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INTERPOLYMER SYSTEMS OF POLY(METHACRYLIC ACID): POLY(4-VINYLPYRIDINE) FOR THE SELECTIVE SORPTION AND DESORPTION OF RHENIUM IONS

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Abstract. *Introduction.* Rhenium is a rare transition metal used in alloys and catalysis. Industrial recovery comes mainly from sulfuric acid effluents formed during copper and molybdenum processing. Sorption is the main method, but the choice of effective sorbents remains an issue. *Aims.* Interpolymer systems (IPS) of poly(methacrylic acid) (PMAA) and poly(4-vinylpyridine) (P4VP) were prepared and tested. IPS samples with different PMAA:P4VP ratios were prepared and tested. Sorption capacity, desorption behavior, and structural as well as thermal characteristics were investigated. *Results and Discussion.* The sorption efficiency varies with polymer composition. The 2:4 PMAA:P4VP ratio gave the highest uptake. Desorption in 2% nitric acid was moderate, with a maximum of about 75.6%. FTIR spectra showed shifts in carboxyl and pyridyl bands, confirming the role of functional groups. TGA/DSC data indicated stabilization of the polymer network after rhenium binding. *Conclusion.* The PMAA:P4VP system at a 2:4 ratio combined high sorption, moderate desorption, and improved structural stability. This system may be applied for rhenium recovery from metallurgical wastewater.

Keywords: interpolymer systems, rhenium extraction, poly(methacrylic acid) (PMAA), poly(4-vinylpyridine) (P4VP), ammonium perrhenate, selective sorption and desorption, polymer-based sorbents

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1. Introduction

Rhenium (Re) the rare transition metalswith a high melting point (3186 °C), mechanical strength, and resistance to extreme conditions. These properties make it important in superalloys for gas turbines, jet engines, and nuclear devices [1–9]. Rhenium also has multiple oxidation states (–1 to +7), which allows its compounds to be used as catalysts in petroleum refining and other processes [10–20].

Primary deposits of rhenium are very limited. The element occurs in trace amounts in copper and molybdenum sulfide ores, usually below 0.2 wt% [1–20]. During roasting, rhenium forms volatile Re_2O_7, which is absorbed from flue gases in sulfuric acid solutions. These acid solutions remain the main industrial source [22,23]. Secondary sources are also used, including spent Pt–Re catalysts, turbine components, and rhenium-containing alloy scrap [11, 12].

Recovery is mainly performed by solvent extraction or sorption. Sorption attracts interest because sorbents can be designed with selectivity toward perrhenate ions [13–16]. Various sorbents have been studied, such as activated carbons, layered double hydroxides, and metal–organic frameworks [17, 18].

Recently, interpolymer systems (IPS) have been tested as alternatives. They combine acidic and basic polymers in one system. Cooperative effects in these systems often result in higher sorption compared with single polymers. One mechanism is remote functional group interaction. Jumadilov et al. (2012) reported that when polyacidic and polybasic hydrogels contact through a shared aqueous phase, long-range electrochemical effects and conformational changes can activate or suppress functional groups. This leads to higher sorption capacity and system response [18–20].

This work investigates IPSs based on poly(methacrylic acid) (PMAA) and poly(4-vinylpyridine) (P4VP) for recovery of rhenium from ammonium perrhenate solutions. Considering the limited natural reserves of rhenium and its strategic importance, these systems can be considered as a sustainable approach for treating metallurgical effluents.

2. Materials and Methods

A total of eight PMAA/P4VP hydrogel–rhenium samples were prepared. Four contained rhenium in the form of perrhenate, and four contained hydroxide species. P4VP (Mw ≈ 160,000; Sigma-Aldrich) was used as the electron-donating polymer.

Hydrogels were obtained either by polymerization of water-soluble monomers or by crosslinking within a gel matrix. PMAA and P4VP gels were synthesized separately.

All reagents were of analytical grade: ammonium perrhenate (NH_4ReO_4, ≥99.9%), sodium hydroxide (≥98%), sulfuric acid, and nitric acid (>90%).

Thermal analysis was performed using TG 209 F3 Tarsus (NETZSCH) and TGA/SDTA 851e (Mettler Toledo). FTIR spectra were recorded on a JASCO FT/IR-6X spectrometer (4000–400 cm^{-1}, KBr). pH values were measured with a pH200EM REX meter. Masses were determined on a Shimadzu AX200 balance. Rhenium concentrations were analyzed by spectrophotometry and AAS-3C.

Model rhenium solutions (100 mg·L^{-1} as Re) were prepared by dissolving 28.8 mg of NH_4ReO_4 in distilled water and diluting to 200 mL. The solutions were kept in the dark to avoid photodegradation. Polymer samples were enclosed in woven semi-permeable bags and immersed for 0.5–48 h. PMAA:P4VP molar ratios ranged from 6:0 to 0:6. Aliquots of 5 mL were withdrawn at fixed intervals and analyzed for residual rhenium. Polymer amounts were calculated from the repeat-unit molar masses of PMAA (86 g·mol^{-1}) and P4VP (105 g·mol^{-1}), keeping the total number of units constant, equal to that in 0.030 g of PMAA at a 6:0 ratio.

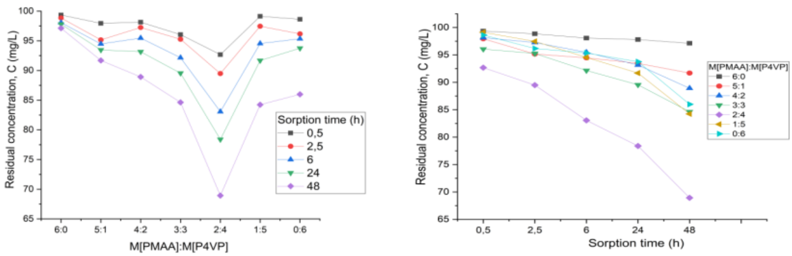
Desorption was performed using freshly prepared 2% HNO_3. After sorption, polymer samples were transferred into the acid solution and kept for 24–48 h. The desorption degree (R, %) was calculated from the residual rhenium concentration.

3. Results and discussion

Sorption behavior

The efficiency of rhenium uptake by the PMAA:P4VP systems depended on polymer ratio and contact time (Figure 1). In the initial period (0.5–2.5 h), residual concentrations of Re(VII) remained high for most system, showing the slow stage of binding. After 6 h, a marked decrease in concentration was observed, especially in systems with balanced polymer ratios.

The 2:4 PMAA:P4VP system was the most effective. After 48 h, the residual concentration decreased to 68.9 mg/L, which corresponded to the maximum sorption capacity. Systems with ratios 6:0 and 0:6 showed much lower efficiency. This confirmed that cooperative interaction of acidic and basic components was necessary for effective sorption.

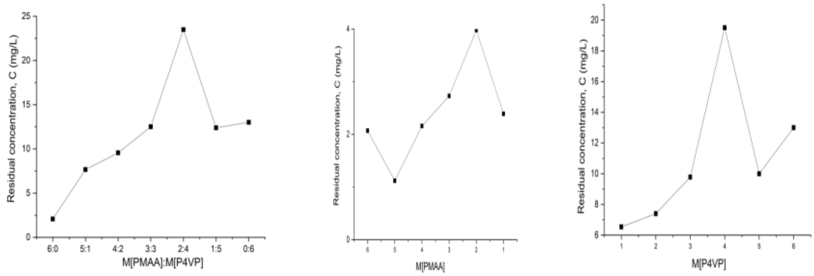


a b

Figure 1 - Dependence of Re(VII) concentration in solution (Ce, mg/L) on (a) the polymer ratio PMAA:P4VP and (b) contact time.

Desorption performance

Desorption with 2% HNO_3 showed clear differences depending on the polymer system (Figure 2). The 2:4 PMAA:P4VP system had the highest desorption degree (23.5 mg/L). Although binding was strong, a fraction of rhenium ions could be recovered under acidic conditions. Separate tests of the polymers showed that P4VP contributed more strongly than PMAA, in line with the donor character of its pyridine groups. The results confirm that P4VP is the key component in the sorption–desorption mechanism.

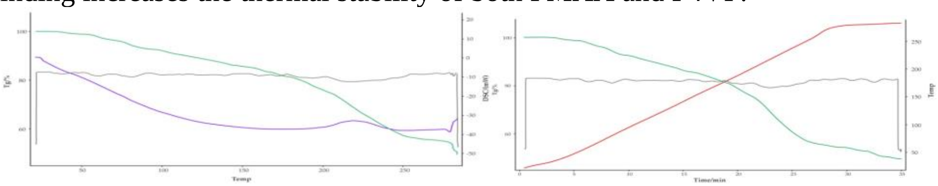


a b c

Figure 2 - Desorption behavior of rhenium ions (Cd, mg/L) as a function of (a) the PMAA:P4VP molar ratios, (b) the PMAA ion-exchange component, and (c) the P4VP ion-exchange component.

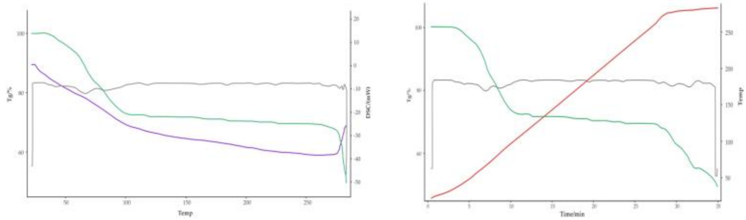
Thermal analysis (TGA/DSC)

Thermal analysis of pristine PMAA and P4VP showed characteristic stages of water loss and polymer degradation (Figure 3, Figure 4). After of rhenium sorption, both polymers demonstrated higher thermal stability in the 2:4 PMAA:P4VP system (Figure 5, Figure 6). This stabilization is attributed to stronger intermolecular interactions and coordination bonds between rhenium ions and polymer functional groups. These interactions restricted polymer chain mobility and shifted the onset of decomposition to higher temperatures. The observed changes in DSC and TGA/DTG thermograms confirm that rhenium binding increases the thermal stability of both PMAA and P4VP.



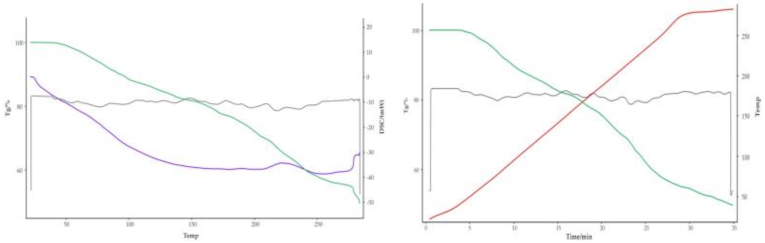
(a) (b)

Figure 3 - DSC/(a) and TGA/ (b) DTG thermogram of PMAA before rhenium sorption.



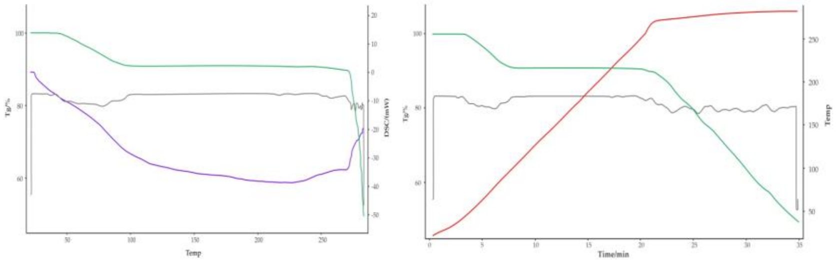
(a) (b)

Figure 4 - DSC/(a) and TGA/ (b) DTG thermogram of P4VP before rhenium sorption



(a) (b)

Figure 5 - DSC/(a) and TGA/ (b) DTG thermogram of PMAA after rhenium sorption in PMAA:P4VP (2:4 molar ratio).

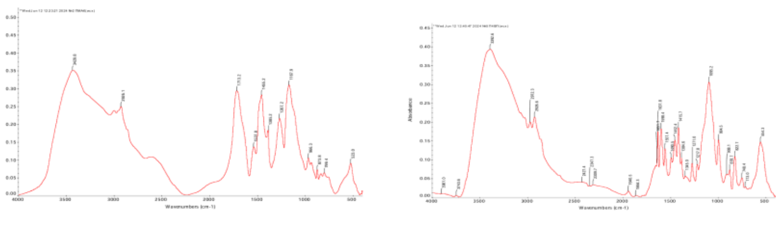


(a) (b)

Figure 6 - DSC/(a) and TGA/ (b) DTG thermogram of P4VP after rhenium sorption in PMAA:P4VP interpolymer system (2:4 molar ratio).

FTIR spectroscopy

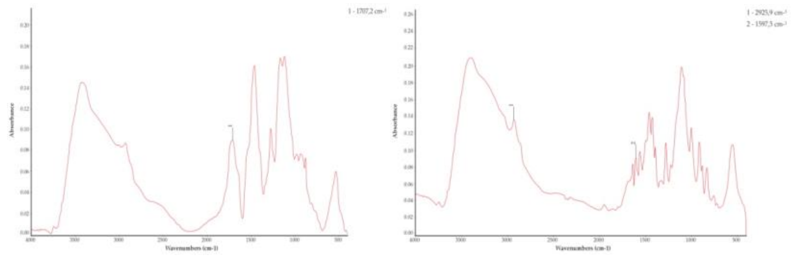
FTIR spectra confirmed the interaction of rhenium ions with functional groups of both polymers. In pristine PMAA, strong absorption bands were observed in the region of carboxyl group vibrations, while P4VP showed characteristic pyridine ring modes (Figure 7). After rhenium sorption, distinct spectral changes were detected (Figure 8). In PMAA, the C=O stretching band shifted, indicating coordination of the carboxyl groups with Re(VII). In P4VP, changes in the pyridine ring vibrations confirmed the donor–acceptor role of pyridyl groups. These observations demonstrate that both polymers take part in binding, with P4VP contributing more strongly due to its electron-donor character.



(a) (b)

Figure 7 - FTIR spectra of pristine PMAA (a) and pristine P4VP (b) in the interpolymer system 2:4 PMAA:P4VP.

The FTIR spectra of the polymer PMAA (a) and P4VP (b) after the sorption of rhenium ions from a model solution are represented in Fig. 8.



(a) (b)

Figure 8 - FTIR spectra of PMAA (a) and P4VP (b) after rhenium ion sorption in the interpolymer system 2:4 PMAA:P4VP.

General interpretation

The combined sorption, desorption, thermal, and spectroscopic results indicate a synergistic effect in PMAA:P4VP systems. The 2:4 ratio gave the best performance. At this composition, acidic and basic groups worked in balance, which increased sorption and improved structural stability. The results confirm that weakly cross-linked interpolymer complexes can act as selective sorbents for rhenium recovery.

4. Conclusion

Sorption and desorption of ReO_4^- in PMAA:P4VP interpolymer systems were investigated. Sorption efficiency was determined mainly by functional group interactions. The 2:4 PMAA:P4VP ratio showed the highest uptake capacity. Desorption was driven largely by the electron-donor contribution of P4VP. Weakly acidic–basic interpolymer systems are suitable as selective and reusable sorbents for rhenium recovery from aqueous solutions.

Spectroscopic (FTIR) and thermal (TGA/DSC) measurements showed that the interaction of carboxyl and pyridyl groups with Re(VII) ions promoted stabilization of the polymer matrix. The structural and thermal changes noted above confirm direct participation of both active elements and show that P4VP takes the front throne, because of electron-donor properties of its pyridine moieties. Thus, cooperating interpolymer effects play an important role in designing advanced sorbents.

From a real-world application perspective, the PMAA:P4VP system is a low-cost, reusable, and environmentally friendly sorbent for rhenium recovery from metallurgical effluents based on a high sorption capacity, moderate desorption and increasing material stability. For a broader application, future studies should confirm their efficiency in real wastewater, study long-lasting sorption–desorption cycles at plants, and apply this strategy to other important metals like molybdenum and tungsten, to expand sustainable polymer-based methods for rare-metal recovery.

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РЕНИЙ ИОНДАРЫН ТАҢДАМАЛЫ СОРБЦИЯ ЖӘНЕ ДЕСОРЦИЯЛАҒАНДАҒЫ ПОЛИ (4-ВИНИЛПИРИДИН):ПОЛИ (МЕТАКРИЛ ҚЫШҚЫЛЫ) ИНТЕРПОЛИМЕРЛІ ЖҮЙЕСІ

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Түйіндеме. *Кіріспе.* Рений – қорытпалар мен катализде қолданылатын сирек кездесетін өтпелі металл. Өнеркәсіпте ол негізінен мыс пен молибденді өңдеу кезінде пайда болатын күкірт қышқылының қалдықтарынан алынады. Негізгі әдіс ретінде сорбция болып табылады, бірақ тиімді сорбенттерді таңдау проблема болып қала береді. *Мақсаттар.* Поли (метакрил қышқылы) (PMAA) және поли (4-винилпиридин) (P4VP) интерполимерлік жүйелер (IPS) алынды және сыналды. PMAA:P4VP қатынасы әртүрлі ЖЗШ үлгілері алынды және сыналды. Сорбциялық қабілеті, десорбциялық қасиеттері, құрылымдық және термиялық сипаттамалары зерттелді. *Нәтижелер және талқылау.* Сорбцияның тиімділігі полимердің құрамына байланысты. PMAA:P4VP қатынасы 2:4 қатынасында ең жақсы сіңіруді қамтамасыз етеді. 2% азот қышқылындағы десорбция орташа, максимум шамамен 75,6% болды. ИҚ спектрлері функционалды топтардың рөлін растайтын карбоксил мен пиридил жолақтарында ығысулар көрсетті. TGA/DSC деректері рениймен байланысқаннан кейін полимерлі тордың тұрақтануын көрсетті. *Қорытынды.* PMAA:P4VP жүйесі 2:4 қатынасында жоғары сорбцияны, орташа десорбцияны және жақсартылған құрылымдық тұрақтылықты біріктіреді. Бұл жүйені металлургиялық кәсіпорындардың ағынды суларынан рений алу үшін қолдануға болады.

Түйін сөздер: интерполимерлік жүйелер, рений экстракциясы, поли (метакрил қышқылы) (pmaa), поли (4-винилпиридин) (P4VP), аммоний перренаты, селективті сорбция және десорбция, полимер негізіндегі сорбенттер

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ИНТЕРПОЛИМЕРНЫЕ СИСТЕМЫ ПОЛИ(МЕТАКРИЛОВАЯ КИСЛОТА): ПОЛИ (4-ВИНИЛПИРИДИН) ДЛЯ СЕЛЕКТИВНОЙ СОРБЦИИ И ДЕСОРБЦИИ ИОНОВ РЕНИЯ

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Резюме. *Вступление.* Рений - редкий переходный металл, используемый в сплавах и катализе. В промышленности его извлекают в основном из сернокислотных отходов, образующихся при переработке меди и молибдена. Основным методом является сорбция, но выбор эффективных сорбентов остается проблемой. *Цели.* Были получены и протестированы интерполимерные системы (ИПС) из поли (метакриловой кислоты) (PMAA) и поли (4-винилпиридина) (P4VP). Были получены и протестированы образцы IPS с различным соотношением PMAA:P4VP. Были исследованы сорбционная способность, десорбционные свойства, структурные и термические характеристики. *Результаты и обсуждение.* Эффективность сорбции зависит от состава полимера. Соотношение PMAA:P4VP в соотношении 2:4 обеспечивает наилучшее поглощение. Десорбция в 2%-ной азотной кислоте была умеренной, максимальная - около 75,6%. ИК-спектры показали сдвиги в карбоксильных и пиридильных полосах, подтверждающие роль функциональных групп. Данные TGA/DSC показали стабилизацию полимерной сетки после связывания с рением. *Вывод.* Система PMAA:P4VP в соотношении 2:4 сочетает в себе высокую сорбцию, умеренную десорбцию и улучшенную структурную стабильность. Эта система может применяться для извлечения рения из сточных вод металлургических предприятий.

Ключевые слова: интерполимерные системы, извлечение рения, поли (метакриловая кислота) (PMAA), поли (4-винилпиридин) (P4VP), перренат аммония, селективная сорбция и десорбция, сорбенты на основе полимеров

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