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REMOVAL OF CADMIUM FROM AQUEOUS SOLUTIONS VIA EMULSION LIQUID MEMBRANE PROCESS: BATCH EXPERIMENTAL INVESTIGATIONS

Abstract: The objective of this study was to evaluate the efficiency of the emulsion liquid membrane (ELM) technique for cadmium removal from aqueous solutions through systematic assessment of key operating parameters. Kerosene was used as the diluent, sorbitan monooleate (Span 80) as the surfactant, Di-(2-ethylhexyl) phosphoric acid (D₂EHPA) as the carrier, and hydrochloric acid (HCl) as the stripping agent. The effects of surfactant concentration, agitation speed, internal to membrane phase ratio, emulsion to feed ratio, and HCl concentration in the internal phase were investigated. The best conditions yielded a maximum cadmium removal efficiency (η_{Ca}) of 97.4 % at 4 % surfactant concentration, 450 rpm agitation speed, 1 : 4 internal to membrane ratio, 1 : 3 emulsion to feed ratio, and 0.25 M HCl. The results of cadmium removal follow the second-order kinetic model. The results demonstrate that the developed ELM system is an effective and rapid method for cadmium removal, with strong potential for application in industrial wastewater treatment and heavy metal remediation.

Keywords: cadmium, emulsion liquid membrane, surfactant, emulsification

Introduction

Cadmium is one of the most often occurring inorganic water contaminants [1]. It primarily originates from industrial activities such as mining, metal smelting, battery manufacturing, phosphate fertiliser use, and production of plastics and pigments [2]. Classified as a Group 1 carcinogen by the International Agency for Research on Cancer (IARC), cadmium is highly toxic even at trace concentrations. It bioaccumulates primarily in the kidneys and bones, causing nephrotoxicity and skeletal damage, including bone demineralisation. Additionally, cadmium's persistence in aquatic environments poses long-term ecological threats and its interactions with water treatment processes may generate secondary carcinogenic byproducts, compounding the risks to human health and ecosystems [3].

Although several conventional methods such as precipitation, adsorption, ion exchange, and membrane filtration have been employed for cadmium removal from wastewater, many of these approaches suffer from significant limitations [4]. These include high operational costs, generation of secondary waste, limited efficiency at low metal concentrations, and poor selectivity in the presence of competing ions [5-7]. Moreover, some of the extractants or carriers used in advanced methods such as supported liquid

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membranes or solvent extraction exhibit toxicity or instability under environmental conditions. Therefore, there is a continuous need to explore and optimise more efficient, selective, and environmentally friendly systems such as the ELM using safer and more stable material combinations [8].

Compared to conventional approaches like adsorption, liquid-liquid extraction, and distillation, membrane separation technologies have emerged as more effective alternatives due to their lower energy demands and higher efficiency [9]. Emulsion liquid membranes (ELMs) have garnered particular attention for their capacity to simultaneously extract and recover contaminants in a single integrated process. ELM represents a significant breakthrough in purification technology by facilitating enhanced separation effectiveness by minimising the diffusion path between the phases [10-12]. ELMs offer unique advantages such as unsteady-state mass transfer, high selectivity, increased flux rates, reusability, and minimal energy requirements [13]. These systems utilise selectively permeable materials that facilitate the targeted transport of solutes, making them suitable for cadmium removal from wastewater. Furthermore, ELM offers substantial operational advantages. It enhances the extraction and stripping processes by enabling continuous and flexible operation, reducing membrane thickness, increasing the available surface area, and improving solute transfer rates [14].

Liquid membranes typically comprise three distinct regions: the external aqueous feed phase, the organic membrane phase, and the internal stripping phase. In ELMs, the double emulsion structure maximises the interfacial area for mass transfer, enhancing the efficiency of extraction and separation processes [15]. ELMs are prepared by first forming a primary emulsion of the internal phase dispersed in the membrane phase using a surfactant, followed by dispersing this emulsion in the external aqueous phase [16]. The membrane phase acts as a selective barrier allowing mass transfer between the two aqueous phases [17].

Although ELMs have shown promise for heavy metal removal, limited studies have focused on the specific combination of Span 80 as the surfactant, D₂EDPA as the carrier, HCl as the internal stripping agent, and kerosene as the diluent in cadmium recovery applications. This study addresses that gap by evaluating the performance and stability of this unique formulation. The novelty lies in enhancing the performance of this underexplored ELM system to achieve efficient cadmium removal from aqueous solutions. The novelty lies in enhancing the performance of this underexplored ELM system to achieve efficient cadmium removal from aqueous solution. The main objective of this study is to achieve a high cadmium removal rate in a short time while maintaining the stability of the emulsion liquid membrane (ELM) structure. The process aims to be both cost-effective and environmentally friendly. Key operational parameters were systematically investigated, including surfactant concentration, agitation speed, internal to membrane phase ratio, emulsion to feed phase ratio, and HCl concentration in the internal phase, to identify the conditions that maximise η_{Cd} .

Experimental work

The ELM process can be broken down into three key steps: emulsification (creating a stable emulsion), extraction (transfer of Cd(II) into the membrane), and settling as shown in Figure 1 [18].

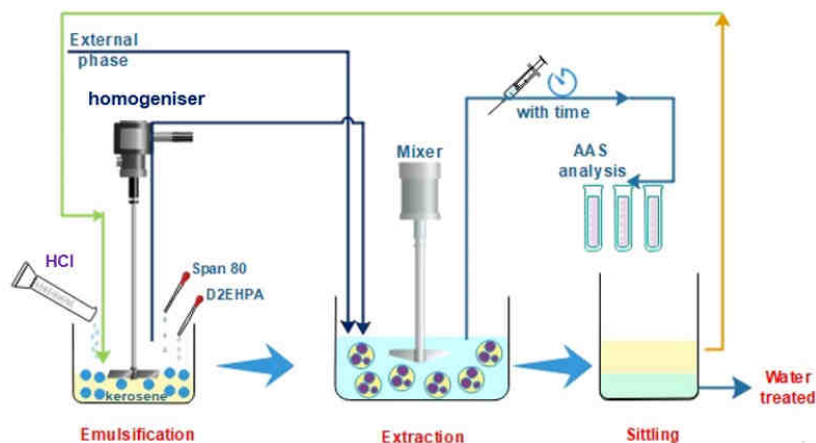


Fig.1. Schematic representation of cadmium removal from wastewater via ELM

Emulsion preparation

For this project, we purchased purified kerosene with a boiling point between 175 °C and 325 °C from Al-Dora Refinery, high-purity, refined D₂EHPA from Sigma Chemicals, Span 80.

1. HCl, and cadmium from Thomas Baker. HCl solutions with concentrations of 0.1 M, 0.25 M, and 1 M were prepared by dilution of concentrated HCl (37 %, approximately 12 M), using the dilution equation.
2. To create the necessary 100 ppm for cadmium, the proper weight of cadmium 0.195 g, was added to distilled water (1 liter).
3. In order to create a water-in-oil emulsion, the internal stripping phase (HCl) was added to a mixture of kerosene and surfactant that contained (2 % to 5 %) v/v Span 80 and a carrier that contained 5 % v/v D₂EHPA. The mixture was intensely mixed using a high-speed homogeniser Ultra Turrax IKA-T45 (Germany) set to 10,000 rpm for 10 minutes in order to produce a thin layer of a milky white liquid, as shown in Figure 2.

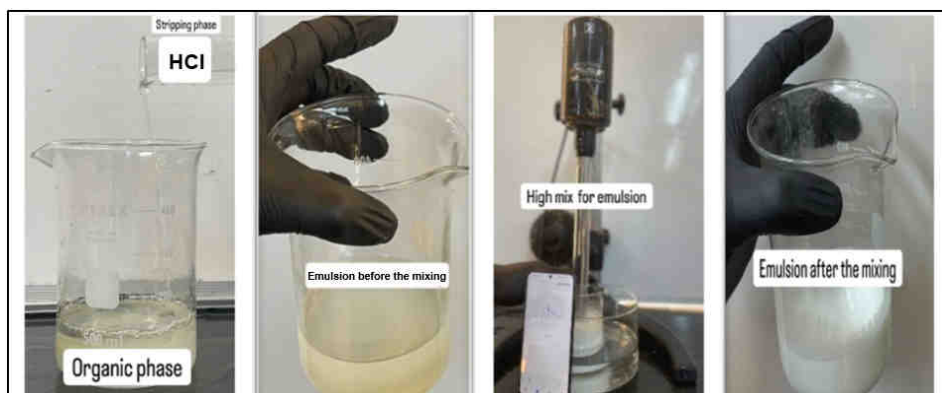


Fig. 2. Preparation steps of ELM

This thin layer should be freshly prepared before the extraction process. Different ratios ($V_I : V_M$) of the internal stripping phase to membrane phase (kerosene) of (1 : 2, 1 : 4, 1 : 6) were prepared.

Extraction experiments

By using an OS20-S Overhead LED Digital stirrer (CHINA), the water phase and the emulsion were mixed with the cadmium feed phase that contained initial concentrations of 100 ppm in a ratio of milky white liquid to external (feed) phase ($V_E : V_F$) of (1 : 3, 1 : 5, 1 : 7) by volume. The stirrer rotated at 250 rpm, 350 rpm, 450 rpm, 650 rpm. A 5 mL nylon syringe was used to draw samples from the disturbed solution at different intervals. (A circular, 125 mm Ø, medium speed, Type 001) filter paper from Thomas Baker was then used to filter the samples.

Optimisation approach and parameter selection

In this study, the optimisation was carried out by investigating one parameter at a time while keeping the other parameters constant, with the objective of achieving the highest η_{Cd} . The parameters selected for evaluation were identified based on their significant influence on the ELM process and their critical role in determining removal efficiency. The selection of parameter levels for testing was guided by preliminary experimental observations, where conditions producing lower efficiencies were replaced with those showing better performance and stability. The chosen parameters and their levels were specifically targeted to achieve the best experimental emulsion stability, minimise internal phase leakage, and maximise η_{Cd} under the conditions of the present work. Although a one-factor-at-a-time (OFAT) approach was employed due to time and resource constraints, the selection of parameter levels was guided by preliminary experiments and literature data to ensure meaningful and comparable results.

Analysis and calculations

The Atomic Absorption Spectrophotometer (AAS) SHIMADZU AA-7000 (Japan) shows that the concentration of cadmium in the external aqueous feed phase diminishes throughout time during the ELM extraction at the wavelengths of 228.85 nm for cadmium. η_{Cd} was then determined:

$$\eta_{Cd} = \frac{C_0 - C_f}{C_0} \cdot 100 \quad [\%] \quad (1)$$

where C_0 represents the initial concentration of cadmium in the external phase, C_f represents the final concentration of cadmium in the sample after extraction.

Settling

To separate the emulsion from the feed phase post-extraction, the mixture is divided using a separating funnel after agitation. The feed phase, being denser than the emulsion phase, accumulated below. After the phase separation, the aqueous phase was meticulously extracted from the membrane layer, resulting in the formation of two distinct layers (the treated water and the emulsion), as shown in Figure 3.

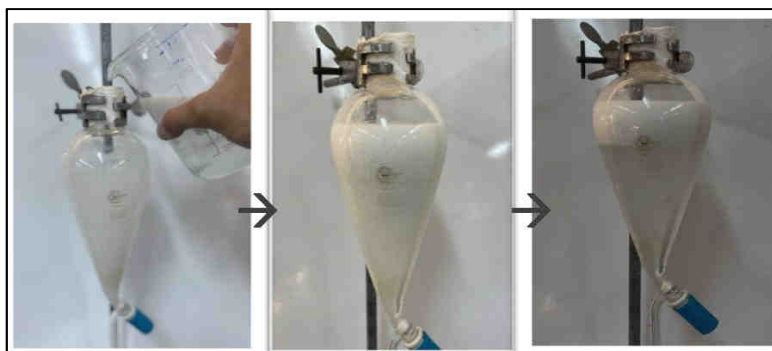


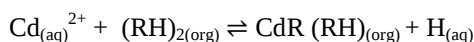
Fig. 3. The emulsion and cadmium solution separate into two distinct layers

Mechanism of ELM

Extraction, diffusion, and stripping constitute a sequence of coupled actions in the transport mechanism of Cd^{2+} in the ELM system. This work uses a water-in-oil (W/O) emulsion where the internal aqueous phase (0.25 M HCl) is emulsified inside an organic membrane phase comprising kerosene, 5 % v/v D_2EHPA as carrier, and 4 % v/v Span 80 as surfactant [2].

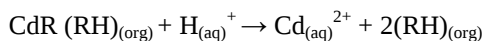
The mechanism involves the following stages [19]:

1. Extraction: from the external aqueous feed solution, cadmium ions $\text{Cd}(\text{II})$ diffuse to the membrane interface and react with D_2EHPA to generate a neutral organometallic complex:



This step takes place at the external membrane interface, where D_2EHPA efficiently extracts cadmium.

2. Diffusion through the Membrane Phase: The complex $\text{CdR}(\text{RH})$ traverses the organic membrane towards the internal interface, propelled by the concentration gradient [20].
3. Stripping: Upon arriving at the membrane-internal interface, the complex interacts with hydrogen ions from the HCl internal phase, liberating free Cd^{2+} into the internal aqueous solution:



The D_2EHPA is regenerated and returns to the extraction interface for reuse.

The transport mechanism of metal ions from the external (feed) phase to the internal phase through the membrane for actual wastewater, as shown in Figure 4.

The adsorption of Cd^{2+} onto the ELM is primarily driven by the strong affinity between the phosphoric acid groups of D_2EHPA and the divalent $\text{Cd}(\text{II})$, forming stable organometallic complexes. Electrostatic attraction and ion-exchange interactions further enhance the uptake of Cd^{2+} at the membrane interface before diffusion into the internal phase. These interactions are consistent with adsorption mechanisms reported in previous studies [18].

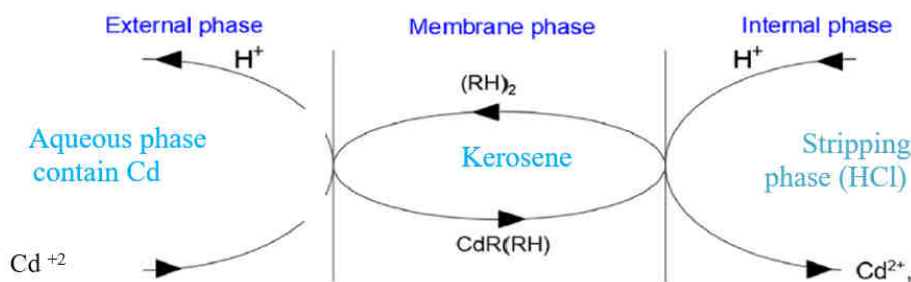


Fig. 4. Schematic representation of the transport mechanism in ELM

Result and discussion

Effect of surfactant concentration

Emulsion stability and integration into the emulsion-based liquid-liquid (ELM) system strongly depend on the surfactant (Span 80). In this work, different concentrations of Span 80 (2 %, 3 %, 4 %, and 5 %) v/v were investigated. The best surfactant concentration was found to be 4 % v/v, achieving a η_{Cd} of 92.6 %, as shown in Figure 5.

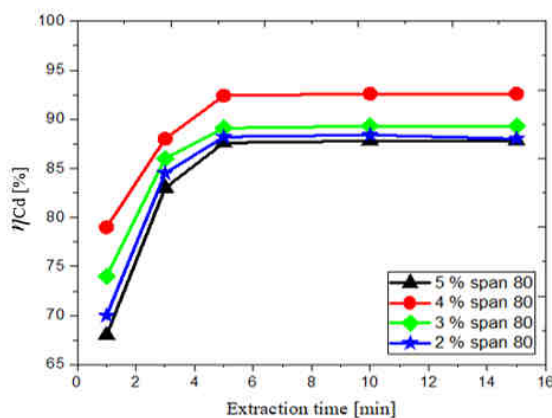


Fig. 5. Effect of surfactant concentration on η_{Cd} in ELM process (experimental conditions: agitation speed = 350 rpm, $V_I : V_M = 1 : 4$, $V_E : V_F = 1 : 5$, $C_{i0} = 0.25$ M, rapidity of homogeniser = 10,000 rpm, initial cadmium concentration: 100 ppm)

Previous research confirms that increasing surfactant concentration up to a certain limit enhances emulsion stability and reduces the breaking rate, thereby improving cadmium transport across the membrane. For example, one study reported that increasing the surfactant concentration from 1 % to 4 % v/v reduced membrane breakdown and improved removal efficiency [20, 21]. This agrees with our findings, where 4 % Span 80 provided the highest removal efficiency. However, at 5 % v/v, the efficiency dropped to 87.8 %, indicating that exceeding the best experimental range can decrease removal performance. This drop is explained by the increased mass transfer resistance at the interface between the

internal and external phases, which limits Cd(II) transport [22]. Moreover, very high surfactant concentrations can cause greater osmotic swelling, leading to emulsion breakdown, increasing the viscosity of the system, and reduced removal efficiency [23]. These results align with the scientific literature, confirming that there is a best surfactant concentration that balances emulsion stability and ion transport efficiency; in our case, this was 4 % v/v Span 80.

Effect of agitation speed

This parameter was tested at various speeds (250, 350, 450, and 650) rpm. As shown in Figure 6, increasing agitation speed improved η_{Cd} , rising from 86.8 % at 250 rpm to 92.6 % at 350 rpm, and then to 94.6 % at 450 rpm. This improvement reflects the role of shear force in reducing emulsion droplet size and increasing the contact area between phases, thereby enhancing mass transfer [24]. The efficiency increased slightly to 95 % at 650 rpm, but a gradual decline was observed over time, likely due to emulsion breakage caused by excessive shear force and osmotic swelling, which reduced the capacity to retain Cd(II) [20, 25].

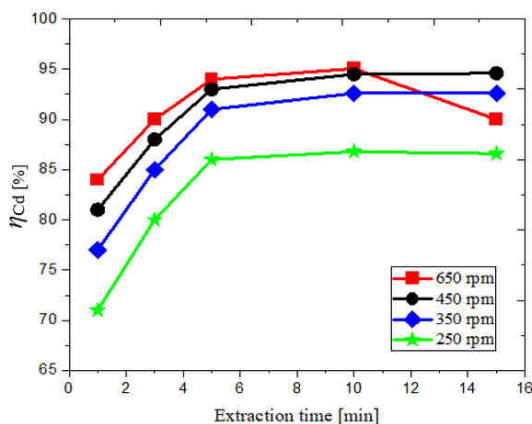


Fig. 6. Effect of agitation speed on η_{Cd} in ELM process (experimental conditions: surfactant concentration = 4 % v/v, $V_I : V_M = 1 : 4$, $V_E : V_F = 1 : 5$, $C_{I0} = 0.25$ M, rapidity of homogeniser = 10,000 rpm, initial cadmium concentration: 100 ppm)

Based on these results, 450 rpm was identified as the best experimental speed, as it provided high efficiency and maintained stability over time without causing emulsion collapse, unlike at 650 rpm. This aligns with previous studies reporting a maximum agitation speed threshold, beyond which emulsion breakdown rates increase, reducing removal efficiency. Moreover, the high speed breaking the emulsion droplet leads to a reduction in the ELM system efficiency [10].

Effect of internal to membrane phase ratio

The results of this work demonstrate a clear effect of the internal to membrane phase ratio ($V_I : V_M$) on η_{Cd} . Ratios of 1 : 2, 1 : 4, and 1 : 6 were investigated. The best experimental ratio was found to be 1 : 4, yielding a η_{Cd} of 94.6 %. As shown in Figure 7,

efficiencies of 93.8 % and 91.0 % were obtained at ratios of 1 : 2 and 1 : 6, respectively. These findings are consistent with previous studies indicating that decreasing the ratio to 1 : 6 reduces removal efficiency.

This reduction is attributed to the higher viscosity of the emulsion, which promotes coalescence of internal droplets and the formation of large bubbles. These bubbles reduce the effective contact area between the internal and external phases, thereby compromising the extraction process [26]. Conversely, increasing the ratio to 1 : 2 reduces removal efficiency and increases membrane breakage because the insufficient membrane phase cannot adequately protect the internal droplets, leading to emulsion rupture. Membrane breakage was observed to be more severe at 1 : 2 than at 1 : 4, significantly impacting removal efficiency [2, 27]. Additionally, ratios above 1 : 4 can cause swelling of the internal droplets, further reducing both extraction and stripping efficiency. Therefore, maintaining a ratio of 1 : 4 ensures both emulsion stability and high removal performance.

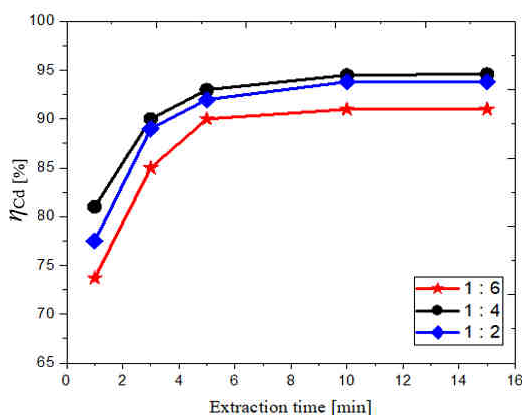


Fig. 7. Effect of the internal to membrane phase ratio on η_{Cd} in ELM process (experimental conditions: agitation speed = 450 rpm, surfactant concentration = 4 % v/v, $V_E : V_F = 1 : 5$, $C_{i0} = 0.25$ M, rapidity of homogeniser = 10,000 rpm, initial cadmium concentration: 100 ppm)

Effect of emulsion to feed phase ratio

η_{Cd} was evaluated at emulsion to feed phase ratios of 1 : 3, 1 : 5, and 1 : 7. The best ratio was 1 : 3, achieving a η_{Cd} of 97.4 %. As shown in Figure 8, the efficiency decreased to 94.5 % at 1 : 5 and further to 85.4 % at 1 : 7.

These results indicate that increasing the treatment ratio from 1 : 7 to 1 : 3 improves removal efficiency, likely due to the higher number of emulsion droplets and the increased contact surface area between the membrane phase and the internal phase, which enhance mass transfer [28].

This finding is consistent with Ghorbanpour and Jahanshahi [29] who reported that the 1 : 3 ratio, with its higher emulsion volume, promotes cadmium transport efficiency through an increased surface area. Similarly, Laki et al. [22] emphasised that increasing the treatment ratio ($V_E : V_F$) can improve removal efficiency. However, excessive emulsion volume may lead to reduced effective transport area due to higher membrane density, and can also increase emulsion breakage, both of which decrease efficiency.

The lower efficiency observed at 1 : 7 compared with 1 : 5 and 1 : 3 may be due to these effects, including high viscosity, emulsion rupture, and reduced transport area when the emulsion volume exceeds a certain limit. Therefore, a ratio of 1 : 3 appears to provide the best balance between maximising emulsion volume and maintaining an effective surface area for cadmium transport.

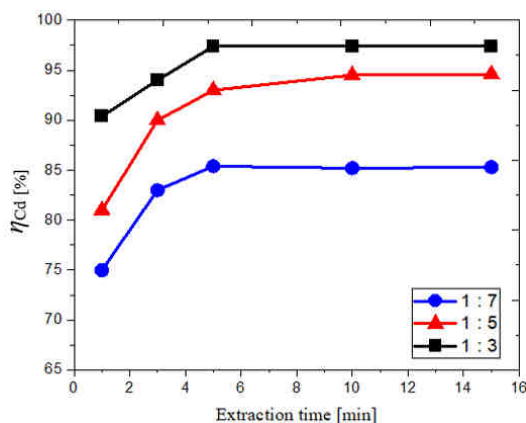


Fig. 8. Effect of the emulsion to feed phase ratio $V_E : V_F$ on η_{Cd} in ELM process (experimental conditions: agitation speed = 450 rpm, surfactant concentration = 4 % v/v, $V_I : V_M = 1 : 4$, $C_{i0} = 0.25$ M, rapidity of homogeniser = 10,000 rpm, initial cadmium concentration: 100 ppm)

Effect of HCl concentration

Three concentrations (0.1, 0.25, and 1.0 M) were investigated to evaluate the effect of HCl concentration on η_{Cd} . As shown in Figure 9, the best concentration was 0.25 M, achieving 97.4 % removal efficiency, compared to 57 % at 0.1 M and 95.7 % at 1.0 M. The improvement from 0.1 to 0.25 M is attributed to the increased ionic driving force between the two phases, enhancing cadmium transfer. This observation is consistent with previous studies, which reported that the primary driver of the extraction process is the difference in the chemical potential of H^+ ions [30].

The reduced efficiency at 1.0 M is likely due to the negative effect of high acid concentration on membrane stability, as it may interact with the surfactant (Span 80), compromising its function and weakening emulsion stability [31]. Additionally, excessive acid concentration can create a large ionic strength difference between phases, causing osmotic swelling, dilution of the internal phase, and reduced removal efficiency [32]. Thus, 0.25 M provides the best balance between ion transport efficiency, membrane stability, and overall η_{Cd} .

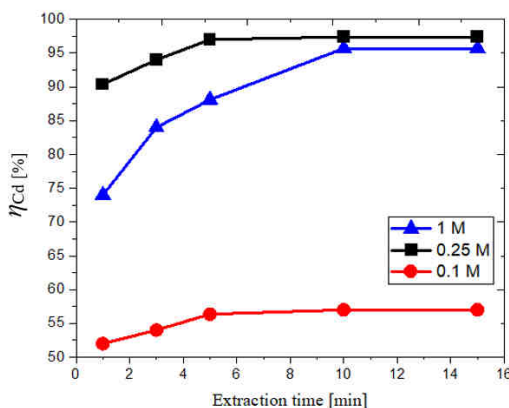


Fig. 9. Effect of HCl concentration on η_{Cd} in ELM process (experimental conditions: surfactant concentration = 4 % v/v, $V_I : V_M = 1 : 4$, $V_E : V_F = 1 : 3$, agitation speed = 450 rpm, rapidity of homogeniser = 10,000 rpm, initial cadmium concentration: 100 ppm)

Kinetics study of cadmium removal via ELM

The kinetics of cadmium removal by the ELM system were evaluated using both the pseudo-first-order and pseudo-second-order models [33]. The analysis was conducted using the following equation:

- The pseudo-first-order model is expressed as:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (2)$$

where: q_e [mg/g] and q_t [mg/g] are the adsorption capacities at equilibrium and at time t , respectively, and k_1 [1/min] is the rate constant. The linear plot of $\ln(q_e - q_t)$ versus t is shown in Figure 10a.

- The pseudo-second-order model is given by:

$$\frac{t}{q} = \frac{t}{q_e} + \frac{1}{k_2 q_e} \quad (3)$$

where k_2 [g/mg·min] is the rate constant. The corresponding plot of t/q_t versus t is presented in Figure 10b.

The results demonstrated that the pseudo-first-order model exhibited a relatively weak correlation ($R^2 = 0.739$), whereas the pseudo-second-order model provided an excellent fit with the experimental data ($R^2 = 0.999$). This indicates that the cadmium removal process in the ELM system follows the pseudo-second-order kinetics, suggesting that chemisorption through valence forces and complex formation between Cd^{2+} ions and the phosphoric acid groups of D_2EHPA is the dominant rate-controlling step. These findings are in strong agreement with the work of Admawi and Mohammed [20], who also reported that cadmium removal via emulsion liquid membranes was best described by the pseudo-second-order model with high reliability.

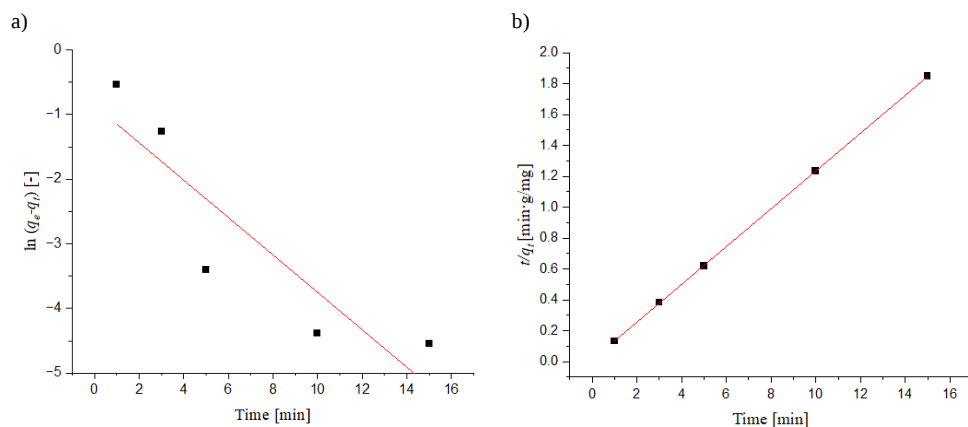


Fig. 10. Kinetic models for cadmium removal by ELM: a) pseudo-first-order kinetic model, b) pseudo-second-order kinetic model

Potential applications and implications

The ELM system developed in this study demonstrates strong potential for practical applications in the treatment of industrial effluents containing cadmium and other toxic heavy metals. Its improved emulsion stability, reduced internal phase leakage and high removal efficiency under the best experimental conditions make it suitable for deployment in sectors such as mining wastewater treatment, electroplating effluent remediation, and recovery of valuable metals from industrial streams. Furthermore, the enhanced operational reliability and cost-effectiveness of the process can facilitate its integration into existing wastewater treatment facilities, particularly in regions where advanced treatment technologies are limited. The findings of this work thus provide a foundation for scaling up the ELM process and adapting it for continuous operation in real-world scenarios.

Based on the investigated parameters in this study, targeted strategies can be proposed to overcome the major challenges commonly encountered in emulsion liquid membrane (ELM) processes, including emulsion instability and phase leakage. Emulsion stability can be enhanced by selecting the best surfactant concentration, which improves interfacial film strength, and by adjusting emulsification speed to achieve uniform droplet formation without inducing excessive shear. Additionally, adjusting the treatment ratio, the internal phase to membrane phase ratio, and the emulsion to feed phase ratio can help maintain structural integrity and reduce droplet coalescence. Phase leakage can be minimised by adjusting the acid concentration in the internal phase to maintain osmotic balance and reduce internal phase migration. Implementing these targeted measures, as demonstrated in this work, can significantly improve both the efficiency and reliability of the ELM system.

Challenges addressed in this study

Previous studies have addressed cadmium removal via the emulsion liquid membrane (ELM) technique but reported persistent technical challenges. For instance, Jabbar and Rushdi [34] applied the Taguchi method to optimise operational variables such as surfactant and carrier concentrations, emulsification speed, and the membrane to external phase ratio. Although their work achieved good removal efficiency, they reported ongoing

issues including emulsion instability, internal phase leakage, and droplet swelling. Similarly, Ahmad [36] employed a “green” ELM with vegetable oils as the diluent, demonstrating that higher carrier and surfactant concentrations enhanced stability but reduced mass transfer rate. Kusumastuti et al. investigated the influence of surfactant concentration, carrier concentration, and internal phase composition, achieving high removal efficiencies under optimal conditions, yet still facing problems such as emulsion swelling, leakage and reduced stability at higher additive concentrations [36].

Compared to these studies, the present work directly addresses the reported challenges by systematically optimising key operational parameters including surfactant concentration, stirring speed, internal phase concentration, internal to membrane phase ratio, and emulsion to feed phase ratio to achieve the most suitable balance between high η_{Cd} , improved emulsion stability, and minimised internal phase leakage. This approach is intended to deliver more practical and applicable operational guidelines than those currently available in the literature.

Conclusion

The ELM process has proven to be highly effective in treating aqueous solutions containing cadmium. Increasing surfactant concentration improved η_{Cd} up to a best concentration of 4 % v/v, which ensured excellent emulsion stability; however, excess surfactant reduced efficiency. Increasing agitation speed enhanced removal efficiency up to 450 rpm, beyond which efficiency declined due to emulsion instability. The concentration of the internal phase reagent had a significant influence, with a best concentration of 0.25 M HCl in the stripping phase providing high efficiency without compromising stability. The internal to membrane phase ratio showed improved efficiency up to 1 : 4, after which performance decreased. The best emulsion to feed phase ratio was 1 : 3, providing a balance between droplet volume and effective mass transfer area. Prolonging emulsification beyond the best duration of 10 minutes reduced efficiency due to droplet coalescence and instability. Under these best experimental parameters, 4 % v/v surfactant concentration, 0.25 M internal agent, 10-minute emulsification, 1 : 4 internal to membrane phase ratio, 450 rpm agitation, and 1 : 3 emulsion to feed ratio. The ELM system achieved a maximum η_{Cd} of 97.4 %. Furthermore, kinetic analysis indicated that cadmium removal followed the pseudo-second-order model ($R^2 = 0.99997$), suggesting that chemisorption through complex formation with D₂EHPA functional groups was the dominant rate-controlling step. These findings highlight the strong potential of the developed ELM system for industrial wastewater treatment and heavy metal remediation. This developed system can be feasibly integrated into existing wastewater treatment facilities for the removal of cadmium and potentially other heavy metals. This provides a cost-effective and environmentally friendly treatment solution for industrial effluents.

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