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SORPTION OF LEAD CATIONS (II) BY ACID-MODIFICATED ZEOLITE IN ALKALINE MEDIUM

Abstract. A systematic analysis of the sorption properties of acid-modified zeolite was carried out in alkaline (pH 8.5) medium with respect to Pb^{2+} cations. The interaction of time, temperature and concentration of lead on the sorption capacity of acid-modified zeolite was established. The sorption curves depending on temperature and concentration of Pb^{2+} ions have extreme character. The appearance of the maximum or minimum on the lead sorption curves probably due to saturation of modified zeolite with lead cations under these conditions, consequence, a decrease in its sorption properties. This process, in turn, is caused by the desire of the system to equilibrium, where the concentration of lead is equalized in both phases. The optimal conditions for sorption of Pb (II) cations in an alkaline medium with an acid-modified zeolite corresponding to the maximum degree of their absorption (99.8-99.9%) were determined.

Keywords: alkaline medium, lead cations (II), heavy metals, acid-modified zeolite, sorption.

Introduction. In recent years sorption methods for the purification of aqueous media and wastewater by using of modified natural zeolites have become widespread [1, 2]. The acid activation of natural zeolites is accompanied by the process of dealumination, as a result, the channels of zeolite framework are unblocked, which leads to an increase in Si/Al ratio, the formation of silanol groups, an increase in the effective size of micropores, and an increase in exchange capacity of zeolite [3-9]. The exchange acid centers appear in the zeolite structure due to acid activation. When acid is activated by appearance of active in the process of sorption of H^+ exchange acid centers and the displacement of aluminum (Al^{3+}) into exchange positions, more favorable arrangement of active sites for the interaction of reacting substances are created. In addition, at acidic treatment of zeolite the silica increases the specific surface area and porosity of the activated samples and removed impurities blocking the channels [10]. The stability of silica-alumina skeleton of high-silica zeolites to the action of acids has increased the possibilities of regulating their properties by decationization and dealumination under various conditions of acid treatment.

It has been found that acid-activated clinoptilolite tuffs are shown high sorption properties, for example with respect to phenylalanine [11, 12] and formaldehyde [13, 14], which is their practical application in the purification of contaminated media. A method for extracting α-tocopherol from vegetable oils based on high selectivity of acid-activated clinoptilolite to α-tocopherol from an ethanol

solution was developed and proposed [15]. Modification of natural zeolites with acids increases their efficiency of extracting NH_4^+ ions from various media [16, 17]. It was shown that acid-activated natural clinoptilolite (Sokirnitsky deposit, Ukraine) shows a growth in sorption capacity with respect to SO_2 molecules [18]. Obviously, this is due to the reduction in the main (donor) surface centers (potential centers of adsorption of acceptor molecules SO_2), as a result of action of protonic acid. Detailed studies by the authors of [19] and carried out in [18] showed that the samples of Bulgarian clinoptilolite, treated by boiling for 4 hours in 2N, 4N, 6N and 8N hydrochloric acid behave differently in the adsorption processes of SO_2. For H-Cl (4N HCl) samples protective action time and amount of adsorbed SO_2 were maximum. However for the sample of H-Cl (8N HCl), these values were lower than natural clinoptilolite. The authors associate the obtained results with an increase in the degree of dealumination and partial destruction of the zeolite crystal lattice [19]. Acid treatment of natural zeolite allows to obtain more efficient sorbents in relation to phosphates, fluorine, cations of iron (III), aluminum (III), and heavy metals [20-22].

A negative influence of temperature on the process of acid activation of clinoptilolite was revealed in [23]. Activation of clinoptilolite of Bigadis deposit (Turkey) with 5 M hydrochloric acid at 25, 40 and 100 °C reduces the content of cations in aluminosilicate, the amount decrease by increase of the temperature. However, there is no complete removal of exchange cations and aluminum.

A number of alkaline production effluents contain in dissolved form inorganic impurities such as Fe, Mg, Pb, As, Cu, Mn, Ni, etc. Lead ions among previous cations-impurities are toxic, which are capable to concentration and accumulation in soil, wastewater, groundwater, and in the human body [24-26]. For the purification of contaminated alkaline media application of acid-modified zeolites is perspective due to an increased exchange capacity.

From the review it is clear that acid modification of natural zeolites of various deposits increases their sorption properties. However, the sorption capacity of obtained sorbents is determined by the nature of used zeolite, temperature and other factors of the process.

The aim of the work was to study the influence of the sorbent norm, time, temperature and concentration of Pb^{2+} cations on the process of their sorption from an alkaline medium by acid-modified zeolite.

EXPERIMENTAL PART

In this research was used zeolite of the Shankanay deposit modified by 15% hydrochloric acid. The sorption capacity of acid-modified zeolite with respect to lead was studied under stirring conditions in thermostat reactor which was evaluated by change in content of Pb^{2+} cations in solution, i.e. with the difference in the initial and residual concentration of lead (after the completion of the process). The degree of sorption (extraction) is the ratio of difference between the initial and reached concentration of Pb (II) cation at fixed time to its original content.

Initial and final concentrations of lead ions in solutions were analysed by using an atomic absorption spectrophotometer (AA-7000, Shimadzu Corporation, Japan), № A30664901456.

Studying of sorption process of lead (II) cations was carried out in lead-containing aqueous solution with pH of 8.5, which created with 1 N sodium hydroxide solution. The predetermined concentration of Pb^{2+} cations was obtained by introducing in the alkaline solution estimated quantity of acetic acid-lead Pb $(CH_3COO)_2 \cdot 3H_2O$.

The effect of the norm of acid-modified zeolite on its sorption capacity was investigated by an alkaline solution at room temperature where concentration of Pb equal to 49.1 mg/l. The sorbent consumption was varied from 2.5 to 30 g per 100 g of purified alkaline solution. The sorption process was carried out at room temperature for 30 minutes.

The degree of purification of various media significantly depends on time, temperature, and concentration of Pb^{2+} cations. Quantitative determination of such a complex dependence carried out by method of mathematical planning of the orthogonal rototable 3 factorial experiment of the 2nd order [27]. Variable (input) factors of the process were: time (Z_1, min), temperature (Z_2, ^oC), and C_{Pb} (Z_3, mg/l). The selected levels of factors and range of their variation are given in table 1.

Table 1 – Coordinate center of the plan, levels of variation

Parameters	Z_1, min	Z_2, ^oC	Z_3, mg/L
Upper level (+1)	48,9	59,9	81,8
Center of the plan - zero level (Z_i^0)	32,5	45	55
Lower level (-1)	16,1	30,1	28,2
Interval of variation along the axis (ΔZ_i)	16,4	14,9	26,8
Star point (+1,682)	60	70	100
Star point (-1,682)	5	20	10

The changes in the concentration of Pb (II) cations indicated in the table correspond to possible range of their presence in contaminated solutions and wastewater.

Defined (output) parameter Y_1 (response) was residual content of Pb^{2+} ions in studied system "Pb^{2+} - alkaline medium - acid-modified zeolite" after sorption of lead by sorbent.

RESULTS AND DISCUSSION

Studying of effect of the norm of acid-modified zeolite on its sorption capacity with respect to Pb (II) cations in an alkaline medium (pH 9) revealed, increasing in the sorbent consumption from 2.5 g to 5 g per 100 g of purified solution the sorption rate rised from 98.28 % to 99.95% (by 1.67%). Further increase in sorbent consumption practically has no effect on the degree of alkaline purification from lead. The optimal ratio "modified zeolite (T): alkaline solution (G)" is 5:100.

Studying of the sorption of Pb^{2+} cations depending on its concentration, time and temperature carried out at constant ratio of S:L equal to 5:100.

Based on the coordinates of the center of plan, variation levels and planning matrix (table 1, 2), experiments carried out with the appropriate conditions for studying of Pb^{2+} cations sorption by an acid-modified zeolite in an alkaline medium. The results are shown in table 2.

Table 2 – The residual content of Pb^{2+} cations and degree of their sorption by acid-modified zeolite in alkaline medium

#	The natural value of experiments			The content of Pb^{2+}	
	X_1, min	X_2, °C	X_3, mg/l	The residual content of Pb^{2+}, mg/L	Ks, %
1				0,03	99,88
2	48,9	30,1	28,2	0,09	99,65
3	16,1	59,9	28,2	0,03	99,88
4	48,9	59,9	28,2	0,03	99,88
5	16,1	30,1	83,7	0,03	99,96
6	48,9	30,1	83,7	0,03	99,96
7	16,1	59,1	83,7	0,79	99,06
8	48,9	59,1	83,7	0,93	98,95
9	5	45	55	1,03	98,19
10	60	45	55	0,93	98,37
11	32,5	20	55	1,06	98,14
12	32,5	70	55	6,78	87,95
13	32,5	45	10	2,25	79,73
14	32,5	45	100	14,8	84,10
15	32,5	45	55	0,06	99,89
16	32,5	45	55	1,21	97,87
17	32,5	45	55	1,02	98,21
18	32,5	45	55	1,18	97,93
19	32,5	45	55	0,86	98,49
20	32,5	45	55	1,65	97,11

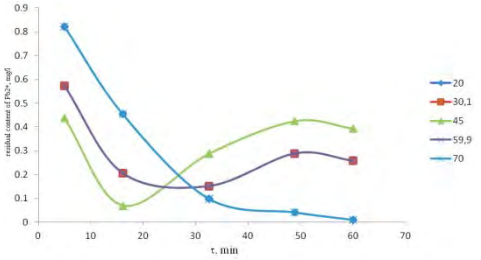
After the processing of the results and the elimination of insignificant coefficients, obtained by regression equation allows calculating the residual content of Pb (II) cations in the alkaline medium after the end of the sorption process by acid-modified zeolite also describes dependence of the residual content of Pb (II) cations on investigated process parameters:

$$Y_{Pb} = 0{,}339+0{,}089X_2+0{,}106X_3+0{,}136X_1^2+0{,}109X_2^2+0{,}158X_3^2+0{,}200X_2X_3$$

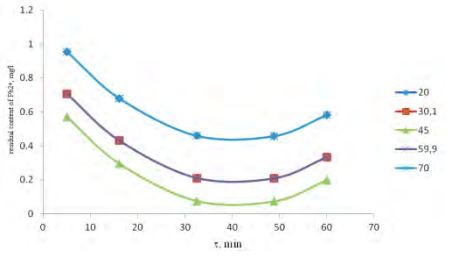
The regression equation checked with Fisher criterion by comparing the variances. It is found that $F<F_{1-p}(f_1,f_2)=Y_{Pb^{2+}}$ 1,204<4,699), so that the equation adequately describes the experiment.

Analysis of regression equation showed that cleaning the solution with an acid-modified zeolite, the residual content in the liquid phase of system "Pb^{2+} - alkaline medium - acid-modified zeolite" is a function of all investigated variables. However their effect is different. Thus, in the regression equation, the time effect is represented only by positive quadratic value (X_1^2). The time factor affects independently on the output parameter and increasing coefficient by value higher than the coefficient of temperature factors (X_2) and concentration of lead cations (X_3).

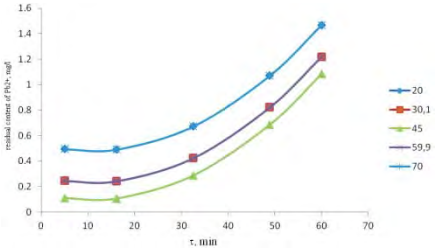
The considered interference of factors and their effect on the response is well demonstrated by following graphs. It can be seen from Figure 1a, b by increasing purification process duration of an alkaline solution with a low concentration of Pb^{2+} (10-28 mg/L) at all temperatures reduces its residual content in liquid phase of the system. The most intensive sorption process takes up to 30 minutes. Increasing of process time from 30 to 50 min has no effect on the change of lead concentration in solution with C_{Pb} = 10 mg/L (figure 1, a), but leads to an increase in its residual content in the solution of S_{Pb} = 28 mg/L (figure 1, b). Thus, at 20°C after 5 min of process there is 0.92 mg/L of Pb^{2+}, after 16 min - 0.72 mg/L of Pb^{2+}, after 32 min - 0.46 mg/L of Pb^{2+}, after 60 minutes - 0.60 mg/L Pb^{2+}. Accordingly, the degrees of sorption are 96.7; 97.44; 98.36 and 97.87%, i.e. with the increase in the duration of the process, there is a tendency to reduce the sorption capacity of sorbent.



a



b



c

Figure 1 –
Influence of time on residual content
of Pb (II) cations
in liquid phase of system "Pb^{2+} - alkaline
medium - acid-modified zeolite":
a – 10 mg/L; b – 28 mg/L; c – 82 mg/L

Increasing of process time has a detrimental effect on the purification of more concentrated lead-containing alkaline solutions (81.8-100 mg/L Pb), which contributes to the growth of the residual content of Pb^{2+} ions in the liquid phase of system (figure 1, c). For example, the process starting from 16-20 minutes and up to 60 minutes in an alkaline solution containing 100 mg/L of Pb cations at 20°C, the degree of lead sorption by an acid-modified zeolite decreases by 1.4%. In this case, increasing the process time has a negative effect on the sorption of lead.

According to the regression equation describing the dependence of the residual content of Pb (II) cations on investigated process parameters, it follows that temperature effect on the degree of sorption of Pb^{2+} ions is complex and is represented by positive values of the linear factor X_2, quadratic X_2^2, double interaction of temperature and concentration of Pb cations (X_2X_3). Since in equation before the coefficient of variable X_2 there is a "plus" sign, the temperature factor affects the output parameter not only as double interaction with the third factor (C_{Pb}), but also independently and directly proportional with. Comparison of coefficients of considered factor X_2 shows its practically equivalent effect along with the first X_1 (time) and the third X_3 (C_{Pb}) factors. A somewhat larger effect is exerted by the factor of double interaction X_2X_3 (temperature and C_{Pb}). Apparently, lead adsorption is exerted by influence of temperature and concentration of Pb^{2+} cations.

Analysis of the obtained results revealed that the curves dependence of residual content of Pb^{2+} cations on temperature have extreme character (figure 2). At the same time, the influence of temperature, lead concentration and time on purification of alkaline solutions with low (10 mg/L) and high (100 mg/L) concentrations are noted (figure 2, a, c). Wherein the sorption curves at 45°C in the range of 5-16 min in solutions with C_{Pb} = 10 mg/L have a minimum, and in the range of 32-60 min have maximum, while in solutions with C_{Pb} = 100 mg/L on sorption curves in the indicated time limits are characterized by the presence of maximum and minimum. In the first case growth of temperature up to 45°C has a positive effect, as the residual content of Pb^{2+} cations decreased (the degree of sorption increases), and in the second case - negative one, since the concentration of Pb^{2+} ions in the system raised (the sorption degree decreases).

In systems the sorption curves throughout the studied time interval are characterized by a minimum at 45°C when C_{Pb} equal to 28-82 mg/L (figure 2, b). Raising of temperature to 45°C has positive effect. Above noted temperature the degree of sorption of lead decreased. For example, 97.63% Pb is sorbed in 16 minutes at 20°C, at 45°C - 98.98% Pb and at 70°C - 97.55% Pb.

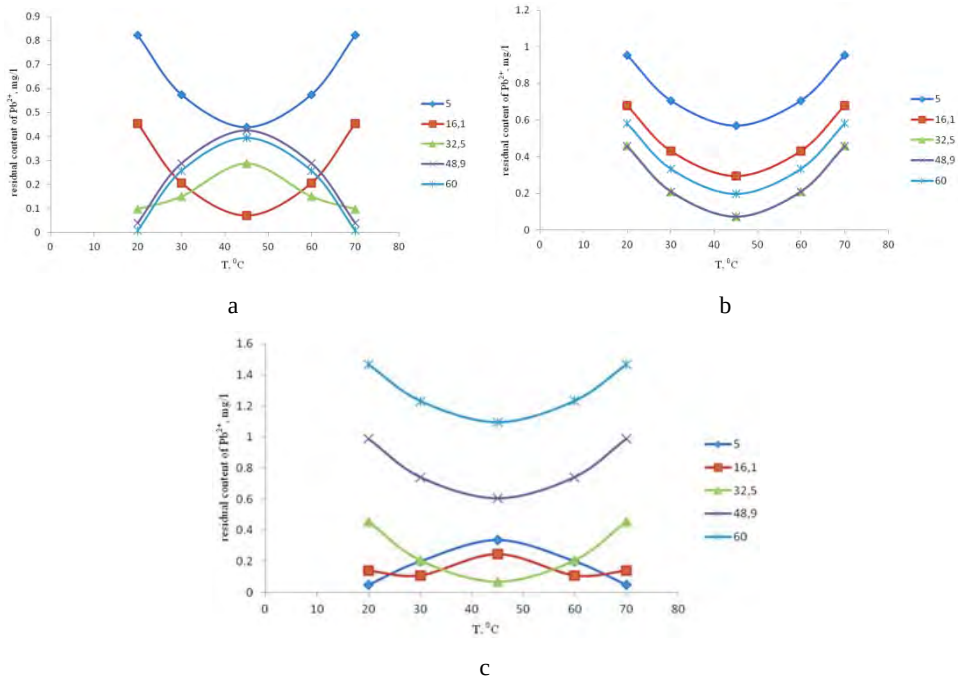
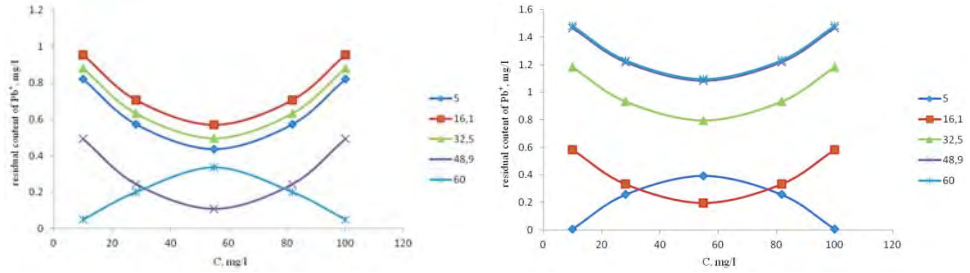


Figure 2 – Effect of temperature on residual content of Pb (II) cations in the system "Pb^{2+} - alkaline medium - acid-modified zeolite": a – 10 mg/L Pb^{2+}; b – 28 mg/L Pb^{2+} ; c – 100 mg/L

Appearance of the maximum or minimum on the sorption curves is probably due to the saturation of acid-modified zeolite with lead cations under these conditions, consequence, deterioration of its sorption properties. This process caused aspiration of system to equilibrium where the concentration of lead in both phases is equalized.

According to the regression equation, the effect of concentration of Pb^{2+} cations on their sorption by an acid-modified zeolite is represented by positive linear (X_3) and quadratic (X_3^2) factors. Compare with contact duration of the sorbent with purification of alkaline solution this suggests a more complex effect of concentration of lead in solution on the process of their sorption. If we compare the values of lead concentration factors X_2 and time X_1^2, then for X_1^2 (time) its value is several higher, but this is somewhat balanced by the close values at coefficients (X_3) and (X_3^2) corresponding to the concentration of lead ions.

The sorption curves of lead depending on their concentration (figure 3) have similar character to the sorption curve by temperature (figure 2). So the curves have extreme character with min or max at 55 mg/L Pb. Moreover, the sorption curves at low temperatures (20°C) obtained in the interval of 5-50 min, which characterized by the presence of min and at 60 min - max (figure 3, a), at higher temperature (70°C) is prescribed min on the curves in the interval of 15 -60 min, and max - at 5 min of the process (figure 3b).



a b

Figure 3 – The effect of Pb (II) cations concentration on its residual content in the system of "Pb^{2+} - alkaline medium - acid-modified zeolite": a – 20°C; b – 70°C

So, at 20°C up to 50 min with increasing of C_{Pb} to 55 mg/L, lead sorption degree (K_c) increased, and with a further increase K_c C_{Pb} decreased. However, simultaneous increase in C_{Pb} and time (60 min) decreased the sorption degree reaching to minimum at 55 mg/l Pb, then increased with raising of C_{Pb}.

At higher temperature (70°C), the maximum degree of Pb sorption is also reached in solutions with their concentration of 55 mg/L. However, for 15-60 min and the minimum - for 5 min of the process.

The presence of maximum or minimum on the sorption curves is due to the saturation of the zeolite, consequence, due to the deterioration of its sorption properties. This process, in turn, may be caused by the desire of the system to equilibrium, where the concentration of copper in both phases is equalized. So that the appearance of maximum on the sorption curves is due to the desorption process, when under these conditions took place exit of the sorbed cations from zeolite back to solution [28].

When determining the optimum conditions for sorption of Pb^{2+} cations by an acid-modified zeolite from an alkaline medium, it is necessary to consider temperature, lead concentration and the process time. Thus, the greatest degree of sorption of Pb cations is achieved at 20 and 70°C in an alkaline solution with C_{Pb} equal to 10 and 100 mg/L, respectively, in 60 and 5 minutes (99.8-99.9%) and with C_{Pb} equal to 55 mg/for 16 and 48 min, respectively, at 20 and 70°C (99.6-99.8%).

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Резюме

Р. М. Чернякова, Р. А. Қайыңбаева, А. А. Ағатаева,
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СІЛТІЛІК ОРТАДА ҚЫШҚЫЛМЕН МОДИФИКАЦИЯЛАНҒАН ЦЕОЛИТ АРҚЫЛЫ ҚОРҒАСЫН (II) КАТИОНДАРЫН СОРБЦИЯЛАУ

Қышқылмен модификацяланған цеолиттің сілтілік ортада (pH 8,5) Pb^{2+} катиондарына қатысты сорбциялық қасиеттерін жүйелі түрде талдау жүргізілді. Уақыт, температура және қорғасын концентрациясының қышқылмен модификацияланған цеолиттің сорбциялық қабілетіне өзара әсер ететіні анықталды. Температура мен Pb^{2+} иондары концентрациясына тәуелді сорбциялық қисықтар айрықша сипатқа ие. Қорғасынның сорбциялық қисықтарында максимум немесе минимумның пайда болуы осы жағдайларда модификацияланған цеолиттің қорғасын катиондарымен қанығуына, соның салдарынан оның сорбциялық қасиеттерінің төмендеуіне байланысты болуы мүмкін. Бұл процесс өз кезегінде жүйенің тепе-теңдікке ұмтылуы салдарынан туындайды, нәтижесінде қорғасын концентрациясы екі фазада да теңеседі. Сілтілік ортада Pb (II) катиондарының қышқылмен модификацияланған цеолит арқылы сорбциялану процесінің оңтайлы жағдайлары анықталды. Pb (II) катиондары ең жоғарғы сорбциялану дәрежесіне Pb концентрациясы 10 және 100 мг/л (99,8-99,9 %) тең ерітіндіде 5 және 60 минутта, сондай-ақ Pb концентрациясы 55 мг/л (99,6-99,8 %) тең ерітіндіде 16 және 48 минутта 20 және 70°С температурада жетеді.

Түйін сөздер: сілтілік орта, қорғасын (II) катиондары, ауыр металдар, қышқылмен модификацияланған цеолит, сорбция.

Резюме

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СОРБЦИЯ КАТИОНОВ СВИНЦА (II)
КИСЛОТОМОДИФИЦИРОВАННЫМ ЦЕОЛИТОМ
В ЩЕЛОЧНОЙ СРЕДЕ

Проведен системный анализ сорбционных свойств кислото- модифицированного цеолита в щелочной (pH 8,5) среде по отношению к катионам Pb^{2+}. Установлено взаимовлияние времени, температуры и концентрации свинца на сорбционную способность кислото-модифицированного цеолита. Сорбционные кривые в зависимости от температуры и концентрации ионов Pb^{2+} носят экстремальный характер. Появление на кривых сорбции свинца максимума или минимума, вероятно, обусловлено насыщением в этих условиях модифицированного цеолита катионами свинца и, как следствие, снижением его сорбционных свойств. Данный процесс, в свою очередь, вызван стремлением системы к равновесию, при котором происходит выравнивание концентрации свинца в обеих фазах. Определены оптимальные условия процесса сорбции катионов Pb (II) кислотомодифицированным цеолитом в щелочной среде. Наибольшая степень сорбции катионов Pb (II) достигается в растворе с C_{Pb}, равной 10 и 100 мг/л (99,8-99,9 %) за 5 и 60 мин и с C_{Pb}, равной 55 мг/л за 16 и 48 мин (99,6-99,8 %) при температурах 20 и 70^{o}C.

Ключевые слова: щелочная среда, катионы свинца (II), тяжелые металлы, кислотомодифицированный цеолит, сорбция.