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REMOVAL OF CESIUM AND STRONTIUM IONS FROM AQUEOUS SOLUTIONS USING METAKAOLIN BASED GEOPOLYMERS

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The aim of presented work was to synthesize geopolymers based on the metakaolin and to determine their adsorption capability in the process of cesium and strontium ions removal from the aqueous solutions. New approaches were proposed for obtaining the two samples of geopolymers in technologically suitable forms. Morphology of materials was investigated by means of X-ray fluorescence analysis (XRF), low-temperature nitrogen adsorption/desorption and scanning electron microscopic studies (SEM). As it follows from the data of XRF analysis, SiO₂ and Al₂O₃ oxides found to be the major components in all samples investigated (~ 54–84 wt. %). As was determined by SEM studies, geopolymers consisted from nanosized particles, amorphous geopolymer binder and unreacted kaolin. It has been found that all samples involve the mesopores with approx. 1–40 nm radii. The greatest specific surface area calculated by the Brunauer-Emmet-Teller (BET) method had the sample obtained in the forms of spherical rods ($S_{BET} = 88 \text{ m}^2/\text{g}$) that about 10 times greater than for initial kaolin taken for synthesis. The ion exchange capacities of materials in the process of Cu²⁺, Cs⁺ and Sr²⁺ removal from water solution were determined and it was found that these properties depend on the method of materials obtaining. Data showed that the geopolymers were more effective for removal of the desired ions than initial kaolin. The greatest adsorption capacity towards cesium ions was received on the samples obtained in forms of pyramids and was reached 1.75 mmol/g. Experimental data were fitted into the Langmuir models and the main Langmuir constants were calculated. When analysing the data of investigation with comparing the literature data it was noted that geopolymers obtained can be used in adsorption technology for purification of water from radionuclides as technologically suitable sorbents.

Keywords: cesium and strontium ions removal, metakaolin based geopolymers, impact of synthetic route, morphology

INTRODUCTION

Due to widespread application of the nuclear energy in the world the removal of hazardous radionuclides, first of all cesium and strontium, attracts the great attention of researchers [1, 2]. Various kinds of sorbents are widely employed in this process such as titanates, vanadates, and tungsten based materials, manganese oxides, hexacyanoferrates, metal sulfides, ammonium molybdophosphates, or hydroxyapatite, etc. [1]. Practice poses certain well-known requirements to sorbents, first of all to low cost of their fabrication, to uniformity parameters of porous structure and to high adsorption capacity towards desired ions.

The potential materials in aspects to their low costs production are the clay minerals [3, 4], cesium and strontium adsorption have been successfully studied on the bentonites [5], clinoptilolites [6], layered clay materials (kaolin)

[7], natural zeolites [8], etc. It is necessary to note that high adsorption capability of these sorbents is reached only due to activation (mechanical or chemical modification) of initial materials [3–8].

Geopolymers are three-dimensional amorphous inorganic polymers formed by the activation of aluminosilicate precursors with an alkali silicate solution at temperatures between 20 and 100 °C [9–13] (structure presented in Fig. 1). Aluminosilicate precursors such as fly ash, blast furnace slag, rice husk ash, volcanic rock powders and kaolin have been used for geopolymers preparation. The main characteristics of these polymers that caused the great interest of researchers are that they can be synthesized at low temperatures (20–100 °C) and have useful technical properties such as high compressive strength, thermal stability at temperatures up to 1300–1400 °C and great durability against various acids and salts.

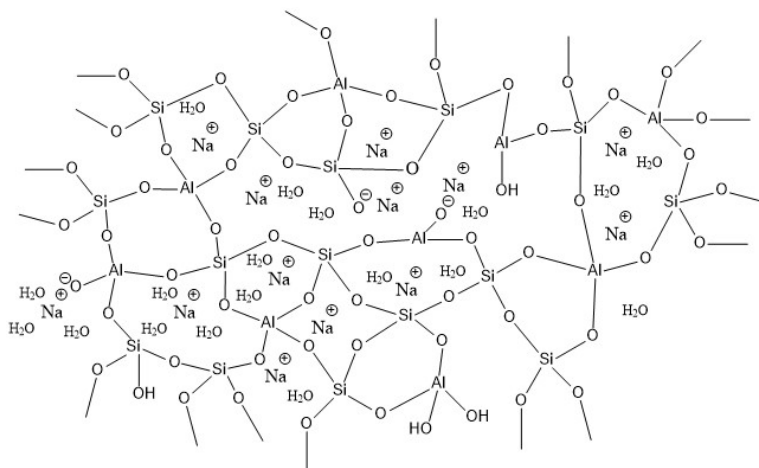


Fig. 1. Three-dimensional network structure of geopolymer inorganic materials

The aim of presented work has been to synthesize the geopolymers using low cost reagents (kaolin and sodium metasilicate produced by Ukrainian enterprises) and to examine their adsorption capacity towards Cu^{2+} , Cs^+ and Sr^{2+} ions. New approaches to the synthesis of geopolymers materials is approved in this work on the last stage of reaction (polymerization) that it has lead to obtaining two samples with significantly greater surface area and adsorption capacities compared the initial kaolin. The solid phases are investigated by XRF analysis, scanning electron microscopic studies, low-temperature nitrogen adsorption/desorption method. The ion-exchange capacity for desired ions have been evaluated and impact of method used on the properties of materials are found. Adsorption experiments have been supported by fitting of isotherms by Langmuir model.

EXPERIMENTAL

Materials. Metakaolin was prepared from kaolin (AKW Ukrainian Kaolin Company, Vynnytsa, Ukraine) which was placed in a muffle furnace and calcined at 800 °C for 2 h. The chemicals, sodium hydroxide (NaOH), cesium chloride (CsCl), strontium chloride ($\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$), copper nitrate $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ were of analytical grade (Ostchem, Ukraine). Initial and equilibrium concentration of metals in solution for adsorption experiment was measured by an atomic absorption spectrometer. The aqueous sodium metasilicate solution of $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio = 3.01 (Na_2O – 9.6 %, SiO_2 – 28 %, density – 1.42 g/L) was produced by Ukrainian enterprise

(PJSC “Zaporizhskloflus”) and analyzed by acid-basic titration method.

Synthesis of geopolymers from metakaolin.

The geopolymers synthesis involved next preparation steps: 22.6 g of NaOH was added to 100 mL of sodium metasilicate (initial modulus = 3.01), the mixed solution was sealed and placed under ultrasonic vibration for 5 min and then was left to cool for 24 h. The modified water glass with the molar ratio of $\text{SiO}_2/\text{Na}_2\text{O} = 1.3$ was obtained. Prepared metakaolin (20.1 g) was added into the as-prepared water glass solution (16.1 mL) according to the molar ratio $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3 = 0.8$ and $\text{SiO}_2/\text{Al}_2\text{O}_3 = 2.96$, then 10.8 mL of deionized water was added with the molar ratio of $\text{H}_2\text{O}/\text{Na}_2\text{O} = 19$, the mixed slurry was mechanically stirred for 15 min.

The last step of geopolymers production was a polymerization of reagents mixture obtained in the previous stage. First sample (named GP 1) was obtained by standard method fabrication of membrane or powder [9] but after maturing for 30 min at room temperature, the mixed viscous solution was cast in a special gride-like form and placed in an oven to cure for 24 h at 60 °C that leads to obtain ion of the pyramids with diameter of faces an about 4 mm (Fig. 2 a). After cooling samples have been washed by deionized water and diluted HCl up to pH = 5 (during 6–7 days).

Second sample (GP 2) was obtained by another way: mixed on the first stage solution had been heated up to 60 °C by microwave oven and then was extruded to NH_4Cl solution ($\text{Na}_2\text{O}/\text{NH}_4\text{Cl} = 1$). Spherical rods obtained as a result are shown in the Fig. 2 b. Samples were

washed up to neutral pH during the shorter time (1 day) than need for washing the samples GP 1.

Characterization of geopolymeric materials.

The chemical composition of the initial kaolin clay, metakaolin and geopolymers obtained was performed with XRF-spectrometer ELVAX CEP-01 (Ukraine).

Specific surface areas and pore size distributions for the synthesized samples were calculated from nitrogen adsorption/desorption curves (NOVA 2200e, Quantachrome, USA) using the Nova Win 2.0 software. The total surface area of the materials S_{total} was calculated by the Brunauer-Emmet-Teller method (BET). The total pore volume (V_{total}) was calculated from the volume of nitrogen adsorbed converted to liquid at a pressure close to $P/P_0 = 1$. To acquire the volume and radii of mesopores (V_{meso} , R_{meso}), Barrett-Joyner-Halenda (BJH) were used. The average pore radii (R_{pore}) was determined from the total pore volume (V_{total}) of the materials and its specific surface area (S_{total}) by equation $R_{pore} = 2V_{total}/S_{total}$. The micropore volume (V_{micro}) was calculated by subtracting the value of V_{meso} from V_{total} . Pore radii distributions were obtained from isotherms in terms of the density functional theory (DFT).

The morphology of kaolin and prepared samples was examined with a scanning electron microscope (SEM) JSM 6700F (JEOL, Japan).

Sorption properties of the samples of magnesium silicate were studied under static conditions: 0.025 g of samples was added to 25 mL of metals aqueous solution with

concentration of 0.1–10 mmol/L. Resulting mixtures are shaken for 4 h, and left for 1 day at 25 °C, after which the sorbents are removed by means of filtration and the metals content in filtrates are determined using a Shimadzu AA 6300 (Shimadzu, Japan) atomic absorption analyzer.

Adsorption capacity (q_e , mmol/g) was calculated by the next equation:

$$q_e = (C_0 - C_e) V/m, \quad (1)$$

where C_0 and C_e are the initial and equilibrium metal concentration in solution, respectively, mmol/L; V is the aliquot volume (L); and m is the mass of the adsorbent (g).

Experimental data were fitted into the Langmuir (2) and Freundlich (3) models, which are commonly used to describe liquid–solid systems [14] by the following equations:

$$q_e = Q_0 K_L C_e / (1 + K_L C_e), \quad (2)$$

$$q_e = K_F C_e^{1/n}, \quad (3)$$

where q_e is the adsorption capacity (mmol/g); C_e is the equilibrium concentration of the adsorbate (mmol/L); Q_0 is the maximum adsorption capacity of the adsorbent (mmol/g); and K_L is the Langmuir sorption equilibrium constant (L/mmol), K_F ((mmol/g)/(L/mmol)ⁿ) and n are the Freundlich adsorption constants. To evaluate the correlation between the experimental data and theoretical models, the coefficient of determination (R^2) was calculated [14].

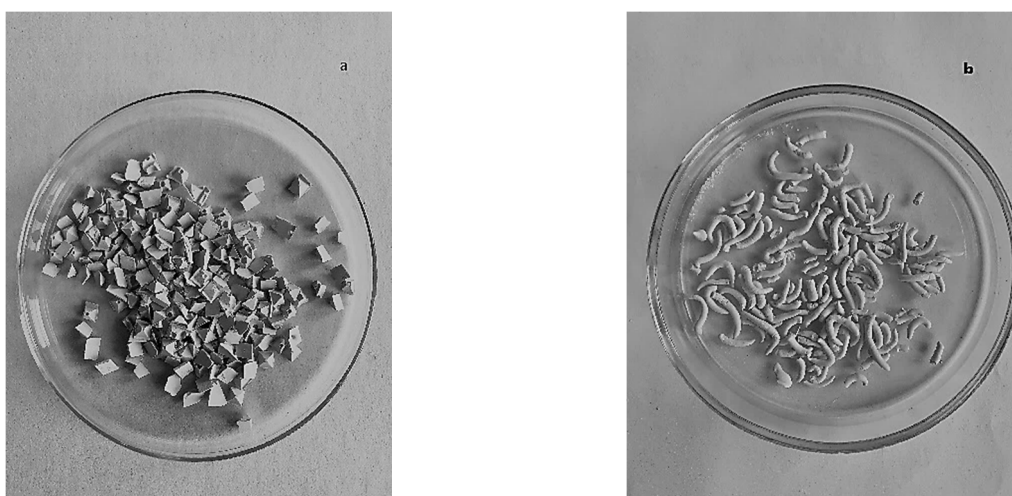


Fig. 2. Photographs of geopolymers obtained as pyramids GP 1 (a) and spherical rods GP 2 (b)

RESULTS AND DISCUSSION

The chemical compositions of the starting and obtained materials determined by XRF analysis are presented in the Table 1. As follow from the data SiO_2 and Al_2O_3 oxides are the major components in all samples (~ 54–84 wt. %); for geopolymers it has been fixed the great increasing of silica contents due to process of polymerization [15–17]. Last line of table includes the content of other oxides such as: MgO , TiO_2 , CaO , Na_2O (< 1 % wt. of each). Its content is lower in geopolymers than in initial kaolin due washing the final products by the water and dilute acid.

It is known that morphology of the materials determines on their adsorption characteristics and greatly depends on the synthetic routes used for its fabrication [18–20]. SEM images of kaolin and two samples of geopolymers obtained are given in the Fig. 3 *a, b, c*, respectively. In accordance with early reports [11, 15, 16, 21], kaolin contents sheets with size an about 1–10 μm (*a*), while the geopolymers are both composed of nano-sized particles and amorphous geopolymers binder (*b, c*). The unreacted kaolin sheets are present in the structure of pyramids (GP 1 *b*). The appearance of geopolymer binder is similar to that one generally observed in the case of metakaolin based geopolymers [16, 21].

Table 1. Chemical composition of the starting and as-prepared geopolymers materials (oxides wt. %)

Composition	Kaolin	Metakaolin	GP 1	GP 2
Al_2O_3	24.7	19.9	25.5	28.3
SiO_2	29.9	40.5	58.7	53.1
Fe_2O_3	0.9	0.9	2.5	1.1
K_2O	0.7	0.6	1.0	1.1
Residue*	43.8	38.1	12.3	16.4

* – other oxides

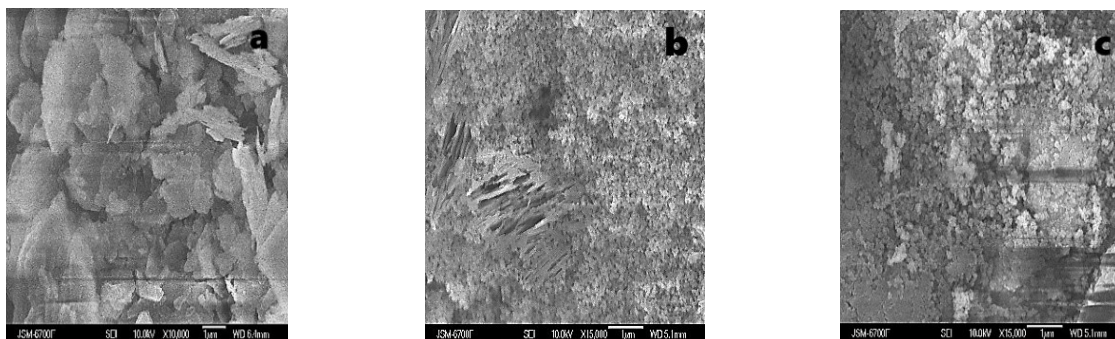


Fig. 3. SEM images of kaolin (*a*) and geopolymers: GP 1 (*b*) and GP 2 (*c*)

Data received by low-temperature nitrogen adsorption/desorption method are presented in the Table 2 and Fig. 4 *a, b*. Nitrogen adsorption/desorption isotherms the kaolin and geopolymers according the IUPAC classification belong to the II type with the hysteresis loop of the H3 type (Fig. 4 *a*). As seen from porosity data, the materials based on the kaolin show the significantly higher than the initial kaolin BET specific surface area, total pore volume and volume of mesopores calculated by the BET and BJH methods (Table 2). Calculated from

isotherms porosity data for geopolymers are in accordance with data published early [9, 13, 17]. Analyzing the pore size distribution calculated from desorption branches of the isotherms in term of DFT method (Fig. 4 *b*), it is found that in all samples are mesoporous with approx. 1–40 nm radii. The most interesting feature found for two geopolymers samples is the absence of mesopores radii less than 5 nm for sample GP 1 (pyramids) while the sample GP 2 has a lot of pores with radii 2.5 nm (Fig. 4 *b*).

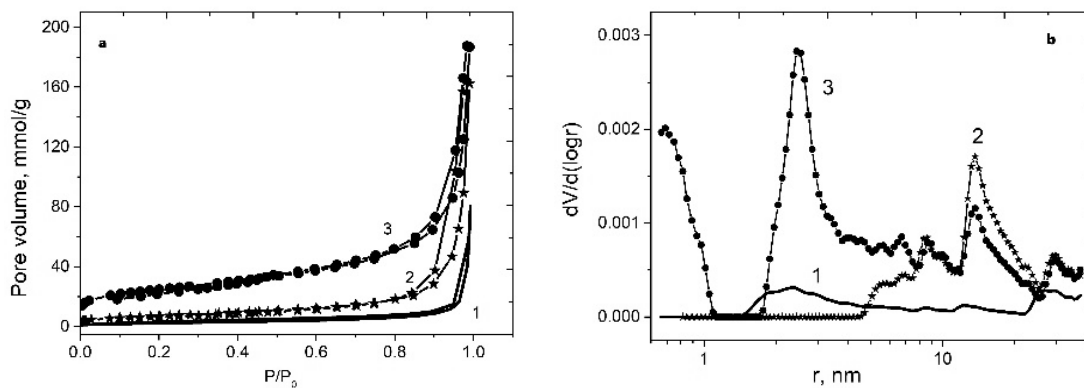


Fig. 4. Nitrogen adsorption/desorption isotherms (a) and pore size distributions obtained in term of DFT method (b) for the kaolin (1) and two samples of geopolymer GP 1 (2) and GP 2 (3)

Table 2. Porosity data for initial kaolin and geopolymers

Sample	Kaolin	GP 1	GP 2
BET surface area, S_{total} (m ² /g)	9	26	88
Total pore volume, V_{total} (cm ³ /g)	0.12	0.25	0.29
Mesopore volume, V_{meso} (cm ³ /g)	0.12	0.25	0.28
Micropore volume, V_{micro} (cm ³ /g)	0	0	0.01
Average pore radius, R_{pore} (nm)	28	19.2	6.6

Investigation of copper ions adsorption has been carried out for choosing the best samples during design of method and for determining the adsorption mechanism realized onto obtained materials. For it the experimental data received at copper ions adsorption are fitted by two theoretical models – Langmuir and Freundlich, result are shown in Fig. 5 and in the Table 3. As

seen from presented data, the Lngmuir model is more acceptable to describe the adsorption of Cu²⁺ on the synthesized geopolymer and the adsorption process is a homogeneous monolayer adsorption. This model is used for further fitting the experimental data received with the target ions.

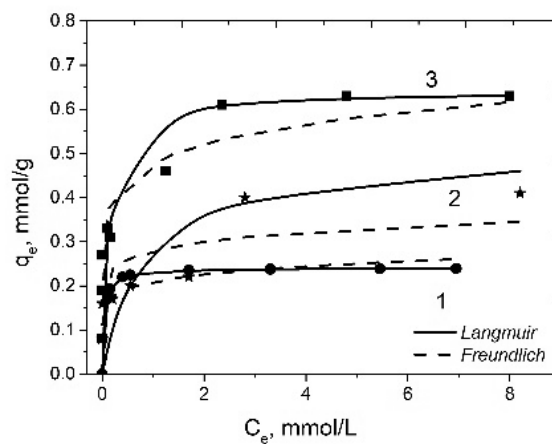


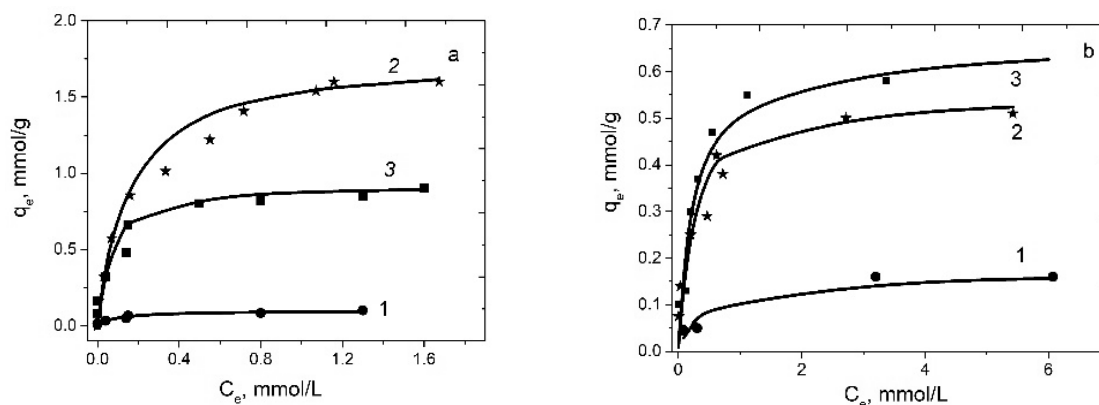
Fig. 5. Experimental data and plots of Langmuir and Freundlich isotherm models for the adsorption of Cu²⁺ by samples: kaolin (1) and geopolymer GP 1 (2) and GP 2 (3)

Table 3. Langmuir and Freundlich isotherm constants for the adsorption of copper ions onto kaolin and kaolin based geopolymers

Samples	Kaolin	GP 1	GP 2
Langmuir			
Q_0 (mmol/g)	0.24	0.50	0.64
K_L (L/mmol)	27.1	1.4	9.2
R^2	0.98	0.99	0.99
Freundlich			
K_F (mmol/g)(L/mmol) ^{1/n}	0.21	0.28	0.48
1/n	0.11	0.10	0.12
R^2	0.97	0.94	0.98

Studies of cesium and strontium ions adsorption on the kaolin and kaolin based geopolymers as isotherms in range $C_0 = 1-10$ mmol/l presented in the Fig. 6 *a, b* and as Langmuir constants calculated and given in Table 4. According the Q_0 values (maximum monolayer coverage capacity, Table 4) geopolymers are most active in adsorption process than initial kaolin. The adsorption capacities for strontium removal are 0.55 and 0.65 mmol/g for GP 1 and GP 2, respectively, while pyramids are most effective in adsorption of cesium (1.75 mmol/g). These fact can be explained by existence in pyramids structure the

mesopores of greater size. Adsorption data indicate that the geopolymer materials have more sorption sites for investigated ions than the kaolin taken to the synthesis. Such result can be assigned to transformation crystalline structure of initial material into amorphous one leading to formation of more porous structure encouraging cesium and strontium ions. Compared to data for geopolymers reported in the literature [23–25], the samples synthesized in this paper show good promise for its use in water and wastewater treatments as technologically suitable sorbents with high adsorption capacities towards cesium and strontium ions.

**Fig. 6.** Sorption isotherms for Cs^+ (*a*) and Sr^{2+} (*b*) adsorbed onto the samples of kaolin (1), GP 1 (2) and GP 2 (3)**Table 4.** Langmuir isotherm constants for the adsorption of cesium and strontium ions onto kaolin and geopolymers obtained

Samples	Kaolin		GP 1		GP 2	
	Cs^+	Sr^{2+}	Cs^+	Sr^{2+}	Cs^+	Sr^{2+}
Q_0 (mmol/g)	0.10	0.17	1.75	0.55	0.93	0.65
K_L (L/mmol)	13.5	2.39	6.64	4.7	19.2	4.02
R^2	0.99	0.99	0.99	0.99	0.99	0.98

CONCLUSIONS

New approaches were proposed for obtaining the two samples of geopolymers based on the kaolin in technologically suitable forms. Morphology of samples synthesized was investigated by means of X-ray fluorescence analysis (XRF), low temperature nitrogen adsorption/desorption method, scanning electron microscopic studies (SEM). As follow from the data of XRF analysis all samples involved the SiO_2 and Al_2O_3 oxides (~ 82–93 wt. %) and other oxides in the quantities less than 1 % of each. SEM studies found that geopolymers consist from nanosized particles and amorphous geopolymer binder, in the case of sample obtained in the form of pyramids the unreacted kaolin was found. Materials synthesized from the kaolin showed the significantly higher than the initial kaolin BET specific surface area, total pore volume and volume of mesopores. The absence was fixed of

mesopores radii less than 5 nm for sample GP 1 (pyramids) while the sample GP 2 had a lot of pores with radii 2.5 nm. The greatest specific surface area calculated by the Brunauer-Emmett-Teller (BET) method was for sample obtained in the forms of spherical rods ($S_{\text{BET}} = 88 \text{ m}^2/\text{g}$) that an about 10 times bigger than for initial kaolin taken for synthesis. The greatest adsorption capacity towards cesium and strontium ions were received on the samples obtained in forms of pyramids and spherical rods – 1.75 (Cs^+) and 0.65 mmol/g (Sr^{2+}), respectively. High adsorption capacities indicated that the geopolymer materials have more sorption sites for investigated ions than the kaolin. Analysing the data of investigation with comparing the literature data it was noted that geopolymers obtained can be used in adsorption technology for purification of water from radionuclides as technologically suitable sorbents.

Вилучення іонів цезію та стронцію з водних розчинів з використанням геополімерних матеріалів на основі метакаоліну

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Метою представленої роботи був синтез геополімерних матеріалів на основі метакаоліну та визначення їхньої адсорбційної здатності у процесах вилучення іонів цезію та стронцію з водних розчинів. Запропоновано нові підходи для одержання двох зразків геополімерів у технологічно придатних формах. Морфологію матеріалів досліджували за допомогою рентгенофлуоресцентного аналізу (РФС), методу низькотемпературної адсорбції/десорбції азоту, скануючої електронної мікроскопії (СЕМ). Як впливає з даних РФС, основними компонентами в усіх досліджуваних зразках виявилися оксиди SiO_2 та Al_2O_3 (~ 54–84 мас. %). Як було встановлено дослідженнями СЕМ, геополімери складаються з нанорозмірних частинок, аморфного геополімерного зв'язуючого і каоліну, що не прореагував. Було встановлено, що всі зразки мають мезопори розміром 1–40 нм. Найбільша питома поверхня, розрахована методом Брунауера-Еммета-Теллера (БЕТ), була виявлена для зразка, отриманого у формі сферичних стрижнів ($S_{\text{BET}} = 88 \text{ м}^2/\text{г}$), що приблизно в 10 разів більше, ніж для вихідного каоліну, взятого для синтезу. Визначено іонообмінну здатність матеріалів у процесі видалення іонів Cu^{2+} , Cs^+ та Sr^{2+} з водних розчинів та встановлено, що ці властивості залежать від способу отримання матеріалів. Дані показали, що геополімери більш ефективні для видалення обраних іонів, ніж вихідний каолін. Найбільша адсорбційна ємність щодо іонів цезію була отримана на зразках у формі пірамід, і досягала 1.75 ммоль/г. Для обробки експериментальних даних була використана теоретична модель Ленгмюра та розраховані основні константи Ленгмюра. Аналізуючи дані досліджень із порівнянням з літературними даними, відзначено, що отримані геополімери можуть бути використані в адсорбційній технології для очищення води від радіонуклідів як технологічно придатні сорбенти.

Ключові слова: вилучення іонів цезію та стронцію, геополімерні матеріали на основі метакаоліну, вплив методу синтезу, морфологія

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