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INVESTIGATION OF RADIATION RESISTANCE OF ADSORBENTS USING THE ^{90}Sr – SOURCE

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Purifying aqueous solutions from radioactive contamination is an extremely relevant scientific topic today. Many organic and inorganic adsorbents can be recommended for the adsorption of heavy metal ions and radionuclides from aqueous solutions, or as carriers for storage and disposal of radioactive waste.

Since radionuclides are sources of ionizing radiation, the radiation resistance of the adsorbent is an important characteristic. These studies aim to investigate the titanium silicate behavior and its adsorption properties' changes or their invariability in the field of intense β-radiation.

Experimental techniques describe the synthesis of titanium silicate adsorbent by sol-gel method and the study of its adsorption capacity toward Ba^{2+} cations. The adsorption of Ba^{2+} cations was investigated under batch conditions with neutral pH of the solution. Initial and residual concentrations of Ba^{2+} cations were controlled by direct complexometric titration with Na-EDTA with Eriochrom Black T as an indicator. The study of the radiation resistance of the adsorbent to high-energy β-radiation was performed using a ^{90}Sr-^{90}Y β^- - source "Sirius" installed in the Microtron Laboratory of the Uzhhorod National University. The distance from the source to the adsorbent samples was 20 cm. The flux of electrons at this distance was 10^8 el/cm^2·per second. The maximum energy of beta particles was 0.456 MeV for ^{90}Sr and 2.28 MeV for ^{90}Y. The maximum duration of exposure was 21 days, which corresponds to 1310 Gy. Raman spectroscopy of irradiated and nonirradiated samples of TiSi was performed using a Raman spectrometer XploRA PLUS installed in the Center for Collective Use of Scientific Equipment "Laboratory of Experimental and Applied Physics" of Uzhhorod National University.

Results consist of kinetic of Ba^{2+} adsorption by titanium silicate and irradiated titanium silicate; isotherm of Ba^{2+} adsorption and Raman spectrum of nonirradiated, irradiated titanium silicate (TiSi) and TiSi after Ba^{2+} adsorption. Results showed that the value of the maximal adsorption was 140.5±9.2 mg/g (6.55 %) under a confidence level of 95 %. The adsorption values of barium ions by irradiated and non-irradiated titanium silicate coincide. This indicates that the adsorption properties of this adsorbent do not change under the influence of such a radiation dose. The Raman spectra of irradiated and non-irradiated titanium silicate coincide, while they do not identify free radicals, or ionic formations, which would indicate a change in the properties of the adsorbent under the influence of beta radiation. It can be argued that this adsorbent is radiation-resistant to beta-radioactivity, with a radiation dose of 1310 Gy.

The main conclusion of the present work is that the studied sample of titanium silicate is radiation-resistant. It can withstand a radiation dose of 1310 Gy without changing its adsorption properties. Titanium silicate can be used for the adsorption of strontium radionuclides, it can be a carrier for the disposal of radioactive waste.

***Keywords**: adsorbent, irradiation, titanium silicate, adsorption, Raman spectroscopy*

Purifying aqueous solutions from radioactive contamination is an extremely relevant scientific topic today. Many organic and inorganic adsorbents can be recommended for the adsorption of heavy metal ions and radionuclides from aqueous solutions, or as carriers for storage and disposal of radioactive waste. Among them are ion exchange resins, adsorbents based on titanium dioxide or titanium silicate, zeolites, metal-organic frameworks, *etc*. However, only a small part of scientific papers is devoted to the investigation of the radiation resistance of adsorbents. Since radionuclides are sources of ionizing radiation, the radiation resistance of the adsorbent is an important characteristic [1–4].

According to publications [5, 6], the radiation resistance of adsorbents is manifested in the invariability of their adsorption properties. The adsorbent is radiation-resistant to a certain radiation dose if the ionizing radiation of a certain type and a given dose does not decrease its adsorption capacity. In our work, the adsorption capacity of titanium silicate toward barium cations (which was measured before and after irradiation) was also chosen as an indicator of TiSi radiation resistance.

These studies aim to investigate the adsorbent's behavior and its adsorption properties' changes or their invariability in the field of intense *β*-radiation.

EXPERIMENT TECHNIQUE

Synthesis of adsorbent and study of its adsorption capacity toward Ba^{2+} cations. Titanium silicate (TiSi) was synthesized in ISPE, NAS of Ukraine, according to the technique described in [1, 7]. For the synthesis of titanium silicates, titanyl sulfate $TiOSO_4$ solution was used. TiSi adsorbents were obtained by mixing two initial solutions: number one was prepared by mixing pure (3.4 M) $TiOSO_4$ and ligand, a mixture with D-sorbitol and L-lactic acid; and the second solution was obtained by mixing a technical solution of liquid glass (3.81 M Si) with 5 M NaOH. This process was performed at room temperature using a magnetic agitator. The sol-gel synthesis of this adsorbent can be described as follows:

$$TiOSO_4 + Na_2O \cdot 2.5SiO_2 + NaOH \rightarrow$$
$$\rightarrow Na_xTi_ySi_kO_m + nH_2O(gel).$$

This solution was taken from the technological sulfate line of production of the rutile white titanium pigment. The heating temperature was not higher than 150 °C, heating was carried out for 48 hours.

The adsorption of Ba^{2+} cations was investigated under batch conditions with neutral pH of the solution. Barium was chosen as the object of investigation because it is an alkali earth element. It is a heavy metal cation with chemical properties close to radium. In addition, barium isotopes, for example ^{141}Ba, can formed as fission radionuclides, like ^{90}Sr or ^{137}Cs. The mass of the adsorbent was 0.05 g; the solution volume (V) was 5 mL in all adsorption experiments. Initial and residual concentrations of Ba^{2+} cations were controlled by direct complexometric titration with Na-EDTA with Eriochrom Black T as an indicator (in addition to Raman analysis of the TiSi surface). Kinetic studies of Ba^{2+} adsorption by irradiated and nonirradiated TiSi were performed using the 0.1 M $BaCl_2$ aqueous solution with the duration of adsorption 5, 10, 15, …, and 60 minutes. The mass of adsorbent and the solution volume were the same, as was mentioned before.

The adsorption values were calculated by equations (1):

$$q_e = \frac{[(C_o - C_e)V]}{m}, \qquad (1)$$

where q_e – the amount of adsorbate uptake at equilibrium, $mg \cdot g^{-1}$; C_o and C_e – adsorbate initial and residual (equilibrium) concentration, $mg \cdot L^{-1}$; V – the volume of the solution, L; m – is the mass of the adsorbent (g).

Study on radiation resistance of TiSi adsorbent. The study of the radiation resistance of the adsorbent in relation to high-energy β^- radiation was performed using ^{90}Sr-^{90}Y β^- - source "Sirius" installed at the Microtron Laboratory of the Uzhhorod National University. This source is a radionuclide of ^{90}Sr in solid form on a ceramic carrier. ^{90}Sr is in secular equilibrium with its daughter radionuclide ^{90}Y. The decay chain of these radionuclides is shown in Diagram 1.

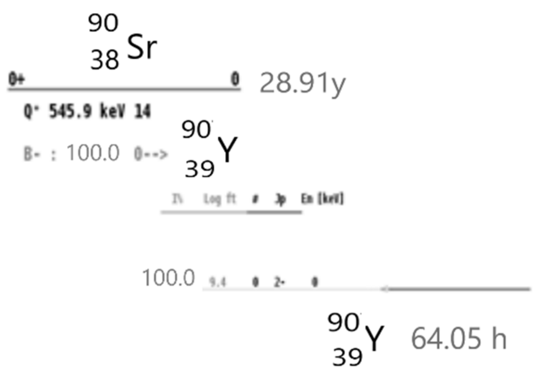


Diagram 1. Radioactive decay of ^{90}Sr. Adapted from resource [8]

The radioactive source of ^{90}Sr – "Sirius" was manufactured in 1980. It consists of 16 cassettes of ^{90}Sr, the initial activity of which on the surface at the time of manufacture was $5.55 \cdot 10^9$ Bq. In other words, the initial activity of each of these sources was close to 1 Ci.

After about 1.5 half-lives (present time), the electron flux at a distance of 20 cm from the source core is 1.10^8 electrons/cm^2 per second. The dose was calculated according to the data (initial activity) indicated in the data sheet and was also controlled by a clinical dosimeter, which is usually used to control the radiation dose at accelerators.

These sours are called ^{90}Sr-^{90}Y because (a) they were made by isolating ^{90}Sr from a mixture of fission radionuclides by the oxalate technique, i.e. precipitation with yttrium oxalate; (2) ^{90}Y is a daughter radionuclide of strontium (Diagram 1).

^{90}Y is always present in the vicinity of ^{90}Sr and if there is a lot of ^{90}Sr, then ^{90}Y radiation cannot be neglected. Since the half-life of ^{90}Y is much shorter than that of ^{90}Sr, they are in a state of secular equilibrium, and the ^{90}Y amount can be calculated using the formula (2) below:

$$N_Y = \frac{\lambda_{Sr}}{\lambda_Y} \cdot N_{Sr}, \quad (2)$$

where N_Y and N_{Sr} are the number of ^{90}Y and ^{90}Sr nuclei, respectively; λ_{Sr} and λ_Y are decay constants of ^{90}Sr and ^{90}Y, respectively.

^{90}Y is always the same amount, while ^{90}Sr is constantly decreasing. The use of such a source for the study of radiation resistance was due to two profits: (1) in this way, the situation of the influence of ^{90}Sr on the adsorbent was simulated since some authors recommend this adsorbent specifically for the adsorption of ^{90}Sr from aqueous solutions [4, 7]. (2) The use of source "Sirius" radiation compared with accelerator radiation saves a lot of electricity.

Thus, in our experiment, the distance from the source to the adsorbent samples was 20 cm. The flux of electrons at this distance was 10^8 electrons/cm^2·per second. The maximum energy of beta particles was 0.456 MeV for ^{90}Sr and 2.28 MeV for ^{90}Y (see in Fig. 1 *a*). The maximum duration of exposure was 21 days. The radioactive source "Sirius" creates a dose of 4 Roentgen per minute, which is equal to 5760 Roentgen per day. Thus, the adsorbent samples were irradiated with doses of $5.76 \cdot 10^3$; $11.52 \cdot 10^3$, $12.10 \cdot 10^4$, and $13.1 \cdot 10^4$ Roentgens (corresponding to 57.6, 115.2, 1210 and 1310 Gray). The maximum radiation dose was 1310 Gy. The IAEA database [8], as well as the programs for calculating the probability of generating bremsstrahlung gamma radiation NPMA Bremsstrahlung simulator, were used in order to perform a qualitative and (if possible) quantitative assessment of the effect of beta radiation on the adsorbent.

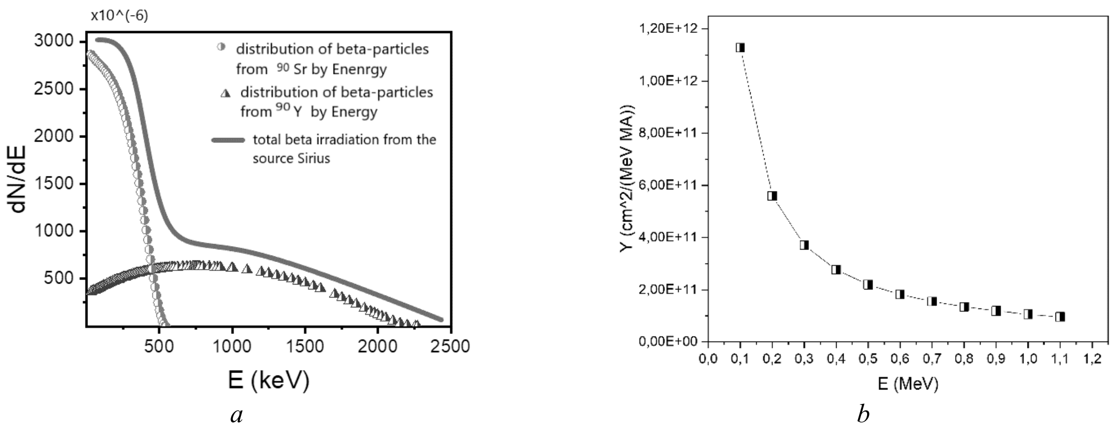


Fig. 1. Total irradiation from the ^{90}Sr-^{90}Y beta source: (*a*) Distribution of β^--particles of the source "Sirius" by the energy; (*b*) Modelling spectrum of bremsstrahlung gamma-ray, which were generated by beta particles of the source "Sirius". This spectrum was constructed using the program NPMA Bremmstraglung

That is, the adsorbent samples were exposed to high-energy electrons with a maximum energy of up to 2.28 MeV (Fig. 1 *a*, as well as bremsstrahlung gamma rays, the energy distribution of which is given in Fig. 1 *b* and in Table 1.

Table 1. Qualitative composition of radioactive radiation, which was used to study the radiation resistance of the adsorbent [8]

nuclide	$E_{\beta-}$ max MeV	E $_{brmss}$ max, keV	Energy [kev/decay]
^{90}Sr	0.5479	320–547	3.988E-3
^{90}Y	2.28	1000–2280	0.25297

All studies were carried out following the Radiation Safety Standards of Ukraine.

After irradiation, TiSi was used for a study of the adsorption of barium ions from an aqueous solution of $BaCl_2$ in a neutral medium. Part of the irradiated adsorbent was left for Raman spectroscopy.

Raman spectroscopy. Raman spectroscopy is commonly used in chemistry to provide a structural fingerprint by which molecules can be identified, therefore this type of spectroscopy was chosen for analysis of TiSi.

Table 2. Specifications of Raman spectrometer XploRA PLUS

Parameters	Value
Acceleration of spectrum mode	SWIFT with a motorized table
Confocal mapping	05 µmXY
Optical microscope	direct
Wavelengths of excitation lasers	532 nм
Sample Dimensions	(5÷10mm) × (5÷10mm) × (0.2÷2 mm)
Drift ACM through XY	2 nm/min

Raman spectroscopy of irradiated and nonirradiated samples of TiSi was performed at the Center for Collective Use of Scientific Equipment “Laboratory of Experimental and Applied Physics” of Uzhhorod National University.

The technical characteristics of the Raman spectrometer XploRA PLUS are given in Table 2.

RESULTS AND DISCUSSION

Adsorption of Ba^{2+} cations by irradiated and non-irradiated titanium silicate. According to publications [9-11], beta radiation is incapable of generating radiation defects of the classical type due to the small mass of beta particles. For example, to shift the atom of the cell into the interstitial space, or to form a Frenkel pair. However, beta radiation with mega-electron-volt Energy is capable of breaking the chemical bonds and changing the oxidation state of the elements. According to [12], this can change the properties of the adsorbent, and even improve them in some cases. For example, the rupture of certain found bonds can lead to the emergence of new adsorption centers. Therefore, the kinetic studies of Ba^{2+} cations of irradiated samples of TiSi and nonirradiated samples were performed simultaneously in parallel experiments.

The results of the adsorption of barium cations by irradiated titanium silicate and an unirradiated sample are shown in Fig. 2.

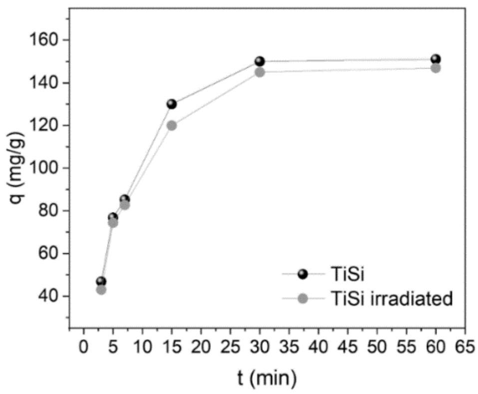
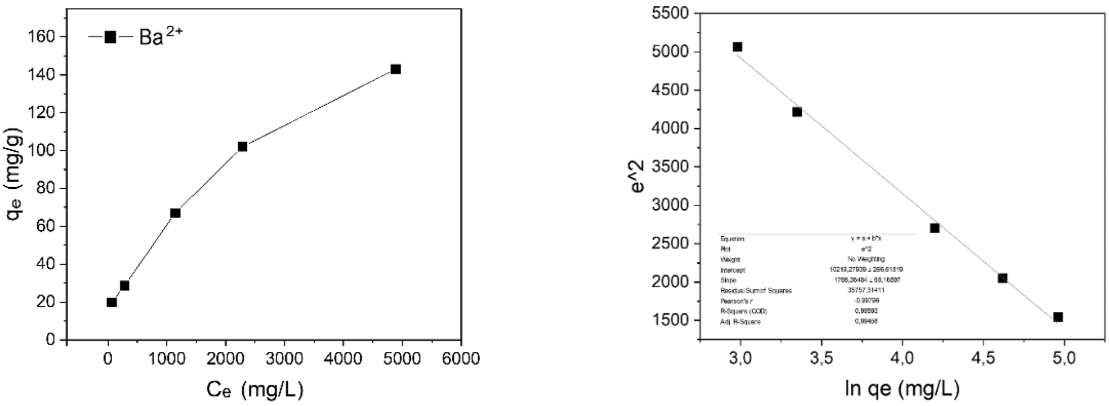


Fig. 2. Adsorption kinetics of Ba^{2+} ions from an aqueous solution of $BaCl_2$ by irradiated (1310 Gy) and non-irradiated titanium silicate. The initial concentration of Ba^{2+} ions was 0.1 M, V_{Ba2+} = 5 mL, m(TiSi) = 0.05 g, pH = 7

The figure shows that the value of the maximum adsorption is 140.5±9.2 mg/g (6.55 %) confidence level of 95 % [1, 13]. The values of adsorption of barium ions by irradiated and non-irradiated titanium silicate coincide. This indicates that the adsorption properties of this adsorbent do not change under the influence of such a radiation dose. There is also no reason to believe that the adsorption mechanism changes. Earlier we showed the adsorption of barium cations to be better described by the theory of Freundlich and Dubinin-Radushkevich, compared to the Langmuir theory [1].

This increases the probability that the surface of titanium silicate cannot be considered homogeneous, i.e. there are different surface groups on the surface of titanium silicate, *e.g.* ≡Ti-OH and ≡Si-OH, which can be adsorption centers. It is necessary, however, to note that not every =Ti-OH- surface group is an adsorption center [14].

Most likely, the adsorption of Ba^{2+} at the pedestrian stage occurs due to the electrostatic interaction of Ba^{2+} cations with the entire surface of titanium silicate. The Dubinin-Radushkevich adsorption theory, which is based on the potential Polanyi theory, is the theory of non-localized adsorption. As we can see, the experimental results of the adsorption of barium ions by this adsorbent are well-described by D-R theory.



a *b*

Fig. 3. Isotherm of Ba^{2+} adsorption by TiSi (*a*); (*b*) Linear fitting of experimental adsorption isotherm using D-R Theory

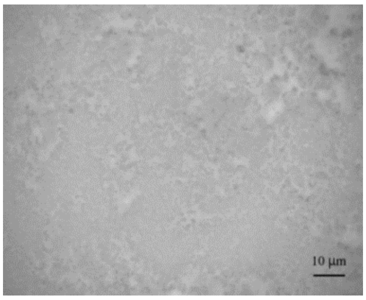
Barium adsorption is influenced by various factors – the presence of mesopores, the developed surface of the adsorbent, van der Waals forces, and electrostatic interaction. It can be assumed that, during irradiation with a dose of 1310 Gy, all these factors do not change, since change neither the magnitude nor the nature of the adsorption process.

Raman Scattering spectra of irradiated and unirradiated titanium silicate. The Raman scattering spectra of irradiated and non-irradiated titanium silicate and the Raman image of the adsorbent sample are shown in Figs. 4 *a*, *b* and 5 *a–c*.

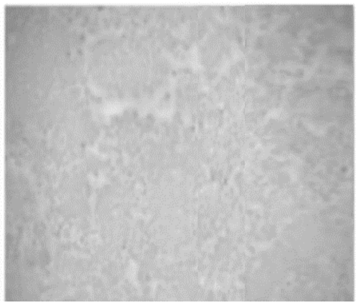
The oscillation of 380 cm^{-1} refers to the oscillations of the Si-OH group. The oscillations, the maximum of which lies in the region of 430–500 cm^{-1}, refer to the oscillations of SiO_2 (Si-O-Si). There are no maxima at 520–620 cm^{-1} that could be attributed to oscillations of radical Si-O* groups on the spectrum. The oscillation of 700–800 cm^{-1} also refers to the oscillations of SiO_2 surrounded by oxygen atoms [15]. Oscillations of 144 and 195 cm^{-1} refer to TiO_2 oscillations, according to the literature [16, 17].

The peak at 300, or rather 274 cm^{-1}, is absent in the Raman spectra of pure silicates and TiO_2 but is manifested in composite materials, for example, TiO_2 nanotubes modified with silicon dioxide [18]. This maximum can belong to Ti-O-Si bonds. However, some authors attribute the maxima of 270 and 304 cm^{-1} to the oscillations of the TiO_6 groups [19]. A small maximum at the 620–650 cm^{-1} position may belong to the oscillations of adsorbed barium. At the same time, this maximum is diagnosed only on the spectrum of titanium silicate (irradiated) after the adsorption of barium cations. According to the authors [20], the Raman spectra of barium-containing glasses and alloys always contain a maximum of about 600–720 cm^{-1} at low, medium, and high barium content in the structure [21, 22]. The oscillation of 810–830 cm^{-1} may refer to the vibration of the Si in the oxygen cage, according to the publication [15]. The small peaks in regions 870–910 cm^{-1} may correspond to the groups $Si_2O_7^{6-}$ [15].

The Raman spectra of irradiated and non-irradiated titanium silicate coincide, while they do not identify free radicals, or ionic formations, which would indicate a change in the properties of the adsorbent under the influence of beta radiation. It can be argued that this adsorbent is radiation-resistant to beta-radioactivity, with a radiation dose of 1310 Gy.

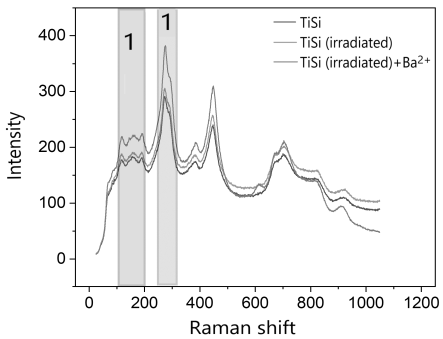


a

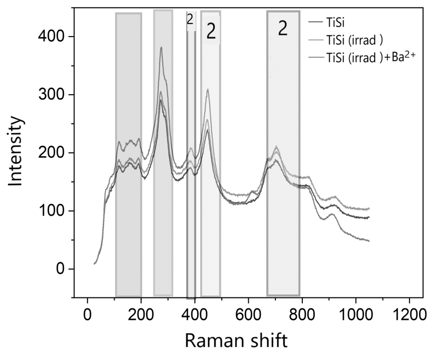


b

Fig. 4. Raman image of titanium silicate before irradiation (*a*); after irradiation by maximal dose (*b*)



a



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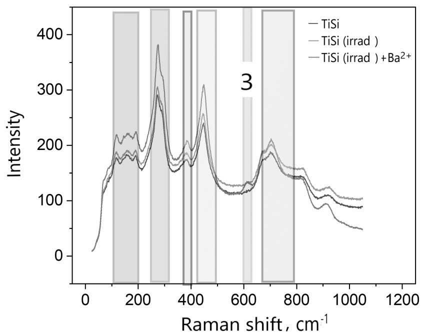

c

Fig. 5. Raman spectra of unirradiated titanium silicate, irradiated titanium silicate, and titanium silicate with adsorbed barium ions on the surface: (*a*) with isolated maxima belonging to the oscillations of the TiO_2 groups (1); (*b*) with highlighted maxima belonging to the oscillations of the groups SiO_2, Si-OH (2); (*c*) with highlighted maxima, which belong to the oscillations of the groups Ba^+-O (3)

CONCLUSIONS

The studied sample of titanium silicate is radiation-resistant. It can withstand a radiation dose of 1310 Gy without changing its adsorption properties.

The Raman scattering spectra of irradiated and unirradiated titanium silicate coincide with high accuracy and do not identify maxima that would belong to ion formations or free radicals.

Titanium silicate can be used for the adsorption of strontium radionuclides, it can be a carrier for the disposal of radioactive waste.

Дослідження радіаційної стійкості адсорбентів з використанням радіоактивного джерела ^{90}Sr

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Очищення водних розчинів від радіоактивних забруднень є надзвичайно актуальною темою сьогодення. Багато органічних і неорганічних адсорбентів пропонують для адсорбції йонів важких металів, радіонуклідів із водних розчинів, або як носії для захоронення радіоактивних відходів. У такому випадку радіаційна стійкість для адсорбентів є важливою характеристикою.

Мета даних досліджень вивчити зміну структурних та адсорбційних властивостей (або незмінність) адсорбентів в полі інтенсивного β-випромінювання створеного ^{90}Sr. Зокрема, було визначено радіаційну стійкість силікату титану.

Експериментальна частина даної роботи складається із опису синтезу адсорбента на основі силікату титану золь-гель методом і досліджень адсорбційної здатності даного матеріалу щодо катіонів Ba^{2+}. Наступна стадія експериментальних досліджень включає в себе дослідження радіаційної стійкості TiSi і Раманівську спектроскопію вихідних зразків адсорбента, опромінених зразків адсорбенту та зразків після адсорбції катіонів Ba^{2+}.

Дослідження радіаційної стійкості відносно високо-енергетичних бета-часток проводили з використанням ^{90}Sr-^{90}Y β^- джерела «Сіріус», встановленого у Мікротронній лабораторії ДВНЗ «УжНУ». Відстань від джерела до зразків адсорбента становила 20 см. Потік електронів на такій відстані був 10^8 е/см^2·с. Максимальна енергія бета-часток стронцію (^{90}Sr) становила 0.456 МеВ, бета-часток ітрію (^{90}Y) 2.28 МеВ. Найдовша тривалість опромінення була 21 добу, що відповідало 1310 Грей. Раманівську спектроскопію досліджуваних зразків адсорбента на основі TiSi проводили з використанням раманівського спектрометра XploRA PLUS у Центрі колективного користування «Лабораторія експериментальної і прикладної фізики» ДВНЗ «УжНУ».

Результати показують, що величина максимальної адсорбції катіонів барію адсорбентом на основі силікату титану після опромінення дозою бета-випромінювання 1310 Грей становить 140.5±9.2 мг/г (6.55 %) при довірчому інтервалі 95 %. Величина адсорбції катіонів Ba^{2+} у таких же самих умовах неопроміненим силікатом титану складає 144 мг/г. Величини адсорбції у межах похибки співпадають. Раманівські спектри опроміненого і неопроміненого силікату титану також є ідентичними, при цьому на них не ідентифікуються вільні радикали або йонні формування, які би свідчили про зміну властивостей поверхні адсорбента під дією дози бета- радіоактивності 1310 Грей.

Основний висновок даної роботи є такий, що досліджений зразок адсорбента на основі силікату титану є радіаційно-стійким. Він може витримувати дозу 1310 Грей без зміни адсорбційних властивостей. Силікат титану може бути використаний для вилучення ^{90}Sr із водних розчинів і як носій для ^{90}Sr при захороненні радіоактивних відходів.

***Ключові слова**: адсорбент, опромінення, силікат титану, Раманівська спектроскопія*

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