among them is occupied by potato snacks. Snack foods are considered healthy if they are low in fat and salt. Eating unhealthy snacks can lead to obesity, hypertension, and cardiovascular disease [1]. Potato snacks are products shaped like three-dimensional objects (shells, horns, spirals, etc.). They are made from a mixture of potatoe-based powdered raw materials fried in oil, with or without salt and various food additives and flavourings (fillers) added [1]. The quality and safety of products are formed at different stages of the technological process, and also depend on the conditions and terms of their storage and sale [2-7]. The paper [4] describes analytical methods of measuring various compounds present in potatoes (dry matter, starch, sugars, etc.) and their effect on the quality of potato snacks. Implementation of the HACCP system in production is mandatory, as it is the most effective of the known methods of obtaining safe products. According to the HACCP scheme provided by the manufacturer, the technological scheme of potato snack production includes: reception of raw materials, pellets, palm olein, spices, and condiments, storage of the raw materials, portioning the raw materials, frying, cooling and separation of oil, flavouring with spices, packaging the finished products. During the technological processes of extrusion (product formation) and frying, under the action of significant rapid changes in the parameters and of high temperatures, mechanical energy transforms into heat. This leads to changes, more or less profound, in the quality of the raw materials (protein denaturation, starch gelatinisation, and other biochemical changes). The nature and depth of the changes and their effect on the product quality depend on the technological mode and its duration [2]. Besides, the chemical composition of snacks is determined by frying (moisture removal): in oil, at above 150°C, the water contained in potatoes transforms into steam, and its expansion in volume expands the product, too, making it porous and crunchy [8]. State Standard of Ukraine (DSTU) 4608:2006 only provides for determination of such components of potato snacks as mass fractions of moisture, fat, and chlorides. In this regard, it is important to improve the existing tests and develop new modern analytical rapid methods of determining the physicochemical characteristics, structure, and composition of potato snacks.

The purpose of this research is to carry out an expert examination of potato snack samples and establish whether they comply with the quality requirements of regulatory documents and with the manufacturer's labelling. The **objectives** of the research:

1) to study the structure of the potato snack samples using optical microscopy and FTIR-spectroscopy;

2) to determine the physicochemical parameters of the potato snack samples;

3) to develop a rapid method of identifying sodium glutamate in the potato snack samples;

4) to develop a rapid method of identifying curcumin adsorbed on snacks.

Research materials and methods

Four commercial samples of potato snacks were selected for the study. They were produced by ITAL FOOD SA, had the flavours *Barbecue* (Sample 1), *Ketchup* (Sample 2), *Cheese* (Sample 3), and *Sour Cream and Onion* (Sample 4) and the net weight 50g in hermetically sealed packages. The manufacture date of the samples tested was the second half of 2019, and the shelf life was nine months.

The substances used for the study were n-butanol p. a. (Merck KGaA, Darmstadt, Germany), acetic acid puriss.p.a. (Azot, Ukraine), ethanol (Lux, Ukraine), n-hexane puriss.p.a. (Germany), ninhydrin to detect amino acids (Sigma-Aldrich).

Monosodium glutamate was identified by thin-layer chromatography using chromatographic plates Sorbfil AF-A-UV (Krasnodar, Russia). The mobile phases were mixtures of ethanol and distilled water in the ratio 7:3, and of n-butanol, acetic acid, and water in the ratio 3:1:1 respectively [9,10]. After the elution, the chromatographic plates were dried in an air stream, ninhydrin solution (0.3g of ninhydrin in 100 ml of n-butanol with 3 ml of acetic acid) was sprayed onto the surface of the plates, the plates were dried with warm air for 10–20 s and heated at 105°C for 5min to detect pink-purple spots of glutamate on the chromatographic plate.

The FTIR spectra were registered with a spectrometer Spectrum One (Perkin-Elmer). The spectrum resolution was 4 cm^{-1}, the number of scans 32.

The luminescence spectra were recorded with a Cary Eclipse spectrofluorometer (Varian, Australia) with a 150 W xenon lamp.

The diffuse reflection spectra in the coordinates F (R)=f (λ, nm), where F (R) is the Kubelka-Munk function, were registered on a spectrophotometer Lambda-9 (Perkin-Elmer) with a special attachment in cuvettes. The layer of the powder material under study was 3 mm thick in relation to the comparison sample MgO.

The morphological characteristics of snacks were examined by optical microscopy using a microscope MBS-10 and a biological microscope SM-6.

The moisture content was determined gravimetrically according to DSTU 7804:2015, the fat content was determined by n-hexane extraction in a Soxhlet extractor according to DSTU 4941:2008, the content of chlorides was determined argentometrically by the Mohr method according to DSTU 4939:2008, the starch content by polarimetry according to DSTU 4953:2008, the protein contentby Kjeldahl according to DSTU 7824:2015, the content of sugars by the permanganatometric method according to DSTU 4954:2008, the mineral impurities content by flotation in water according to DSTU 4913:2008.

Results of the research and their discussion

According to the manufacturer, potato snacks consist of 35% of dehydrated potatoes, potato starch, salt, turmeric extract, vegetable oil, palm olein, and condiments: 6% of barbecue, 6%of ketchup seasoning, 4% of cheese seasoning, and 5% of sour cream and onion seasoning.

According to the manufacturer, the samples of potato snacks contain turmeric extracts. Turmeric extracts are those containing the natural yellow food colourant curcumin E100 (i). The colourant is obtained by extraction from a powdered turmeric rhizome (*Curcuma longa*). The colourant consists of such curcuminoids as curcumin (the main component), demethoxycurcumin, and bisdemethoxycurcumin, which are polyphenols by their chemical nature [11].

Samples of snacks have the form of fragile hollow parallelepipeds (Fig. 1). The product is yellow, with orange and brown inclusions on the surface. The samples have the taste of fried potatoes and flavours corresponding to the additives, and are slightly salty.

Optical microscopy has revealed that the samples have a porous structure, which appears during intensive water evaporation, the pores are distributed throughout the whole mass of snacks, which is typical.

The walls of the pores formed are hard and become fragile. When snacks are powdered, sharp-cornered particles are formed, which is characteristic of the glassy state of starch (see Fig. 2).

Heat treatment of snacks in the course of extrusion and frying changes the structure of starch: only single whole starch grains are found in the samples [12,13].

To assess the quality of the potato snack samples studied, their physicochemical parameters have been quantified: the content of fats, proteins, carbohydrates, moisture, sugars, starch, etc.

The physicochemical parameters of the snacks are shown in Table 1.

As can be seen from the table, the moisture content in the test samples ranges from 4.4 to 4.7%. The content of chlorides does not exceed 2.5%. There are no standards for the starch content in potato snacks. Its mass fraction has been determined polarimetrically and ranges from 66.1% (Sample 2) to 66.7% (Sample 3). Sugars in the samples do not exceed 1.9%. Thin-layer chromatography has revealed that all samples contain monosodium glutamate as a flavour enhancer. In all samples, there is no foreign material and mineral impurities. The tests conform to the manufacturer’s indication that the product contains such ingredients as starch and potatoes (starch and proteins are part of potatoes), maltodextrin (a product ofstarch breakdown into oligosaccharides, maltose, glucose), salt, monosodium glutamate, oil [14].

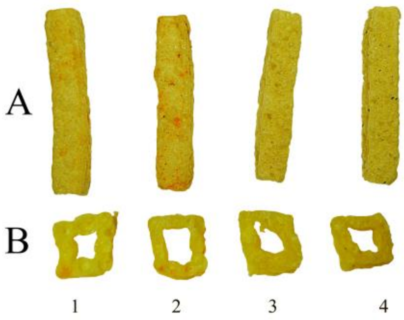

Fig. 1. Outward appearance of the samples (A), and their cross-sections (B): *Barbecue* (Sample 1), *Ketchup* (Sample 2), *Cheese* (Sample 3), and *Sour cream and onion* (Sample 4)

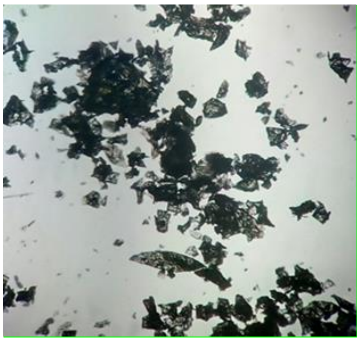

Fig. 2. Microphoto of a powdered potato snack, magnification 28x

Table 1 – Physico-chemical parameters of the snacks (n=2, P=0.95)

Name of the indicator	Requirements DSTU 4608:2006	Sample 1	Sample 2	Sample 3	Sample 4
Moisture content, %	≤5,0	4,6±0,1	4,4±0,1	4,6±0,1	4,5±0,1
Fat content, %	≤33,0	25,56±0,04	25,23±0,04	24,80±0,03	25,17±0,05
Chlorides content, %	≤3,0	2,51±0,03	2,46±0,01	2,44±0,04	2,37±0,04
Starch content, %	-	66,4±0,2	66,1±0,2	66,7±0,2	66,2±0,2
Proteins content, %	-	3,24±0,18	3,41±0,09	3,09±0,18	3,37±0,13
Sugars content, %	-	1,88±0,01	1,92±0,01	1,75±0,01	1,83±0,01
The presence of monosodium glutamate		present			
The presence of impurities	not allowed	absent			
Mineral impurities content, %	≤0,01	absent			

Fig. 3 presents the IR spectra of oil extracts of the samples, which were obtained by determining the mass fraction of fat according to Soxhlet. The IR spectra contain absorption bands of fatty acid triglycerides (oils): absorption bands with maxima at 3005 cm^{-1}, 2924 cm^{-1} (asymmetric stretching vibrations of aliphatic CH_2-groups), 2853 cm^{-1} (symmetric stretching vibrations of aliphatic CH_3- and CH_2-groups), 1746 cm^{-1} (stretching vibrations of –C=O group), 1465 cm^{-1} (deformation vibrations of aliphatic CH_3- and CH_2-groups), 1377 cm^{-1} (deformation vibrations of CH_3- groups), 1237 cm^{-1} (stretching vibrations of C-O group), 1164 cm^{-1} (stretching vibrations of C-O group), 1100 cm^{-1} (stretching vibrations of the C-O group in –C-O-C-), 723 cm^{-1} (rocking vibrations of CH_2-groups) [15].

Fig. 4 shows the IR spectra of powdered potato snacks defatted with n-hexane and dried at 105°C until complete removal of the solvent.

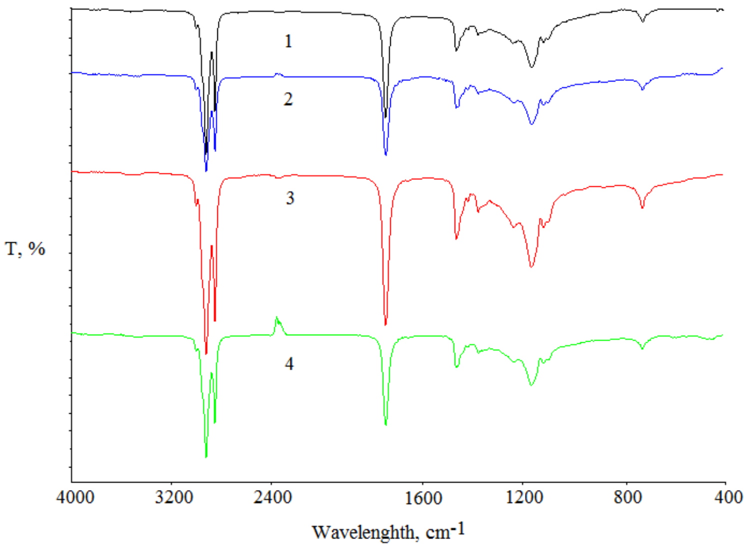

Fig. 3. IR spectra of oil extracts from the potato snacks *Barbecue* (1), *Ketchup* (2), *Cheese* (3), and *Sour Cream and Onion* (4)

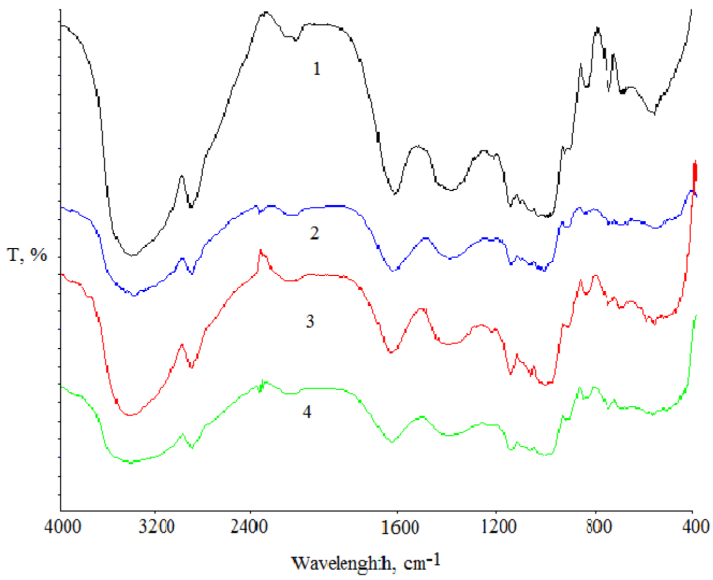

Fig. 4. IRspectra of defatted powdered potato snacks *Barbecue* (1), *Ketchup* (2), *Cheese* (3), and *Sour Cream and Onion* (4)

The IR-spectra of defatted snack powders contain absorption bands of polysaccharides (mashed potatoes, potato starch and maltodextrin): absorption bands with maxima at 3420 cm^{-1} (stretching vibrations of -OH groups), 2927 cm^{-1} (stretching vibrations of CH-groups), 1645 cm^{-1} (vibrations of OH groups of water and torsional vibrations of CH_2 -groups), 1460 cm^{-1} (deformation vibrations of CH-groups), 1381 cm^{-1} (symmetrical deformation vibrations of CH-groups), 1242 cm^{-1} (deformation vibrations of -OH groups), 1157 cm^{-1} (asymmetric stretching vibrations of -C-O-C- groups), 1080 and 993 cm^{-1} (stretching vibrations of C-O groups), absorption bands in the range between 1080 and 990 cm^{-1} are characteristic absorption bands of polysaccharides related to deformation vibrations of C-O-C groups and OH groups, 929, 858, 763 cm^{-1} (vibrations of C-O-C groups of the carbohydrate ring) [15-17].

The turmeric extract in the samples analysed has been identified by means of the diffuse reflection spectra (Fig. 5) and luminescence spectra of curcumin (Fig. 6). Since the diffusion reflection spectra and the luminescence spectra of potato snack samples are identical, in the figures, they are only shown for Sample 1. Depending on the nature of the organic solvent, the absorption maxima of the curcumin solutions are 410–430 nm, whereas the luminescence spectra of curcumin are 460–560 nm [18,19].

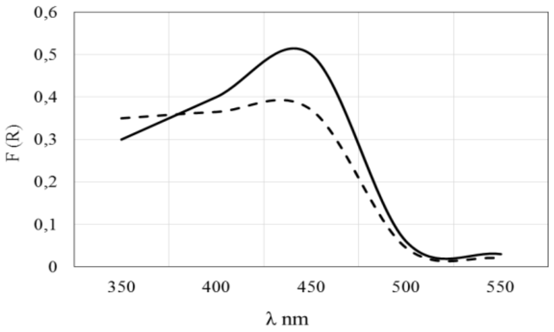

Fig. 5. Diffuse reflection spectra of the turmeric extract applied onto the potato starch powder (1) and the powdered defatted potato snack of Sample 1 (2) (λ_{max}=430nm)

The diffuse reflection spectra of the defatted powdered potato snack are broad bands with the maxima 430 nm and coincide with the maxima of the spectra of the curcumin extract applied onto the potato starch powder (curcuminoids were applied onto the potato starch from ethanolic solutions by removing the solvent). The intensity of the absorption bands of adsorbates, which is expressed in the units of the Kubelka-Munk function F(R), is proportional to the content of adsorbate molecules.

The luminescence spectra of the powdered defatted potato snack are broad bands with the maxima 524 nm and coincide with the maxima of the luminescence spectra of the curcumin extract applied onto the potato starch powder. Thus, it has been shown that diffuse reflectance spectroscopy and luminescence spectroscopy can be used as a rapid non-destructive method of qualitative determination of curcumin in potato snack samples.

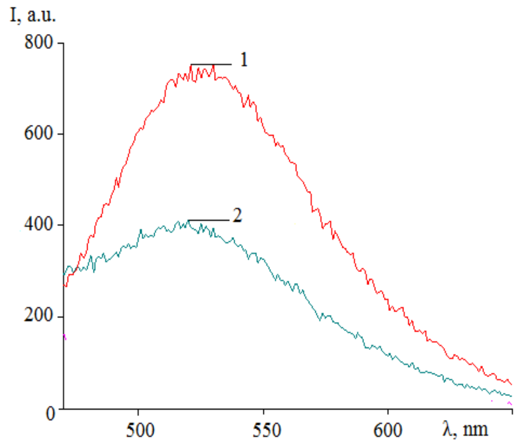

Fig. 6. Luminescence spectrum of the turmeric extract applied onto the potato starch powder (1) and the powdered defatted potato snack of Sample 1 (2) (λ_{ex}=450nm)

Conclusion

Expert examination of four samples of potato snacks with the flavours *Barbecue*, *Ketchup*, *Cheese*, *Sour Cream and Onion* has been carried out with the use of analytical physicochemical methods. The composition of the samples has been studied, and the content of nutrients and food additives has been determined. The product's physicochemical quality parameters established by the tests conform to the manufacturer's indication that the product contains such ingredients as starch, proteins, oil, potatoes, sugars, salt. Based on characteristic absorption bands in the composition of snacks, FTIR has allowed detecting the presence of the two main components: triglycerides of fatty acids (oils) and polysaccharides (starches and maltodextrins). Optical microscopy has revealed that the samples have a porous structure, with pores distributed throughout the whole mass, which is typical of snacks. The dietary supplement monosodium glutamate has been identified by thin-layer chromatography. It has been shown that the turmeric extract E100(i), a natural yellow food colourant, being adsorbed on potato snacks, can be identified in them by its own spectra of diffuse reflection and luminescence spectra, without destroying the samples.

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ЕКСПЕРТИЗА КАРТОПЛЯНИХ СНЕКІВ МЕТОДАМИ ОПТИЧНОЇ МІКРОСКОПІЇ, FTIR-СПЕКТРОСКОПІЇ, СПЕКТРОФЛУОРІМЕТРІЇ ТА ТОНКОШАРОВОЇ ХРОМАТОГРАФІЇ

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Анотація. Проведено експертизу аналітичними фізико-хімічними методами чотирьох зразків картопляних снеків зі смаками «Барбекю», «Кетчуп», «Сир», «Сметана та цибуля». Вивчено склад зразків, визначено вміст поживних речовин та харчових добавок. Встановлено, що масова частка вологи у досліджуваних зразках коливається в межах 4,4–4,7%, хлоридів – не перевищує 2,5%, поляриметричним методом визначено масову частка крохмалю, яка складає від 66,1% (зразок 2) до 66,7% (зразок 3). Масова частка цукрів у зразках картопляних снеків не перевищує 1,9%. У всіх зразках відсутні сторонні і мінеральні домішки. Тести на фізико-хімічні показники якості продукції не суперечать інформації виробника товару про присутність таких інгредієнтів як крохмаль та картопля (крохмаль та білки входять до складу картоплі), олія, мальтодекстрин, цукри, сіль, білкові речовини. Методом FTIR-спектроскопії по характеристичним смугам поглинання у складі снеків встановлено присутність двох основних компонентів: тригліцеридів жирних кислот (олії) та полісахаридів до яких відносяться крохмалі, а також мальтодекстрини. Методом оптичної мікроскопії встановлено, що зразки мають пористу (комірчасту) структуру, пори розподілені по всій масі, що характерно для снеків. При термообробці під час екструзії та обжарюванні снеків змінюється структура крохмалю: у складі зразків виявлені лише поодинокі цілі зерна крохмалю. Основна маса крохмалю знаходиться у склоподібному стані. Харчову добавку глутамат натрію Е621 ідентифіковано методом тонкошарової хроматографії на хроматографічних платівках Sorbfil AF-A-UV, в якості мобільних фаз використовували суміші етанолу та дистильованої води у співвідношенні 7:3 та н-бутанолу, оцтової кислоти та води у співвідношенні 3:1:1. Показано можливість ідентифікації натурального харчового жовтого барвника – екстракту куркуми Е100(і) в картопляних снеках по власним спектрам дифузного відбиття та спектрам люмінесценції без руйнування зразків, в адсорбованому на снеках стані.

Ключові слова: експертиза, картопляні снеки, оптична мікроскопія, FTIR-спектроскопія.

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