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Functionalized Chitosan-Modified PVDF-co-HFP Polymer Inclusion Membrane for Enhanced Au(III) Extraction

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ABSTRACT

Gold recovery from aqueous solutions requires environmentally sustainable alternatives to conventional cyanide-based extraction processes. This study aimed to develop the polymer inclusion membrane (PIM) by incorporating functionalized chitosan into a poly (vinylidene fluoride-co-hexafluoropropylene) (PVDF-co-HFP) membrane to enhance the transport and extraction efficiency of Au(III). The membrane consisted of PVDF-co-HFP as the base polymer, Aliquat-336 as the carrier, and dioctyl phthalate (DOP) as the plasticizer, while functionalized chitosan was incorporated at concentrations ranging from 0.5 to 2.5 wt.%. The fabricated membranes were prepared using the solvent-casting techniques and characterized using scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), contact angle, and ion exchange capacity (IEC). The membrane containing 2.0 wt.% functionalized chitosan (M5) exhibited the highest Au(III) extraction efficiency of 98.19%.The improved extraction performance was attributed to enhanced membrane hydrophilicity, increased ion exchange capacity, and more efficient carrier-mediated transport. These findings demonstrate that the developed PIM system is a promising and sustainable membrane-based technology for high-efficiency gold recovery.

Keywords: extraction, chitosan, membrane, PVDF-co-HFP, Aliquat-336.

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Introduction

Gold is one of the most valuable metals because of its wide range of applications in the jewellery, electronics, aerospace, medical, and financial industries. The high global demand for gold has increased the need for efficient methods to extract gold from low-grade ores and secondary resources. Cyanide leaching has been the most widely used hydrometallurgical process for gold extraction because of its high recovery efficiency. However, the use of cyanide raises environmental concerns because of its high toxicity and the risk of environmental contamination. These concerns have encouraged researchers to develop more sustainable and environmentally friendly alternatives for gold recovery [1].

Membrane-based separation technologies have emerged as a promising alternative because they offer high selectivity, lower chemical consumption, and reduced secondary generation [2]. Among these technologies, polymer inclusion membranes (PIMs) have gained attention for metal ion separation and recovery. A typical PIM consists of a base polymer, a carrier, and a plasticizer. The carrier selectively binds with the target metal ion and transports it across the membrane, while the polymer provides mechanical support and the plasticizer improves membrane flexibility and transport performance [3].

Compared with conventional solvent extraction, PIMs combine extraction and stripping into a single membrane system. This reduces solvent loss and simplifies the overall separation process. Previous studies have successfully applied PIMs for the recovery of various metal ions, including Pb(II), Cd(II), Zn(II), Cr(VI), As(V), and Au(III). The results showed that PIMs have potential as an environmentally friendly technology for hydrometallurgical applications [2].

The selection of membrane materials is important because it directly affects the membrane

performance during metal ion transport. Poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-co-HFP) is commonly used as the base polymer due to its good chemical resistance, thermal stability, and mechanical strength. It is also compatible with hydrophobic carriers such as Aliquat-336. Aliquat-336, a quaternary ammonium salt, has been widely used as a carrier for Au(III) extraction because of its strong affinity for anionic metal complexes and good transport performance [4]. Dioctyl phthalate (DOP) is added as a plasticizer to improve membrane flexibility and enhance carrier mobility within the polymer matrix [5].

Chitosan is a natural biopolymer derived from chitin and has been widely studied for membrane application because of its biodegradability, biocompatibility, and the presence of hydroxyl and amino groups. These functional groups provide active sites for metal ion binding and help improve membrane hydrophilicity. However, the use of native chitosan is limited because of its poor stability in acidic media and relatively weak mechanical properties [4]. To overcome these limitations, chitosan can be functionalized to improve its stability and introduce additional active sites. Compared with native chitosan, functionalized chitosan has better compatibility with the polymer matrix and can enhance ion exchange capacity and metal transport. For these reasons, functionalized chitosan has become a promising additive for PIMs used in metal separation.

In this study, functionalized chitosan was incorporated into a PVDF-co-HFP-based membrane that consisted of Aliquat-336 as the carrier and DOP as the plasticizer for Au(III) extraction. The membrane was prepared using the solvent-casting method and characterized using Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), thermogravimetric analysis (TGA), contact angle measurement, and ion exchange capacity (IEC). The effects of functionalized chitosan concentration on the PIMs' properties and Au(III) extraction performance were then evaluated.

Experimental part

Materials

Poly(vinylidenefluoride-co-hexafluoropropylene) (PVDF-co-HFP) pellets (Sigma-Aldrich, USA) were used as the base polymer. Aliquat-336 (tricaprylmethylammonium chloride, Sigma-Aldrich, USA) was employed as the ionic carrier. Dioctyl phthalate (DOP, purity > 99.5%, Sigma-Aldrich, USA) served as the plasticizer. Chitosan (Sigma-Aldrich, USA) was used as the membrane additive and was functionalized via the ionic gelation method.

Functionalized Chitosan Preparation

0.3 g chitosan was dissolved in 1 wt.% dilute acetic acid solution under continuous magnetic stirring until a clear and homogeneous solution was obtained. Magnesium sulphate was then added dropwise as an ionic crosslinking agent to induce the functionalization process through ionic gelation. Upon completion of the reaction, the resulting functionalized chitosan was separated by centrifugation (6000 rpm, 50 mins) and repeatedly washed with distilled water to remove residual reactants. The purified product was subsequently dried in a laboratory oven at 50 °C until a constant weight was achieved [6].

Membrane Fabrication

50 wt.% of PVDF-co-HFP was dissolved in THF under continuous magnetic stirring until a clear and homogeneous polymer solution was obtained. Subsequently, the specific amount of DOP and Aliquat-336 was added according to the membrane compositions listed in Table 1 below. Functionalized chitosan was then incorporated into the casting solution at 0.5-2.5 wt.% and stirred continuously to ensure its uniform dispersion throughout the polymer matrix.

The prepared casting solution was uniformly cast onto a clean glass plate using a casting knife with a controlled thickness of 200 µm. Solvent evaporation was carried out at ambient conditions for 24 h, resulting in the formation of self-supporting membranes. The dried membranes were carefully peeled from the casting plate, cut into circular discs, and stored in a desiccator before physicochemical characterization and Au(III) transport experiments [4].

Membrane Characterization

The fabricated PIMs were characterized using various microscopic methods. The surface morphology of the fabricated PIMs was characterized using a JEOL JSM-IT100 scanning electron microscope (SEM, JEOL Ltd., Japan). The fabricated PIMs were cut into 5 x 5 mm sections and sputter-coated with Au-Pt under vacuum conditions. SEM micrographs were obtained at an accelerating voltage of 20.0 kV [7].

Table 1 – Membrane formulation with different functionalized chitosan concentrations

Acronyms	PVDF-co-HFP (wt.%)	Aliquat-336 (wt.%)	DOP (wt.%)	Functionalized Chitosan (wt.%)
M1	50	20	10	0.0
M2	50	20	10	0.5
M3	50	20	10	1.0
M4	50	20	10	1.5
M5	50	20	10	2.0
M6	50	20	10	2.5

The functional groups of the fabricated PIMs were characterized using FTIR-ATR (Thermo Scientific Nicolet iZ10, Thermo Fisher Scientific, USA) over the spectral range of 4000-400 cm^{-1} [8].

The thermal stability was examined using a TGA/DSC 2 HT thermogravimetric analyzer (METTLER TOLEDO, Switzerland). Approximately 5-10 mg of each membrane sample was heated from room temperature to 800 ℃ under a nitrogen atmosphre at a heating rate of 10 ℃ min^{-1} [9].

The membrane hydrophilicity was determined using a DataPhysics OCA15plus contact angle goniometer (DataPhysics Instruments GmbH, Germany). A 5 µL droplet of deionized water was deposited onto the membrane surface, and the contact angle was measured in triplicate [10].

2.5 Rate of Performance

The membrane that showed the best overall properties was selected for Au(III) extraction experiments. A standard Au(III) (Sigma-Aldrich, USA) feed solution was prepared by diluting a 1000 mg/L solution with 0.1 M hydrochloric acid (HCl, Mer, Germany) to obtain the 50 mg L^{-1}. The receiving solution was prepared by dissolving 1 g of thiourea (Thermo Fisher Scientific, USA) in 1% HCl to produce a 1.0 M thiourea solution.

The transport performance of the fabricated PIMs was evaluated using H-cell compartments. The fabricated PIMs were positioned between the feed and receiving compartments, each containing 100 mL of solution. Continuous stirring at 400 rpm was maintained throughout the experiment. Samples were collected at 4 h, 8 h, 12 h, 16 h, 20 h, and 24 h, and the Au(III) concentration was analyzed using inductively coupled plasma optical emission spectrometry (ICP-OES, Agilent Technologies Inc., USA).

Results and Discussion

Figure 1 shows the FTIR spectra of the fabricated PIMs. The spectra confirmed the successful incorporation of functionalized chitosan into the fabricated PIMs. An absorption band at 3200-3500 cm^{-1} was observed. This band corresponds to the stretching vibration of hydroxyl (-OH) and amino (-NH_2) groups [11]. Also, the additional peaks at 1550-1650 cm^{-1} were assigned to the amide and amino groups of chitosan [12]. The increase in intensity at 1100-1130 cm^{-1} were attribute to the asymmetric stretching vibration of sulphate groups, indicating the presence of magnesium sulphate in the membrane [13].

No additional absorption peaks were observed in the functionalized chitosan matrix membrane (M2-M6) compared with the pure PIMs (M1). This indicates that no new chemical bonds were formed during the preparation process [14]. Instead, the small shifts in peak position and changes in intensity. This is due to the membrane interacting mainly through hydrogen bonding [11]. These interactions help improve membrane properties while maintaining the chemical stability of PVDF-co-HFP and Aliquat-336 [15].

Figure 2 shows the SEM images of the fabricated PIMs, illustrating the effect of functionalized chitosan on the membrane surface morphology. M1 has a smooth and compact surface with a dense polymer matrix.

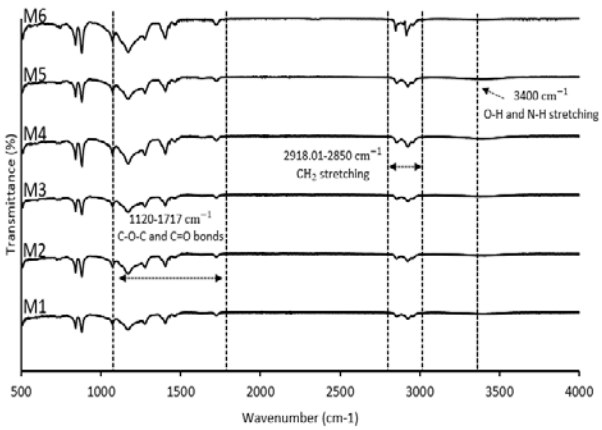

Figure 1 – FTIR spectra of PVDF-co-HFP-based PIMs with different functionalized chitosan (M1-M6)

After the addition of functionalized chitosan, the membrane surface became slightly rougher while maintaining a uniform structure. No obvious cracks, pinholes, or large particle agglomeration were observed, suggesting the functionalized chitosan was well distributed throughout the PVDF-co-HFP matrix. During solvent evaporation, the interaction between PVDF-co-HFP and functionalized chitosan promoted the formation of microstructural irregularities that increased the effective surface area available for ion exchange [16]. A uniform membrane structure was beneficial because it helps reduce transport resistance and provides continuous pathways for ion movement [4].

Among the fabricated membranes, M5 showed the most suitable surface morphology, which could facilitate the carrier-mediated transport of Au(III) [17].

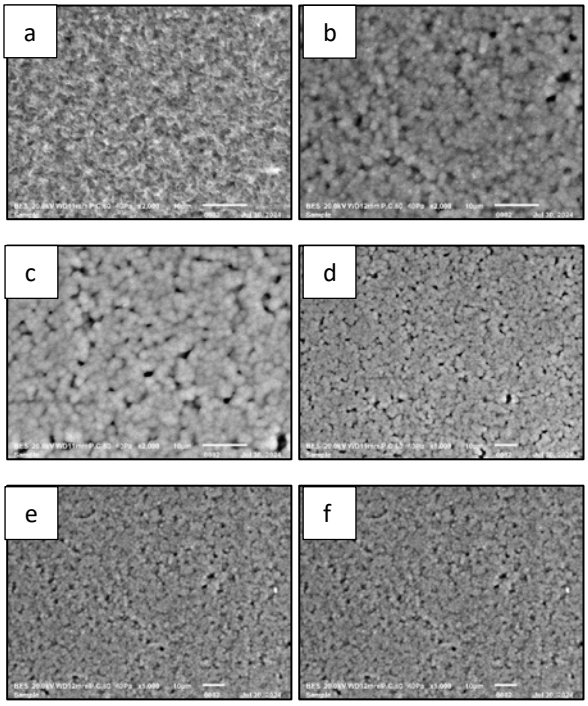

Figure 2 – SEM images of the surface morphology of PVDF-co-HFP-based PIMs with different functionalized chitosan: (a) M1, (b) M2, (c) M3, (d) M4, (e) M5, and (f) M6

Figure 3 shows the TGA curves of the fabricated PIMs. All membranes exhibited a similar thermal degradation pattern. The initial weight loss below approximately 160-200 °C was relatively small and can be attributed to the evaporation of residual moisture and solvent trapped within the membrane during fabrication [13]. This indicates that most of the solvent had been successfully removed during the membrane preparation process.

The second degradation stage was observed at around 250 °C, which is mainly associated with functionalized chitosan and DOP. Membranes with higher functionalized chitosan showed slightly greater weight loss. This is due to the thermal decomposition of the hydroxyl and amino groups [14]. However, all membrane maintained at high degradation temperatures. This means that the functionalized chitosan did not significantly affect the thermal stability of the membrane.

The largest weight loss occurred at approximately 450 °C. This corresponds to the degradation of the PVDF-co-HFP polymer backbone [18]. Similar degradation profiles were observed for all membrane formulations. This is due to the membrane structure remaining stable after the addition of the functionalized chitosan [19].

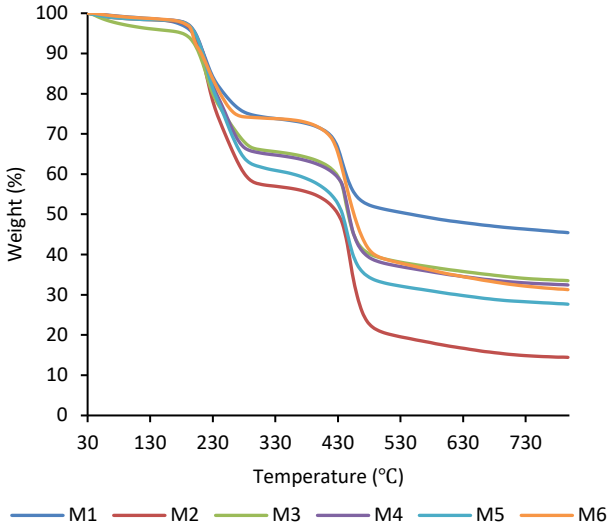

Figure 3 – Thermogravimetric analysis curves of PVDF-co-HFP-based PIMs with different functionalized chitosan (M1-M6), showing the thermal behaviour of the fabricated PIMs

Figure 4 shows the contact angle of fabricated PIMs. M1 exhibited the highest contact angle value of 57.41˚. This behaviour was attributed to the hydrophobicity of the fluorinated polymer backbone, where the presence of C-F bonds in the PVDF-co-HFP-based limits surface interaction with water molecules [4].

The contact angle values decreased with the increasingly functionalized chitosan concentration. This enhancement in the membrane surface hydrophilicity is due to the introduction of the hydroxyl and amino groups. These polar groups promote stronger interaction with water through hydrogen bonding. This can affect the affinity of the membrane surface [4].

The reduction in contact angle confirmed that functionalized chitosan successfully modified the membrane surface by increasing the surface free energy and wettability [20]. Enhanced surface wettability facilitates the adsorption and diffusion of water molecules at the membrane-solution interface. This can contribute to improved hydration of the membrane surface. As a result, the interfacial mass transfer resistance between the feed phase and the interfacial membrane surface was reduced. This can allow a greater accessibility of Au(III) to Aliquat-336. Therefore, the modified PIMs were expected to promote more efficient Au(III) transport during the extraction process.

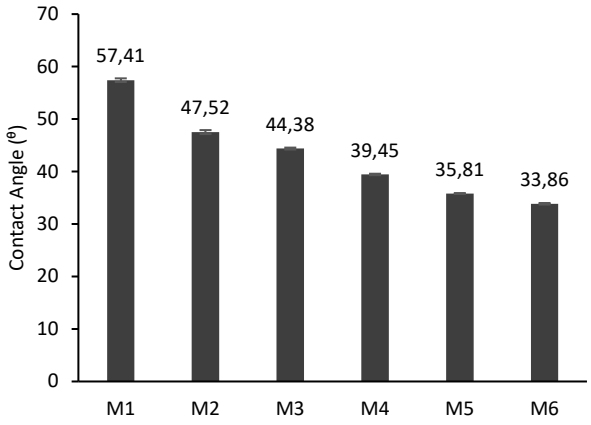

Figure 4 – Contact angle of PVDF-co-HFP-based PIMs with different functionalized chitosan (M1-M6), showing changes in surface hydrophilicity

Figure 5 shows the ion exchange capacity of fabricated PIMs. M1 exhibited the lowest IEC because PVDF-co-HFP is hydrophobic and contains limited availability of ionizable functional groups within the structure.

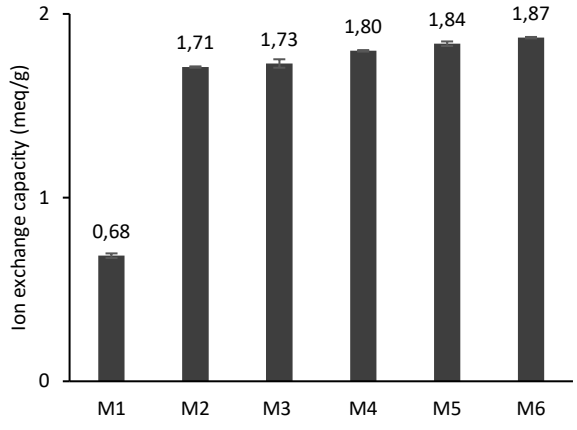

Figure 5 - Ion exchange capacity of PVDF-co-HFP-based PIMs with different functionalized chitosan

Following the addition of functionalized chitosan, the IEC increased because of the amino and hydroxyl groups that provide additional active sites for ion interaction and facilitate ion exchange for Au(III) transport [4]. The increased IEC is due to the higher density of available sites, which can enhance the movement of Au(III) complexes within the membrane [4].

Rate of Performance

Figure 6 shows the Au(III) extraction efficiency of fabricated PIMs with different concentrations of functionalized chitosan. The extraction efficiency increased with the increasing functionalized chitosan up to 2.0 wt.%, achieving a maximum extraction of 98.19%. This is due to the enhanced membrane hydrophilicity and higher ion exchange capacity by the additional hydroxyl and amino groups. This can increase the membrane's affinity and surface wettability, and provide more active sites for ion interaction. Therefore, it can promote the transport of Au(III).

Au(III) diffused toward the membrane surface under the concentration gradient between the feed and receiving phase. As demonstrated by SEM images, the addition of functionalized chitosan produced a more homogeneous membrane with increased surface roughness. It provides a larger effective contact area between the membrane and the solution. At the membrane interface, Au(III) was selectively extracted into the membrane via an ion-exchange reaction with quaternary ammonium groups of Aliquat-336 [21].

Upon reaching the receiving solution interface, Au(III) encounters thiourea in the receiving phase, where Au(III) preferentially forms a stable Au-thiourea complex. The receiving reaction releases Au(III) into the receiving phase while simultaneously regenerating Aliquat-336, allowing the carrier to diffuse back toward the feed phase and participate in transport cycles [22].

However, when the functionalized chitosan concentration was increased to 2.5 wt.%, the extraction decreased slightly. This is because the excessive functionalized chitosan affected the memrbane structure by increasing the density of the polymer matrix and restricting carrier mobility. It may reduce the free volume available for carrier diffusion, increase membrane viscosity during casting, and promote stronger intermolecular interactions that restrict carrier mobility. Consequently, the diffusion of Au(III)-complexes through the membrane becomes slower. Therefore, M5 was selected for subsequent optimization experiments.

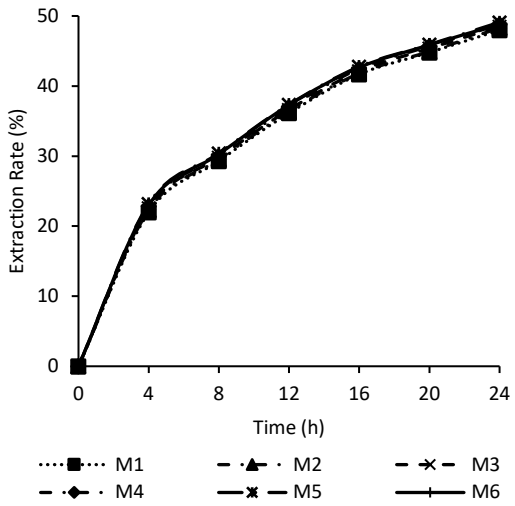

Figure 6 – Receiving phase Au(III) concentration over time of fabricated PIMs

Conclusions

Functionalized chitosan was successfully incorporated into PVDF-co-HFP to enhance Au(III) extraction. Membrane characterization demonstrated that the addition of functionalized chitosan improved membrane morphology, increased surface hydrophilicity, and enhanced ion exchange capacity. These physicochemical improvements facilitated carrier-mediated transport and reduced mass transfer resistance.

Among the fabricated membranes, M5 (2.0 wt.% functionalized chitosan) achieved 98.19% Au(III) extraction. However, further increasing the functionalized chitosan resulted in a decline in extraction percentages. It is indicated that the excessive loading of functionalized chitosan can increase the membrane hydration and diffusion resistance, thereby limiting the Au(III) transport.

Overall, functionalized chitosan is effective for improving the transport and extraction of Au(III) in PVDF-co-HFP. The membrane offers a promising and environmentally friendly alternative that can contribute to the advancement of sustainable separation technologies for metals.

Conflicts of interest. If you agree, you should not delete this statement: On behalf of all authors, the corresponding author states that there is no conflict of interest.

CRediT author statement: **N.Z. Nor'Azmi:** Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing – original draft, Writing – review & editing, Visualization, Investigation; **N.F. Shoparwe:** Supervision, Writing – Review & Editing.

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Функционалдандырылған хитозан-модификацияланған PVDF-co-HFP полимерлі инклюзивті мембрана Au(III) экстракциясын күшейту үшін

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Мақала келді: *19 шілде 2026*
Сараптамадан өтті: *25 шілде 2026*
Қабылданды: *5 тамыз 2026*

ТҮЙІНДЕМЕ

Сулы ерітінділерден алтынды алу дәстүрлі цианид негізіндегі экстракция процестеріне экологиялық тұрғыдан тұрақты баламаларды қажет етеді. Бұл зерттеудің мақсаты Au(III) тасымалдау және экстракция тиімділігін арттыру үшін поли (винилиденфторид пен-гексафторпропилен) (PVDF-пен-HFP) мембранасына функционалданған хитозанды қосу арқылы полимерлік мембрананы (PIM) әзірлеу болды. Мембрана негізгі полимер ретінде PVDF-ко-HFP, тасымалдаушы ретінде Aliquat-336 және пластификатор ретінде диоктилфталат (DOP), сондай-ақ 0,5-тен 2,5 салмақтық % -ға дейінгі концентрацияда функционалдандырылған хитозан қосылды. Дайындалған мембраналар ерітінді құю әдістерін қолдана отырып дайындалды және сканерлеуші электронды микроскопия (SEM), Фурье түрлендіруінің инфрақызыл спектроскопия (FTIR), термогравиметриялық талдау (TGA), жанасу бұрышын өлшеу және ион алмасу сыйымдылығы (IEC) арқылы сипатталды.

	Құрамында 2,0% салмақтық% функционалдандырылған хитозан (M5) бар мембрана Au(III) экстракциясының ең жоғары тиімділігін көрсетті, ол 98,19% құрады. Экстракция өнімділігінің жақсаруы мембрананың гидрофильділігінің жоғарылауына, ион алмасу қабілетінің артуына және тасымалдаушы арқылы тасымалдаудың тиімдірек болуына байланысты болды. Бұл зерттеулер әзірленген PIM жүйесінің жоғары тиімді алтын алу үшін перспективалы және тұрақты мембраналық технология екенін көрсетеді.
	Түйін сөздер: экстракция, хитозан, мембрана, pvdf-co-hfp, aliquat-336.
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Функционализированная хитозан-модифицированная полимерная мембрана на основе PVDF-co-HFP для повышения эффективности извлечения Au(III)

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АННОТАЦИЯ

Для извлечения золота из водных растворов необходимы экологически устойчивые альтернативы традиционным процессам экстракции на основе цианида. Целью данного исследования было разработать полимерную мембрану с включениями (PIM) путем включения функционализированного хитозана в мембрану из поли(винилиденфторида-со-гексафторпропилена) (PVDF-co-HFP) для повышения эффективности переноса и экстракции Au(III). Мембрана состояла из PVDF-co-HFP в качестве базового полимера, Aliquat-336 в качестве носителя и диоктилфталата (DOP) в качестве пластификатора, при этом функционализированный хитозан был включен в концентрациях от 0,5 до 2,5 мас.%. Изготовленные мембраны были получены методом литья из раствора и охарактеризованы с помощью сканирующей электронной микроскопии (SEM), инфракрасной спектроскопии с преобразованием Фурье (FTIR), термогравиметрического анализа (TGA), измерения краевого угла смачивания и ионообменной емкости (IEC). Мембрана, содержащая 2,0 мас.% функционализированного хитозана (M5), продемонстрировала наивысшую эффективность извлечения Au(III) — 98,19%. Улучшение эффективности извлечения объясняется повышенной гидрофильностью мембраны, увеличенной ионообменной емкостью и более эффективным транспортом с помощью носителя. Эти результаты показывают, что разработанная система PIM является перспективной и устойчивой мембранной технологией для высокоэффективного извлечения золота.

Ключевые слова: экстракция, хитозан, мембрана, PVDF-CO-HFP, аликвата-336.

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