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RADIATION INDUCED DEFECTS IN MONOCRYSTALS AND GLASSES OF $Li_2B_4O_7$

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The subject of the work is a study on the influence of irradiation on defect level of $Li_2B_4O_7$ obtained in the form of single crystals and glasses. In order to obtain $Li_2B_4O_7$ monocrystals the Czochralski method was adapted. The substrates were H_3BO_3 and Li_2CO_3 both of high level of purity (4N). $Li_2B_4O_7$ was synthesised according to the following equation:

$$4H_3BO_3+Li_2CO_3 \rightarrow Li_2B_4O_7+6H_2O+CO_2 \quad (1)$$

From bigger monocrystals and pieces of glasses small plates were cut out of the shapes appropriate to conduct further measurements. The plates were polished and then investigated by microscope and conescope observations. Optical transmission of plates at room temperature has been measured as well with a Lambda-2 Perkin Elmer spectrophotometer within the range 200-1100 nm, while with a FTIR Perkin Elmer apparatus within the range 1400-25000 nm. Optical transmission of monocrystals was measured also after radiation treatment (^{60}Co gamma source Issledovatel, doses from 10 to 1000 kGy) at room temperature and after warming of plates at 450°C. Numerical values of the changes of optical absorption were estimated according to the equation:

$$\Delta K = \frac{1}{d}\ln\frac{T_1}{T_2} \quad (2)$$

where: K - coefficient of absorption; T_1, T_2 - transmission of plate before and after irradiation; d - thickness of the plate.

The defect level related to the changes of absorption in $Li_2B_4O_7$ single crystals and glasses were determined before and after the irradiation at the doses from 10 to 1000 kGy.

On the basis of the obtained results it can be concluded that in the case of monocrystals the irradiation at highest doses of 1000 kGy seems very advantageous from the point of view of their optical quality. It is because monocrystals after the irradiation at high doses become more transparent within the range 190-1100 nm. In other words their optical quality becomes much better.

On the contrary, in the case of glasses the effect of better transparency is achieved by warming them in air at a temperature of 450°C.

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ANALYTICAL METHODS FOR THE DETECTION OF IRRADIATION IN FOOD COMMODITIES ACCREDITED IN THE INCT LABORATORY FOR DETECTION OF IRRADIATED FOODS

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During the past two years the Laboratory for Detection of Irradiated Foods of this Institute was involved in the implementation of the quality assurance system according to the PN-EN 45001 standard and the ISO/IEC 25 guide for the methods applied in testing foodstuffs for irradiation. The following documentation of the Quality Assurance System implemented in the Laboratory has been prepared: General Book of Quality Assurance, General Procedures for Quality Assurance and the set of appropriate executive instructions.

For detection of irradiation in foods the following analytical procedures have been adapted and tested for their consistence with appropriate CEN European Standards:

1) the EPR method of detection of irradiated foods containing bone (related to BS-EN 1786),
2) the EPR method of detection of irradiated foods containing cellulose (related to BS-EN 1787),
3) the thermoluminescence method for the detection of irradiation in food from which silicate minerals can be isolated (related to BS-EN 1788).

Upon completion of required actions and procedures (organisation, methodology, documentation) and their preliminary approval, the Laboratory was subjected to the audit executed by the experts from the Polish Centre for Testing and Certification. After introducing all the suggested corrections accepted by the decisive body of the Centre the Certificate of Testing, Laboratory Accreditation for the above mentioned methods has been issued on 25th October 1999.

The scope of accreditation compiles the following categories of foods in which earlier irradiation treatment can be detected:

- food containing bones (poultry, pork, beef, fish, eggs): Qualitative analysis for the presence of the specific radiation induced paramagnetic substances by the EPR spectroscopy method (Documented testing procedure PB SLINŻ 01 edition 1);

- food containing cellulose (nuts, spices, fruits): Qualitative analysis for the presence of the specific radiation induced paramagnetic substances by the EPR spectroscopy method (Documented testing procedure PB SLINŻ 02 edition 1);
- food containing silicate minerals (spices, herbs, fruits, vegetables): Qualitative analysis of silicate minerals for the presence of radiation induced centres detected by the thermoluminescence measurement method (Documented testing procedure PB SLINŻ 03 edition 1).

The accredited methods as well as other methods for the detection of irradiated food presently adapted in our Laboratory have been described in earlier publications [1]. Here, we would like to point out to the highlights of the methods which have been accredited. Food, containing paramagnetic species, characterised by the presence of unpaired electrons is detected by electron paramagnetic resonance spectrometry (EPR/ESR). Many years of investigations have shown that unpaired electrons are present in many stable components of irradiated foodstuffs such as bones, stones, dried fruits, spices etc. The high sensitivity of the method allowing the detection of 10^{-12} mol/sample and its reproducibility enables to detect efficaciously a small concentration of paramagnetic centres in irradiated foodstuffs, being in the order of 10^{-8} mol per sample isolated for measurement. Detection of irradiation by EPR in all kinds of meat containing bones, eggshells, fishbones and scales is based on finding in the analysed sample a characteristic singlet g_a=2.003, g_2=1.997 and Δh_{pp}=0.85 mT. In food containing cellulose, in turn, as, for example, nuts (husks), strawberries and some spices, detection of irradiation is based on finding in the EPR signal a symmetrical triplet which has its centre at about g_b=2.004-2.006 and the distance between the outer satellite peaks (g_a=2.020, g_c=1.983) of 6.0 mT. The presence of these outer peaks in an EPR signal is taken as evidence that food has been irradiated. Such peaks have never been found in non-irradiated samples.

In foodstuffs containing silicate minerals (as inherent components or admixtures) irradiation can be detected by measuring the intensity of luminescence of isolated minerals subjected to heating (thermoluminescence-TL). Irradiation produces free electrons which are trapped in the crystalline lattice of minerals. During heating electrons absorb thermal energy and pass from the excited to basic state emitting photons. The intensity of luminescence at a chosen temperature interval is compared with the luminescence of the same sample subjected to reirradiation with a standard dose of gamma radiation (usually 1 kGy). The value obtained from relation of these two TL measurements is taken as indication of irradiation, when it is higher than 0.5. When this value is lower than 0.1 the foodstuff has not been irradiated. When the intermediate values are obtained an additional evaluation, taking into consideration the shape of luminescence curve is to be made. Glow curves for unirradiated samples have peaks at higher temperature interval while that for irradiated ones have their peaks around 150-200°C. Before accreditation a comprehensive investigation of thermoluminescence of irradiated various herbs and spices has been made in this Laboratory [2].

In the course of certification procedure the testing analyses of unirradiated and irradiated foodstuffs have been performed according to the procedures indicated above. The results obtained fully confirmed the reliability of these methods and their applicability for detecting whether a given food has been irradiated or not.

Recently in the Laboratory a new EPR spectrometer MINI-10 has been installed addressed exclusively to the accredited methods of the detection of irradiated foods based on the EPR spectroscopy. The purchase of the instrument was financed by the Foundation for Polish Science under the Agreement "SUBIN" No 13/99.

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DETECTION OF IRRADIATED FOODS WITH THE USE OF GAS CHROMATOGRAPHY - EXPERIMENTS WITH POULTRY CARCASSES

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The treatment of foodstuffs, which contain fats, with ionising radiation induces a series of chemical changes. These changes cannot be classified as radiation-specific since they also appear in oxidation processes. There is, however, a preferential cleavage of certain chemical bonds in triglycerides when treated with ionising radiation. Among others two types of volatile hydrocarbons produced in this matter can be detected in fairly large quantities: hydrocarbons which have one C atom less than the original fatty acid (C_{n-1}) and those which have two C atoms less and an additional double bond in 1-position ($C_{n-2:1}$). The main fatty acids of chicken carcasses, pork and beef are palmitic acid, stearic acid, oleic acid and linoleic acid. After the treatment, the following radiation induced hydrocarbons appear in abundance (a)