Розглянуто визначення токсичних елементів методами атомно-абсорбційної спектрометрії з атомізацією у полум'ї, колориметричним атомно-абсорбційним методом. Проведено визначення значень питомої об'ємної активності γ-випромінюючих радіонуклідів у напівкопчених ковбасах з використанням м'яса птиці, сочевиці, чебрецю та ялівцю. Результатами дослідження встановлено, що за вмістом токсичних елементів та радіонуклідів розроблені напівкопчені ковбаси відповідають вимогам чинної нормативно-технічної документації України

Ключові слова: пробо підготовка, мінералізація, фоновий розчин, атомно-абсорбційна, спектрофотометр, важкі метали, радіонукліди, рецептури, напівкопчені ковбаси, сочевиця, чебрець, ялівець

Рассмотрены определения токсичных элементов методами атомно-абсорбционной спектрометрии с атомизацией в пламени, колориметрическим атомно-абсорбционным методом. Проведено определение значениях объемной активности γ-излучающих радионуклидов в полукопченых колбасах с использованием мяса птицы, чечевицы, чабреца и можжевельника. Результатами исследования установлено, что по содержанию токсичных элементов и радионуклидов разработанные колбасы соответствуют требованиям действующей нормативно-технической документации Украины

Ключевые слова: подготовка проб, минерализация, фоновый раствор, атомно-абсорбционная, спектрофотометр, тяжелые металлы, радионуклиды, рецептуры, полукопченые колбасы, чечевица, тимьян, можжевельник

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A STUDY OF TOXIC ELEMENTS AND RADIONUCLIDES IN SEMI-SMOKED SAUSAGES MADE WITH LENTILS, THYME, AND JUNIPER

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1. Introduction

In making environmentally friendly products, an important factor is the microelement composition, which used to be considered mainly from the point of view of biological necessity. Human activity has led to an increase in the pollution of the environment, a redeployment of trace elements, and contamination of agricultural products with toxic substances. This has fostered a new direction in the study of trace elements to determine their toxicity and to search for ways of reducing their content in slaughter products and meat products from them [1].

Heavy metals are common toxic pollutants. They are widely used in a variety of industries. However, measures to prevent their entry into the environment are insufficient, so compounds of heavy metals penetrate into industrial waste water. A significant number of these compounds enter the water through the atmosphere. The ecological danger of heavy metals is that they are actively absorbed by phytoplankton and then transmitted to the human body through the food chain [2].

There are two groups of metals, which differ in ecological significance. The first group consists of elements the maximum allowable concentrations (MACs) of which are close to the background values in natural water. It is iron, manganese, and strontium. The second group includes metals of the MACs that significantly exceed the natural background values (copper, zinc). Metals such as vanadium, iron, cadmium, calcium, cobalt, magnesium, manganese, molybdenum, sodium, nickel, tin, chromium, and zinc are necessary for the human body, but their increased amount in the human body leads to health problems. The toxicity in the corresponding gratifications for man is produced by aluminum beryllium, cadmium, copper, arsenic, nickel, lead, silver, strontium, mercury, and chromium. For the normal functioning of a person, it is necessary to achieve a balance of trace elements the violation of which can cause serious diseases and poisoning [3].

According to the sanitary norms of quality of food raw materials and food products, toxic elements that are controlled in food products include lead, cadmium, arsenic, mercury, copper, and zinc [4, 5]; after getting into the human body, heavy metals can produce toxic effects. Therefore,

sausage products should comply with the safety indices of the normative documents, and research on the contents of semi-smoked sausages that include beef, poultry, lentil flour, thyme, and juniper is essential.

2. Literature review and problem statement

Research on the characteristics of specific toxic metals indicates that in agriculture mercury is associated with the use of fungicides; emissions into the atmosphere also happen in the zones of manufacturing cellulose. The first manifestation of mercury toxicity in humans is acute renal failure [6]. Cadmium is associated with the widespread use of phosphates in agriculture and emissions of wastes from the electronic and paint industries. When cadmium enters the body, it concentrates in the liver and kidneys [7]. Lead is one of the most widespread hazardous pollutants in the environment. Annual industrial and transport emissions account for about 400,000 tons [6, 7]. Lead disrupts the nervous, digestive, cardiovascular and endocrine systems, causing a disorder of many metabolic processes [6, 7].

The issue of control of meat products for the content of heavy metals receives special attention. Concentrations of elements of aluminum, arsenic, cadmium, chromium, cobalt, copper, lead, mercury, nickel, selenium, and zinc penetrate into meat with contaminated air and vegetation [8]. In particular, according to the results of researching meat of wild and livestock [9] animals, it has been established that the presence of these elements does not present any danger to the health of consumers as their number does not exceed the maximum permissible concentrations [10].

The level of accumulation of certain heavy metals, in particular Pb, Cd, Hg, and As, was also determined in sheep meat and liver. According to the results of the study of the longest muscle in back and liver samples, it has been established that the age of sheep decreases the content of water in meat, but the contents of protein, fat and ash increase; the accumulation of Cd and Pb depends on the age of animals [11].

An easy and fast method for extracting solids to separate and determine the pre-concentration of heavy metals in food products is atomic absorption spectrometry [12].

With this method, food research on the content of Pb, Cd, Hg, and As is carried out in the trading networks in Serbia and Spain. It has been found that the presence of these elements in products leads to their consumption of 72.30 μg per day by an adult; As and Cd are consumed in the amounts of 21.89 μg and 11.51 μg [13, 14].

No less important is research on heavy metals in sausage products; in particular, the control of dry fermented sausages is carried out in Lisbon (Portugal), where samples for research are taken out on local markets. According to the research results, heavy metals are found in sausages, but they do not pose a danger to consumers [15].

In the Kingdom of Saudi Arabia, the concentration of heavy metals was estimated using an atomic absorption spectrophotometer. According to the research findings, the concentrations of basic metals exceed the recommended maximum acceptable levels proposed by the Joint FAO/WHO Committees and the EU Committee. The sausage products were found to contain the highest values of Fe, Mn, Cu, Zn, Pb, Cd, and Hg [16, 17]. Also, the control of sausage products for compliance with safety indicators is carried out in Ukraine, in particular, by Lviv Oblast State Laboratory of the State Consumer Protection Service in Lviv Oblast (Ukraine) [18].

3. The aim and objectives of the study

The aim of the research is to determine the content of toxic elements and radionuclides in semi-smoked sausages made with the use of beef, poultry, lentils, thyme, and juniper, using techniques developed by Lviv Oblast State Laboratory of the State Consumer Protection Service in Lviv Oblast. This will detect the concentration of heavy metals and radionuclides in semi-smoked sausages at minimal cost.

The main objectives of the work are the following:

– to determine the contents of toxic elements in sausages, using SHF-mineralization for further studying by atomic absorption spectrometry with electrothermal atomization;

– to specify the content of radionuclides by recording the scintillation spectra of γ-radiation, which isolates the test substances with subsequent processing by means of computer equipment;

– to establish the conformity of the produced semi-smoked sausages with lentil flour, thyme, and juniper to normative documents on the content of toxic elements and radionuclides.

4. Materials and methods for studying toxic elements and radionuclides

Toxic elements in the semi-smoked sausages were determined by the method developed in [18] by Lviv Oblast State Laboratory of the State Consumer Protection Service in Lviv Oblast (Ukraine) on the basis of the existing regulations, GOST 30178-96, GOST 2692 9-94, and MVI 77-12-97.

The method was developed by LOSL [18] of the State Consumer Protection Service in Lviv Oblast on the basis of GOST 30178-96, GOST 2692 9-94, and MVI 77-12-97 by using Varian AA240FS Atomic Absorption Spectrophotometer (Agilent Technologies, USA) and Shimadzu AAS-6300 Atomic Absorption Spectrophotometer (Shimadzu, Japan).

In a more detailed way, the method for studying toxic elements and radionuclides is presented in [19].

The needs of the population in meat products of high quality with attractive appearance, taste, technological properties, and high nutritional value are increasing. It stimulates the production of high-quality sausage products that are able to satisfy the demand of various categories of consumers.

In the recipes for new types of semi-smoked sausages, we offer the use of trimmed beef and veal of grade 1 in the amount of 40 kg per 100 kg of meat and poultry. For improvement of technological parameters and enrichment of the chemical composition of the finished products, flour of non-sprouted and sprouted lentils was used in the amounts of 1 kg, 1.5 kg, and 2 kg per 100 kg of meat raw material. Since the previous research confirmed the antioxidant properties of thyme and juniper and the expediency of their use, in order to improve the taste qualities of sausage products, spices were supplemented with minced thyme and juniper in the following ratios: 80:10, 70:20, and 60:30 g per 100 kg (Table 1).

Table 1

The recipes for semi-smoked sausages using flour of non-sprouted and sprouted lentils as well as minced thyme and juniper

Raw materials	Sample 1 – *Osoblyva Simeyna*	Sample 1. 1 – *Osoblyva Simeyna Spiced*	Sample 2 – *Osoblyva Sambirska*	Sample 2. 1 – *Osoblyva Sambirska Spiced*	Sample 3 – *Osoblyva Stryiska*	Sample 3. 1 – *Osoblyva Stryiska Spiced*
	kg per 100 kg of meat					
Beef, trimmed, grade 1	40	40	40	40	40	40
Poultry meat	29	29	28.5	28.5	28	28
Pork breast, in pieces not exceeding 6 mm	30	30	30	30	30	30
Flour of sprouted lentils	1	–	1.5	–	2	–
Flour of non-sprouted lentils	–	1	–	1.5	–	2
Total	100	100	100	100	100	100
Spices	kg per 100 kg of meat					
Salt	2.000	2.000	2.000	2.000	2.000	2.000
Sugar	135	135	135	135	135	135
Pepper, black or white, minced	90	90	90	90	90	90
Thyme minced	80	80	70	70	60	30
Juniper, minced	10	10	20	20	30	30
Garlic, fresh, peeled, minced	200	200	200	200	200	200
Sodium nitrite	0.075	0.075	0.075	0.075	0.075	0.075

In the technology of sausage production, sodium nitrite is used in its minimum amount, which is necessary to create a normal color of the product, because the pH of the cooled meat and the oxidation-reducing potential are low. Sugar is used to improve the color of the product and the aroma content of spices.

According to the developed recipes, the technological scheme of the new types of semi-smoked sausages is as follows.

In the process of preparing the raw material, the reception, cleaning and disassembly of the semi-carcasses is carried out. The raw material is taken from semi-carcasses, which are subject to mandatory weighing. The meat is taken to be cooled at the stage of technological maturity, when the temperature in the thickness of the muscles is 25–28 °C. The semi-carcasses are divided into three parts: front, middle and rear.

The meat is deboned manually with a knife on standard conveyor tables. During trimming, small bones, cartilages, and coarse connective tissues (fat in pork) are removed. Chicken is used cooled.

For mincing the raw material, beef is cut into pieces up to 1 kg. In order to increase the moisture binding capacity, cold meat is salted in the amount of 2 kg of salt per 100 kg of meat. Salted meat is kept at a temperature of 0–4 °C in pieces for up to 48 hours.

Beef and chicken are minced on the mincer (with a diameter of a grate opening being of 8–12 mm). After pork breast is added, the other added ingredients are prepared lentil flour and sodium water with nitrite. The temperature of the prepared minced meat is monitored not to exceed 8–10 °C for the stirring time of 6 minutes. The prepared minced meat is used to stuff natural casings by using a vacuum syringe. Particular attention should be paid to stuffing the mixture into natural casings the benefits of which are elasticity, the ability to shrink, retention of properties in a wet state, optimal adhesion properties, good moisture and smoke penetrability, as well as resistance to the necessary thermal regimes. The minced stuffing in the loaves should be dense without air cavities and bubbles. Excessive filling density of a loaf may lead to a rupture of the casing during the heat treatment process.

The loaves are tied in accordance with the product characteristics. In this case, the contents of one edge of the loaf are pushed to the middle of the casing with a twine that is tied to its end. For hanging the loaves on a stick, loops are made on one of the edges of the loaves. The labels are made with trademarks by means of crosswise twine manually. The minimum length of the loaves should not be less than 15 cm, and the free ends of the casing and the twine should be no more than 2 cm. In places of air accretion, the loaves are punctured with a special metal puncture that has 4–5 metal needles.

The loaves are hung on round and smooth sticks so that they do not touch one another. The sticks are placed on universal frames, which are moved to the chamber for deposition for 2–4 hours at a temperature of 2–4 °C.

Roasting is carried out in special universal thermal chambers with the control of temperature and time parameters. Roasting is done at a temperature of 100 °C for 70 minutes. After the roasting, the temperature in the middle of a

loaf is not less than 40 °C. As a result, the casing acquires a light brown color and becomes compacted.

Roasted loaves are subjected to cooking at a temperature of 75–85 °C for 60–80 minutes. (The temperature inside the loaf is 71 °C).

After the heat treatment, part of the microflora remains in the finished products, and with sufficiently high temperature of the meat products, the microorganisms begin to develop actively. To prevent this, the cooked sausages are cooled in a shower for 15 minutes. The water temperature is 15–16 °C.

After that, the sausages are cooled for 2 hours by air for the temperature in the middle of a loaf to become 15 °C at a temperature of 0–8 °C and a relative humidity of 96 %.

In order to check the products for compliance with the standard, quality control was carried out. The cooled sausages were placed in the refrigerator for storage in a hanging condition at a temperature of 0–8 °C and a relative humidity of 75–80 %.

5. Results of studying the semi-smoked sausages for safety indicators

As for many consumers important criteria for choosing products are quality and safety, the state standards for any raw material for the production of food and finished products set force the criteria for compliance with the content of toxic elements and radionuclides. The consumption of products contaminated with heavy metals leads to serious diseases and high mortality among the population.

The semi-smoked sausages are produced from traditional raw materials of beef and poultry meat with adding lentil flour, thyme, and juniper. In order to determine the safety of the proposed plant materials and to confirm the possibility of their use in sausage production, they were researched for the contents of lead, cadmium, mercury, arsenic, copper, zinc, and a specific amount of radionuclides. Lentils, thyme, and juniper contain no more than the permissible levels of the contents of these toxic elements and radionuclides; thus, they are safe for using them in the semi-smoked sausages [20]. The developed samples of the semi-smoked sausages with the modified recipes have also been studied for their physical and chemical parameters; their amino acid and fatty acid compositions have been studied along with histological research [21–24].

The contents of toxic substances in these semi-smoked sausages should not exceed the maximum permissible concentrations provided by DSTU 4435:2005. In particular, the semi-smoked sausages should contain no more than [4] 0.5 and 0.05 mg/kg of lead and cadmium, no more than 0.03 and 0.1 mg/kg of mercury and arsenic, and no more than 5 and 70 mg/kg of copper and zinc. The specific activities of cesium-137 and strontium-90 should not exceed 200 and 20 Bq/kg (Table 2).

According to the results of the research, it was found that the content of lead in the semi-smoked sausages, mg/kg, was the following: 0.0890 in *Osoblyva Sambirska Spiced*, 0.1040 in *Osoblyva Stryiska Spiced*, and <0.010 in the other experimental samples. Cadmium (mg/kg) was found in the amounts of 0.0160 in *Osoblyva Simeyna* and *Osoblyva Stryiska*, 0.0190 in *Osoblyva Simeyna Spiced* and *Osoblyva Sambirska*, and in the range from 0.0200 to 0.0250 in *Osoblyva Stryiska Spiced* and *Osoblyva Sambirska Spiced*. The content of mercury was <0.005 mg/kg in all the experimental samples. (The recipes for the sausages are given in Table 1).

The contents of copper and zinc were different in all types of the semi-smoked sausages and, in mg/kg, they amounted to the following: in *Osoblyva Simeyna* and *Osoblyva Simeyna Spiced* – 2.2770/30.1970 and 3.0860/26.4120; in *Osoblyva Sambirska* and *Osoblyva Sambirska Spiced* – 2.8880/26.4460 and 2.7910/30.6600; and in *Osoblyva Stryiska* and *Osoblyva Stryiska Spiced* – 2.1730/38.0790 and 2.2350/35.2880.

Table 2

The content of toxic elements and radionuclides in the semi-smoked sausages, mg/kg and Bq/kg

The name of the toxic element	The marginally admissible level, not more than	The name of the sample of the semi-smoked sausages					
		Osoblyva Simeyna	Osoblyva Simeyna Spiced	Osoblyva Sambirska	Osoblyva Sambirska Spiced	Osoblyva Stryiska	Osoblyva Stryiska Spiced
Toxic elements, mg/kg							
Lead	0.5	<0.010	<0.010	<0.010	0.0890	<0.010	0.1040
Cadmium	0.05	0.0160	0.0190	0.0190	0.0250	0.0160	0.0200
Mercury	0.03	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Arsenic	0.1	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010
Copper	5	2.2770	3.0860	2.8880	2.7910	2.1730	2.2350
Zinc	70	30.1970	26.4120	26.4460	30.6600	38.0790	35.2880
Radionuclides, Bk/kg							
Specific activity of cesium-137	200	<6.66	<3.79	<3.75	<6.58	<3.75	<3.75
Specific activity of strontium-90	20	<9.17	<8.61	<13.62	<6.92	<7.69	<8.97

6. Discussion of the results of studying toxic elements and radionuclides in the semi-smoked sausages made with lentils, thyme, and juniper

Any marketed products must comply with the requirements of state standards and technical specifications, including safety indices. These indices include the content of toxic elements, radionuclides and microbiological counts. The developed semi-smoked sausages with the modified recipes, namely using beef, poultry, lentils, thyme, and juniper, were investigated according to the safety methodology developed by Lviv Oblast State Laboratory of the State Consumer Protection Service in Lviv Oblast [18] by the method of atomic absorption spectrometry, using Varian AA240FS Atomic Absorption Spectrophotometer (Agilent Technologies, USA) and Shimadzu AAS-6300 Atomic Absorption Spectrophotometer (Shimadzu, Japan). The use of the developed techniques and this equipment made it possible to determine the contents of lead, cadmium, mercury, arsenic, copper, zinc, and radionuclides and to confirm conformity of the sausages according to these parameters to the requirements of DSTU 4435:2005. Semi-smoked sausages [4]. The research results allowed registering the technical specifications and technological instructions for the semi-smoked sausages *Osoblyva Simeyna*, *Osoblyva Simeyna Spiced*, *Osoblyva Sambirska*, *Osoblyva Sambirska Spiced*, *Osoblyva Stryiska*, and *Osoblyva Stryiska Spiced*.

7. Conclusions

1. It has been established that the content of toxic elements in the researched semi-smoked sausages does not exceed the maximum permissible levels, which comply with the requirements of the standards. The contents in the experimental samples are the following: lead ranges from <0.010 to 0.1040 mg/kg, cadmium – from 0.0160 to 0.0250 mg/kg, mercury – <0.005 mg/kg, arsenic – <0.010 mg/kg, copper – from 2.1730 to 3.0860 mg/kg, and zinc – from 26.4120 to 38.0790 mg/kg.

2. The contents of the radionuclides cesium-137 and strontium-90 do not exceed the maximum permissible levels – that is, 200 and 20 Bq/kg make in the semi-smoked sausages.

3. The semi-smoked sausages that are made using beef, poultry meat, lentil flour, thyme, and juniper conform to the safety standards – that is, in particular, the contents of toxic elements and radionuclides comply with the requirements of the regulatory documents.

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