Features of extraction of neodymium ions by interpolymer systems based on salt forms of industrial ionites

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ABSTRACT

This research aimed to study the effect of remote interaction and mutual activation between salt forms of AB-17-8(Cl$^-$) and KU-2-8(Na$^+$) ion exchange resins on the sorption of neodymium ions. The concentration of neodymium ions was determined using a spectrophotometer based on the interaction with the colored arsenazo (III) reagent. The degree of extraction of neodymium ions during sorption and the degree of extraction of this metal during desorption from the polymer matrix was calculated. The influence of the choice of sorption mode on the efficiency of neodymium extraction is studied: dynamic (with mixing of the solution from which the metal was extracted) and static (without mixing). Based on the obtained dynamics of processes, it is established that when mixing the solution (the range of mixing speed is 40-80 rpm) the equilibrium between sorption and desorption of the metal is established after 6 hours of interaction. Also, in the dynamic mode, the target metal was sorbed much better than in the static mode, affecting the amount of metal ions obtained during desorption. It was found that the maximum degree of neodymium ion sorption in the interpolymer system is observed at a 5:1 hydrogel ratio and 48 hours of remote interaction without mixing. It is 42.8 mg/l of the residual concentration. In the dynamic mode, the maximum sorption is observed for the initial cationite, with a residual concentration of 8.28 mg/l. When calculated per 1 mol of cationite, the effect of mutual activation of hydrogels is visible, which is proved by the tendency to increase the degree of neodymium sorption from the ratio of 6:0 (the initial cationite) to 1:5. A significant increase in the sorption of neodymium ions is observed in various molar ratios of ionites. These results indicate the appearance of ionized structures that form conformations that provide optimal conditions for the sorption of neodymium ions from an aqueous solution of its salt, which can serve practical purposes for its selective extraction from industrial mixtures. The necessity of metal extraction by hydrogels in dynamic mode is established for faster operation of industrial plants and more complete metal binding by polymer matrices.

Keywords: interpolymer system, neodymium ions, mutual activation, AB-17-8(Cl-) and KU-2-8(Na$^+$) ion exchangers, remote interaction.

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Introduction

Rare earth metals (REM) are a strategic raw material due to their unique properties. Kazakhstan has significant reserves of rare-earth minerals [1]. Neodymium is a white plastic metal and its ion Nd^{3+} has a purple-pink color. Neodymium compounds are chemically similar to compounds of lanthanum and other rare earth elements [2]. Due to its close ionic radius, it is difficult to selectively isolate neodymium from a mixture with other rare-earth metals, but modern production cannot do without this metal. Small and powerful magnets are used in computer speakers and hard drives, magnets for wind turbines and hybrid cars, and electric motors in conventional electric vehicles, in addition, permanent magnets based on neodymium iron and boron are also used in sound system speakers. The production of colored glass and fluorescent lighting, laser rangefinders, and guidance systems in the defence industry are also relevant areas, where this metal is used [3].

Previous studies on intergel systems have shown that the mutual activation of hydrogels significantly affects the change in electrochemical, volume-gravimetric, and sorption properties concerning REM [[4], [5], [6], [7]]. This work aims to find out the conditions for maximum sorption of

neodymium ions from solution for its possible subsequent selective extraction from industrial mixtures.

Experimental part

Equipment. The mass of the sorbents was determined by weighing on a Shimadzu TX423L electronic analytical balance. The optical density of neodymium nitrate solutions was determined using a CPC-3 photocolorimeter.

Materials. The studies were performed in solutions of 6-water neodymium nitrate (Nd^{3+} concentration = 100 mg/l). Industrial ion exchangers in the salt form were used: strong-base AB-17-8(Cl^-) and strong-acid KU-2-8(Na^+). To conduct the study, these hydrogels were used to make interpolymer systems with different molar ratios of cationite and anionite.

Experiment. The experiments were performed at room temperature. Ion exchange resins KU2-8 (Na^+) and AB-17-8 (Cl^-) were used in the dried state to study the sorption of neodymium ions. The presence of moisture in KU-2-8 (Na^+) was 17%, and AB-17-8 (Cl^-) was 32.34%. Studies of the interpolymer system were performed in the following order: each ion-exchange resin in dry form was placed in separate polypropylene meshes, in different molar ratios. Then polypropylene meshes with ion-exchange resins KU2-8 and AB-17-8 were placed in glasses with neodymium nitrate solutions (200 ml each). The first experiment was conducted in dynamic mode with active mixing of the working solution (mixing speed range: 40-80 rpm) with polypropylene meshes containing hydrogels. To study the dynamics of metal sorption, aliquots were selected after 1, 6, 24, and 48 hours to determine the residual concentration of the metal. Desorption was performed using nitric acid (2%) for 72 hours. The same procedure was performed in static mode (without active mixing).

Method of determination of neodymium ions. The method for determining neodymium ions in solution is based on the formation of a colored complex compound of the organic analytical reagent arsenazo III with rare earth metal (REM) ions [8].

Results and discussion

Today, a lot of research is aimed at finding new ways to efficiently and selectively recover rare earth metals from primary and secondary sources. In the review work [9], the authors note that REMs are the most important strategic resources and the role of recycling for the production of these metals has increased. Hydrometallurgical methods and extraction are used all over the world to concentrate rare earth metals, but ion exchange sorption processes are becoming increasingly popular due to their simplicity and environmental safety. The authors also provided an overview of existing ion exchangers that have already been successfully used to separate target metals from impurities, emphasizing the cost-effectiveness of sorption in modern production.

Study [10] presents a promising process for extracting rare earth metals from silicate ores. Autors provided direct leaching and acid baking using sulfuric acid. The influence of sulfuric acid concentration, leaching temperature and the influence of solid to liquid ratios were studied. As a result, leaching with sulfuric acid in the presence of hydrogen peroxide made it possible to isolate 80-90% of rare earth metals from silicate ore. This study is important since few works are devoted to the extraction of rare earth metals from this type of raw material, while further development is necessary related to the heat treatment of silicate raw materials before leaching with acid.

In another article [11] a method was developed for the recovery of rare earths from the green lamp phosphor by dissolution in concentrated methanesulphonic acid. Using this reagent, it was possible to achieve high leaching efficiency: (74% Tb, 78% Ce and 95% La) in a relatively short time (1 h). The process itself was carried out under milder conditions (low temperatures) in comparison with existing methods. Further extraction procedures are required for further separation of the obtained rare earths.

Methods for using ionic liquids for isolating rare earth elements and selectively separating them from each other have been actively developed recently. For example, article [12] describes the separation of lanthanum, samarium, and neodymium ions from cobalt and nickel ions using an ionic liquid trihexyl(tetradecyl)phosphonium nitrate. According to the authors' conclusions, using an ionic liquid it is possible to separate rare earth metals from cobalt and nickel up to 99%. In addition, the ionic liquid itself can be regenerated using distilled water. The authors also provided a theoretical basis for the extraction mechanism. This technology will have a major impact on the

recycling of samarium-cobalt magnets and nickel metal hydride (NiMH) batteries.

The effectiveness of various factors on the extraction of both individual ions and mixtures of rare earth metals was studied in [13]. Researchers used ionic trihexyltetradecylphosphonium 3-hydroxy-2-naphthoate, and water solutions of La, Ce, Nd, Ho and Lu. By changing the extraction conditions, it was found that the best extraction was observed at pH 2.5. An increase in temperature from 20 to 30 degrees also had a positive effect on the efficiency of extraction, while the process of establishing equilibrium occurred quickly (6 hours). According to the authors, during desorption (0.5 M nitric acid), satisfactory results were observed for lanthanum and lutetium. Further improvements to such methods are needed, especially to increase desorption rates and selectivity.

Sorption is a promising process in the isolation of rare earths due to its simplicity, economic feasibility and environmental tolerance. For example, article [14] describes the sorption of lanthanum ions using oxidized and non-oxidized multi-walled carbon nanotubes. According to the data obtained, both sorbing materials are suitable for industrial use for the production of lanthanum. Various factors, such as solution pH, temperature and stirring speed, influenced the time to establish equilibrium and the amount of target metal recovery. Desorption can be carried out with sulfuric acid.

The most affordable and simple sorbents are industrial ion exchangers. Thanks to the huge variety of ion exchangers on the market, it is possible to select such systems and conditions (pH, temperature, ionic strength, movement speed, etc.) that will be most effective for maximum sorption of target metals, and in some cases, their selective separation from each other. A lot of work has been done in this area [[15], [16], [17], [18]].

In research [19], cationites with various functional groups were compared, as well as bifunctional cationites in REM sorption. Acidic mine waters acted as a secondary source of rare earth metals. At the same time, it was necessary to separate the target metals from the transition metals, in particular from the excess of iron. As a result, the authors found that cationites with a sulfogroup extract REM more selectively, especially at low pH values. At the same time, bifunctional resins (sulfonic and phosphonic functional groups) showed better results than monofunctional aminophosphonic resins.

Sparsely cross-linked polyelectrolytes were used in this study. Polymer networks of hydrogels have a three-dimensional structure, the structure of which is partly determined by the degree of crosslinking with special compounds. As a result of crosslinking between linear chains, covalent bonds are formed and the material becomes elastic, which is quite important in the technical practice of metal extraction, it becomes insoluble in water. KU-2-8 and AB-17-8 are representatives of strongly acidic and strongly basic polyelectrolytes (PE). These PE elements are dissociated in an aqueous medium, as a result of which charges of the same name are formed on the polymer matrix and repel each other. In this regard, the polymer changes its conformation and swells due to the unfolding of the chain [20]. The change in the conformation and swelling of PE occurs up to certain limits and depends on many factors (the degree and constant of dissociation of PE, ionic strength of the solution, temperature, pH of the medium). It is also possible to assume that the restriction of further changes in the polymer matrix is a cloud of counterions that have a screening effect. Therefore, when these stabilizing ions are removed, the conformation of the material can be further changed. Such a procedure is possible based on the" remote interaction effect", which consists of the effect of two hydrogels of different nature placed in a common water environment on each other, and separated to exclude their direct interaction.

Based on this phenomenon, it is possible to influence the conformational properties of two hydrogels by changing their molar ratios in the aqueous medium where the target ions are located that need to be sorbed. The objectives of this paper are: to find out how conformational changes based on the "long-range effect" affect the sorption efficiency of neodymium ions; to find out the features of sorption of this ion in static and dynamic modes. The results obtained in studies of the pH, electrical conductivity, and degree of swelling of samples in in the works mentioned earlier, can be used as evidence of changes in the properties of polyelectrolytes during mutual activation. The results obtained can be explained by the formation of uncompensated charges at the interstitial links of individual hydrogels as a result of their mutual activation. Such changes increase the potential of PE in the sorption of REM ions. Thus, it

was confirmed in [21] that the remote interaction of polymers in the intergel system KU 2-8 cationite and AB-17 anionite provides mutual activation of these macromolecules with subsequent transition to a strongly ionized state. The maximum sorption of yttrium ions was observed at molar ratios of KU2-8:AB-17-8=3:3 in comparison with individual sorbents. In another study [22], electrochemical properties of polyacrylic acid and polyethyleneimine were studied by conductometry and pH metry to predict the possibility of mutual activation of polyacrylic acid and polyethyleneimine. When pH is measured after 24 hours, the acidity index has the lowest values, indicating a high content of H^+ ions in the aqueous medium. Accordingly, the specific electrical conductivity reached its maximum value at the ratio of 3:3 (gPAK: gPEI), which coincides with the result of the pH measurement. The obtained data indicate that significant changes in the electrochemical and conformational properties of the initial macromolecules occur in this interpolymer system.

Tables 1 and 2 show the results of sorption of neodymium ions from its solution with a concentration of 100 mg/l. The residual concentration was determined every 1, 6, 24, and 48 hours after the start of sorption. According to numerical data, there is a clear difference in the rate of metal sorption depending on the choice of the mode (static or dynamic). The best indicator of neodymium sorption in both modes is observed at the ratio of cationite and anionite 5:1 (Fig. 1, 2). This may mean that it is precisely at this molar ratio that the PE conformation acquires the optimal structure for neodymium sorption.

Table 1 - Dependence of neodymium sorption on time in static mode (residual concentration) $C_{init.}$ = 100 mg/l.

Ratios of KU2-8(Na$^+$): AB-17-8(Cl$^-$)	1 h.	6 h.	24 h.	48 h.
6:0	80.96	83.4	69.57	56.96
5:1	79.75	77.75	58.95	42.8
4:2	81.96	82.18	73.66	62.71
3:3	81.29	84.17	75.87	67.9
2:4	82.18	81.74	76.989	68.79
1:5	82.73	83.95	78.09	73.55
0:6	82.29	87.71	84.16	83.62

Table 2 - Dependence of neodymium sorption overtime in dynamic mode (residual concentration) $C_{init.}$ = 100 mg/l.

The ratios of KU2-8(Na$^+$): AB-17-8(Cl$^-$)	1 h	6 h	24 h	48 h
6:0	45.896	8.727	8.727	8.28
5:1	42.79	12.157	12.046	11.825
4:2	65.9	30.96	29.74	27.86
3:3	52.42	38.15	38.6	38.04
2:4	74.325	47.334	50.98	47.99
1:5	82.06	71.227	69.789	75.76
0:6	84.72	84.39	86.05	84.059

In the dynamic mode of neodymium sorption, the equilibrium is established after 6 hours, and the metal concentration in the solution practically does not change. In static mode, the concentration gradually decreases during the entire 48 hours of the process, while the completeness of extraction does not reach the same values as with mixing.
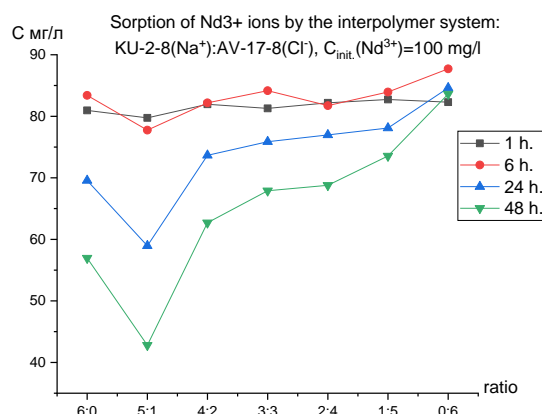

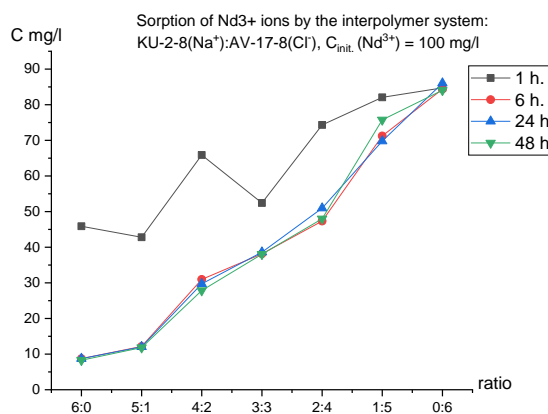**Figure 1**-Residual concentration after static neodymium ion sorption

Figure 2 -Residual concentration after dynamic neodymium ion sorption

Figures 3 and 4 show the results of desorption of the target ion. In this case, the maximum tendency in the polymer ratio of 5:1 is also clearly traced, which confirms the increased sorption from the solution in this molar ratio. In this case, the amount of desorption also depends on the mode of conducting. When mixing, the desorption rate is higher. One possible explanation is the following assumption: KU-2-8, which is mainly desorbed, is a strongly acidic cationite. When desorbed with nitric acid, the equilibrium should shift towards the formation of a weaker acid, but KU-2-8 is also a relatively strong electrolyte, so the equilibrium will occur more slowly. Consequently, in the static mode, the small amount of metal ion extraction is explained by the kinetic factor, i.e. slower protodesorption from the polymer matrix. After 72 hours. The maximum amount of desorption during stirring is 42.46 mg/l, and without stirring-10.28 mg/l. The second more obvious factor is the greater sorption of the target ion during mixing, which means that more metal can be obtained during desorption.
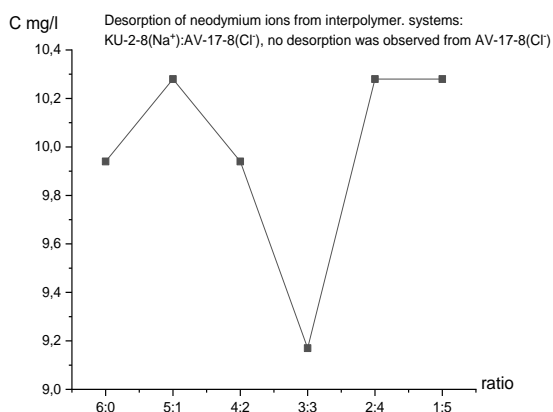

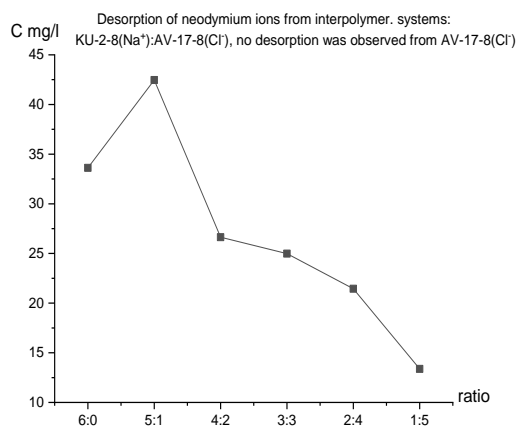**Figure 3** - Static desorption of neodymium ions from KU-2-8 (Na⁺)

Figure 4 - Dynamic desorption of neodymium ions from KU-2-8 (Na⁺)

The degree of extraction (sorption) of neodymium ions was calculated by the formula:

$$n = \frac{C_{init.} - C_{resid.}}{C_{init.}} * 100\%,$$

where C_{init} is the initial concentration of neodymium ions in solution, mg/l; $C_{resid.}$ is the residual concentration of neodymium ions in solution, mg/l.

Figure 5 shows graphs of the degree of sorption in various modes. Under static conditions, the maximum degree of desorption is observed in the ratio of hydrogels of 5:1, while in dynamics the ratio of 6:0 (only KU-2-8 (Na⁺) cationite prevails. This is based on the assumption that only cationite is sorbed, therefore, a larger amount of it leads to greater extraction. But comparing the ratios of 6:0 and 5:1, it can be seen that their numerical values are not very different. Despite the lower amount of cationite, high neodymium sorption is also observed due to the mutual activation of hydrogels in the 5:1 ratio. Mutual activation and thus the effect on the swelling and conformation of PE on each other is also proved by the view of the graph itself. In the absence of the long-range effect, the degree of extraction with a gradual decrease in cationite would decrease monotonically and represent a straight line (not ideal, since the effect of "polyelectrolyte swelling" will affect).
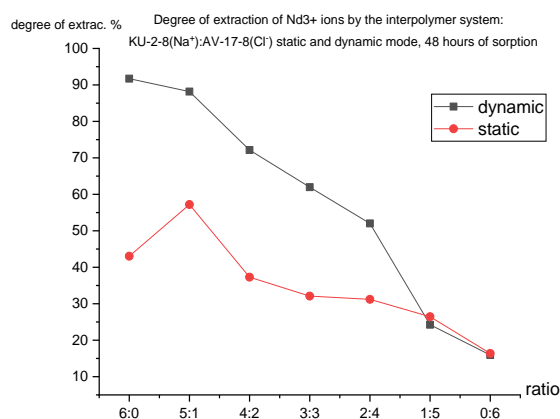

Figure 5 - The degree of neodymium ion extraction under various conditions

Another important indicator for the industrial practice of REM extraction is the ability to effectively desorb these metals from sorbent matrices. For this purpose, the indicator of the degree of neodymium desorption from the polymer matrix KU-2-8(Na+) was calculated in this

study[+](assuming that the main amount of neodymium is absorbed by cationite since the presence of metal was not detected by the method during desorption from AB-17-8(Cl[-]) with nitric acid). This value was calculated using the following formula:

$$\omega = \frac{C_{desor.}}{C_{sorb.}} * 100\%$$

where ω is the degree of desorption, C_{desor} is the concentration of desorbed metal, and C_{sorb} is the concentration of absorbed metal. C_{sorb} was calculated by subtracting the residual ion concentration (determined on a spectrophotometer) from the initial ion concentration.

Figure 6 shows the graph and dependences of the degree of desorption on the molar ratios of cationite and anionite. Desorption was performed for 72 hours with or without stirring. In the 5:1 ratio with constant mixing, a peak is also observed, and high values of the degree of extraction during desorption from the polymer matrix in the ratios of 3:3, 2:4, and 1:5. It can be numerically explained by the small amount of sorption and relatively good desorption. For example, in the ratio 6:0, low desorption is numerically explained by good sorption and retention of metal in the polymer structure.
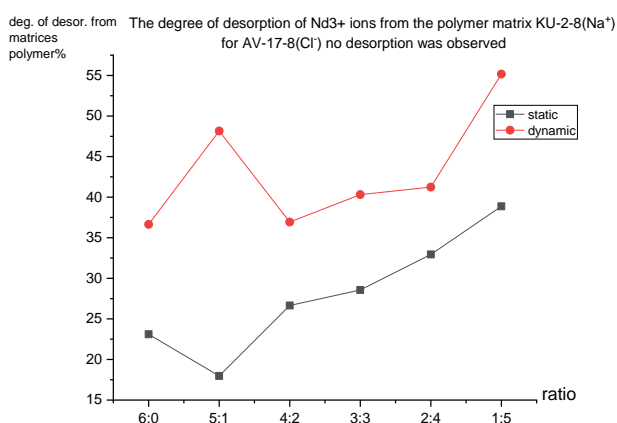

The degree of desorption of Nd3+ ions from the polymer matrix KU-2-8(Na+) for AV-17-8(Cl-) no desorption was observed

Figure 6 - Degree of neodymium desorption from cationite in different modes

Since different molar ratios of cationite and anionite were used in the experiment, it is necessary to recalculate by 1 mol of the polymer in order to see the real picture of the degree of sorption. The need for such a recalculation is clear

from simple logic - if the absorption of a metal ion occurs mainly due to cationite, then a larger amount of it will lead to better results. Therefore, with active mixing in Figure 2, the graph clearly shows that the lowest residual concentration falls on the ratio of 6:0, i.e. individual KU-2-8 (Na[+]). The highest residual concentration in the dynamic regime is observed in the ratio 1:5, but this does not directly mean "worse" sorption, but only shows that with a gradual decrease in cationite, the residual concentration increases. When converted to 1 mol, a completely different picture appears. Figures 7, and 8 show an inverse trend of increasing the sorption degree from 6:0 to 1:5 in both modes. This fact confirms that due to the mutual activation of hydrogels, their ionization increases, which contributes to better metal binding. Otherwise, when converted to 1 mol, the sorption degree values at 6:0 and 1:5 would have to coincide or at least be close in values.
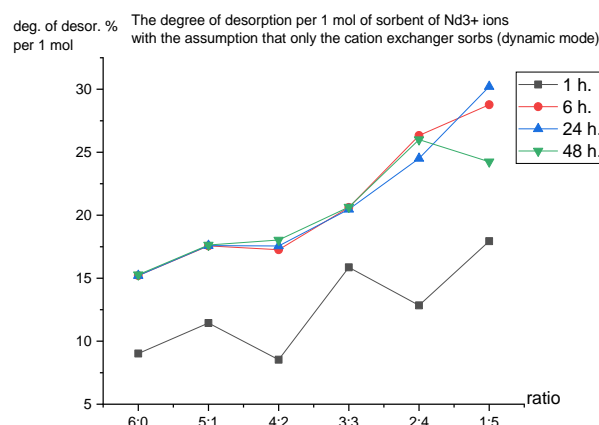

The degree of desorption per 1 mol of sorbent of Nd3+ ions with the assumption that only the cation exchanger sorbs (dynamic mode)

Figure 7-Degree of desorption from cationite in terms of 1 mol (dynamic mode)

The degree of desorption per 1 mol of sorbent of Nd3+ ions with the assumption that only the cation exchanger sorbs (static mode)
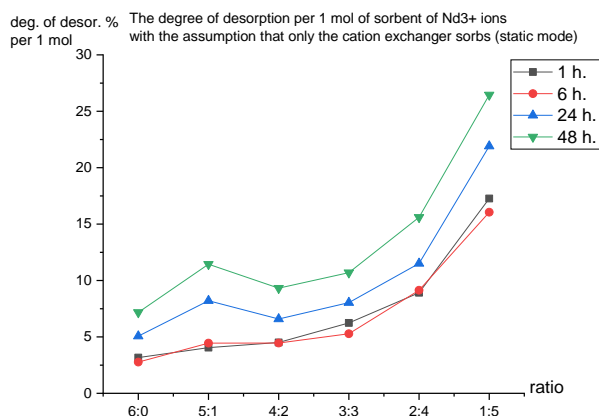
Figure 8-Degree of desorption from cationite in terms of 1 mol (static mode)

Conclusions

The results obtained reveal several important points. First, the mutual activation of hydrogels (cationite and anionite) generally results in more efficient sorption of neodymium ions compared to individual samples. This fact is especially clearly visible when converting sorbents to 1 mol, which eliminates the difference in sorption values due to different mass ratios of cationite. This is due to an increase in the ionization of the cationite, and consequently, its conformational changes, which contributes to the energetically and sterically more profitable binding of metal ions from the solution of its salt. At the same time, the maximum degree of recovery was observed at the ratio KU-2-8 (Na$^+$): AB-17-8(Cl$^-$) 5:1 in static mode, and at 6:0 in dynamic mode. The second important fact is the need to extract rare-earth metals in a dynamic mode since in comparison with static, equilibrium occurs faster, and at the same time, numerically high sorption of the target metal is observed.

Өнеркәсіптік ионалмастырғыштардың тұз формалары негізіндегі интерполимерлік жүйелермен неодим иондарын алу ерекшеліктері

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ТҮЙІНДЕМЕ

Бұл зерттеу АВ-17-8(Cl$^-$) және КУ-2-8(Na$^+$) ион алмастырғыш шайырларының неодим иондарының сорбциясына қашықтықтан өзара әрекеттесу мен өзара белсендірудің әсерін зерттеуге бағытталған. Неодим иондарының концентрациясы боялған арсеназо (III) реагентімен әрекеттесу негізінде спектрофотометрмен анықталды. Сорбция кезінде неодим иондарының бөліну дәрежесі және полимер матрицасынан десорбция кезінде осы металдың алыну дәрежесі есептелді. Сорбция режимін таңдаудың неодимді алу тиімділігіне әсері зерттелді: динамикалық (металл алынған ерітіндіні араластыра отырып) және статикалық (араластырусыз). Алынған процестердің динамикасына сүйене отырып, ерітіндіні араластыру кезінде (араластыру жылдамдығының диапазоны 40-80 айн. мин.) металдың сорбциясы мен десорбциясы арасындағы тепе-теңдік 6 сағаттық өзара әрекеттесуден кейін орнатылады. Сондай-ақ, динамикалық режимде мақсатты металл статикалық режиммен салыстырғанда әлдеқайда жақсы сорбцияланды, бұл десорбция кезінде алынған металл иондарының санына әсер етті. Интерполимер жүйесіндегі неодим иондарының максималды сорбция дәрежесі гидрогельдердің 5:1 және 48 сағаттық араластырусыз қашықтықтан әрекеттесу қатынасында байқалатыны және 42,8 мг/л қалдық концентрациясында болатыны анықталды. Динамикалық режимде бастапқы катионит үшін максималды сорбция байқалады, қалдық концентрациясы 8,28 мг/л. 1 моль катионитке есептегенде гидрогельдердің өзара активтенуінің әсері айқын көрінеді, бұл неодимнің 6:0 (бастапқы катионит) 1:5 қатынасынан сорбция дәрежесінің жоғарылау тенденциясымен дәлелденеді. Иониттердің әртүрлі моль қатынастарында неодим иондарының сорбциясының айтарлықтай өсуі байқалады. Бұл нәтижелер оның тұзының сулы ерітіндісінен неодим иондарының сорбциясы үшін оңтайлы жағдайларды қамтамасыз ететін конформацияларды құрайтын иондалған құрылымдардың пайда болғанын көрсетеді, бұл оның өнеркәсіптік қоспалардан селективті алу үшін практикалық мақсаттарға қызмет ете алады. Өнеркәсіптік қондырғылардың жылдам жұмыс істеуі және металды полимерлі матрицалармен толық байланыстыру үшін металды динамикалық режимде гидрогельдермен алу қажеттілігі анықталды.

Түйін сөздер: интерполимерлік жүйе, неодим иондары, өзара активтену, АВ-17-8(Cl-) және КУ-2-8(Na$^+$) ион алмастырғыштары, қашықтықтан өзара әрекеттесу.

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Особенности извлечения ионов неодима интерполимерными системами на основе солевых форм промышленных ионитов

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АННОТАЦИЯ

Данное исследование было направлено на изучение влияния дистанционного взаимодействия и взаимной активации между солевыми формами ионообменных смол AB-17-8(Cl^-) и КУ-2-8(Na^+) на сорбцию ионов неодима. Концентрация ионов неодима определялась с помощью спектрофотометра на основе взаимодействия с окрашенным реагентом арсеназо (III). Были рассчитаны степень извлечения ионов неодима при сорбции и степень извлечения этого металла при десорбции из матрицы полимеров. Изучено влияние выбора режима сорбции на эффективность извлечения неодима: динамического (с перемешиванием раствора из которого извлекался металл) и статического (без перемешивания). Исходя из полученной динамики процессов установлено, что при перемешивании раствора (диапозон скорости перемешивания 40-80 об. в мин.) равновесие между сорбцией и десорбцией металла устанавливается после 6 часов взаимодействия. Также при динамическом режиме целевой металл сорбировался намного лучше по сравнению со статическим режимом, что повлияло и на количество ионов металла полученного при десорбции. Установлено, что максимальная степень сорбции ионов неодима у интерполимерной системы наблюдается при соотношениях гидрогелей 5:1 и 48 часов дистанционного взаимодействия без перемешивания, и составляет 42,8 мг/л остаточной концентрации. В динамическом режиме максимальная сорбция наблюдается для исходного катионита, остаточная концентрация 8,28 мг/л. При рассчете на 1 моль катионита отчетливо виден эффект взаимной активации гидрогелей, который доказывается тенденцией увеличения степени сорбции неодима от соотношения 6:0 (исходный катионит) к 1:5. В различных мольных соотношениях ионитов наблюдается значительный рост сорбции ионов неодима. Данные результаты указывают на возникновение ионизованных структур, формирующих конформации, которые обеспечивают оптимальные условия для сорбции ионов неодима из водного раствора его соли, что может служить практическим целям его селективного извлечения из промышленных смесей. Установлена необходимость извлечения металла гидрогелями в динамическом режиме для более быстрой работы промышленных установок и более полного связывания металла полимерными матрицами.

Ключевые слова: интерполимерная система, ионы неодима, взаимная активация, ионообменники AB-17-8(Cl-) и КУ-2-8(Na^+), дистанционное взаимодействие.

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