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Features of the extraction of yttrium and lanthanum with an intergel system based on hydrogels of polyacrylic acid and poly-4-vinylpyridine

To predict the sorption activity and selectivity of hydrogels, the effect of mutual activation of polymer networks in the intergel system was studied. The intergel system of the hydrogel of polyacrylic acid (hPAA) and the hydrogel of poly-4-vinylpyridine (hP4VP), which was studied at a distance through the volume of the solvent in the absence of direct contact between the polymer networks, were chosen as the object of study. Intergel systems have been investigated using methods for measuring electrical conductivity, pH, and gravimetry. The mutual activation of La³⁺ and Y³⁺ ions with the intergel system in an aqueous medium was also studied. It has been established that as a result of the remote interaction of the studied hydrogels, their mutual activation occurs, leading to a significant change in their electrochemical and conformational properties. At certain ratios of acidic and basic hydrogels, a significant increase in the sorption of lanthanum and yttrium ions is observed in comparison with the initial hydrogels. These results indicate the appearance of ionized structures with optimal conformation, providing an optimal ligand environment around lanthanum and yttrium ions.

Keywords: intergel systems, polyacrylic acid, poly-4-vinylpyridine, hydrogels, remote interaction, La³⁺ ions, Y³⁺ ions, sorption, desorption.

Introduction

It is known that polymer hydrogels having functional groups capable of binding metal ions are classified as highly selective polymer structures. This contributes to the fact that such polymers have a sorption ability with respect to metal ions, including ions of rare earth elements.

According to the phenomenon of the remote interaction of polymer hydrogels in an aqueous medium, nonionic macromolecules that are unable to undergo ionization and subsequent dissociation cannot participate in the process of remote interaction. Therefore, the remote interaction of polymers of various structures leads to their functionalization.

Previous studies showed that the remote interaction of polymer hydrogels leads to a significant change in their electrochemical and conformational properties. As a result of remote interaction, functional groups are formed without counter ions, stabilized by intramolecular interactions; electrochemical and conformational changes in macromolecules lead to a significant increase in the sorption ability of the intergel system, consisting of two hydrogels; the «long-range effect» under certain conditions leads to high selectivity with respect to metal ions [1–4].

In this regard, the aim of this work is to study the sorption ability using an example of an intergel system consisting of hydrogels of polyacrylic acid and poly-4-vinylpyridine with respect to lanthanum and yttrium ions, as well as predicting the possibility of using them to extract rare and rare-earth metals.

Experimental

To measure the electrical conductivity of the solutions, a MARK-603 conductometer (Russia) was used; the concentration of hydrogen ions was determined using a Metrohm 827 pH-Lab pH meter (Switzerland). The mass was determined on an analytical electronic balance SHIMADZUAY220 (Japan). The concentration of La^{3+} and Y^{3+} ions was determined on spectrophotometers Perkin Elmer Lambda 35 (USA), Jenway-6305 (Japan).

The studies were carried out in an aqueous medium and in a 0.005 M solution of 6-aqueous lanthanum and yttrium nitrate. Polyacrylic acid hydrogels were synthesized in the presence of a crosslinking agent N,N -methylene-bis-acrylamide and a redox system $\text{K}_2\text{S}_2\text{O}_8 - \text{Na}_2\text{S}_2\text{O}_3$ in an aqueous medium. The synthesized hydrogels were crushed into fine dispersions and washed for a long time with distilled water until a constant value of the electrical conductivity of aqueous solutions was established. A Sigma-Aldrich crosslinked divinylbenzene hydrogel of poly-4-vinylpyridine (hP4VP) was used.

Electrochemical studies

Experiments to determine the pH and conductivity were carried out at room temperature. The studies of the intergel system were performed in the following order: each hydrogel in dry form was placed in separate cells from a plastic bag, the pores of which are permeable to low molecular weight ions and molecules, but impermeable to dispersion of hydrogels. Then the cells with dry hydrogels were placed in glasses with aqueous solutions and solutions of lanthanum nitrate. The electrical conductivity and pH of the supra gel were determined in the presence of hydrogels in solution. Adsorption behavior in relation to La (III) in aqueous conditions: studies using periodic and column methods under various conditions: experimental studies of the kinetic and thermodynamic characteristics of the adsorption process, studied in the work, which are indicated in the reference literature and were considered [5–6].

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

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

$$\eta = (C_{\text{int}} - C_{\text{res}}) / C_{\text{res}} * 100 \%,$$

where C_{int} is the initial concentration of the metal in solution, g/l; C_{res} is residual concentration of metal in solution, g/l [7–9].

Results and Discussion

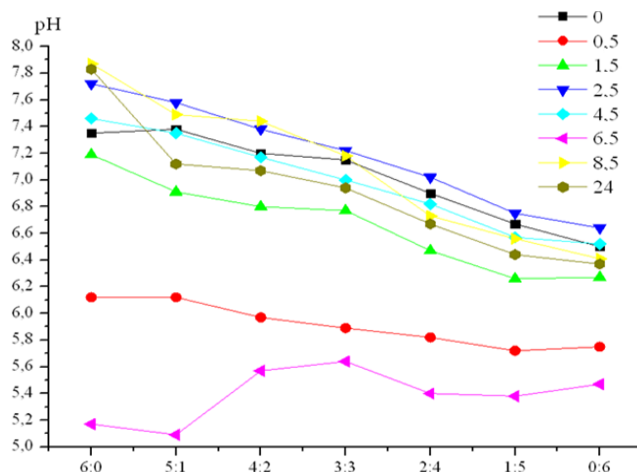
Study of the mutual activation of hPAA:hP4VP hydrogels in the intergel system

The presence of an intergel system in an aqueous solution leads to various processes that affect the electrochemical balance in the solution.

As a result of these interactions, a state arises in which there are no counterions in some of the charged functional groups of hydrogels. The concentration of ionized groups that lack counterions depends on the initial molar ratios of the polymer networks and other factors.

Figure 1 shows the time dependence of the pH of aqueous solutions on the molar ratios of hP4VP. As polyacid predominates, a decrease in the concentration of hydrogen ions with time is observed. Distinct minima are observed in the presence of only polyacids (hPAA:hP4VP = 6:0) after 6.5 hours of interaction. The maximum pH values after 24 hours are observed at a ratio of hPAA:hP4VP = 6:0. As the proportion of polybase increases, the pH of the solution gradually decreases. The lowest pH value is observed in a 5:1 ratio after 6.5 hours of interaction.

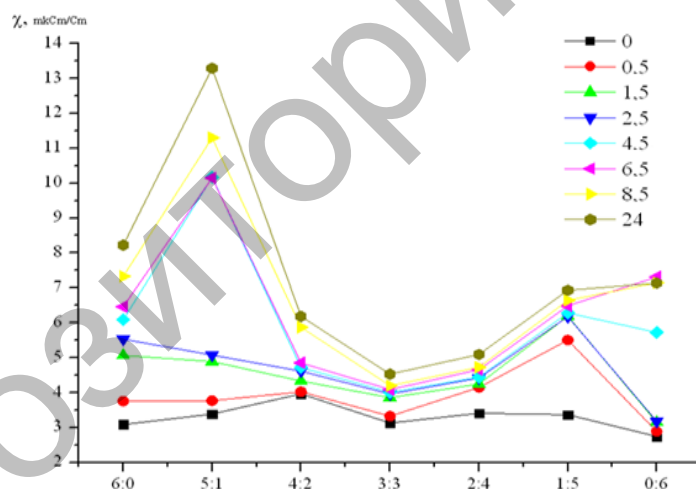
The appearance of an excess of H^+ ions is due to the high swelling rate and dissociation of COOH groups, as well as the insufficient swelling rate of the main groups and their low concentration. An increase in the content of OH^- ions in the aqueous medium is associated with a low swelling rate and a low concentration of COOH groups, as well as a high swelling rate and the interaction of the main functional groups with H^+ ions. This is possible in the case of a second reaction in which hydroxyl anions are released into the solution. In parallel, the third reaction is taking place, as a result of which the free proton binds to the pyridine ring and the concentration of positively charged ions in the solution decreases sharply.



1 — 0 h; 2 — 0.5 h; 3 — 1.5 h; 4 — 2.5 h; 5 — 6.5 h; 6 — 6.5 h; 7 — 8.5 h; 8 — 24 h

Figure 1. Dependence of the pH of aqueous solutions on the molar ratio of hydrogels hPAA:hP4VP over time

Figure 2 shows the dependence of changes in the electrical conductivity of solutions on the molar ratio of hydrogels over time. During the remote interaction, regions of minimum and maximum electrical conductivity appear, and at the beginning of the interaction, the conductivity of the solutions increases. With a ratio of 5:1, high values of electrical conductivity are observed after 4.5; 6.5; 8.5 and 24 hours. The maximum conductivity values are reached after 24 hours. As can be seen from the figure, the minimum electrical conductivity of aqueous solutions is observed at a ratio of 3:3 for all the time of distance interaction of hydrogels.



1 — 0 h; 2 — 0.5 h; 3 — 1.5 h; 4 — 2.5 h; 5 — 6.5 h; 6 — 6.5 h; 7 — 8.5 h; 8 — 24 h

Figure 2. Dependence of the electrical conductivity of aqueous solutions on the molar ratio of hydrogels hPAA:hP4VP in time

Comparing the values of electrical conductivity and pH, we can conclude that as a result of mutual activation of the hydrogels go into a more highly ionized state. The area of maximum activation of hydrogels is the ratio of hPAA:hP4VP = 5:1. The highest ionization of gP4VP hydrogel occurs at a ratio of 5:1.

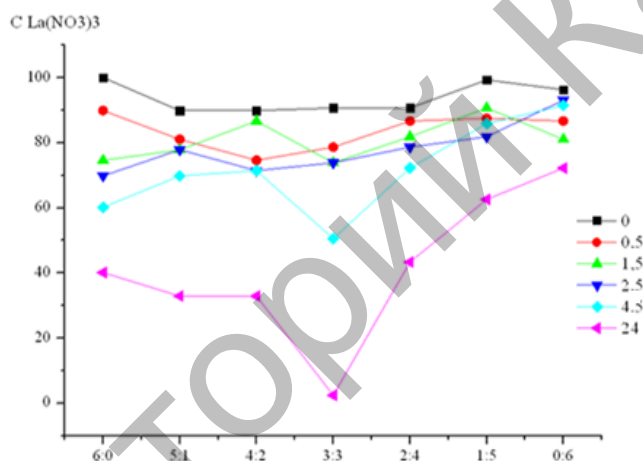
The minimum electrical conductivity of the hPAA:hP4VP intergel system is due to the binding of the proton cleaved from the carboxyl group by the vinylpyridine heteroatom. When immersed in an aqueous medium, both hydrogels begin to swell as a result of interaction with water molecules. Carboxyl groups are first ionized, then anions — COO⁻ and hydrogen ions (protons) H⁺ are dissociated into the carboxylate. The ionization of the cationic hydrogel of poly-4-vinylpyridine in an aqueous medium occurs as a result of the addition of hydrogen ions formed during the dissociation of carboxyl groups and water molecules into H⁺, OH⁻

ions. The long-range effect of hydrogels leads to the fact that during these interactions the total content of positive charges in the aqueous medium decreases.

High values of electrical conductivity, in turn, indicate that, at certain ratios of two hydrogels, the dissociation of carboxyl groups prevails over the process of proton association by polybasic nitrogen heteroatoms. The reason for this may be a conformational change in the links in the interstitial chains. At certain concentrations, charged NH^+ groups can form intramolecular cross-links $\geq \text{N} \dots \text{H}^+ \dots \text{N} \equiv$, which lead to the folding of macromolecular tangles and a decrease in proton binding.

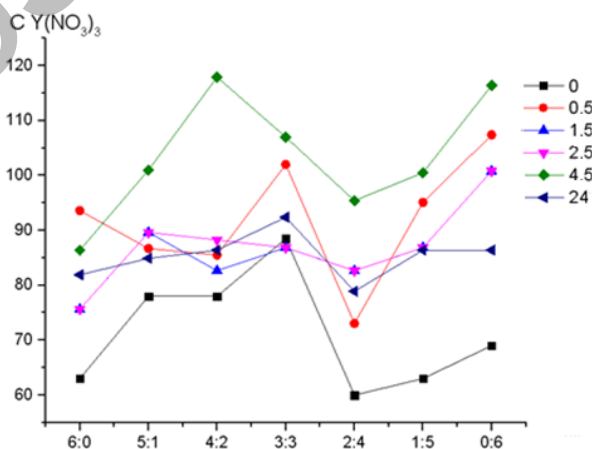
From the above results, it can be expected that the phenomena of mutual activation of functional hydrogels should be reflected in activation processes with metal ions. To verify this assumption, the mutual activation of the intergel system with respect to lanthanum and yttrium ions was studied.

Figure 3 reflects the change in the concentration of lanthanum ions during its sorption by the hPAA:hP4VP intergel system. First, in all ratios, we observe that the amount of lanthanum ions in the solution is high. As can be seen from the figure, in comparison with the ratios when only polyacid or polybase is present in the solution (ratios 6:0 and 0:6), the ratios at which there are 2 hydrogels in the solution have a much greater sorption ability. A much higher degree of extraction of La^{3+} ions at a ratio of 3:3. Moreover, the greatest sorption occurs when the ratio of hPAA:hP4VP = 3:3 after 24 hours of interaction. This is due to the fact that, at this ratio, the hydrogels of polyacrylic acid and poly-4-vinylpyridine are in a highly ionized state due to the mutual activation of hydrogels in intergel systems.



1 — 0 h; 2 — 0.5 h; 3 — 1.5 h; 4 — 2.5 h; 5 — 4.5 h; 6 — 24 h

Figure 3. Dependence of the concentration of lanthanum ions on the molar ratio of hydrogels in the hPAA:hP4VP intergel system in the environment of 6-aqueous lanthanum nitrate



1 — 0 h; 2 — 0.5 h; 3 — 1.5 h; 4 — 2.5 h; 5 — 4.5 h; 6 — 24 h

Figure 4. Dependence of the concentration of yttrium ions on the molar ratio of hydrogels in the hPAA:hP4VP intergel system in a medium of 6- yttrium nitrate

The dependence of the concentration of yttrium ions on the molar ratio of hydrogels in time in the hPAA:hP4VP intergel system in a medium of 6-³yttrium nitrate is shown in Figure 4. The results obtained indicate that mutual activation leads to a significant increase in the sorption properties of polymer hydrogels in the intergel system.

The starting hydrogel of polyacrylic acid has a higher degree of extraction of yttrium ions. As can be seen from Figure 4, the highest activation of yttrium ions occurs at a ratio of 2:4 hPAA:hP4VP. In the figure, one can observe the general regularity of the degree of activation of yttrium ions in the intergel system. The lowest activation point corresponds to a 4:2 ratio after 4.5 hours of interaction. And in a 2:4 ratio of the intergel system, a decrease in the concentration of yttrium ions is observed. The main reason for such a high degree of extraction is the high ionization of polymer structures as a result of their mutual activation. Further, the extraction of yttrium ions by the intergel system decreases and the polybase present in the solution shows the lowest activation with yttrium ions. These results indicate the appearance of ionized structures with optimal conformation, providing an optimal ligand environment around lanthanum and yttrium ions.

Table 1 presents the values of the degree of extraction of lanthanum ions by the hPAA:hP4VP intergel system. It can be seen that in the hPAA:hP4VP intergel pairs, the degree of sorption is much higher compared to individual hydrogels. This is due to the high degree of ionization of the starting polymers in the intergel system during their remote interaction as a result of mutual activation. The highest ionization of hPAA:hP4VP occurs at a ratio of 50 % hPAA – 50 % hP4VP, as a result of which the degree of extraction of lanthanum ions reaches maximum values (97.6 %).

Table 1

Degree of extraction of lanthanum ions with hydrogels PAA, P4VP

τ, h	$\eta(\text{La}), \%$						
	hPAA:hP4VP, %						
	100	83:17	67:33	50:50	33:67	17:83	100
0	0	10.2	10.2	9.4	9.4	0	3.7
0.5	10.2	19	25.4	21.4	13.4	12.6	13.4
1.5	25.4	22.2	13.4	26.2	18.2	9.4	19
2.5	30.2	22.2	28.6	26.2	21.4	18.2	7
4.5	39.9	30.2	28.6	49.5	27.8	14.2	8.6
24	59.8	67.1	67.1	97.6	56.7	37.5	27.8

Table 2 shows the values of the degree of extraction of lanthanum ions by the hPAA: hP4VP intergel system. The highest ionization of hPAA:hP4VP occurs at a ratio of 33 % hPAA – 67 % hP4VP, as a result of which the degree of extraction of yttrium ions reaches maximum values (26.9 %).

Table 2

Degree of extraction of yttrium ions with hydrogels PAA, P4VP

τ, h	$\eta(\text{Y}), \%$						
	hPAA:hP4VP, %						
	100	83:17	67:33	50:50	33:67	17:83	100
0	37	22	10.2	9.4	9.4	0	3.7
0.5	6.4	13.3	14.5	3.2	26.9	4.9	2.6
1.5	24.3	10.3	17.3	13.1	17.3	13.1	9.2
2.5	24.3	10.3	11.7	13.1	17.3	13.1	9.2
4.5	13.6	9.9	2.1	3	4.6	4.5	3.6
24	18.1	15.1	13.6	1.6	21.1	13.6	13.6

Conclusions

Based on the obtained data on the electrical conductivity, pH, and swelling coefficient, we can conclude that sorption of lanthanum ions by polymer hydrogels occurs. When the molar ratio of hPAA: hP4VP hydrogels is equivalent, the intergel system has the highest sorption ability with respect to lanthanum ions in

comparison with the initial hydrogels. The maximum degree of binding of the polymer chain to yttrium ions is manifested when the ratio of hPAA:hP4VP = 33 %:67 %. The mutual activation of hPAA and hP4VP hydrogels in the intergel system allows one to achieve a significantly higher degree of sorption for the intergel pair than for individual hydrogels. As can be seen from the data obtained, most of the lanthanum is recovered within 24 hours. In this case, the highest sorption occurs at the ratios of 67 % hPAA – 33 % hP4VP and 33 % hPAA – 67 % hP4VP, 67.1 and 56.7 % of lanthanum are recovered, respectively. The largest amount (97.6 %) of lanthanum is recovered at a ratio of 50 % hPAA – 50 % hP4VP at 24 h [10–13].

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Полиакрил қышқылы мен поли-4-винилпирдин гидрогельдері негізіндегі интергельді жүйеден иттрий мен лантанды алу ерекшеліктері

Гидрогельдердің сорбциялық белсенділігі мен селективтілігін болжау үшін интергельді жүйедегі полимерлік торларды өзара активтендірудің әсері зерттелген. Зерттеу объектісі ретінде полиакрил қышқылының гидрогелі (гПАК) және поли-4-винилпирдиннің (гП4ВП) гидрогелі таңдалды. Интергельді жүйенің рН, электр өткізгіштігі гравиметриялық өлшеу әдістерімен анықталды. Сондай-ақ сулы ортада интергельді жүйемен La^{3+} және Y^{3+} иондарының өзара активациясы зерттелді. Ең жоғары электр өткізгіштік 24 сағаттан кейін орын алды. Электр өткізгіштігінің ең төменгі мәндері гПАК:гП4ВП = 0:6 интергельді жүйесінде

байқалған. Ол интергельді жүйенің әлсіз диссоциациясымен байланысты. гПАК:гП4ВП интергельді жүйесінде сутегі иондарының концентрациясының ұлғаюы гПАК:гП4ВП = 5:1 арақатынасында көрінген. Зерттелген гидрогельдердің қашықтықтан өзара әрекеттесуі нәтижесінде олардың электрохимиялық және конформациялық қасиеттерінің айтарлықтай өзгеруіне әкеліп соқтыратыны және өзара активтендіру жүргізілгені анықталды. Егер біз осы деректерді электр өткізгіштігі туралы деректер бойынша салыстырсақ, бұл ретте карбоксильді топтардың диссоциациясы процесі протондардың винил пиридинге қосылу процесінен басым болады деген қорытынды жасауға болады. Қышқылдық және негіздік гидрогельдердің белгілі бір арақатынасында бастапқы гидрогельдермен салыстырғанда лантан және иттрий иондарының сорбциясының айтарлықтай өсуі байқалған. Бұл нәтижелер лантан және иттрий иондарының айналасында оңтайлы лигандты ортаны қамтамасыз ететін оңтайлы конформациясы бар иондалған құрылымдардың пайда болуын көрсетеді.

Клт сөздер: интергельді жүйе, полиакрил қышқылы, поли-4-винилпиридин, La^{3+} ионы, Y^{3+} ионы, сорбция, десорбция.

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Особенности извлечения иттрия и лантана интергелевой системой на основе гидрогелей полиакриловой кислоты и поли-4-винилпиридина

Для прогнозирования сорбционной активности и селективности гидрогелей было исследовано влияние взаимной активации полимерных сеток в интергелевой системе. В качестве объекта исследования была выбрана интергелевая система «гидрогель полиакриловой кислоты (гПАК) – гидрогель поли-4-винилпиридина (гП4ВП)», которая изучалась на расстоянии через объем растворителя при отсутствии непосредственного контакта между полимерными сетками. Интергелевые системы были изучены методами измерения электропроводности, pH и гравиметрии. Также рассмотрена взаимная активация ионов La^{3+} и Y^{3+} с интергелевой системой в водной среде. Зависимость удельной электропроводности от молярного соотношения гидрогелей во времени, увеличение электропроводности происходит при соотношении гПАК:гП4ВП = 5:1 на протяжении всего времени удаленного взаимодействия. Максимальная электропроводность была достигнута через 24 ч. Минимальные значения электропроводности отмечены в области интергелевой системой гПАК:гП4ВП = 0:6, что связано с его слабой диссоциацией. Зависимость концентрации ионов водорода гПАК:гП4ВП, увеличение концентрации ионов водорода происходит при соотношении гПАК:гП4ВП = 5:1. Установлено, что в результате дистанционного взаимодействия изучаемых гидрогелей происходит их взаимная активация, приводящая к значительному изменению их электрохимических и конформационных свойств. Эти результаты указывают на возникновение ионизованных структур с оптимальной конформацией, обеспечивающих оптимальное лигандное окружение вокруг ионов лантана и иттрия.

Ключевые слова: интергелевая система, полиакриловая кислота, поли-4-винилпиридин, сорбция, десорбция, ионы La^{3+} , ионы Y^{3+} .

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