

RESEARCH ON THE PROPERTIES OF SORBENTS AND DECONTAMINANTS IN THE PROCESSES WITH SMALL ISOTOPE EFFECTS

V.V. Levenets, A.Yu. Lonin, O.P. Omelnik, A.O. Shchur

National Science Center "Kharkov Institute of Physics and Technology", Kharkiv, Ukraine

E-mail: levenets@kipt.kharkov.ua

The low values of isotope effects in the sorption/decontamination processes make it possible to investigate the properties of the sorbing and decontaminating agents with the use of stable isotope simulators. In the work presented here the element content was determined from the characteristic X-ray emission induced by the proton beam (PIXE), by gamma-ray emission from the radioactive source, and by emission from the X-ray tube. Based on the results of efficiency determination for different kinds of sorbents and decontaminants, recommendations have been given for the choice of materials having the wanted performance characteristics.

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High requirements placed upon the ecological safety and the personnel safety of nuclear power facilities cause the development of new materials and refining of the existing ones, which are used for trapping and decontamination of ^{235}U fission products such as ^{131}I , ^{137}Cs , ^{90}Sr , ^{60}Co . For capture of ^{131}I and its compounds in air purification filters, of possible use is the application of both impregnated and unimpregnated carbon of the grades "skt-3", "Electrode-D", "DGF-2", etc. [1 - 3]. For removal of the ^{137}Cs , ^{90}Sr , ^{60}Co isotopes from liquid radioactive wastes, the most popular practice lies in application of ion-exchange sorbents, e.g., natural and synthetic zeolites [4 - 7]. These zeolites represent a complex aluminum-silicate structure, which is characterized by a combined sorption mechanism [8, 9]. To clean the equipment of ^{137}Cs , ^{90}Sr , ^{60}Co contaminants, use can be made of the detergents comprising complexing agents, viz., alginates or pectins [10, 11].

The aim of the present work has been to investigate the properties of sorbing and decontaminating agents in order that the materials with higher decontamination/cleanup factor values could be created.

ISOTOPE EFFECTS

When performing the studies of sorption processes by the use of nuclear physics research methods, it is of importance to estimate the values of isotope effects. These values are determined by differences in the nuclear masses of certain isotopes and are responsible for the distinctions between such properties of isotope compounds as the density, viscosity, diffusivity, heat capacity, thermal conduction, heat of evaporation, melting heat, boiling/fusion temperatures, etc. The isotope effect value decreases with a decrease in the relative difference between the atomic weights of the isotopes. For the studied elements (cobalt, strontium, cesium, iodine), the relative difference in the atomic weights of isotopes (stable and with a half-life of more than 1 h) is a few percent. The values of kinetic and thermodynamic isotope effects for these elements have similar values [12, 13]. A major part of theoretical and experimental investigations into isotope effects was dedicated to the elements of the first three periods of the Periodic Table of the Elements. However, the number of these investi-

gations is substantially less for the elements in the middle of the periodic table. In addition to theoretical and experimental laboratory studies of isotope effects [14 - 20], the results of these effects in the surrounding world are also studied over a period from several years (for example, in the biosphere) to millions and billions of years (the geological environment of the Earth and space objects) [21 - 25]. These great time ranges, which make it possible to detect and register the isotope effects, are required because of their extremely small values. The absorption of iodine and its compounds in the types of unimpregnated carbon occurs mainly due to the surface absorption process, whereas in the potassium iodide-impregnated kinds of carbon the processes of radioactive iodine absorption are supplemented with the isotope exchange. In the case of zeolites, the compounds of cobalt, strontium, and cesium are absorbed due to chemisorption and ion exchange, while in cleaning means the process of absorption goes owing to complex formation with alginates and pectins. With application of carbon air filters, zeolite and detergent-based liquid filters, the interaction time of the absorbent/detergent agents with the substance under decontamination is sufficiently small for the isotope effects to manifest themselves.

To determine the content of the elements under study in the materials, various physical and nuclear physics methods are of considerable current use [26 - 27]. Among them, preference is given to the methods based on detection of characteristic X-ray emission (CXE) that can be excited on using accelerated charged particles, radioactive source emission, X-ray tube emission. Unlike the neutron activation analysis, or the analysis based on nuclear reaction radiation, the detected X-ray emission is essentially independent of the isotope of the element. For example, it has been demonstrated in ref. [28] that the experimentally measured relative value of the isotopic shift in the energy of the K-series emission from Sn, Hf, W, Hg, Pb, U and some lanthanides amounted only to a few ppm. The analysis of theoretical descriptions and empirical data [29 - 33] in order to estimate the isotopic shift of photon/ion ionization cross-sections (and also, the CXE yield) for K- and L-shells gives much the same order of magnitude.

Thus, the investigations aimed at developing sorbing and cleaning materials with improved decontamination properties have proved that the elemental analysis results for iodine, cobalt, strontium and cesium and the data determined by the X-ray emission detection method are independent of the isotopic composition of the elements. Hence, the investigations of this type permit the usage of stable isotopes instead of radioactive analogs for the corresponding elements.

EQUIPMENT AND MATERIALS

The analysis of accelerated proton-excited elements was carried out with the use of the NSC KIPT analytical nuclear-physical complex “SOKOL”. The main characteristics of the accelerator and its output devices have been described in ref. [34]. Measurements were performed at the output device of channel 1 located on the left at an angle of 45 degrees to the ion beam direction (Fig. 1).



Fig. 1. Channel 1 output of the “SOKOL” complex

The output device includes a vacuum chamber, soft and hard X-ray detection units with Si(PiN) and high-purity germanium crystals as the base. The radiation is extracted from the chamber through vacuum-tight beryllium windows.

To conduct X-ray radiometric measurements, we have designed and manufactured the measuring module of the “SKIF” test bench [35]. Its parameters were optimized so as to determine the iodine content in a light-weight 10 cm³ matrix, though the module can be also applied for determining the content of elements having atomic numbers ranging from 48 to 56, which are comprised in such objects as carbon, plant ashes, etc. The external view of the module without the detection unit is shown in Fig. 2.

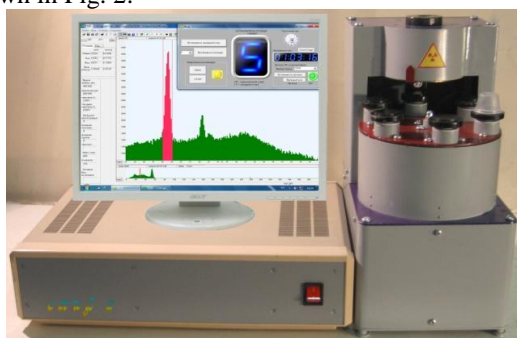


Fig. 2. External view of the “SKIF” measuring module (without the detection unit)

The X-ray fluorescence in the material under study is induced by 59.54 keV radiation from the 3.9 GBq radioactive source ²⁴¹Am (simultaneous arrangement of

up to three similar sources is admissible). The lift-and-turn device, serving to deliver the subjects to the irradiation zone, can carry up to 6 containers to be measured, each container being rotated during the measurement. To increase the emission detection efficiency in the energy range from 20 to 30 keV, the detection unit with high-purity Ge crystal was used [36].

In the X-ray fluorescence studies, use was made of the small-size NSC KIPT facility “ELEAN”, devised for X-ray fluorescent elemental analysis [37]. The background in the channel for detection of X-ray emission coming from the primary radiation of the X-ray tube is suppressed in the facility owing to the effect of partial polarization of the radiation during its reflection from the sample surface. For this purpose, the X-ray optics (Fig. 3) was used with mutually perpendicular arrangement of three axes for sampling the emission from the system: X-ray tube anode – secondary radiator – sample under analysis – X-ray detector. In this case, the detector lies in the plane other than 1-2-3.

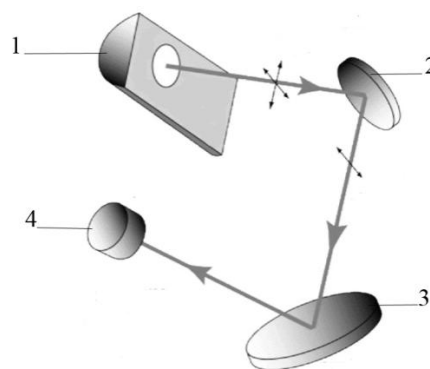


Fig. 3. X-ray optics of the “ELEAN” facility:
1 – X-ray tube; 2 – secondary radiator;
3 – measured sample; 4 – detection unit crystal

For use in the investigations, samples of carbon sorbents “Ehlektrod-D” (Ukraine-manufactured) and “DGF-2” (Germany-manufactured) were subjected to dynamic pumping with the vapors of stable iodine isotope [38]. The external appearance of “Ehlektrod-D” sorbent granules is presented in Fig. 4.



Fig. 4. “Ehlektrod-D” sorbent granules

To determine the iodine content in the carbon by the external standard method, calibration samples were prepared, which comprised sodium iodide in ultrapure carbon with ¹²⁷I mass concentrations ranging from 0.01 to 0.5%.

In the studies into decontamination properties of the developed detergent agent with regard to ¹³⁷Cs, ⁹⁰Sr, and ⁶⁰Co, stable isotope-based simulators of these elements were used. Human hairs were put into nitrate solutions

of stable cobalt, strontium, cesium isotopes taken separately, and then were dried. After that, a part of each weighed portion was successively washed with the detergent-water solution and was dried again. The weighed portions prepared in this way were pressed to make samples for the measurements in the tableted form.

The sorption properties of zeolites and composite sorbents were investigated under static and dynamic conditions. The solutions under study comprised a mixture of cesium, strontium and cobalt ions. During the experiments, stable compounds of the mentioned elements were used. The targets contained dry residues of the solution on the paper base (filter paper).

RESULTS AND DISCUSSION

DETERMINATION OF THE DETERGENT DECONTAMINATION FACTOR

The detergent decontamination factor was determined from measurements of the stable isotope content with the use of the NPC "SOKOL". The parameters of the accelerated proton beam were chosen to be: 140 keV beam energy, 50 kA average beam current. The analytical lines for cobalt and strontium were provided by the X-ray fluorescence K-series of these elements.

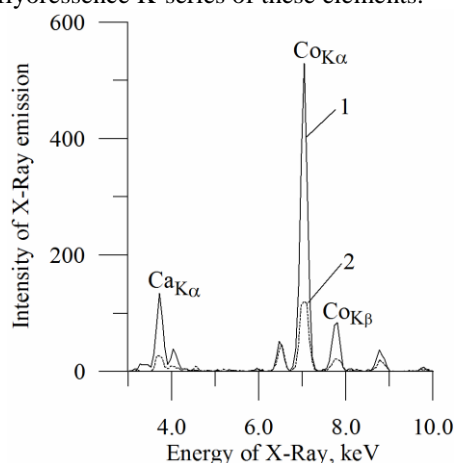


Fig. 5. Spectra from the cobalt-containing human hair before (1) and after (2) detergent decontamination

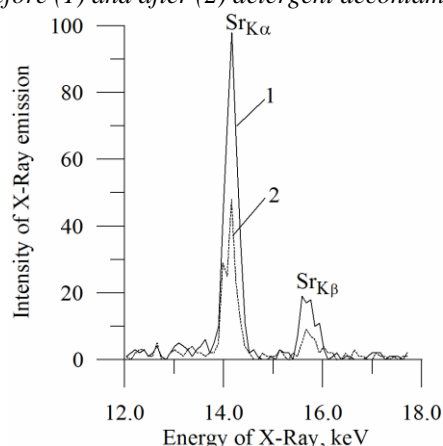


Fig. 6. Spectra from the strontium-containing human hair before (1) and after (2) detergent decontamination

The registration of these lines was performed with the detection units based on Si(PiN) crystals and high-purity germanium, respectively (Figs. 5 and 6). As regards cesium, its analytical line was taken from the L-series, which registered by the Si(PiN) detector (Fig. 7).

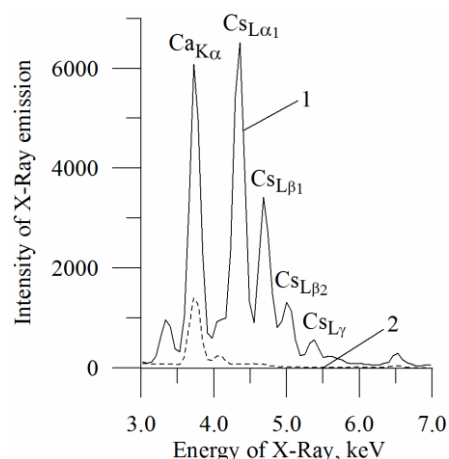


Fig. 7. Spectra from the cesium-containing human hair before (1) and after (2) detergent decontamination

The mathematical processing of the spectrometric information was carried out using the IAEA WinQXAS program. The spectrum processing data were used to determine the decontamination factor. It was calculated as a ratio of the analytical peak areas of the elements under study in the samples before and after treatment with the detergent. The resulting data are listed in the Table.

Detergent decontamination factors for cesium, strontium and cobalt

Elements	Decontamination factor
Cs	574.0
Sr	3.1
Co	3.6

SORPTION PROPERTIES OF CARBON SORBENTS

The sorption properties of carbon sorbents were investigated by the X-ray radiometry method with the use of the measuring module "SKIF" [39]. Based on the spectra of calibration samples, we could determine the parameters of the calibration straight line by the least squares method. The spectra taken from the carbon sorbents "DGF-2" and "Ehлектrod-D" were used to calculate the iodine content distribution in the filter mockup. The spectral region with the K-series of iodine in the carbon sorbent is shown in Fig. 8.

The sorption properties of the adsorbing agent were estimated by its sorption capacity index α , s^{-1} , which was calculated as $\alpha = \ln(C_{in}/C_{out})/\tau$, where τ is the time taken by the to pass in through the adsorption layer (conditioned contact time), s; C_{in} and C_{out} are, respectively, the iodine mass concentrations in the initial and final layers of carbon in the filter mockup, % [40, 41].

According to the IAEA guidelines, as well as the USA and European standards [42, 43], the minimum value of the ratio of the iodine content of air at the inlet of the model adsorber to the iodine value at the outlet of the model adsorber must be no less than 100: $C_{in}/C_{out} > 100$ (or 4.605 in logarithmic representation: $\ln(C_{in}/C_{out}) > 4.605$).

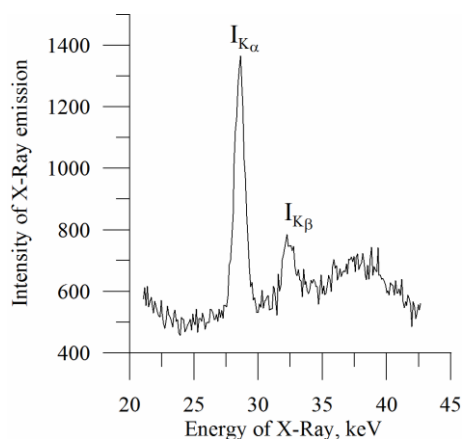


Fig. 8. Spectral region of the carbon sorbent "Ehlektrod-D" under investigation

The processing of the obtained spectrometric information has demonstrated that the logarithm of the ratio of the output iodine concentration to the iodine concentration in the layer at the input of the model filter amounts to 13.29 and 6.99 for "DGF-2" and "Ehlektrod-D" sorbents, respectively.

SORPTION PROPERTIES OF ZEOLITES

The content of Co, Cr, Cs in the prepared samples subjected to decontamination of solutions with zeolites was determined by means of the "ELEAN" facility. The anode voltage of the X-ray tube was set to be 50 kV, and the anode current was equal to 20 mA. Germanium was used as a secondary radiator. The content of Co and Sr was determined using X-ray emission the K-series, while the Cs content was found using the X-ray emission L-series. Fig. 9 shows the spectral region for the strontium-containing test sample after zeolite-based sorption.

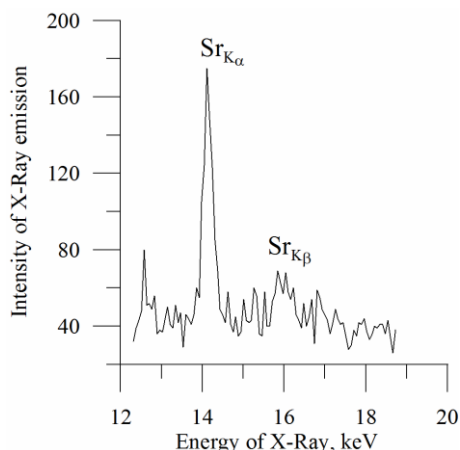


Fig. 9. Spectral region for the strontium nitrate-containing sample subjected to sorption

For quantitative estimation of the sorption properties of zeolites we have used the sorption coefficient ($K_s, \%$), which was calculated by:

$$K_s = \frac{(C_0 - C_p) \cdot 100\%}{C_0},$$

where C_{init} and C_{eq} are, respectively, the initial and equilibrium concentrations of the solution, mole/dm³.

The obtained results bear witness to high sorption properties of natural zeolite-clinoptilolite in relation to

cesium. As regards cobalt and strontium, clinoptilolite shows some deterioration of its sorption properties. On the contrary, the synthetic zeolites exhibit higher sorption properties with relation to cobalt and strontium [44].

CONCLUSIONS

The present investigations have demonstrated that the isotopic composition of contaminants has little or no effect on the determination of decontamination/cleanup factors for materials of different grades used in atomic energy industry.

A high value of the cleanup factor of the developed detergent agent has been found with respect to cesium.

It has been established that the decontamination factors of the carbon sorbents intended for air purification filters of nuclear power plants meet the IAEA requirements.

The analytical studies have confirmed the data that the natural zeolite (clinoptilolite) exhibits a high selectivity to cesium, but shows a substantially lower efficiency in the cases of cobalt and strontium. The fact that high sorption properties with respect to the ions of these elements are shown by synthetic zeolites, opens the prospect for the combined use of both natural and synthetic zeolites.

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ИЗУЧЕНИЕ СВОЙСТВ СОРБЕНТОВ И ДЕЗАКТИВИРУЮЩИХ СРЕДСТВ В ПРОЦЕССЕ С МАЛЫМИ ВЕЛИЧИНАМИ ИЗОТОПНЫХ ЭФФЕКТОВ

В.В. Левенец, А.Ю. Лонин, А.П. Омельник, А.А. Щур

Благодаря малым значениям величин изотопных эффектов у процессах сорбции и дезактивации возможно изучение свойств исследуемых нами сорбентов и дезактивирующих средств по использованию имитаторов со стабильными изотопами. В представленных исследованиях элементное содержание определялось с использованием характеристического рентгеновского излучения, возбуждаемого пучком протонов, гамма-излучением от радиоактивного источника, излучением от рентгеновской трубки. По результатам определения эффективности различных типов сорбентов и дезактивирующих средств даны рекомендации по выбору материалов с необходимыми характеристиками.

ВИВЧЕННЯ ВЛАСТИВОСТЕЙ СОРБЕНТІВ І ДЕЗАКТИВУЮЧИХ ЗАСОБІВ У ПРОЦЕСАХ З МАЛИМИ ВЕЛИЧИНАМИ ІЗОТОПНИХ ЕФЕКТІВ

В.В. Левенець, О.Ю. Лонін, О.П. Омельник, А.О. Щур

Завдяки малим значенням величин ізотопних ефектів у процесах сорбції та дезактивації можливо вивчення властивостей досліджуваних нами сорбентів і дезактивуючих засобів з використання імітаторів зі стабільними ізотопами. У представлених дослідженнях елементний вміст визначався з використанням характеристичного рентгеновського випромінювання, що порушувався пучком протонів, гамма-випромінюванням від радіоактивного джерела, випромінюванням від рентгеновської трубки. За результатами визначення ефективності різних типів сорбентів і дезактивуючих засобів надано рекомендації щодо вибору матеріалів з необхідними характеристиками.