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SORPTION OF Ce(III) ON Na-MODIFIED TRANSCARPATHIAN CLINOPTILOLITE

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The sorption properties of the Na-form of Transcarpathian clinoptilolite towards trace amounts of Ce(III) under dynamic conditions were investigated . The optimal conditions for Ce(III) sorption were determined depending on the pH of the solution, concentration of cerium salt, and the thermal treatment of the sorbent. The sorption capacity of the Na-form of clinoptilolite towards Ce(III) under optimal conditions is 34.5 mg cerium per 1 g of sorbent.

Keywords: sorption, Na-clinoptilolite, solid phase extraction, cerium(III), sample preparation.

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1. Introduction

Cerium is the most common rare earth element. Its content in the Earth's crust is $6.8 \cdot 10^{-3}$ % [1]. Cerium, along with other lanthanides, is widely used in many modern high-tech industries, such as nuclear energy, metallurgy, electronics, optics, and medicine, due to its unique magnetic, catalytic, electrical, and optical properties [2, 3]. Due to the increasing use of rare earth elements in industry, significant amounts of them end up in the environment. When analyzing natural objects and technological solutions for Ce content, it is usually necessary to preconcentrate, separate, and extract this lanthanide. There is also the problem of removing Ce from technological solutions. For such purposes, the solid-phase extraction method can be used, therefore, the search for effective sorbents for trace amounts of Ce is relevant. This method has a number of advantages over liquid extraction and other concentration methods: high enrichment and extraction factors, rapid phase separation, no or minimal use of toxic organic solvents, possibility of combining with various methods of determining REE, low cost, possibility of automating sample preparation, and ease of operation. Natural zeolites are promising sorbents for lanthanides [3–6]. In particular, methods for concentrating many rare earth elements have been developed based on the most common natural zeolite – Transcarpathian clinoptilolite [7–13]. Compared to activated carbon and ion exchange resins, clinoptilolite is ~2000 and ~5000 times cheaper, respectively [14].

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The advantage of natural zeolites lies not only in the low cost of the material, but also in their high efficiency and selective sorption.

To improve the physicochemical and analytical properties of zeolites, they are often subjected to chemical modification, which involves increasing the size of internal cavities and channels or creating adsorption-active centers on the surface. Modification can be carried out using various inorganic and organic substances [15–20]. It is known that the Na-form of Transcarpathian clinoptilolite differs significantly in its sorption properties from the natural form and usually has a higher sorption capacity for heavy metal ions [21–24].

Previously, we investigated the sorption properties of natural [25] and acid-modified [26] forms of Transcarpathian clinoptilolite with respect to Ce(III). Based on the natural form of Transcarpathian clinoptilolite, a selective sorption-luminescence method for determining trace amounts of Ce(III) has been developed [27].

The purpose of our article was to study the sorption properties of the Na-form of Transcarpathian clinoptilolite towards trace amounts of Ce(III) in aqueous solutions and to investigate the potential possibility of using this sorbent, obtained on the basis of natural aluminosilicate, in the solid-phase extraction method during sample preparation for analysis and the removal of Ce(III) from solutions.

2. Materials and methods

The clinoptilolite from the deposit in the village of Sokyrnytsia, situated in the Zakarpattia region of Ukraine contained 85–90 % (mass fraction) of the main component. Its specific surface area, determined by water sorption, was equal to 59 $m^2 \cdot g^{-1}$ [28]. The clinoptilolite formula in the oxide form (mass fraction) is as follows: SiO_2, 67.29; Al_2O_3, 12.32; TiO_2, 0.26; Fe_2O_3, 1.26; FeO, 0.25; MgO, 0.99; CaO, 3.01; Na_2O, 0.66; K_2O, 2.76; H_2O, 10.90 [29].

The selected batch of natural clinoptilolite was ground in a ball mill and sieved through a sieve with a grain diameter of 0.20–0.31 mm. Zeolite with the appropriate grain size was washed with distilled water and dried at room temperature to an air-dry state. The Na-form of clinoptilolite was obtained according to [24] as follows: clinoptilolite grains were pretreated with a 0.25 M HNO_3 solution for 4 hours at room temperature. Then, the zeolite fraction was separated, washed with distilled water to remove traces of acid, and treated with a 1 M $NaNO_3$ solution for 1–1.5 hours (repeated 7–8 times). The obtained Na-form of clinoptilolite was dried at room temperature. Thermogravimetric studies showed that the TG and DTG curves of uncalcined samples of natural clinoptilolite and its Na-form are practically identical [21]. However, Na-modified clinoptilolite has a more developed specific surface area (65 $m^2 \cdot g^{-1}$), which is occupied by physically and chemisorbed molecular water [23].

The Na-clinoptilolite samples were calcined at appropriate temperatures for 2.5 hours in a WSU 200 drying oven (Germany) or SNOL 7.2/1100 muffle furnace (Lithuania), and then cooled in a desiccator.

All reagents were of analytical grade. Standard aqueous solutions of cerium nitrate (concentration: 1.0 $mg \cdot mL^{-1}$) were prepared by dissolving the metallic cerium (99.9 % purity) in the nitric acid solution (1:1). The working solutions of Ce(III) were prepared by the appropriate dilution of the standard solution. The 0.05 % solution of sulfarsazene was prepared using 0.05 M aqueous solution of $Na_2B_4O_7$. Solutions of all other reagents were prepared by dissolving weights in distilled water.

The sorption characteristics of Na-clinoptilolite were studied under dynamic conditions in solid-phase extraction mode. For this purpose, a peristaltic pump of the NP-101 brand (Ukraine) was used, to which a sorption cartridge was connected. To determine the dynamic sorption capacity, solutions of Ce(III) of various concentrations were passed through a sorption cartridge filled with 0.60 g of sorbent at a rate of 5.0 $mL \cdot min^{-1}$ using a peristaltic pump. The passage point of cerium(III) was registered spectrophotometrically using the reaction that leads to a formation of orange complex of cerium(III) with sulfarsazene. Sulfarsazene changes its color from yellow to orange starting from the cerium(III) concentration of 100 $ng \cdot mL^{-1}$. The passage moment of Ce(III) was determined using a DR/4000V spectrophotometer (HACH) at 540 nm.

The sorption processes were studied at 20 ± 1 °C.

3. Results and Discussion

The sorption capacity of clinoptilolite may vary when solutions with different initial concentrations of Ce(III) ions are passed through it. To study the dependence of sorption on the concentration of Ce(III) ions on the Na-form of clinoptilolite, solutions with concentrations of 0.5 $\mu g \cdot mL^{-1}$, 1.0 $\mu g \cdot mL^{-1}$, and 2.0 $\mu g \cdot mL^{-1}$were used. The results are shown in the table.

Dependence of the sorption capacity of Na-clinoptilolite on concentration of Ce(III), pH 8.0

Concentration of Ce(III), $\mu g \cdot mL^{-1}$	Sorption capacity, $\mu g \cdot g^{-1}$
0.5	34 500
1.0	24 670
2.0	13 700

The data show that the sorption of lower concentrations of Ce(III) ions is much more efficient. This dependence is very important in the sorption of microquantities of elements and is observed both in natural [25] and H-form clinoptilolite with respect to Ce(III) ions [26].

Probably, the different sorption capacity of Na-clinoptilolite with respect to low and high concentrations of Ce(III) is associated with the different ability to form aqua- and hydroxocomplexes. As the concentration of Ce(III) in the solution increases, polynuclear cationic hydroxocomplexes of the composition $Ce_3(OH)_5^{4+}$ and $Ce_2(OH)_2^{4+}$ are formed. Since the sizes of polynuclear hydroxocomplexes are larger than those of conventional hydroxocomplexes, their access to zeolite pores is difficult, which decreases the sorption capacity of the Na-form of clinoptilolite.

One of the important stages in selecting the optimal conditions for concentrating Ce(III) on the Na-form of clinoptilolite is to study the dependence of the sorption capacity of zeolite on the acidity of the aqueous solution. The required pH value of Ce(III) salt solutions was created by adding 0.1 M NaOH or HNO_3. The results of the study are shown in Fig. 1.

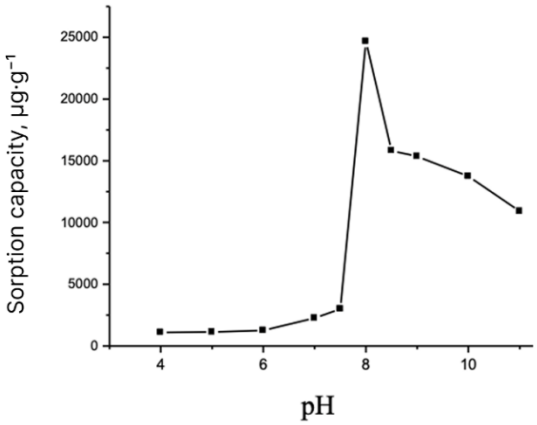


Fig. 1. Dependence of the sorption capacity of Na-modified clinoptilolite towards Ce(III) on the pH value of the aqueous solution ($C_{Ce(III)}$ = 1.0 μg·mL^{-1})

The results obtained indicate that the sorption of Ce(III) ions on Na-clinoptilolite is most effective in a weakly alkaline medium. In the pH range from 4.0 to 7.5, the sorption capacity of zeolite gradually increases. With a further increase in pH to 8.0, the sorption properties increase sharply and reach their maximum value at pH 8.0. At higher pH values of the solution, there is a gradual decrease in the sorption of Ce(III) ions.

It is known [18, 30] that the adsorption-active centers of Transcarpathian clinoptilolite with respect to metal ions are mainly OH-groups. Therefore, we believe that the nature of the sorption process of Na-clinoptilolite with respect to Ce(III) ions is determined both by the peculiarities of the chemistry of the zeolite surface and the forms of Ce(III) in aqueous solutions at different pH. At low pH values, the dissociation of hydroxyl groups with H^+ eliminatoin on the surface of mineral grains, primarily responsible for the sorption of heavy metals from solutions, is almost completely suppressed, which determines the low adsorption capacity of Na-clinoptilolite with respect to Ce(III) ions at pH < 6. At low pH values, a competing reaction of ion exchange for protons also occurs. As the pH increases, the dissociation of surface groups increases, while sorption increases simultaneously. Also, with an increase in pH, the form of existence of Ce(III) in solution changes. In particular, with a decrease in the content of cationic forms of aquacomplexes $[Ce(H_2O)_9]^{3+}$, an increase in the proportion of cationic aquahydroxocomplexes $[Ce(OH)(H_2O)_8]^{2+}$, $[Ce(OH)_2(H_2O)_7]^+$ is observed. We assume that under optimal sorption conditions (pH = 8.0), the Na-form of clinoptilolite most effectively sorbs cationic forms of aqua and aquahydroxocomplexes of Ce(III). With an increase in the pH of the medium (pH > 8.0), a decrease in sorption capacity is observed, since anionic complexes prevail in an alkaline medium, and, as is known, these complexes are not sorbed on zeolites.

To ensure pH stability, it is advisable to use a buffer solution. This improves the metrological characteristics of concentration and also reduces the influence of various impurities on the concentration process, as it ensures a stable ionic strength of the solution. We tested Tris buffer solution and urotropin buffer solution with a pH of 8.0. When using a Tris buffer solution (C_{buffer} = 4·10^{-4} M), the sorption capacity of Na-clinoptilolite is 5.33 mg·g^{-1}, which is 4.6 times less than the sorption capacity of Na-clinoptilolite with respect to Ce(III) without using a buffer solution. When using urotropin buffer solution, the sorption capacity of Na-clinoptilolite was ~20 % lower compared to Tris buffer solution.

The sorption properties of Transcarpathian clinoptilolite and its modified forms significantly depend on their preliminary heat treatment [7–13, 16–19, 21–28]. The Na-clinoptilolite was heat treated for 2.5 hours at different temperatures and, after cooling in a desiccator, its sorption capacity with respect to Ce(III) ions was determined. The results of this study are shown in Fig. 2.

Fig. 2 shows that preliminary calcination of Na-clinoptilolite to 250 °C leads to a decrease in sorption capacity, while further calcination of zeolite to 500 °C results in a significant increase in sorption efficiency. According to source [30], physically adsorbed and ligand water are released by clinoptilolite in this temperature range. It is known that during the dehydration of zeolites, exchange cations are displaced, which affects the cross-section of the channels. Perhaps such blocking leads to a weakening of the sorption properties of Na-clinoptilolite with respect to large Ce(III) hydroxocomplexes, which is observed in the temperature range from 20 to 250 °C in Fig. 2.

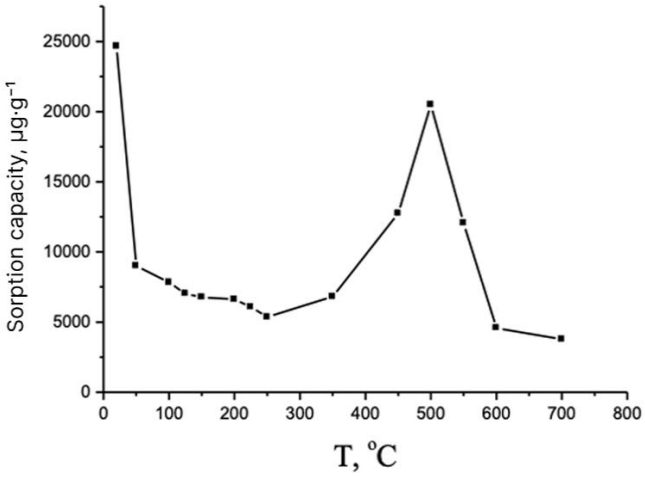


Fig. 2. Dependence of the sorption capacity of Na-clinoptilolite towards Ce(III) on thermal treatment (pH 8.0; $C_{Ce(III)} = 1.0$ μg·mL^{-1})

In the range from 250 °C to 500 °C, the sorption capacity increases sharply and reaches its maximum at 500 °C. Since the sorption-active centers of Transcarpathian clinoptilolite and its modified forms with respect to rare earth elements are mainly surface OH-groups, the increase in the sorption efficiency of Na-clinoptilolite samples in this area is probably associated with an increase in the surface OH-groups of zeolite water molecules, as well as surface silanol groups (Si–OH).

A significant decrease in the sorption properties of zeolite with respect to Ce(III) ions at temperatures > 500 °C is explained by the processes of deep dehydroxylation of the zeolite surface and its amorphization. It is under such conditions that the Na-form of clinoptilolite begins to break down.

As shown in Fig. 2, samples of uncalcined Na-form clinoptilolite demonstrate the highest sorption capacity.

Based on the generalized research results, we see that the maximum sorption capacity of the Na-form of Transcarpathian clinoptilolite with respect to Ce(III)

is 34.5 $mg \cdot g^{-1}$ (see table), which exceeds the sorption capacity of natural [25] and H-form [26] of this zeolite by 5.4 and 5.9 times, respectively.

4. Conclusions

The sorption properties of the Na-form of Transcarpathian clinoptilolite with regard to trace amounts of Ce(III) under dynamic conditions have been investigated. The optimal sorption conditions are as follows: pH 8.0; flow rate of Ce(III) solution with a concentration of 0.5 $\mu g \cdot mL^{-1}$ – 5 $mL \cdot min^{-1}$; sorbent grain diameter – 0.20–0.31 mm. When using Tris buffer and urotropin buffer solutions (pH 8.0), the sorption capacity of the Na-form of clinoptilolite is significantly decreased. The maximum sorption capacity is found in uncalcined samples of Na-clinoptilolite with respect to Ce(III) – 34.5 $mg \cdot g^{-1}$, which exceeds the sorption capacity of the natural and H-forms of this zeolite by 5.4 and 5.9 times, respectively.

The ability of the Na-form of Transcarpathian clinoptilolite to sorb both low and high concentrations, its high sorption capacity give reason to propose this sorbent for the concentration of Ce(III) ions in solid-phase extraction mode at the stage of water preparation for analysis, as well as for the removal of Ce(III) ions from technological solutions.

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СОРБЦІЯ Ce(III) НА Na-ФОРМІ ЗАКАРПАТСЬКОГО КЛИНОПТИЛОЛІТУ

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Методи визначення рідкісноземельних елементів здебільшого потребують попередньої підготовки зразків, яка охоплює концентрування, розділення та вилучення металів з природних об'єктів і технологічних розчинів. Одним із способів вирішення проблем підготовки зразків для аналізу є метод твердофазної екстракції з використанням різних сорбентів, зокрема природних цеолітів. Найпоширенішим із природних цеолітів є закарпатський клиноптилоліт.

Досліджено сорбційні властивості Na-форми закарпатського клиноптилоліту стосовно слідових кількостей Ce(III) у динамічних умовах. Визначено оптимальні умови сорбції Ce(III) залежно від концентрації солі церію, pH розчину та температури попереднього прожарювання сорбенту. Для забезпечення сталості pH доцільно застосовувати буферний розчин. Це поліпшує метрологічні характеристики концентрування, а також зменшує вплив різних домішок на процес концентрування, оскільки забезпечує сталу йонну силу розчину. Апробовано трис-буферний розчин та уротропіновий буферний розчин з pH 8,0. Однак використання цих буферних розчинів призвело до зниження сорбційної здатності Na-клиноптилоліту стосовно Ce(III).

Оптимальні умови сорбції такі: pH 8,0; швидкість пропускання розчину Ce(III) з концентрацією 0,5 мкг/мл – 5 мл/хв; діаметр зерен сорбенту – 0,20–0,31 мм. Максимальне значення сорбційної ємності проявляють непрожарені зразки Na-клиноптилоліту стосовно Ce(III) – 34,5 мг/г, що перевищує значення сорбційної ємності природної та H-форми цього цеоліту в 5,4 та в 5,9 рази, відповідно.

Здатність Na-форми закарпатського клиноптилоліту сорбувати як низькі, так і високі концентрації, його висока сорбційна ємність дають підставу пропонувати цей сорбент для концентрування іонів Ce(III) в режимі твердофазової екстрації на стадії підготовки вод до аналізу, а також для вилучення іонів Ce(III) з технологічних розчинів.

Ключові слова: сорбція, Na-клиноптилоліт, твердофазова екстракція, церій(III), пробопідготовка.

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