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DEVELOPMENT AND IMPLEMENTATION OF A PROTOCOL FOR COUNTRYWIDE CONTROL AND MONITORING OF IRRADIATED FOOD IN THE REPUBLIC OF NORTH MACEDONIA

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A b s t r a c t: The national monitoring of irradiated food was realized in the Laboratory for detection of irradiated food, as a part of the governmental food safety-monitoring program. The protocol for implementation of countrywide control and monitoring of irradiated food was developed by the Laboratory and provides a selection of test methods and paths with the highest efficiency and economy of the procedure, while ensuring maximum accuracy of the results. As a part of the monitoring program, 140 samples, comprising dried herbs, spices, vegetables and seasonings were tested. All samples were tested according to PPSL method, while 25 were additionally tested according to TL method. The results showed that 6.43 % of the samples have indications that they have been treated with ionizing radiation, 92.86 % of the samples have no indications that they have been treated with ionizing radiation, one sample cannot be identified as irradiated or unirradiated since not enough minerals could be isolated.

Key words>: monitoring; thermoluminescence; photostimulated luminescence; irradiated food

РАЗВОЈ И СПРОВЕДУВАЊЕ ПРОТОКОЛ ЗА КОНТРОЛА И МОНИТОРИНГ НА ХРАНА ТРЕТИРАНА СО ЈОНИЗИРАЧКО ЗРАЧЕЊЕ ВО РЕПУБЛИКА СЕВЕРНА МАКЕДОНИЈА

А п с т р а к т: Националниот мониторинг на храна третирана со јонизирачко зрачење беше реализиран во Лабораторијата за контрола на храна третирана со јонизирачко зрачење, како дел од владината програма за следење на безбедноста на храната. Протоколот за спроведување на контрола и мониторинг на озрачената храна е развиен во Лабораторијата и обезбедува избор на методи и патеки за тестирање со највисока ефикасност и економичност на постапката, притоа обезбедувајќи максимална точност на резултатите. Како дел од програмата за мониторинг беа тестирани 140 примероци, составени од сушени билки, зачини и зеленчук. Сите примероци беа тестирани според методот ПОСЛ, а 25 беа тестирани и дополнително според методот ТЛ. Резултатите покажаа дека 6,43 % од примероците имаат индикации дека биле третирани со јонизирачко зрачење, 92,86 % немаат индикации дека биле третирани со јонизирачко зрачење. Кај еден примерок озрачувањето не можеше да се идентификува бидејќи не можеше да се изолираат доволно минерали.

Клучни зборови: мониторинг; термолуминисценција; оптички стимулирана луминисценција; храна третирана со јонизирачко зрачење

INTRODUCTION

To enforce correct labeling or to detect non-authorized products, countrywide control and monitoring of irradiated food is an essential tool. The countrywide control and monitoring of irradiated food was realized in the Laboratory for detection of irradiated food at Faculty of Electrical Engineering and Information Technologies, Ss. Cyril and Methodius University in Skopje, as part

of the government food safety monitoring program in the Republic of North Macedonia.

Countrywide control and monitoring of irradiated food in the Republic of North Macedonia began as a tool for full implementation of the Law on Food Safety [1, 2] and Rulebook on special requirements for food safety produced by ionizing radiation. This Rulebook has harmonized national legislation with Directive 1999/2/ EC [3] and Directive 1999/3/EC [4]. According to these directives, the European Parliament requires countries to conduct wide control and monitoring and report the results of checks carried out at the product marketing stage. The Rulebook prescribes special requests for food safety produced with ionizing radiation, as well as the rules for its labeling. According to the Rulebook, the words "irradiated" or "treated with ionizing radiation" should be marked on the packaging in a certain place, depending on whether it is sold in individual packages, in bulk, or used as an ingredient, even if the content of such ingredients is less than 25 % of the end product. Moreover, according to this Rulebook, irradiation is allowed only for herbs, spices and seasonings.

The Accreditation Certificate of Testing Laboratory No. LT–074 has been issued by the Accreditation Institute of the Republic of North Macedonia to the Laboratory for detection of irradiated food. Laboratory for detection of irradiated food is accredited according to standard MKC EN ISO / IEC 17025:2018, with fixed scope of Accreditation of two standard testing methods:

- Thermoluminescence (TL) method for the detection of food from which silicate minerals can be separated according to MKC EN 1787:2011 Standard. This method allows examination of herbs and spices, plant extracts, shrimps. The application of the method of thermoluminescence depends on the content of minerals in the sample and the experience of the performer in the techniques for mineral separation [5].
- Pulsed photostimulated luminescence (PPSL) method is a fast and economical method, which does not require prior preparation of the test material, but may strongly depend on the environmental conditions in which the sample has been stored prior to testing. This method is used for examination of spices, herbs and shrimps [6].

PROTOCOL FOR IMPLEMENTATION OF COUNTRYWIDE CONTROL AND MONITORING OF IRRADIATED FOOD

The protocol for implementation of countrywide control and monitoring of irradiated food was developed by the Laboratory for detection of irradiated food. The Laboratory is obliged to present the protocol and to conduct training of the authorized inspectors from the Food and Veterinary Agency who are in charge of implementing parts of the same. The protocol provides a selection of test methods and paths with the highest efficiency and economy of the procedure, while ensuring maximum accuracy of the results (Figure 1).

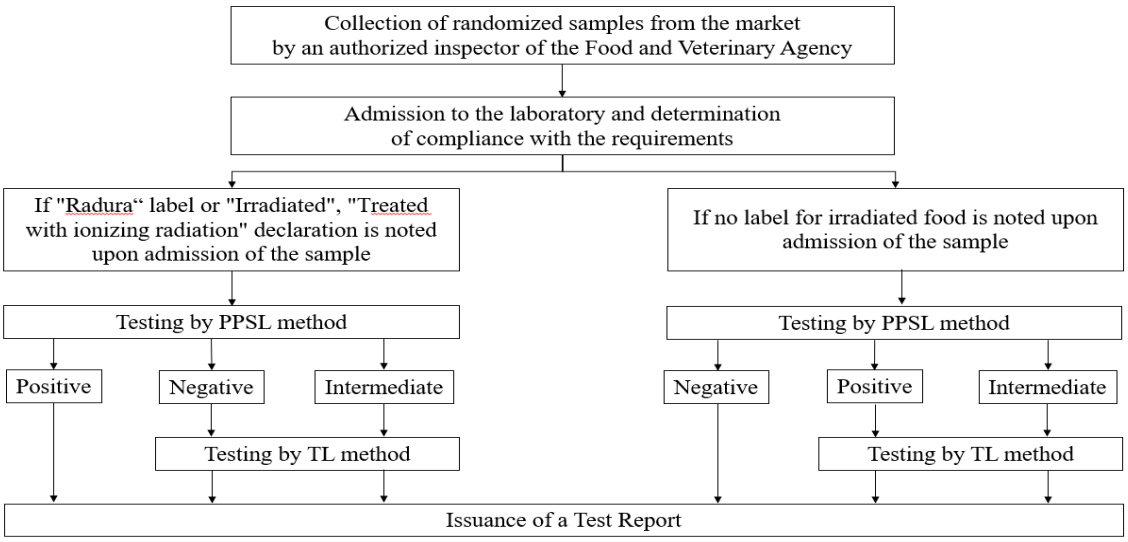

Fig. 1. Protocol for implementation of countrywide control and monitoring of irradiated food

Collection of randomized samples from the market is performed by authorized inspectors according to the sampling procedure. Whenever possible, the sample should be taken from a place protected from light. Care should be taken to avoid contamination. If the sample is taken with a tool (bulk), it is recommended that the sample is taken from the middle of a larger package. Samples are placed in a light protected box. Samples should be stored in a place protected from light before testing. Avoid exposing samples to temperatures above 50 °C.

The samples accompanied with the Sample Minutes for each sample are taken by the Laboratory for detection of irradiated food. Upon receipt, the compliance of the sample data with the Sample Minutes is checked, and whether the sample complies with the testing methods available in the laboratory. During the visual examination and checking of the minutes, it is determined if there are any notifications about the sample irradiation history.

Depending on whether any labels or tags are found, there are two possible test paths according to the protocol, thus samples are divided into two main groups:

- First group in which "Radura" label or "Irradiated" or "Treated with ionizing radiation" declaration is noted upon admission of the sample.
- The second group where no label for irradiated food is noted upon admission of the sample.

In the next phase, the samples are examined using the PPSL method for both groups of samples. Depending on the obtained results and the present declarations, the issuance of a Test Report or additional testing by the TL method is undertaken. If a positive result is obtained according to the PPSL method in the first group of samples, the Test Report is issued, while in the samples with a negative or an intermediate result, an additional examination is performed with the TL method. In the second group of samples, additional analyses are performed with the TL method if according to the PPSL method a positive or an intermediate result is obtained, while with the negative result the Test Report is issued right away.

Measurements with the PPSL method are performed by the SUERC irradiated food screening system designed and developed at the Scottish Universities Research and Reactor Centre. The procedure has been set according to EU standard for detection of irradiated food: EN 13751:2011 Foodstuffs – Detection of irradiated food using photostimulated luminescence. Samples were put in disposable plastic Petri dishes in the form of a thin layer of powder. During measurements samples are stimulated by an array of infra-red light emitting diodes which are pulsed symmetrically on and off for equal periods of time. Luminescence is measured using a patented digital lock-in photon counting method by using a cathode photomultiplier tube. Every measurement lasts for 60 s and results are obtained once in a second. For evaluation of results included in this work, by the recommendations given in EN 13751:2011 Foodstuffs – Detection of irradiated food using photostimulated luminescence, low threshold is set to 700 counts/60 s, whereas high threshold is set to 5000 counts/60 s.

Measurements with the TL method are performed by Riso TL/OSL reader DA-20, developed at the RISO Campus in Danish Technical University. The procedure is set according to EU standard for detection of irradiated food – EN 1788:2011 Foodstuffs – Thermoluminescence detection of irradiated food from which silicate minerals can be isolated. Thermoluminescence analysis is done twice for each sample. The first measurement is done after isolation of silicate minerals, which includes a thorough chemical preparation. Food samples are mixed with water and most of the organic material is removed by decantation. Sodium polytungstate solution is used for density separation of minerals and organic material, whereas hydrochloric acid is used for dissolving carbonates adhering to the silicate minerals. Extracted silicate minerals are dried with acetone and additionally dried overnight at room temperature. Prepared samples are put on steel cups suitable for the reader. Measurements are performed in nitrogen atmosphere with nitrogen gas at a constant flow rate. The initial temperature is set to 70 °C and final temperature is set to 400 °C, with a heating rate of 2 °C/ s. During measurement, the first glow curve is recorded, giving the variation of TL intensity with temperature. Because of the known heating rate, the temperature is known at every second of the measurement. The second measurement is done after exposing the sample to a certain dose of ionizing radiation and detecting the signal for the second time, thus obtaining the second glow curve. Because measurement is done right after irradiation, preheat of sample is included in the measurement procedure. Glow ratio gives the ratio of the integral luminescence intensity from the first and the second measurement, evaluated over the temperature interval 150 °C–250 °C.

After the completed analyses, a Test Report is issued, containing the following data:

- information on sample identification;
- date of sampling;
- date of delivery;
- date of examination;
- test results;
- reference to the European Standard EN 13751, i.e. EN 1788.

The test results are accompanied by an appropriate interpretation as follows:

- Positive – there is an indication that the sample has been treated with ionizing radiation.
- Negative – there is no indication that the sample has been treated with ionizing radiation.
- Can not be determined.

RESULTS AND ANALYSIS

A total of 140 samples were tested in the frame of the countrywide control and monitoring program. In the period from 2018 to 2020, three cycles of testing of 50 samples each in 2018 and 2019 were performed, while 40 samples were tested in 2020.

The assortment of consumable products in 2018 compiles 29 (58 %) samples of spices, 6 (12%) samples of seasonings and 15 (30 %) samples of dried herbs. In 2019, 28 (56 %) of the received samples are spices, while 4 (8%) are seasonings and 18 (36 %) are dried herbs. In 2020 the last cycle of countrywide control and monitoring, 30 (75 %) were spices, 3 (7 %) were seasonings and 7 (18 %) were dried herbs. The total distribution of products is 87 (62 %) spices, 13 (9 %) seasonings and 40 (29 %) dried herbs (Figure 2).

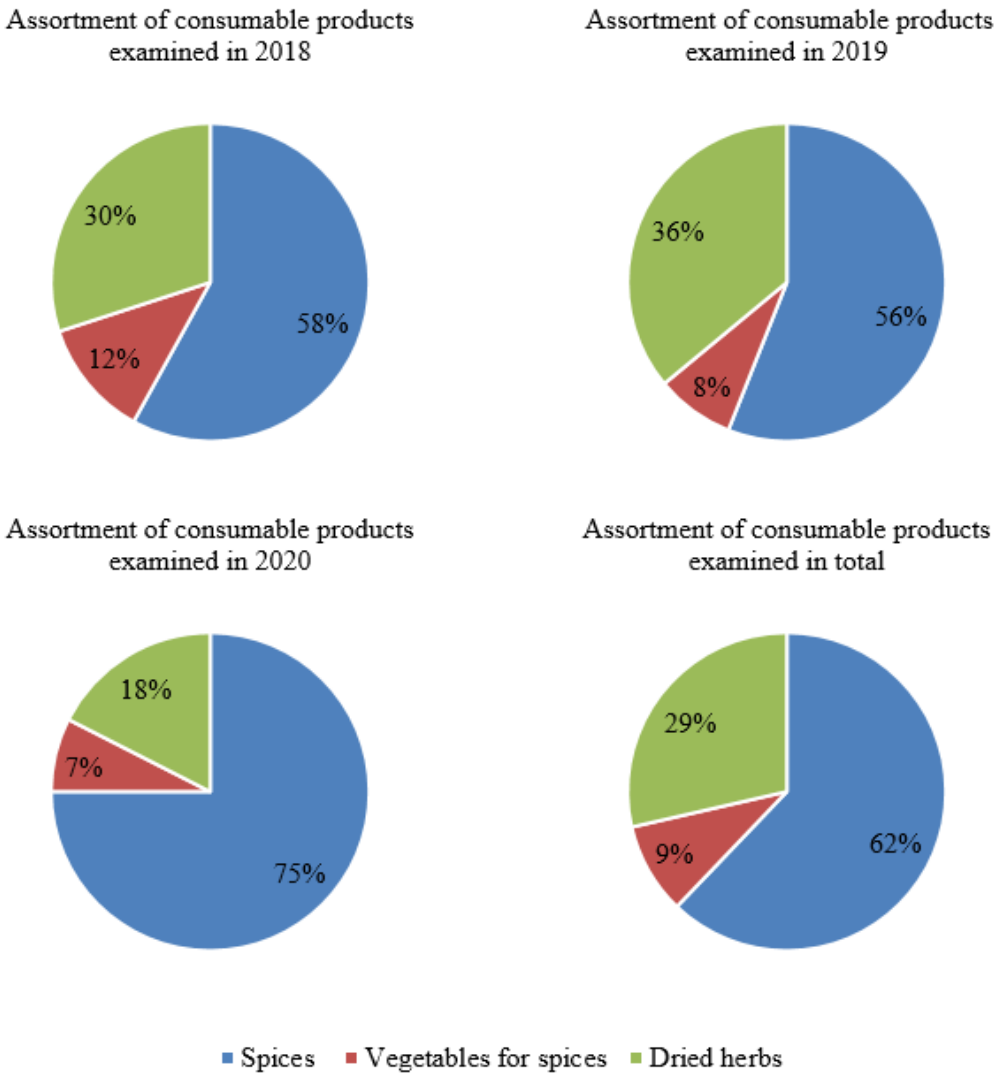

Fig. 2. Assortment of consumable products examined by year and in total

Following the accepted protocol for selection of methods and issuance of a test report, all 140 (100%) samples were tested with the PPSL method, while 25 (17.85%) were additionally tested with the TL method due to indications of irradiation treatment or due to an intermediate PPSL result (Figure 3).

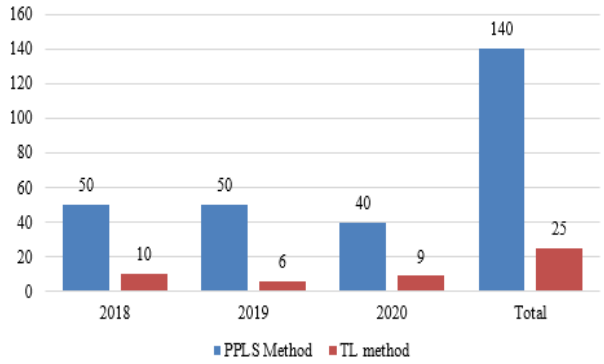

Fig. 3. Number of tested samples by method

All samples for which an intermediate result was obtained by the PPSL method belong to the group of samples that do not have any label indicating irradiation treatment. Such a result in 2018 was obtained in 6 (12 %) samples, in 2019 in 3 (6 %) samples, while in 2020 in 6 (12 %) samples. Six (40 %) of these samples are a mixture of spices that have a declared ingredient of table salt that has a high threshold of luminescence, while 3 (20%) samples are ginger. All 15 (10.71 %) samples for which the intermediate result was obtained by PPSL method were identified as negative after additional testing with the TL method.

In the first group of samples with a declaration of the irradiation treatment, there are 7 (14 %) samples in 2018, 3 (6 %) samples in 2019 and there are no such samples in 2020. All samples of the first group were labeled with the symbol "Radura" which is used for labeling irradiated food according to the General Standard for Labeling of Food Packaging – Standard for the Labeling of Prepackaged Foods CODEX STAN 1-1985 of the Organization for Nutrition and Agriculture at the United Nations – FAO, and the World Health Organization – WHO. However, this symbol is not accepted by national and European regulations. All samples marked with "Radura" belong to the group of spices.

In 2018, 4 (8 %) samples were detected as irradiated, all of them labeled with Radura which is a legally unrecognized mark, which means that all samples are incorrectly marked. Additionally, 3 (6 %) samples that were determined as unirradiated were marked with Radura. In 2019, 2 (4 %) samples were detected as irradiated, one of them labeled with Radura;, 2 (4 %) samples that were determined as unirradiated were marked with Radura. In 2020, 3 samples were detected as irradiated.

From the total of 140 samples, 14 (10 %) were not compliant and 1 sample (0.71%) gave inconclusive results. The non-compliance observed was mainly incorrect labeling and use of legally unacceptable labels for food treated with ionizing radiation (Figure 4).

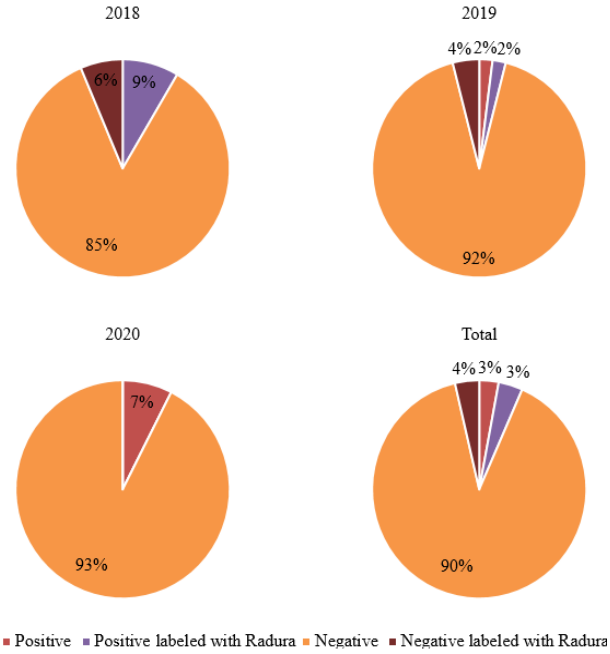

Fig. 4. Results by year and in total

CONCLUSION

The developed protocol for implementation of countrywide control and monitoring of irradiated food ensures economy and maximum accuracy of the obtained results.

In the countrywide control and monitoring program, 9 (6.43%) of the tested samples have indications that they have been treated with ionizing radiation, 130 (92.86 %) of the tested samples have no indications that they have been treated with ionizing radiation, while 1 sample (0.71%) cannot be identified as irradiated or unirradiated since not enough minerals could be isolated from it.

Five (55.56%) of the 9 samples identified as irradiated were labeled with the international label "Radura". There were also 5 (3.85%) of 130

samples labeled with "Radura", but there are no indications that they have been treated with ionizing radiation. If all these samples are taken as samples that do not meet the requirements, a total of 14 (10%) are non compliant samples.

In addition to the government's commitment to following and implementing EU directives, this countrywide control and monitoring program is important from two other aspects, one of which is to protect customers and their rights and the other one is to help companies become able to follow the rules and their implementation.

The countrywide control and monitoring program did not provide results for food that is prohibited to be treated with ionizing radiation, thus there is a lack of data of this type. It is necessary for the laboratory in the future to introduce new methods for examination of samples that are not on the list for food that is approved for irradiation, such as milk, meat etc.

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[6] MKC EN 1788:2011 Foodstuffs – Thermoluminescence detection of irradiated food from which silicate minerals can be isolated.