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Новосібір мемлекеттік техникалық университеті
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**«ФУНКЦИОНАЛДЫҚ МАТЕРИАЛДАРДЫҢ
ХИМИЯЛЫҚ ТЕХНОЛОГИЯСЫ»**
атты 6-шы Халықаралық Ресей-Қазақстандық
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МАТЕРИАЛДАРЫ
15-16 маусым 2020 жыл

МАТЕРИАЛЫ
6-ой Международной Российской-Казахстанской
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**«ХИМИЧЕСКИЕ ТЕХНОЛОГИИ
ФУНКЦИОНАЛЬНЫХ МАТЕРИАЛОВ»**
15-16 июня 2020 года

MATERIALS
of the 6th International Russian-Kazakh
scientific and practical conference
**«CHEMICAL TECHNOLOGY
OF FUNCTIONAL MATERIALS»**
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ортопедических имплантациях), грамотрицательным бактериям *E.coli* и бактериям *K.pneumonia*, а также и к дикому штамму *K.pneumonia*. Антимикробный эффект был оценен и продемонстрирован.

Работа выполнена по проекту МОН РК ГФ ИРН AP05131647 «Физико-химические основы получения многофункциональных биомедицинских материалов (нанопленок) с антибактериальными и противовоспалительными свойствами» 2018-2020 гг.

PHYSICOCHEMICAL AND ADSORPTION PROPERTIES OF NATURAL ZEOLITE AND CHAMOTTE CLAY

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Heavy metals (HM) are one of the most dangerous pollutants of the environment. They can accumulate in the human body causing severe disruption of different organs. Lead is among the most toxic HM for people, especially children. Lead poisoning can cause problems with cardiovascular, enzyme and reproductive systems, dysfunction of kidneys and liver, both the peripheral and the central nervous system are influenced by lead exposure [1].

Adsorption methods of water treatment from lead ions have proven their efficiency [2]. Natural zeolites and clays are effective and accessible materials to use as adsorbents of different pollutants [3, 4, 5, 6].

The current study is dedicated to the study of the adsorption capacity of natural zeolite (from the Shankanay deposit of Almaty region) and Chamotte clay (from Ukrainian deposit and provided by Teplosvet Inzhiniring, LLC, Kiev) for Pb^{2+} -ions removal.

Adsorption experiments were performed in static mode at room temperature and pH=6. Initial and equilibrium concentrations of Pb^{2+} were determined using Atomic Absorption Spectrometer Shimadzu 6200, Japan.

SEM images of the natural zeolite and Chamotte clay are presented in the Figure 1.

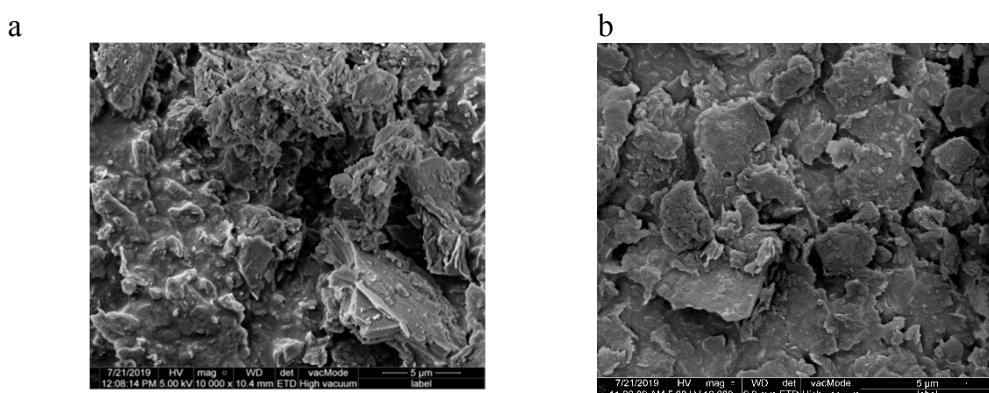


Fig. 1. SEM-image of (a) natural zeolite; (b) chamotte clay

The morphology of natural zeolite is represented by dense agglomerates of various shapes and sizes. As it is known, zeolites have a three-dimensional crystal lattice [7], which accounts for their porosity. The presence of pores can also be seen in the SEM image of the zeolite. Particles of Chamotte clay are represented by layers of flocculent formations of irregular shape and various sizes. Clays have a layered structure, as a result of which they are also porous [5].

The XRD analysis of the samples suggests that the crystalline structure of natural zeolite consists of clinoptilolite, quartz and anorthite; and the structure of clay consists of kaolinite, mullite and quartz.

The results of FTIR-spectra of zeolite and chamotte clay are shown in the Figure 2(a,b). The following bands are present in the spectrum of Chamotte clay (Fig.2(a)): at 3692 and 3621 cm^{-1} assigned to the stretching vibrations of OH-groups adsorbed on the surface of the mineral; at 1639 cm^{-1} due to the inner-layer OH(Al-OH) stretching mode; at 1027 and 1005 cm^{-1} to the Si-O planar stretching; at 910 cm^{-1} assigned to the inner-surface Al-OH deformation vibrations; at 789 and 746 cm^{-1} due to the symmetric stretching vibrations of Si-O-Al bonds [8]; and at 692 cm^{-1} assigned to Mg(Al-OH) vibrations [9].

As it can be seen from the figure 2(b), FTIR-spectrum of the zeolite has the following bands: 760, 984, 1637, 3395, 3575 cm^{-1} . The weak bands in region 750-790 cm^{-1} correspond to the symmetric stretching vibration of the T—O bonds [10]. The most intensive peak at 900-1100 cm^{-1} corresponds to O-T-O (where T=Si, Al) bonds [11]. The bands at 3000–4000 cm^{-1} and ~1600 cm^{-1} are due to vibrations related to the presence of OH- groups and zeolite water in the structure [12].

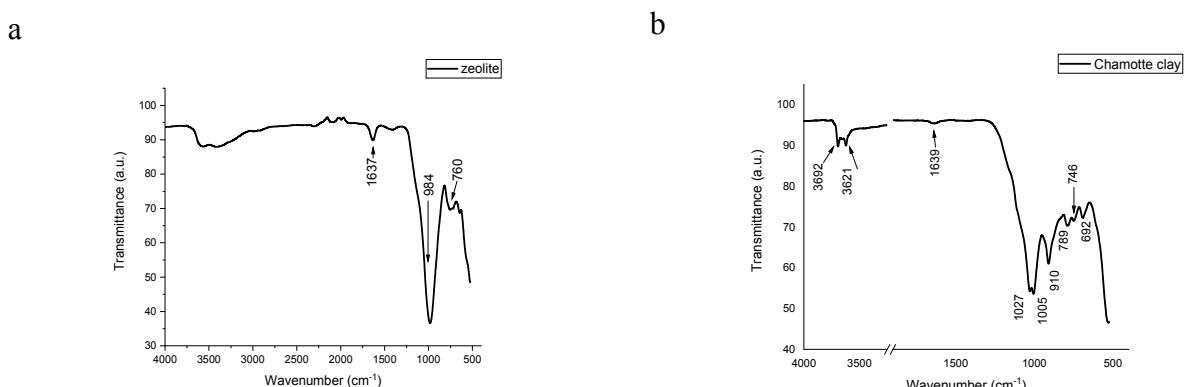


Fig. 2 (a) FTIR spectrum of zeolite; (b) – FTIR spectrum of chamotte clay

The Table 1 shows the results of specific surface area, average pore size and total pore volume obtained with BET N_2 adsorption method. Thereby, specific surface area is 4,1 m^2/g and 8,4 m^2/g for zeolite and clay respectively.

Table 1 – BET N_2 adsorption results of zeolite and Chamotte clay

| No | Sample | Surface area, m^2/g | Average pore size, nm | Total pore volume, cm^3/g |
|----|---------------|-------------------------------------|-----------------------|---|
| 1 | Zeolite | 4.1 | 0.75 | 0.015 |
| 2 | Chamotte clay | 8.4 | 0.69 | 0.029 |

A study of the adsorption properties of natural zeolite and chamotte clay was carried out with respect to Pb^{2+} ions, one of the most toxic heavy metals. The results of studying the process of sorption of lead ions from solutions with concentrations of 5, 10, 20, 50, 100, 200, 500 mg/L by the investigated materials are presented in the Figure 6.

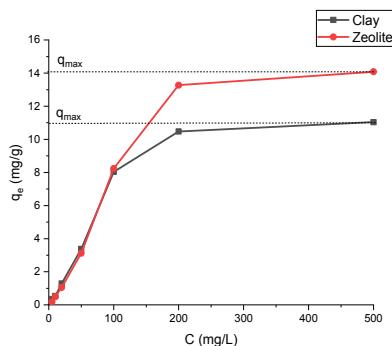


Fig 3. Pb^{2+} ions adsorption isotherm on the natural zeolite and Chamotte clay

According to the IUPAC classification, the adsorption isotherms of lead (II) ions by the studied zeolite and clay are of type I isotherm, which 'approaches a limiting value' and usually is used to describe adsorption on microporous solid adsorbents having relatively small external surfaces (e.g. activated carbons, molecular sieve zeolites and certain porous oxides), the limiting uptake being governed by the accessible micropore volume rather than by the internal surface area [13]. Also, such isotherms are called Langmuir isotherms. The adsorption capacity q_{\max} was 14 mg/g for zeolite and 11 mg/g for clay.

When describing the sorption process, adsorption models of sorption isotherms are of great importance. They show how metal ions are distributed between the adsorbent and the liquid phase when they are equilibrium depending on the concentration (adsorbent adsorbate). In the present work, sorption isotherms were calculated according to the theories of Langmuir and Freundlich.

Table 2 – Parameters obtained when fitting the experimental data to the Langmuir and Freundlich isotherms.

| Theory | Parameters | Zeolite | Clay |
|------------|-------------------------|--|---|
| Langmuir | R^2 | 0,999 | 0,999 |
| | $K_L, \text{L/mg}$ | $(0,4297 \pm 0,0496)$ $(14,2027 \pm 1,056)$ | $(0,8502 \pm 0,0491)$ $(11,056 \pm 1,056)$ |
| | $q_{\max}, \text{mg/g}$ | 0,0372 | 0,0282 |
| Freundlich | R^2 | 0,886 | 0,819 |
| | β | $(0,4463 \pm 0,3377)$ $(0,0715 \pm 0,0710)$ | $(22,4739 \pm 28,7010)$ |
| | K_F | 2,9483) | 2,9483) |

As can be seen from the Table 2, the Langmuir isotherm better describes the process of adsorption of lead ions by the studied materials, since the correlation coefficients were $R^2 = 0.999$ for zeolite and clay. Also, the values of adsorption capacity q_{\max} obtained graphically almost coincide with the values obtained from the graph $q_e = f(C)$ (Fig. 3), which also proves the relevance of this model.

It was reported in the work [14], that according to [15] it is possible to calculate from the Langmuir isotherms the separation factor (K_R) (Eq.1), which is useful to determine whether the separation is or not favorable in batch systems.

$$K_R = \frac{1}{1 + K_L C_0} \quad (1)$$

where K_L is the Langmuir constant (L/mg), C_0 is the initial concentration of Pb^{2+} (mg/L). If $K_R > 1$ the adsorption is not favorable; if $K_R = 1$ the adsorption is linear; if $0 < K_R < 1$ the adsorption is favorable; and if $K_R = 0$ it is irreversible. As it can be seen from fig. 4, separation factors for both

zeolite and clay are within $0 < K_R < 1$, i.e. adsorption is favorable. It can also be assumed that adsorption is more favorable on zeolite because the K_R values for zeolite are greater than for clay. It also corresponds to the fact, that the values of q_{\max} are bigger for zeolite as well. With an increase in the concentration of metal ions, the separation factor $K_R \rightarrow 0$, i.e., tends to be irreversible.

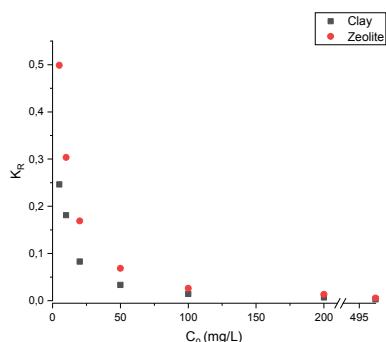


Fig. 4. Separation factors determined from Langmuir isotherms applied to zeolite and clay.

Conclusions.

In the present study, adsorption ability of the natural zeolite and Chamotte clay towards Pb(II) ions has been studied. The maximum uptake of lead ions (q_{\max}) was determined as 14 mg/g for zeolite and 11 mg/g for clay. Langmuir and Freundlich isotherm models were used to describe the adsorption mechanism. It was observed that Langmuir isotherm model best describes the adsorption of Pb^{2+} on natural zeolite and clay, the correlation coefficients R^2 were 0.999 for both adsorbents. It means that a monolayer of adsorbate is formed on a heterogeneous surface. The separation factors (K_R) were calculated from the Langmuir isotherm: $0 < K_R < 1$ for both zeolite and clay, i.e. adsorption is favorable. It may be concluded that both natural zeolite and Chamotte clay can be used as low-cost adsorbents for wastewater treatment from lead (II) ions.

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NEW PILLARED CLAYS PREPARED FROM DIFFERENT DEPOSITS OF KAZAKHSTAN

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1. Introduction

Kazakhstan is one of the largest countries in the world and have hundreds of chemical industry plants that generates daily wastewater containing pollutants requiring treatment. Catalytic agents based on natural clays are proposed for solving problems with water purification. Natural resources are among the main wealth of the Republic of Kazakhstan, including abundant and cheap natural clays in the southern region of the country. This work aims to explore natural clays in the synthesis of low cost pillared clays to be used as catalysts in oxidation technologies for the treatment of wastewaters [1]. Since nitro phenols are commonly found in many types of industrial wastewaters (e.g. plastic, pharmaceutical, paper or pesticides industries), 4-nitrophenol (4-NP) was used as representative model compound in catalyst screening studies [2].

2. Experimental

Zirconium pillared clays were synthesized using natural clays, from Akzhar (Clay A), Karatau (Clay B) and Kokshetau (Clay C) regions (Kazakhstan) zirconium tetrachloride as pillarizing agent. The pillarizing solution was prepared by slow addition of NaOH (0.2 M) to the solution containing Zr, at room temperature, until pH = 2.8. The pillarizing process was then performed keeping a ratio of 10 mmol of Zr per gram of clay. The final material was calcined for 2 h at 823 K. The elemental content of the clays was determined by Electron Microprobe (EMP) analysis. The oxidation experiments were performed in a 250 mL well-stirred glass reactor at 50 °C, initial pH = 3,5 g/L of 4-NP, 2.5 g/L of catalyst and 17.8 g/L of hydrogen peroxide.

3. Results and Discussion

Natural clays of Karatau and Akzhar, Kokshetau prepared for pillarization with Zr cations possess excellent catalytic properties in the 4-NP oxidation reaction in Fig.1. In the catalytic oxidation experiments, the total removal of the pollutant is reached after 4 h when Zr pillared clays are used as catalyst, instead of natural clays [3]. The conversions of TOC (X_{TOC}) after 24 h, with each material,