

## IMPROVED SORPTION OF PRASEODYMIUM IONS FROM NITRATE SOLUTIONS BY ACTIVATED ION EXCHANGERS

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**Abstract.** *Introduction.* In this research, we investigated the praseodymium ions sorption by the ion exchangers KU-2-8 (H<sup>+</sup> form) and AV-17-8 (OH<sup>-</sup> form) as an interpolymer system called "KU-2-8:AV-17-8" (X:Y) with the molar ratio of ionic groups X:Y (6:0, 5:1, 4:2, 3:3, 2:4, 1:5, and 0:6) in praseodymium nitrate solution. To be clear, two cross-linked polymers having active functional groups that are in the same solution but not in direct contact form an interpolymer system. *The purpose* of our research to demonstrate how activating ion exchangers as functional polymers by remote contact in an aqueous medium enhances the sorption of a variety of valuable metals and their compounds from industrial waste solutions, including praseodymium. *Objects.* Ion exchangers that are mutually activated in an aqueous medium undergo conformational and electrochemical changes that lead to a shift in their state from neutral to highly ionized and an increase in their sorption activity toward target metal ions. *Results.* The experimental findings showed that the sorption activity of the interpolymer system KU-2-8:AV-17-8 (5:1) increased significantly when compared to praseodymium ions (43 %), but the sorption activity of the raw KU-2-8 (6:0) and raw AV-17-8 (0:6) had the same values of 25 % and 5 %, respectively. *Conclusion.* This research sheds light on a novel and promising approach to improve the efficiency of ion exchangers, opening possibilities for the effective recovery and removal of valuable metals from complex solutions. Additionally, it holds practical implications for sustainable waste management and resource recycling, contributing to the advancement of environmental protection and resource conservation in various industrial sectors. Further investigations and optimization of the interpolymer system could yield substantial benefits for greener and more resource-efficient future sorption technologies.

**Key words:** ion exchangers, interpolymer system, activation, nitrate solution, process, technology, sorption, ICP-OES, residual concentration, praseodymium

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## 1. Introduction

Praseodymium is a rare earth element with various applications, including magnet production [1], lighting [2], lasers [3], and more. The increasing demand for its manufacturing has driven research into alternative sources of praseodymium [4, 5]. Industrial aqueous waste could serve as an additional source for its production [6]. Currently, several methods for recovering metals from solutions have been developed, such as chemical precipitation [7], membrane filtration [8], and adsorption [9]. Among these approaches, adsorption stands out as one of the most promising due to its high efficiency and low cost [10].

Ion exchangers are being long employed as adsorbents in order to remove various metal ions from solutions [11-15]. These porous materials contain exchangeable functional groups (e.g., carboxylic, sulfonic, or amino groups) [16] that can interact with metal ions in an aqueous environment through electrostatic attraction. The activation step is necessary to modify the conformational and electrochemical characteristics of the ion exchangers, rendering them highly ionized [17]. To achieve this, polypropylene meshes with polymers inside were put at some distance from each other within a glass container filled with double-distilled water. This setup established an interpolymer system KU-2-8:AV-17-8 (X:Y), where X:Y represents the molar ratio of ionic groups (ranging from 6:0 to 0:6) (see figure 1).

In hydrometallurgy, an urgent need for the formation of highly efficient sorption technology aimed at recovering valuable rare earth elements (REEs) from waste solutions [18, 19]. Nevertheless, despite numerous attempts to create polymeric sorbents, only a limited number have found practical applications [20, 21]. This is largely attributed to the low selectivity exhibited by most ion exchangers, alongside the challenges in adapting them to intricate industrial solutions [22]. Recent times have witnessed a growing interest in the utilization of interpolymer systems for the sorption of rare earth metal ions from aqueous solutions [23]. The reciprocal activation of these ion exchangers induces conformational and electrochemical modifications, leading to their transformation into a highly ionized state and subsequently enhancing sorption activity [24].

The novelty of the present research lies in the implementation of the activated ion exchangers KU-2-8 and AV-17-8 as the interpolymer system for the extraction of praseodymium cations from an aqueous medium.

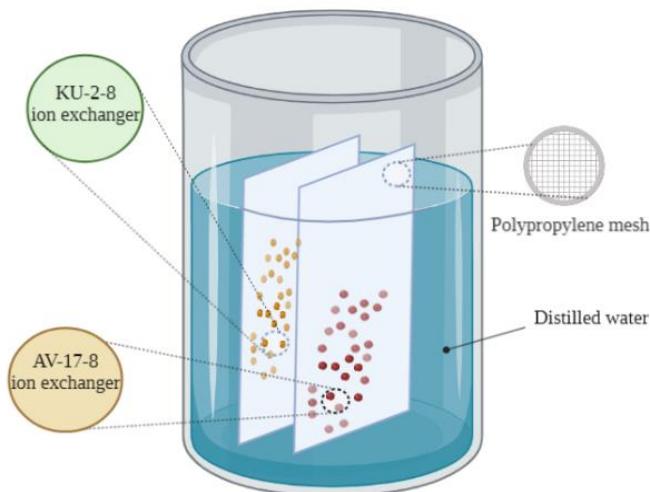
## 2. The experimental part

In this study, we employed various instruments and equipment to meticulously assess the diverse properties of the solutions. For discerning the specific electrical conductivity and effectively characterizing the polyelectrolyte dissociation equilibrium, we utilized the MARK-603 conductometer (Vzor, Russia). To quantify the concentration of hydrogen ions essential for investigating the solution's acid-base attributes, we employed the Metrohm 827 pH-meter pH-Lab (Metrohm AG, Switzerland). For the determination of polymer sample mass, we turned to the Shimadzu AY220 (Shimadzu Corporation, Japan). To ascertain

the concentration of praseodymium (III) ions within the test solutions, we conducted optical density measurements using the Jenway-6305 spectrophotometer (Jenway, UK). Each measurement was performed in triplicate, and the precision of our measurements maintained a margin of error not exceeding 1 %.

*Materials.* In the ongoing research, the subsequent materials were employed: (1) Strongly acidic KU-2-8 ( $\text{H}^+$  form) obtained from Azot, Ukraine – a crosslinked ion exchanger based on styrene and divinylbenzene (with granule size ranging from 0.40 to 0.55 mm); (2) Strongly basic AV-17-8 ( $\text{OH}^-$  form), also acquired from Azot, Ukraine – a gel-type anion exchanger based on a copolymer of styrene and divinylbenzene (with granule size spanning 0.3 to 1.2 mm).

The reagents employed were as follows: for the introduction of praseodymium ions into the solution, praseodymium (III) nitrate hexahydrate (Sigma-Aldrich, Germany) with a purity of 99.9 % trace metals base was utilized. To quantify the praseodymium concentrations within the examined materials, the powdered form of the arsenazo III metal indicator reagent (Sigma-Aldrich, Germany) served as the color-forming agent. Additionally, perchloric acid ( $\text{HClO}_4$ ) (70 % ACS reagent, Sigma-Aldrich, Germany) was employed to produce the standard solution.



**Figure 1** - Illustration of the activation process of the interpolymer system KU-2-8:AV-17-8 (X:Y) in an aqueous medium

*Initiation of the Interpolymer System.* Our earlier studies [24] have shown that polymers may be converted into a highly ionized state more easily when polyelectrolytes are mutually activated during their distant contact. The sorption efficiency of target metal ions is therefore noticeably increased as compared to the raw ion exchangers. Ion exchanger-containing polypropylene meshes were

arranged around 1-2 cm apart within a glass container filled with distilled water to start this activation process. The configuration shown in figure 1 represented the interpolymer system KU-2-8:AV-17-8 (X:Y, signifying the molar ratio of ionogenic groups).

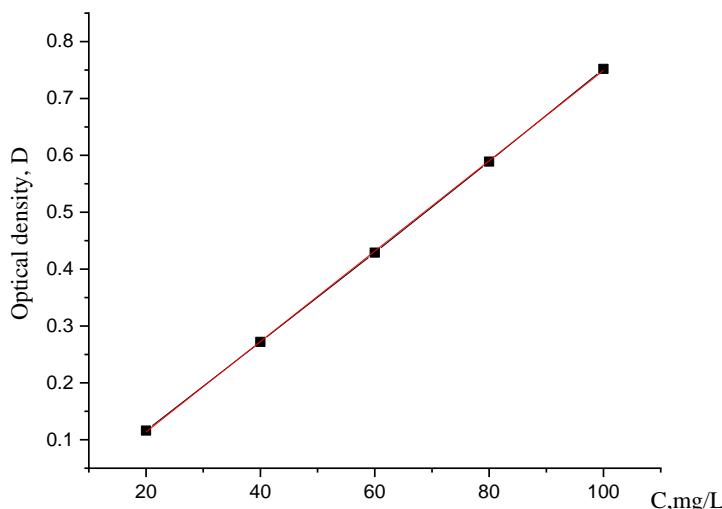
*Determination of the Praseodymium Ion Concentration.* To conduct the experiments, a solution (1000 mL) containing praseodymium ions at a concentration of 100 mg/L was meticulously prepared. The objective was to investigate the sorption performance of the interpolymer system KU-2-8:AV-17-8 (X:Y) across varying ratios (6:0, 5:1, 4:2, 3:3, 2:4, 1:5, and 0:6) concerning praseodymium ions. This solution was carefully divided into seven individual glasses (each containing 100 mL). Subsequently, the ion exchangers KU-2-8 and AV-17-8 were introduced into separate polypropylene meshes, each in accordance with their respective molar ratios denoted as X:Y.

A Jenway-6305 spectrophotometer was used to determine the residual amounts of praseodymium ions in the solution. Further, the sorption degree ( $\eta$ ) of the interpolymer system KU-2-8:AV-17-8 (X:Y) was calculated using Equation (1):

$$\eta = \frac{C_{initial} - C_{residual}}{C_{initial}} \times 100\% \quad (1)$$

where  $C_{initial}$  and  $C_{residual}$  are the initial and residual concentrations (in g/L) of praseodymium ions in the test solutions, respectively.

*Calibration Curve Plotting.* To establish the calibration curve, an initial solution of praseodymium (III) nitrate hexahydrate was prepared, with a concentration of 100 mg/L. This solution was subsequently diluted to create a working solution of 10 mg/L. Following this, a sequence of five standard solutions was meticulously prepared. Each standard solution contained 10, 20, 30, 40, and 50 µg of the target analyte, within 50 mL of solution.



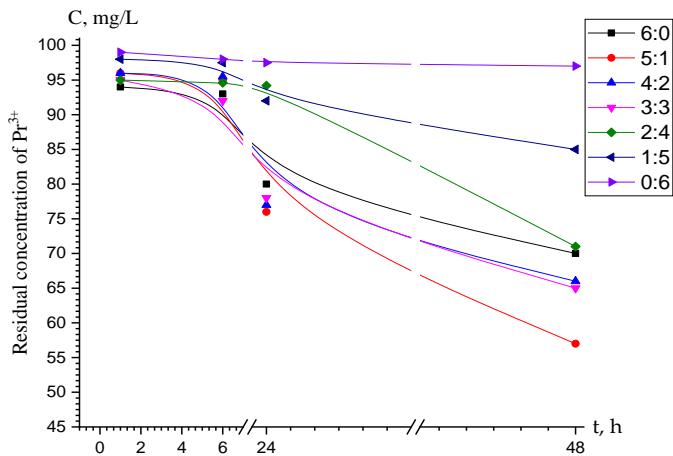
**Figure 2** - The calibration curve for determining the praseodymium concentrations in the test solutions

These standard solutions were then transferred into 50 mL volumetric flasks. In each flask, 12 mL of a 0.015% arsenazo III reagent and 2 mL of 0.08M perchloric acid were introduced. Distilled water was added to each flask to achieve a final volume of 50 mL. After allowing the mixtures to stand for 15 minutes, the measurement process was initiated. The optical density (D) of the formed praseodymium ion complexes within the solutions was determined using a Jenway-6305 spectrophotometer. The calibration curve (figure 2) was plotted by the Origin Pro software (OriginLab Corporation, Massachusetts, USA) ( $r^2$  value was 0.99605).

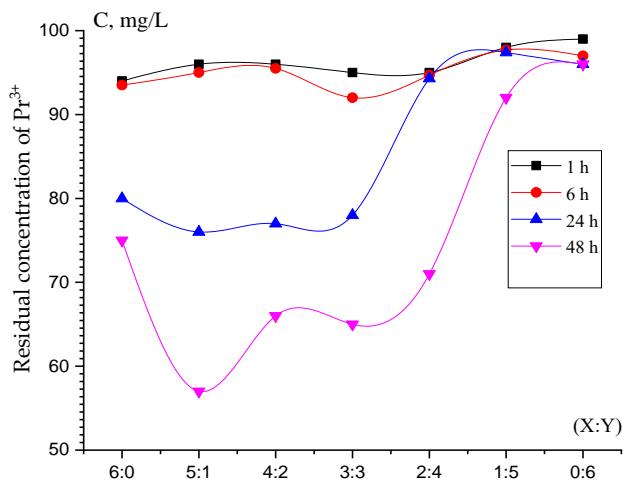
A polymer sorption degree (when customized to a given application) can be used to improve its performance. Understanding a polymer's sorption properties, for example, might allow for changes to its surface chemistry to improve selectivity for certain compounds or stability in different settings.

### 3. Results and their discussion

Figure 3 shows that the interpolymer system KU-2-8:AV-17-8 (X:Y) demonstrated the highest sorption activity towards praseodymium ions at molar ratios 5:1. As a result, the above-mentioned in-terpolymer system was chosen for further detailed comparison with the raw KU-2-8 (6:0) and raw AV-17-8 (0:6) ion exchangers (figure 5).

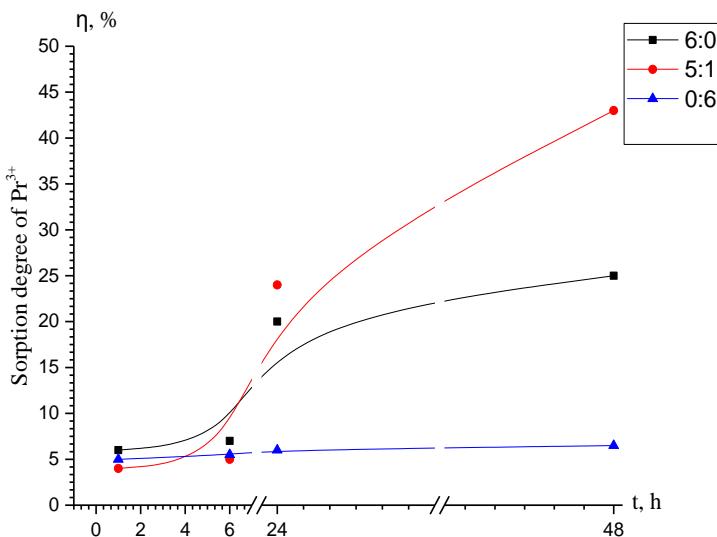


**Figure 3** - The residual concentration of praseodymium ions in a solution after sorption as a function of time



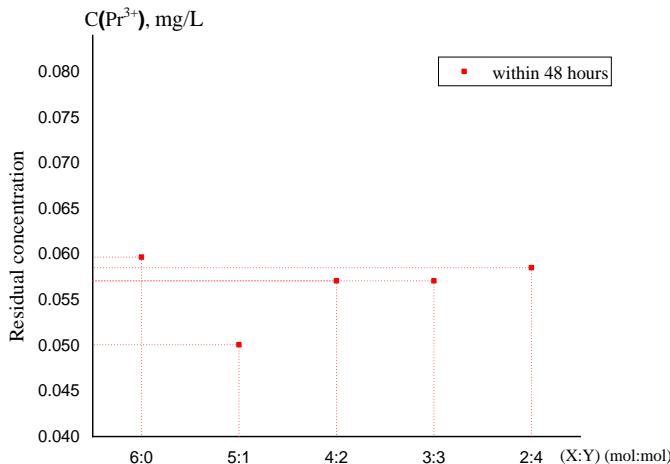
**Figure 4** - The residual concentration of praseodymium ions in a solution after sorption as a function of molar ratios (X:Y)

Figure 4 presents the data on the residual concentration of praseodymium ions in a solution after sorption as a function of molar ratios (X:Y). The data (figure 4) demonstrates that the interpolymer system KU-2-8:AV-17-8 (X:Y) shows the highest sorption activity toward praseodymium ions at molar ratios 5:1 after 48 hours of interaction.



**Figure 5** - Sorption degree of praseodymium ions by the interpolymer system KU-2-8:AV-17-8 (5:1) in comparison with the raw ion exchangers (6:0 and 0:6) over time

Figure 5 demonstrates that after 48 hours of interaction, the interpolymer system KU-2-8:AV-17-8 (5:1) showed the maximum degree of praseodymium ions sorption (43 %), while the similar values for the raw KU-2-8 (6:0) and raw AV-17-8 (0:6) ion exchangers were 25 % and 5 %, respectively.



**Figure 6** - ICP-OES analysis of the residual praseodymium ions concentration in the test solutions after 48 hours of sorption

Furthermore, we used the ICP-OES analytical technique (figure 6) to validate the previously obtained data. At a molar ratio of 5:1 from figure 3, we observed low values (indicating high sorption) for the residual concentration of praseodymium ions in the solution. The experimental results not only corroborate the improved sorption activity of the KU-2-8:AV-17-8 interpolymer system (5:1) but also highlight its superiority over the initial (raw) KU-2-8 (6:0) and AV-17-8 (0:6) ion exchangers when it comes to sorbing praseodymium ions.

#### 4. Conclusion

The effectiveness of employing interpolymer systems as effective sorbents for the removal of different metal ions from liquid solutions is underlined in this work. By producing strong ionization, we increased the sorption activity of both acidic and basic ion exchangers in aqueous media. According to the experimental results, the interpolymer system KU-2-8:AV-17-8 (5:1), with initial (raw) ion exchangers KU-2-8 (6:0) and AV-17-8 (0:6), registered values of 25% for the optimum praseodymium ion sorption.

Future research should prioritize a detailed examination of sorption and desorption mechanisms, a complete examination of the economic situation, a study of techniques for regenerating interpolymer systems, and a broadening of the scope of applied research. These efforts are necessary for a comprehensive assessment of the potential for extracting praseodymium ions from secondary sources and the introduction of large-scale industrial sorption processes.

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**Conflict of Interest:** All authors declare that they have no conflict of interest.

#### БЕЛСЕНДІРЛГЕН ИОН АЛМАСТЫРҒЫШТАРМЕН НИТРАТ ЕРІТІНДІЛЕРИНЕН ПРАЗЕОДИМ ИОНДАРЫНЫҢ ЖАҚСАРТЫЛГАН СОРБЦИЯСЫ

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**Түйіндеме.** *Kіріспе.* Бұл зерттеу «КУ-2-8:АВ-17-8» интерполимерлік жүйесінде орналасқан КУ-2-8 ( $\text{H}^+$  пішіні) және АВ-17-8 ( $\text{OH}^-$  пішіні) сияқты белсендірліген ион алмасу шайырларымен празеодим иондарының сорбциясын зерттеді. Иондық топтардың молярлық қатынасы X:Y (6:0, 5:1, 4:2, 3:3, 2:4, 1:5, және 0:6), содан кейін празеодим нитратының ерітінділеріне батырылады. Түсіндіру үшін интерполимер жүйесі-бул бір сулы ерітіндіде орналасқан, бірақ тікелей байланыссыз белсенді функционалды топтары бар екі өзара байланыскан полимерлердің тіркесімі. **Жұмыс мақсаты.** Біздін зерттеуіздің мақсаты - металдардың кен спектрінін (сонын ішінде празеодим) және олардың өнеркәсіптік калдық ерітінділерінен қосылыстарының сорбциясын жақсарту үшін су ортасындағы кашықтықтан өзара арекеттесу арқылы функционалды полимерлер ретінде ион алмасу шайырларын белсендірудің әсерін көрсету. *Әдістер.* Сулы ортадағы ион алмастырғыштардың өзара белсендірілуі конформациялық және электрохимиялық өзгерістерге әкелді, бұл олардың жоғары иондалған күйге өтуіне әкелді, содан кейін мақсатты металл иондарына қатысты сорбциялық белсенділік жақсарды. *Нәтижелер және талқылау.* Біздің

эксперименттік нәтижелеріміз КҮ-2-8:АВ-17-8 (5:1) интерполимерлік жүйесі празеодим иондарына катысты сорбциялық белсенділіктің (43 %) айтарлықтай өскенін көрсөтті, ал бастапқы КҮ-2-8 (6:0) және АВ-17-8 (0:6) сәйкесінше 25 % және 5 % күрады. *Тұжырым.* Бұл зерттеу күрделі ерітінділерден құнды металдарды тиімді алу мүмкіндіктерін аша отырып, ион алмастырыш шайырлардың тиімділігін артырудың жаңа және перспективалы тәсіліне жарық түсіреді. Сонымен катар, зерттеу қалдықтарды тұрақты басқару және ресурстарды қайта өңдеу үшін практикалық маңызыға ие, бұл әртүрлі салаларда коршаған ортаны қорғау мен ресурстарды үнемдеуді жақсартуға ықпал етеді. Интерполимерлік жүйелерді одан ері зерттеу және онтайланыруды экологиялық және ресурстарды үнемдейтін сорбциялық технологияларының дамуына айтарлықтай үлес коса алады.

**Түйінді сөздер:** ион алмастырыштар, интерполимер жүйесі, активация, нитрат ерітіндісі, процесс, технология, сорбция, ICP-OES, қалдық концентрациясы, празеодим

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## УЛУЧШЕННАЯ СОРБЦИЯ ИОНОВ ПРАЗЕОДИМА ИЗ НИТРАТНЫХ РАСТВОРОВ АКТИВИРОВАННЫМИ ИОНООБМЕННЫМИ СМОЛАМИ

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**Резюме.** Введение. В данном исследовании изучалась сорбция ионов празеодима такими активированными ионообменными смолами как КҮ-2-8 ( $H^+$  форма) и АВ-17-8 ( $OH^-$  форма), находящихся в интерполимерной системе "КҮ-2-8:АВ-17-8" с молярными соотношениями ионных групп равных X:Y (6:0, 5:1, 4:2, 3:3, 2:4, 1:5, и 0:6), далее погружаемых в растворы нитрата празеодима. Для пояснения, интерполимерная система представляет собой комбинацию двух смешанных полимеров с активными функциональными группами, находящимися в одном и том же водном растворе, но без прямого контакта. Цель нашего исследования - демонстрация эффекта активации ионообменных смол как функциональных полимеров путем их дистанционного взаимодействия в водной среде для улучшения сорбции широкого спектра целевых металлов (включая празеодим) и их соединений из растворов промышленных отходов. Методология работы заключалась в проведении взаимной активации ионообменников в водной среде для приведения их к конформационным и электрохимическим изменениям, с последующим переходом во высокоионизированное состояние и улучшенной сорбционной активностью по отношению к ионам целевого металла празеодима. Результаты и обсуждение. Экспериментальные результаты показали, что интерполимерная система КҮ-2-8:АВ-17-8 (5:1) продемонстрировала значительное увеличение сорбционной активности (43 %) по отношению к ионам празеодима, в то время как аналогичные значения для исходных КҮ-2-8 (6:0) и АВ-17-8 (0:6) составили 25 % и 5 %, соответственно. Заключение. Данное исследование проливает свет на новый и многообещающий подход к повышению эффективности ионообменных смол, открывая возможности для эффективного извлечения ценных металлов из сложных растворов. Кроме того, исследование имеет практическое значение для устойчивого управления отходами и вторичной переработки ресурсов, способствуя улучшению охраны окружающей среды и ресурсосбережения в различных отраслях промышленности. Дальнейшие исследования и оптимизация интерполимерных систем могут привнести существенный вклад в развитие более экологических и ресурсосберегающих сорбционных технологий.

**Ключевые слова:** ионообменные смолы, интерполимерная система, активация, нитратный раствор, процесс, технология, сорбция, ICP-OES, остаточная концентрация, празеодим

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