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Kinetic Modeling of Electromembrane Extraction of Copper using a Novel Electrolytic Cell Provided with a Supported Liquid Membrane

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Abstract

The aim of this study is to investigate the kinetics of copper removal from aqueous solutions using an electromembrane extraction (EME) system. To achieve this, a unique electrochemical cell design was adopted comprising two glass chambers, a supported liquid membrane (SLM), a graphite anode, and a stainless-steel cathode. The SLM consisted of a polypropylene flat membrane infused with 1-octanol as a solvent and bis(2-ethylhexyl) phosphate (DEHP) as a carrier. The impact of various factors on the kinetics constant rate was outlined, including the applied voltage, initial pH of the donor phase solution, and initial copper concentration. The results demonstrated a significant influence of the applied voltage on enhancing the rate of copper mass transfer across the membrane. As the applied voltage increased, the rate constant also increased. Additionally, increasing the pH of the solution led to an initial elevate in the rate constant, reaching a maximum value at pH 5, after which it started to decline. Moreover, higher initial copper concentrations had an adverse effect on the rate constant. Notably, the concentration decay profiles observed under different operating conditions followed first-order kinetics, with correlation coefficients exceeding 0.99. The elucidation of this discovery emanated from a remarkable and striking congruence between the experimental data and the mathematical underpinnings of the first-order kinetics model. This serendipitous alignment profoundly reinforced the robustness, veracity, and unwavering reliability of meticulously obtained results, amplifying the credibility and trustworthiness of the present comprehensive study.

Keywords: Copper, Kinetic modeling, Mass transfer, Electromembrane extraction, wastewaters.

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1- Introduction

Copper serves as a crucial trace element for humans and other living organisms, playing a vital role in the proper functioning of numerous enzymes and proteins. Moreover, it has various societal uses, such as the production of conductive strands, flow control components, kitchenware, coins, and construction resources. However, it is important to acknowledge that copper, classified as a heavy metal, presents notable environmental risks. Industries involved in metal finishing, textile production, and electronics manufacturing can produce wastewater containing copper concentrations of up to 500 mg/L. Consequently, it becomes necessary to treat copper-containing wastewater prior to its release into the environment [1]. In compliance with European Union regulations, the acceptable range for copper concentration in drinking water sources, including rivers, is set between 1.5 and 2 mg/L. Therefore, it is advisable to ensure that discharged effluents remain below this limit for adhering to the prescribed guidelines [2]. Numerous techniques have been employed for the removal of copper from waste streams, offering a wide array of approaches including adsorption, chemical precipitation, ion exchange, biosorption, reverse osmosis, membrane separation with ion exchange, and electrochemical precipitation. Each method presents a distinct and effective means of eliminating copper from waste streams. However, these techniques have drawbacks due to their high material and operational costs [3].

Over recent decades, electromembrane extraction (EME) has emerged as a prominent method employed in the analysis of pollutants resulting from use in various medical and environmental groups, offering preconcentration and separation capabilities. EME has exhibited remarkable effectiveness, demonstrating its efficiency and reliability in various applications. Notably, when compared to other microextraction systems, EME distinguishes itself by its ability to achieve substantial enrichment factors while maintaining remarkably short extraction durations [4]. During the early stages of this discipline, scholarly inquiries predominantly concentrated on the analysis of metallic elements within diverse biological specimens[5-10]. After a considerable period, a

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*Corresponding Author: Noor R. Kadhim, Email: noor.jabbar2011d@coeng.uobaghdad.edu.iq prominent research article surfaced, presenting a groundbreaking initiative of utilizing EME for the purpose of analyzing metallic constituents in solutions [11]. The successful extraction of heavy metals through EME is influenced by several variables, including the type of solvent, selection of carrier, pH levels of the donor phase and receiving phase solutions, stirring rate, extraction time, and more. The optimization of these variables plays a crucial role in attaining successful and efficient extraction of heavy metals using this technique[4]. In EME, SLM comprised of long-chain alcohols such as 1-heptanol and 1-octanol have conventionally been deployed in electromembrane extraction endeavors [12].

The extensive literature on EME has comprehensively documented its application in metal ion extraction, elucidating a myriad of methodologies and practical applications. Notably, Basheer et al. (2008) undertook an investigation focusing on the extraction of lead from biological fluids through EME[5]. In their work, toluene was employed as a solvent in the SLM, and a potential of 300 V was employed for a duration of 15 minutes. In a remarkable study, Kubáň et al. (2011) revealed the exceptional potential of EME in extracting diverse heavy metal ions[11]. Their research successfully applied EME to remove heavy metal ions such as manganese, cadmium, zinc, cobalt, copper, nickel, and lead. Their findings not only underscored the effectiveness of EME in surmounting the challenges associated with varying sample matrices but also paved the way for its comprehensive utilization in heavy metal ion analysis and extraction. In a noteworthy investigation conducted by Hosseiny Davarani et al.(2015), highly efficacious EME of various heavy metal cations, such as cadmium, zinc, cobalt, copper, and silver, were achieved[13]. Their approach involved the utilization of a SLM comprising 1-octanol infused with 0.5% v/v Bis(2-ethylhexyl) phosphate and 0.5% v/v tris (2-ethylhexyl) phosphate. They demonstrated the effectiveness of EME in selectively extracting heavy metal cations from aqueous solutions, thus making noteworthy contributions to the advancement of analytical methods employed in heavy metal analysis and environmental monitoring [13]. Collectively, these studies underscore the suitability of EME as a technique for metal ion extraction and shed light on its potential in heavy metal removal and kinetic studies[14].

Within the realm of electrochemical processes conducted under galvanostatic conditions, the removal of heavy metals presents two discernible scenarios: the first scenario pertains to electron transport control, characterized by a linear metal concentration profile as a function of time, while the second scenario corresponds to mass-transport control, wherein the concentration undergoes exponential decay over time [15]. The electromembrane process plays a crucial role in the kinetics of heavy metal removal. It serves as a vital means to gather valuable insights into process dynamics, including reaction rate, reaction order, and mass transfer properties. Through this process, essential information that sheds light on the behavior and characteristics of heavy metal removal can be acquired.

Within the confines of a batch reactor, particularly in the context of first-order reactions, the rate at which the reaction proceeds exhibit a direct proportionality to the concentration of the reagent involved. Consequently, this implies that the rate of alteration in reagent concentration, denoted by C, relative to time possesses a proportionality to the reagent concentration itself at that specific moment in time. In situations where the reaction entails the removal of the constituent, the corresponding equation governing this phenomenon can be briefly articulated as follows [16]:

$$\frac{dC}{dt} = -k\,C \tag{1}$$

Integrating Equation 1 with C = C0 at t = 0 leads to:

$$C_t = C_O\, e^{-kt} \tag{2}$$

$$\text{Or} \quad \ln (C/C_0) = -K \cdot t \tag{3}$$

Here, C symbolizes the concentration of the constituent undergoing removal (mg/L). C_0 corresponds to the initial concentration of the constituent at the onset of the extraction process(mg/L). The variable (t) signifies the duration of the extraction process, (hr). Lastly, the parameter K represents the reaction rate constant, (hr^{-1}). This study focuses on the studying the kinetics of electromembrane extraction of copper (II) from a simulated wastewater, where effects of different parameters such as voltage input, pH levels and initial content amounts of copper on the kinetic rate constant were investigated. No such study was performed previously based on our knowledge.

2- Experimental

2.1. Chemicals

Deionized water was utilized as the solvent for both the source phase and acceptor solutions in all experiments. The cation, Cu^{2+}, originated from a stock solution of $CuNO_3$ obtained from Central Thomas Baker (Mumbai, India). The acceptor solution was prepared using HCl acid acquired from Thomas Baker (Ambernath, India). For the electromembrane extraction (EME) procedure, 1-octanol, sourced from Central Drug House (Delhi, India), served as the organic solvent.

In the EME process for copper removal, bis(2-ethylhexyl) phosphate (DEHP), was used as the carrier and procured from Shanghai Macklin Biochemical (Shanghai, China). The pH value was controlled using NaOH obtained from POCH (Poland). A polypropylene membrane sheet, supplied by Jian City Qing Feng Equipment Material Co. China, with a thickness ranging from 0.18 to 0.22 mm and an average pore size of 0.1 microns, was employed as the solid material for the liquid membrane.

2.2. Electro-membrane extraction system

The electromembrane extraction cell, depicted in Fig. 1, comprises two chambers with a total volume of 137.44 cm^3 for each. The first chamber is cylindrical, accommodating the donor phase. It features a diameter of 5 cm and a length of 7 cm. This chamber contains a volume of 100 ml of the donor phase Adjacent to this chamber, positioned on the lateral side, is a cylindrical horizontal channel measuring 3 cm in diameter and 3 cm in length. This channel is situated 2 cm above the base of the donor chamber. The second chamber same as the design of the first chamber and contains the acceptor phase, also with a volume of 100 ml. Both chambers are equipped with Teflon covers, measuring 4.8 cm in diameter and 10 mm in thickness. These covers feature two holes: one for securing the electrode and the other for solution feeding. Within the cell, an anode made of graphite was employed. The graphite anode possesses dimensions of 5 cm in length, 3 cm in width, and 5 mm in thickness. The choice of graphite as the material for the anode is attributed to its capability to facilitate the anodic oxidation of water [20]. In the electromembrane extraction (EME) system, a stainless steel 316L plate with dimensions of 5 cm in length, 3 cm in width, and 2 mm in thickness served as the cathode. The use of stainless-steel electrodes in the EME system is notable due to its cost advantages compared to platinum electrodes. To facilitate the extraction process, a circular polypropylene membrane sheet with an active surface area of 7 cm^2 was impregnated with 1-octanol and a suitable carrier for a duration of 15 minutes. Subsequently, the membrane sheet was positioned between the channels of the two chambers and securely fastened using airtight cups surrounding each channel. Four screws and nets were employed for enhanced stability. Before assembling the membrane with the channels, the edges of each channel were meticulously covered with RTV silicone rubber to ensure a leak-free setup. To ensure continuous stirring and effective mixing of the solution, two magnetic bars were placed in both the acceptor and donor chambers. A magnetic stirrer with a variable speed of rotation ranging from 0 to 1000 rpm (Metrohm AG Herisau, Switzerland, Type: 18490010) was utilized for this purpose. The EME system operated at various voltages within the range of 20-60 V, with power supplied by a UNI-T power supply (Hong Kong, Type: UTP3000). To accurately measure the current passing through the EME system, an ammeter with a maximum current of 0.2 A (UNI-T, Kowloon, Hong Kong) was connected in series.

The electromembrane extraction (EME) experiments were carried out under ambient room temperature conditions. To ensure accurate measurements and avoid any interference resulting from the solvent used for SLM and the absorption of copper ions during atomic absorption measurements, the acceptor and donor solutions were collected separately in glass containers after each run. After collection, the solutions were subjected to a heat treatment process to evaporate all the aqueous components, ensuring the isolation of the desired analytes for further analysis. [13]. Following that, the vials were emptied, and deionized (DI) water was added to refill them. Subsequently, samples were extracted from these vials to identify the presence of copper using atomic absorption spectroscopy (AAS).

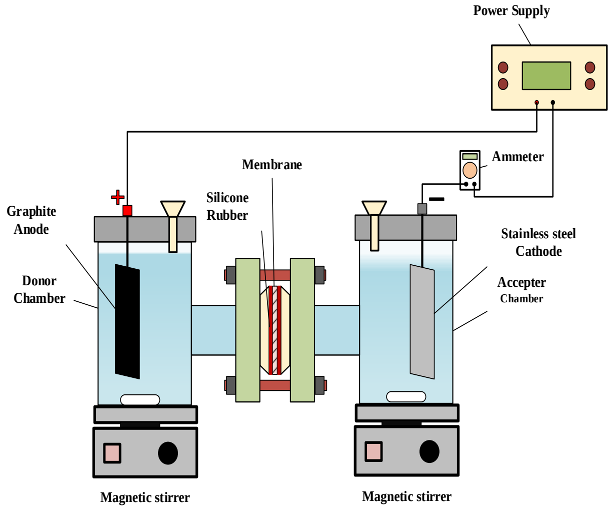

Fig. 1. EME System

2.3. The analysis and measurements

To analyze the samples, an AA-7000 atomic absorption spectroscopy instrument manufactured by SHIMADZU (Japan) was utilized, employing an air-acetylene flame configuration. The specific wavelength of 240.7 nm was selected for the detection of copper cations in the atomic absorption spectroscopy analysis. For pH measurement, a portable pH meter manufactured by HANNA (Romania) was employed, ensuring accurate and precise pH readings.

The efficiency of copper removal was calculated based on the following equation [17-18]:

$$RE(\%) = \frac{C_i - C_f}{C_i} \times 100 \qquad (4)$$

In the realm of electrochemical processes, specifically in the context of Cu(II) electrolysis, the final concentration of Cu(II), denoted as C_f and measured in parts per million (ppm), corresponds to the concentration achieved at the end of the electrolysis period Δt. Conversely, the initial Cu(II) concentration at the onset of the electrolysis, denoted as Ci and also measured in parts per million (ppm), represents the concentration prevailing prior to the commencement of the electrochemical reaction.

In the present work the electrolysis time was taken as 6 hr. based on preliminary experiments that showed extraction time should not exceed 6h. to confirm there is no jump in the current would occur in which the supported liquid membrane starts to release from the membrane to the solution [19].

3- Results and Discussion

3.1. Effect of the Applied Potential

Fig. 2 shows the copper concentration profile with time at different applied voltages, the corresponding relation between ln(C/C_o) vs. time is displayed in Fig. 3. It can be seen that heightening applied voltage yields decreasing in the final concentration of copper reaching 1.8 mg/l at 60 volt. Fig. 3 and Table 1 confirm that the relation between ln (C/C_o) and time is almost linear with a correlation coefficient R^2 not less than 0.992 confirming that the system obeyed a first-order kinetic and it was under mass transfer control. However, it needs a longer time to reach the final concentration within the acceptable limit since the process solely depends on the transfer across the SLM. Meanwhile, the transfer across the SLM is selective which cannot be obtained using traditional electrochemical methods. This reflects the suitability of EME as an extraction method for the target analyte (copper ion). Table 1 demonstrates that the rate constant increases as the applied voltage increases from 20 volts to 60 volts and its value at 60 volts becomes triple its value at 20 volts. This is an indication that the applied voltage is a significant and major factor in EME [19].

In a comprehensive investigation carried out by Khattab et al.[15] , an analogous relationship was established, albeit with a different focus. Specifically, their study delved into the kinetics of copper removal employing a packed bed cathode and traditional electrodeposition methods, emphasizing the correlation between current and rate constant rather than the applied voltage. The primary objective of their research was to gain profound insights into the intricacies of copper removal dynamics and elucidate the interplay between current and rate constant within the context of electrodeposition techniques.

In a prior research endeavor, attention was directed, on the other hand, towards the separation of nickel and cadmium from laboratory-made wastewater utilizing a bio-electrochemical reactor equipped with a packed bed rotating cylinder cathode. Interestingly, our observations revealed a striking parallelism between the applied voltage and the rate constant. This intriguing revelation suggested a discernible correlation between the applied voltage and the velocity at which nickel and cadmium were effectively eliminated from the wastewater. Our findings thus bestowed valuable illumination upon the intricate dynamics of the bio-electrochemical reactor system, underscoring its efficacy in efficiently removing heavy metals from wastewater[17,20]. It is noteworthy, however, that in the context of the present investigation, the impact of the applied voltage on the rate constant assumes a more prominent role and exhibits a nonlinearity, as depicted in Table 1. This pronounced nonlinearity can be attributed to the utilization of higher voltages within the framework of electromembrane extraction (EME), thereby accentuating the influence of applied voltage on the rate constant.

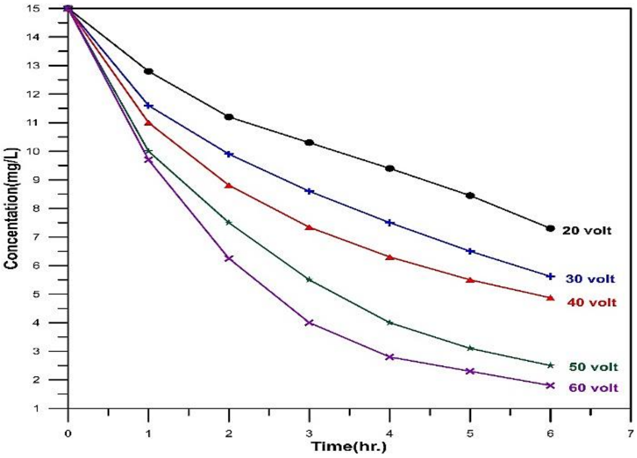

Fig. 2. Concentration Profile with Time at Different Applied Voltage. Initial copper concentration: 15 ppm; Carrier: DEHP (1 % v/v); feed solution: DI water; acceptor solution: 0.1 M HCl; SLM: 1-octanol; extraction time: 6 hrs.; stirring rate: 1000 rpm; pH: 5

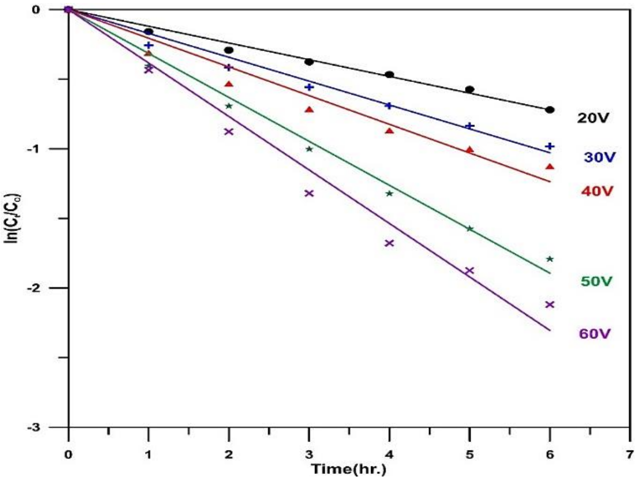

Fig. 3. Plots of ln(C/Co) vs Time at Different Applied Voltage

Table 1. Rate Constant at Different Applied Voltage

Parameters	Value	K(hr^{-1})	K(min^{-1})	R^2
Applied voltage (Volts)	20	0.1200945055	2.000 $x10^{-3}$	0.9959
	30	0.1714285714	2.857 $x10^{-3}$	0.9936
	40	0.2060791209	3.434 $x10^{-3}$	0.9873
	50	0.3156043956	5.260 $x10^{-3}$	0.9968
	60	0.3841318681	6.400 $x10^{-3}$	0.9927

3.2. Effect of the initial pH of the donor

To ensure the efficiency of EME process and considering previous studies, the pH range selected for the experiments was between 3 and 8. An initial pH value of 1 in the acceptor solution was used by employing 0.1 M hydrochloric acid (HCl). The decision to utilize HCl as the acceptor medium was guided by prior empirical findings [13]. Fig. 4 shows the copper concentration profile with time at different pH, the corresponding relation between ln(C/Co) vs. time is displayed in Fig. 5. It is apparent that raising pH yields a rate rise constant to reach a maximum value at pH of 5 then starts to decline beyond that. Fig. 5 and Table 2 confirm that the relation between ln (C/Co) and time is almost linear with a correlation coefficient R^2 not less than 0.992 confirming that the system obeyed first-order kinetic and it was under mass transfer control. Besides, Table 2 demonstrates that the rate constant at pH=5 becomes triple its value at pH=3. This is an indication that high acidity is not recommended for EME as well as high alkalinity [19]. It seems that operating between 5 and 6 is the preferred condition for copper extraction because the value of the rate constant is higher in this range leading to higher mass transfer. A similar observation was not in the traditional electrochemical methods and our previous works confirm that in the removal of both Ni and Cd [20, 21].

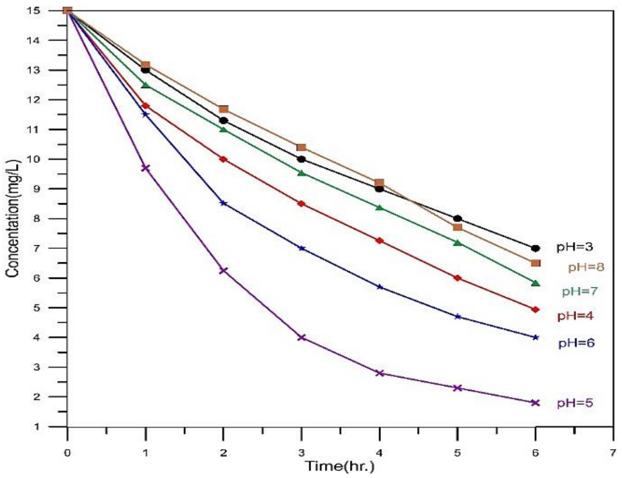

Fig. 4. Concentration Profile with Time at Different Initial pH of the Donor. Initial copper concentration: 15 ppm; carrier: DEHP (1 % v/v); feed solution: DI water; acceptor solution: 0.1 M HCl; SLM: 1-octanol; extraction time: 6 hrs.; stirring rate: 1000 rpm; voltage: 60 V

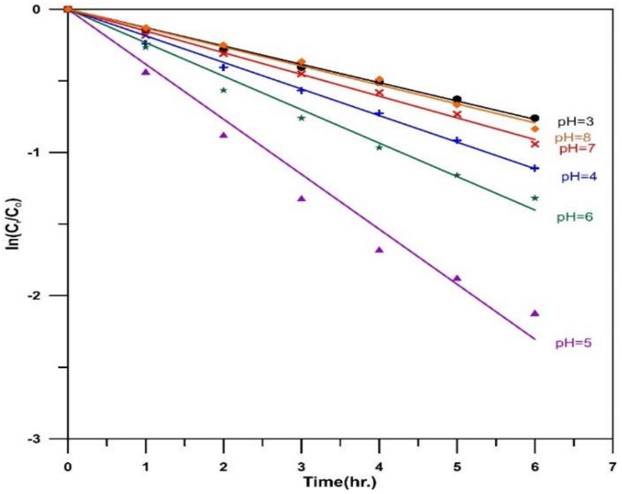

Fig. 5. Plots of ln(C/Co) vs Time at Different pH

Table 2. Rate Constant at Different pH

Parameters	Value	K(hr^{-1})	K(min^{-1})	R^2
pH	3	0.1282197802	2.137 x10^{-3}	0.9989
	4	0.1856813187	3.095 x10^{-3}	0.9985
	5	0.3841318681	6.400 x10^{-3}	0.9927
	6	0.2337472527	3.896 x10^{-3}	0.9955
	7	0.1517835165	2.529 x10^{-3}	0.9984
	8	0.1322417582	2.204 x10^{-3}	0.9971

3.3. Effect of Copper Initial Concentration

Assessing the consequence of varying Initial-stage copper concentrations on the electromembrane extraction (EME) process is valuable and should be considered to demonstrate if system behavior departs from the mass transfer regime. In pursuit of this objective, an investigation was conducted, analyzing a series of copper concentrations at a range from 5 to 25 ppm. Fig. 6 shows the copper concentration profile with time at different initial concentrations of copper, the corresponding relation between ln(C/Co) vs. time is displayed in Fig. 7. It is notable that amplifying initial concentration beyond 15 ppm results in decreasing the rate constant. Fig. 7 and Table 3 confirm that the relation between ln (C/Co) and time is almost linear with a correlation coefficient $R^{2,}$ not less than 0.991 and the system is still under mass transfer control within this range. At higher concentrations, the driving force is high leading to the transfer higher amount of copper ions from the donor phase to the accepter phase. However, the removal efficiency had an adverse effect where it was decreased as high concentration was used as shown in Fig. 6. For example, amount of copper transfer when starting with a concentration of 25 was 18.71 mg/L at 6 hrs. resulting in removal efficiency of 74.84% while the amount of copper transfer when starting with a concentration of 15 was 13.2 mg/L at 6 hrs. resulting in removal efficiency of 88% Similar observation was not at the traditional electrochemical methods and our previous works confirms that in removal both of Ni and Cd [20, 21].

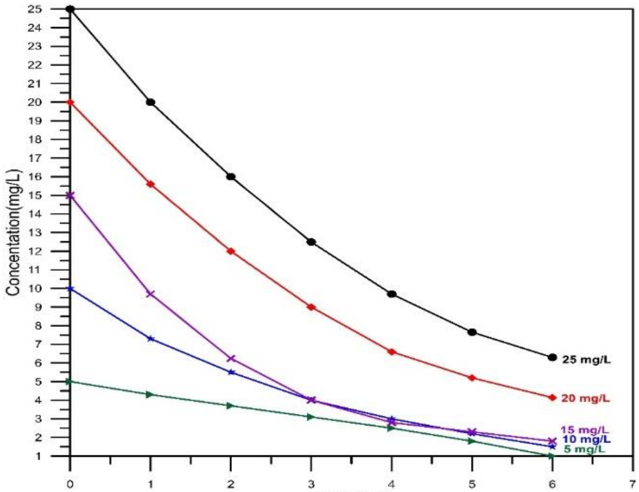

Fig. 6. Concentration Profile with Time at Different Initial Concentration of Copper. Carrier: DEHP (1 % v/v); feed solution: deionized water; acceptor solution: 0.1 M HCl; SLM: 1-octanol; extraction time: 6 hrs.; stirring rate: 1000 rpm; voltage: 60 V; pH: 5

Table 3. Rate Constant at Different Initial Concentration of Copper

Parameters	Value	K(hr^{-1})	K(min^{-1})	R^2
Copper Concentration (mg/L)	5	0.1896593407	3.161 x10^{-3}	0.9911
	10	0.3079560440	5.133 x10^{-3}	0.9994
	15	0.3841318681	6.400 x10^{-3}	0.9927
	20	0.2668241758	4.447 x10^{-3}	0.9994
	25	0.2326593407	3.877 x10^{-3}	0.9997

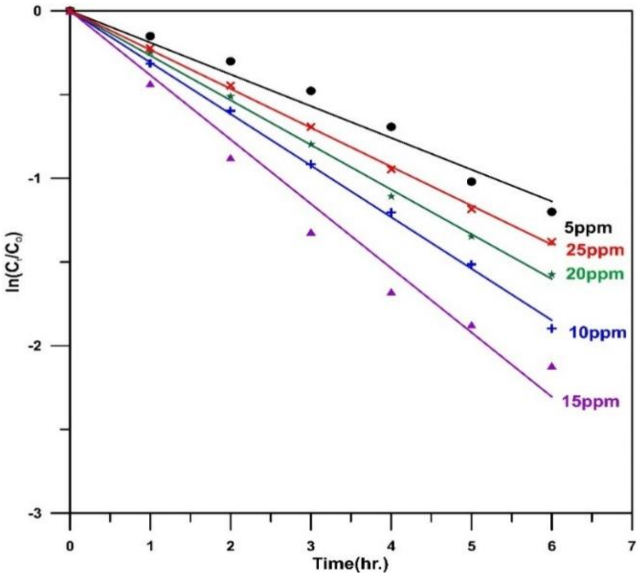

Fig. 7. Plots of ln(C/Co) vs Time at Different Initial Concentrations of Copper

4- Conclusion

This study focused on examining the kinetics of copper removal using electromembrane extraction (EME). By applying an external electrical field, copper ions were successfully transported from a donor solution through an organic liquid membrane to an aqueous receiving solution. The role of applied voltage was crucial, with a threefold increase resulting in a proportional tripling of the rate constant, emphasizing its impact on EME efficiency. Optimal pH (around 5) was vital for the membrane's performance, ensuring efficient copper transfer. Higher copper concentrations (>15 ppm) and deviations from optimal pH negatively affected the rate constant. Despite this, the system achieved an impressive copper removal efficiency exceeding 70%, which could be further enhanced through consecutive operations. The logarithmic profiles of C/Co vs. time consistently showed a strong linear relationship, confirming the validity of first-order kinetics in representing the mass transfer-controlled behavior of the system.

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النمذجة الحركية لاستخراج النحاس باستخدام الغشاء الكهربائي باستخدام خلية التحليل الكهربائي الجديدة المزودة بغشاء سائل مدعوم

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الخلاصة

الهدف من هذه الدراسة هو فحص حركية إزالة النحاس من المحاليل المائية باستخدام نظام استخراج الغشاء الكهربائي (EME). لتحقيق ذلك، تم اعتماد تصميم فريد للخلايا الكهروكيميائية يشتمل على غرفتين زجاجيتين، وغشاء سائل مدعوم (SLM)، وأنود جرافيت، وكاثود من الفولاذ المقاوم للصدأ. يتكون SLM من غشاء مسطح من مادة البولي بروبيلين مملوء بـ ١-أوكتانول كمذيب وثنائي (٢-إيثيل هكسيل) فوسفات (DEHP) كحامل. تم تحديد تأثير العوامل المختلفة على معدل ثابت الخواص الحركية، بما في ذلك الجهد المطبق، ودرجة الحموضة الأولية لمحلول الطور المانح، وتركيز النحاس الأولي. أظهرت النتائج أن الجهد المطبق له تأثير كبير في تعزيز معدل نقل كتلة النحاس عبر الغشاء. كلما زاد الجهد المطبق، زاد معدل ثابت أيضًا. بالإضافة إلى ذلك، أدت زيادة الرقم الهيدروجيني للمحلول إلى ارتفاع أولي في ثابت المعدل، ووصل إلى قيمة قصوى عند الرقم الهيدروجيني ٥، وبعد ذلك بدأ في الانخفاض. علاوة على ذلك، كان لتركيزات النحاس الأولية الأعلى تأثير سلبي على ثابت المعدل. والجدير بالذكر أن ملامح انحلال التركيز التي لوحظت في ظل ظروف تشغيل مختلفة اتبعت الخواص الحركية من الدرجة الأولى، مع معاملات الارتباط التي تتجاوز ٠,٩٩. انبثق توضيح هذا الاكتشاف من تطابق ملحوظ ومذهل بين البيانات التجريبية والأسس الرياضية لنموذج الحركية من الدرجة الأولى. عززت هذه المواءمة المفاجئة بعمق من المتانة والصدق والموثوقية التي لا تتزعزع لنتائجنا التي تم الحصول عليها بدقة، مما زاد من مصداقية دراستنا الشاملة وموثوقيتها.

الكلمات الدالة: النحاس، النمذجة الحركية، الانتشار الجزيئي، استخلاص الغشاء الكهربائي، مياه الصرف الصحي.