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Electrochemical Extraction with Supported Liquid Membrane for Removing Copper from Synthetic Wastewater: Optimisation through Response Surface Methodology

Noor R. Kadhim[*], **Hussain M. Flayeh**[**], **Ali H. Abbar**[***]
and Hasan F. Al-azzawie[****]
[*,**]*Environmental Engineering Department, College of Engineering, University of Baghdad, Iraq*
[***]*Biochemical Engineering Department, Al-Khwarizmi College of Engineering, University of Baghdad, Iraq*
[****]*Lincoln university, Malysia*
[*]Email: noor.jabbar2011d@coeng.uobaghdad.edu.iq (Corresponding Author)
[**]Email: Hussain-altemmimi@coeng.uobaghdad.edu.iq
[***]Email: ali.abbar@kecbu.uobaghdad.edu.iq
[****]Email: hazzawie2@yahoo.com

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Abstract

In this study, copper was removed from an aqueous solution through electromembrane extraction (EME), a new approach that utilises a two-chamber electrochemical cell. It consists of two electrodes (stainless steel cathode and graphite anode) and a solid liquid membrane (SLM). SLM is composed of supporting polypropylene membrane impregnated with1-octanol as an organic solvent and bis(2-ethylhexyl) phosphate (DEHP) as a carrier. The effects of process parameters such as applied voltage, pH and copper concentration on the removal of copper were investigated. Response surface methodology (RSM) was applied to optimise these parameters and their interactions. Results indicated that pH has the major effect on the removal of copper, followed by applied voltage. The squared interaction term in the RSM model has the highest contribution (70.5%), followed by the linear term, thereby confirming the significant interaction among the variables. The optimum conditions include an applied voltage of 60V, pH of 5.18 and initial copper concentration of 5 ppm, which yield to a removal efficiency of 80% after 6 hours of operation. The findings demonstrate the use of electromembrane extraction as an efficient method for the removal of heavy metals and provide valuable insights for future application to other environmental and water treatment processes.

Keywords: electromembrane extraction, copper; SLM; RSM, DEHP;1-octanol

1. Introduction

Increasing environment pollution in the international ecosystem is a major concern that should be resolved [1]. Main contaminants of water are heavy metals, which cause several public health issues. Most heavy metals are present in the industrial wastewater and are highly resistant to degradation [2,3]. In addition to entering the body through the skin, these metals could enter through food and drink [4,5]. They are poisonous and can harm the brain and central nervous system as well as the kidneys, liver, lungs, blood and other essential organs [6].

Copper is a heavy metal that generates serious environmental hazards [6]. Wastewater from metal finishing, textile and electronic industries may contain copper at concentrations up to 500 mg/L. Based on the global environmental regulations, this concentration is higher than the permissible level; hence, wastewater containing copper must be treated before being discharged into the environment [7].

Copper ions are removed using different methods, such as adsorption, chemical precipitation, membrane separation and electrochemical methods (electrocoagulation and/or cathodic deposition) [8]. Each method has its own advantages and disadvantages, but most of them can be costly and incapable of treating low concentrations of copper ions [8,9].

Recently, membrane extraction technology is regarded as an effective separation technique. It combines the advantages of extraction and membrane separation. In this method, heavy metals can diffuse from a high-concentration chamber (donor phase) through a membrane to a low-concentration chamber (acceptor phase) as a result of concentration difference across the membrane. Liquid–liquid extraction underlies the operation of liquid membrane (LM) technique and exhibits advantages such as excellent selectivity, energy efficiency and environmental protection [10]. This method offers a high extraction efficiency for metal ions. Different types of membranes include bulk liquid membranes (BLM), emulsion liquid membranes (ELM), supported liquid membranes (SLM) and polymer inclusion membranes (PIM) [11,12].

SLM is based on solid polymer materials and does not require the extensive use of extraction agents or intricate steps involved in traditional approaches, such as demulsification and emulsion preparation. Its advantages in selectively separating and enriching low concentrations of heavy metals in high-matrix background are numerous, energy-efficient, and environmentally friendly [13].

Fast heavy metal transport from the donor phase to the acceptor phase can occur when an electric field is applied over SLM [14]. The apparatus is known as electromembrane extraction (EME), where the donor and acceptor phases are submerged in two platinum electrodes that are powered by a power source [13,14]. The two types of EME used for extraction of heavy metals are hollow fibre and flat membrane, and the latter is preferred for scale up [15]. In the removal of heavy metals, EME is applied to remove diverse cations for different solutions [16,17]. However, literature on the application of flat membranes for removing copper is limited. Moreover, the most suitable type for scale up is rare and has limited supply.

This work aims to apply a flat EME for removing copper from an aqueous medium on a laboratory scale. A brand-new two-chamber cell with a flat sheet membrane was adopted as an easy and cost-effective cell design because it used inexpensive electrodes (graphite anode and stainless-steel cathode) and SLM, instead of platinum electrodes. The effect of several parameters including applied voltage, donor pH and initial copper concentration on removal efficiency were investigated through response surface methodology (RSM). In this powerful experimental design, the interactions among variables can be recognised as impossible to be determined by the traditional one factor at one time approach, which requires a high number of runs.

2. Experimental

2.1 Chemicals

All chemicals used were of analytical grade. $CuNO_3$ was purchased from Central Thomas Baker, Mumbai, India. Deionised water was used as solvent to create acceptor and donor phase solutions. 1. Octanol was utilised as organic solvent in the liquid membrane and was obtained from Central Drug House, Delhi, India. Bis(2-Ethylhexyl) phosphate (DEHP) was purchased from Shanghai Macklin Biochemical and used as the carrier in SLM (Shanghai, China). A polypropylene membrane sheet with a pore diameter of 0.1 microns and a thickness of 0.18–0.22 mm was utilised as supportive liquid membrane. It was supplied by Jian City Qing Feng Equipment Material Co. in China. HCl and NaOH were provided by Thomas Baker, Ambernath, India.

2.2 Electromembrane extraction system

Figure 1 depicts the electrochemical apparatus, which consists of two symmetrical chambers with a volume of 137.44 cm^3 each. The first chamber was utilised as a donor phase, while the second chamber was utilised as an acceptor phase and had an active volume of 100 cm^3. Both chambers were 5 cm in diameter and 7 cm in length and had a lateral channel (3 cm in diameter and 3 cm in length) positioned 2 cm above the chamber base. Each chamber has a Teflon cup (4.8 cm in diameter and 10 mm in thickness). Graphite was adopted as the anode because it facilitates water decomposition by anodic oxidation[18]. Stainless steel, which is less expensive than platinum electrodes, was used as a cathodic material. SLM is composed of polypropylene membrane sheet with an active surface area of 7 cm^2 and was impregnated for 15 minutes with 1-octanol solvent and a suitable carrier. SLM was fixed in the space between the chambers and secured via two Teflon cups, four screws and netting. Two magnetic stirrers (Metrohm AG Herisau, Switzerland, Type: 18490010) were placed inside the chambers for mixing at 1000 rpm. The EME was run at various applied voltages using a UNI-T/UTP3000 power supply (Hong Kong). The exact current value was determined with a UNI-T connected ammeter (Kowloon, Hong Kong) [15].

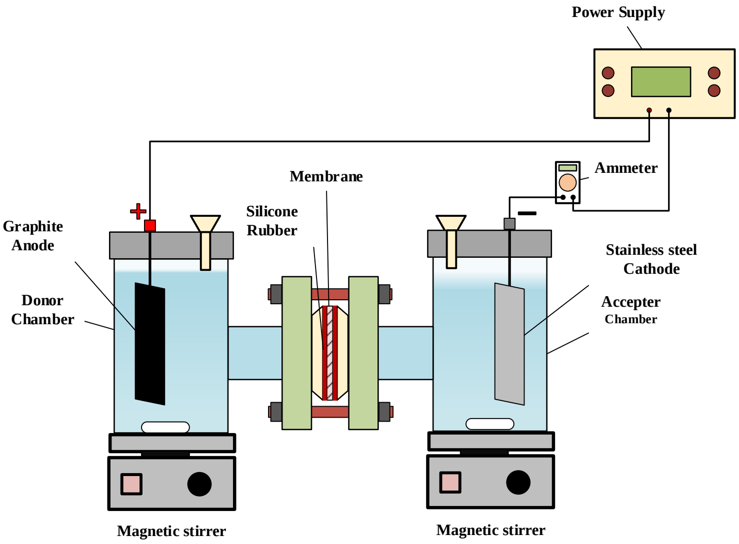


Fig. 1. EME system

2.3 Analysis and measurements

The samples were analysed using an AA-7000 atomic absorption spectroscope (SHIMADZU, Japan) to ascertain the concentration of copper ions.

The efficiency of copper removal was evaluated using Eq. 1 [18]:

$$RE(\%) = \frac{C_i - C_f}{C_i} \times 100 \qquad \dots (1)$$

where C_i is the initial concentration of Cu in part per million, and C_f is the final concentration of C_u measured after completing the run at time Δt.

In this work, the electrolysis time was set to 6 hours based on a preliminary run conducted at the midpoint, during which a jump did not occur in the current. This finding demonstrated that the liquid membrane did not begin to leak into the solution [19].

2.4 Experimental design

RSM based on Box–Behnken design (BBD) is a widely used experimental tool to correlate process variables and response in statistical and mathematical settings. Compared with the conventional one factor at a time strategy, RSM requires fewer runs, resulting in an experimental program that is more economical and time efficient [20].

BBD, which is based on three-level parameters, was used to optimise the process parameters. RE percent was used as response, and process variables included voltage (X1), pH (X2) and initial copper concentration (X3). The maximum value of each process variable was set at (+1), the middle value was set at (0) and the lower value was set at (-1) [21]. The process variables and their chosen values are shown in Table 1. An array of runs based on BBD was created using Minitab-17 software.

Table 1,
Process factors (coded and real levels).

Process factors	**Range in BBD**		
Coded levels	Low(-1)	Middle(0)	High(+1)
Applied voltage (Volt),X1	20	40	60
pH,X2	3	5	7
Concentration of copper (ppm),X3	5	15	25

A quadratic equation that relates the process factors with responses is shown in Eq. 2[22].

$$Y = a_0 + \sum a_i x_i + \sum a_{ii} x_i^2 + \sum a_{ij} x_i x_j \quad \ldots(2)$$

The intercept term is a_0, and j and i stand for the index numbers for the patterns. a_i and a_{ii} represent the first-order (linear) and second-order effects. The interaction effects are represented by a_{ij}. the coded process factors are $x1.,x2..,xk$. Finally, *Y* is the response term. Based on ANOVA, a regression coefficient (R^2) can be used to evaluate the fitting of Eq. 2.

3. Results and Discussion

3.1 Statistical results

RSM with BBD was used to simulate and optimise the removal of copper by EME. Fifteen runs were completed, and the results are displayed in Table 2. A second-order quadratic model was built using RSM in terms of real values of the parameters (Eq. 3). Table 2 also presents the anticipated values of RE derived from Equation 3.

$$RE\% = -37.62 + 0.134\,x_1 + 44.02\,x_2 - 2.062\,x_3 + 0.00301\,x_1 * x_1 - 4.050\,x_2 * x_2 + 0.04931\,x_3 * x_3 - 0.0378\,x_1 * x_2 + 0.00165\,x_1 * x_3 + 0.0336 x_2 * x_3 \quad \ldots(3)$$

The results showed that RE% ranged from 40.98% to 80%. As a preliminary evaluation of the effects of parameters, increasing the applied voltage increased RE% (compared between runs 5 and 12). Increasing the concentration of copper decreased RE% (compared between runs 5 and 11). Increasing the pH from 3 to 7 led to higher removal of RE% (compared between runs 6 and 2). However, the real effects of these parameters and their interaction were only assessed using ANOVA combined with response surface plots.

Table 2,
Optimisation of copper removal by RSM

Run Order	**Pt Type**	**Blocks**	**Applied potential (V)**	**pH**	**Initial concentration (ppm)**	**RE%**	
						Actual	**Predicted**
1	2	1	40	7	5	64.3	64.080
2	2	1	60	7	15	61.03	60.241
3	0	1	40	5	15	67.53	67.510
4	0	1	40	5	15	70	67.510
5	2	1	60	5	5	80	81.009
6	2	1	60	3	15	53.33	53.209
7	2	1	40	3	5	56.26	55.373
8	2	1	20	3	15	40.98	41.769
9	2	1	20	7	15	54.72	54.841
10	2	1	40	3	25	46.84	47.060
11	2	1	60	5	25	74.8	74.701
12	2	1	20	5	5	73.15	73.249
13	0	1	40	5	15	65	67.510
14	2	1	40	7	25	57.57	58.458
15	2	1	20	5	25	66.63	65.621

ANOVA was used to assess the acceptability of BBD. Fisher’s F and P tests were adopted to establish the significance of the model and its parameters [22]. In general, a high significance for the coefficient terms is indicated by large F values and small p values [23]. Table 3 presents the ANOVA for the response surface model.

Contr. % indicates the percentage contribution of each variable; DF denotes the degree of freedom of the model and its parameters; the terms “the adjusted mean of square (Adj. MS)”, “adjusted sum of square (Adj. SS)” and “sum of square (Seq. SS)” represent the statistical terms for the model. The estimated F value of 50.09 and P value of 0.0001 indicated the high significance of the regression model. The regression R^2 was 0.989 which is statistically significant and only (0.011) of the total variance not confirmed by the model [24].

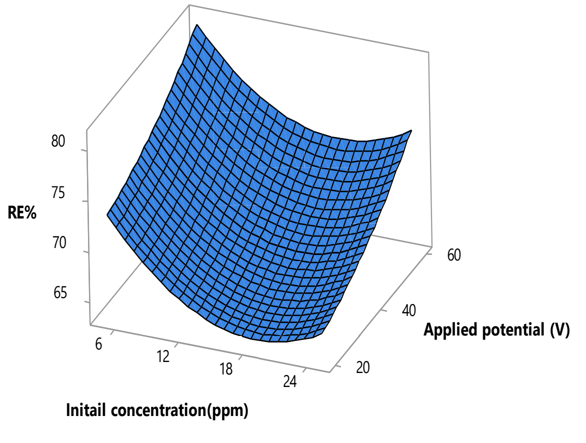
Table 3,
Analysis of variance for copper removal

Source	DF	Seq. ss	Contr.%	Adj. ss	Adj. Ms	F-value	P-value
Model	9	1578.01	98.90	1578.01	175.335	50.09	0.0001
Linear	3	440.99	27.64	440.99	146.997	42.00	0.001
(X1)	1	141.79	8.89	141.79	141.793	40.51	0.001
(X2)	1	202.11	12.67	202.11	202.106	57.74	0.001
(X3)	1	97.09	6.09	97.09	97.092	27.74	0.003
Square	3	1125.66	70.55	1125.66	375.218	107.20	0.0001
"X1 ∗ X1"	1	15.06	0.94	5.35	5.350	1.53	0.271
"X2 ∗ X2"	1	1020.81	63.98	968.86	968.860	276.80	0.0001
"X3 ∗ X3"	1	89.79	5.63	89.79	89.787	25.65	0.004
2-Way lnter.	3	11.37	0.71	11.37	3.788	1.08	0.436
X1 ∗ X2	1	9.12	0.57	9.12	9.120	2.61	0.167
X1 ∗ X3	1	0.44	0.03	0.44	0.436	0.12	0.739
X2 ∗ X3	1	1.81	0.11	1.81	1.809	0.52	0.504
Error	5	17.50	1.10	17.50	3.500		
Lack of Fit	3	5.00	0.31	5.00	1.667	0.27	0.847
"Pure-Error"	2	12.50	0.78	12.50	6.250		
Total	14	1595.51	100.00				
Model-summary		S.	R^2	R^2(adj.)	PRESS	R^2(pred.)	
		1.87088	98.90%	96.93%	108.133	93.22%	

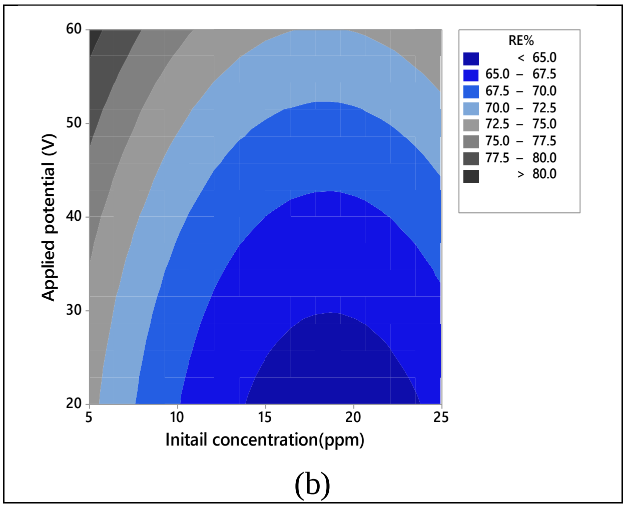
Table 3 shows that pH has a high contribution to the linear part of the model, followed by applied voltage and initial copper concentration. However, the model's second term showed a significant contribution of pH with high contribution of 63.98%. The applied voltage had a small impact with a contribution of 0.94%. The interaction term was the most dominant in the model, confirming that the relation between removal efficiency and operating parameters is not linear and a significant interaction exists. In this experiment, the lack of fit was statistically non-significant. The model produced reliable predictions that match well with the response values [25].

3.2 Influence of Process Factors on copper removal Efficiency

The effects of particular variables on responses can be seen visually with the use of RSM graphics. The effects of applied voltage and initial copper concentration on the removal efficiency of copper at a constant pH of 5 are displayed in Figures (2-a and 2-b). Figure 2-a shows the surface response plot, while Figure 2-b shows the contour plot. The surface plot verifies that for all copper concentrations tested, an increase in RE percent occurs as the applied voltage increases, and the relationship appears to be linear. This phenomenon may be explained by the enhancement in the mass transfer of copper across the membrane between the two phases due to the formation of more Cu (II)-DEEHPA complexes inside SLM, where their movements increased by applying higher voltages. Similar findings were published by Meng et al. in 2021 [26] when they used EME for cadmium removal. They also discovered that 6.0 V was the optimal applied voltage. Similar observation was found by Kadhim et al. in 2023[9] for the removal of zinc by using EME with SLM.



(a)



(b)

Fig. 2. Effect of applied potential and initial concentration on copper removal efficiency: a) surface plot, b) contour plot (hold: pH=5)

The percentage of RE decreased when the initial copper concentration decreased from 25 ppm to 15 ppm, after which it began to gradually increase as the concentration further decreased down to 5 ppm. An exponential relationship was found between RE percent and starting concentration when the copper concentration decreased from 5 to 15 ppm. These findings are consistent with the findings of Swain et al. (2007) [27] and are true even when in the absence of an electrical current. This work also demonstrated how well the chosen carrier, DEHP, facilitated the diffusion-driven passage of copper ions across the membrane. The results provide evidence for the effectiveness and suitability of DEHP as a carrier for EME [27]. Based on the associated contour plots, an area with an applied voltage between 58 and 60 V and an initial concentration of (5–6 ppm) led to 80% RE.

Figures 3-a and b show the effects of applied voltage and pH at an initial copper concentration of 15 ppm. The percentage of RE increased as the pH increased from 3 to 5 and then started to decrease thereafter (Figure 3-a). These results imply that the most apparent effective copper–DEHP bonding occurred at pH 5, suggesting that this is the ideal pH in 1-octanol [28]. Interestingly, DEHP undergoes more protonation when the pH is below 5, preventing it from effectively interacting with ions [26]. At high pH values, Cu (II) tends to form complex species, which hinders its passage across the membrane. Previous studies confirmed that releasing heavy metals into an acidic medium is better than releasing it in a neutral and/or an alkaline medium [28]. Meng et al. [26] reported the same behaviour in removal of Cd. Kadhim et al. [9] also stated similar behaviour in the removal of zinc using EME with SLM. Davarani et al. [15] found that an alkaline medium is preferred for the removal of Cd^{2+}, Zn^{2+} and Co^{2+} by using membrane containing mixed carriers (DEHP and TEHP).

The corresponding contour plots (3-b) indicate that RE% ≥70% occurred in a limited area with an applied voltage of 50–60 V and a pH range of 4.5–5.5.

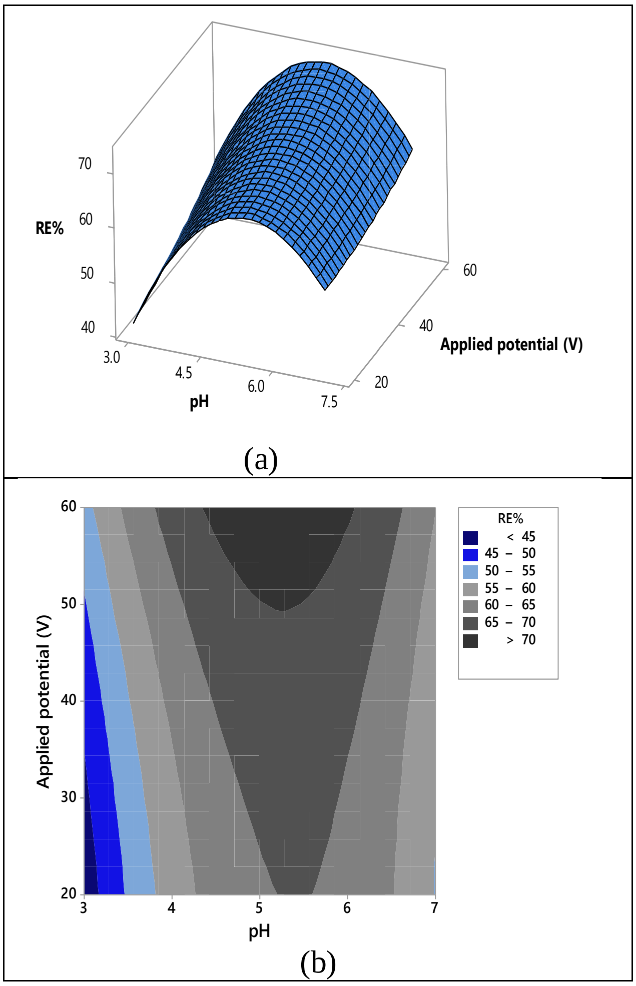


Fig. 3. Effect of applied potential and pH on copper removal efficiency: a) surface plot, b) contour plot (Hold value: copper intail concentration of 15 ppm)

Figures (4-a and b) shows the effect of initial concentration of copper and pH on RE%. A clear interaction was found between the pH and concentration of copper. In this regard, the ideal conditions differ from those found when applying the conventional one component at a time method. The corresponding contour plot (Fig. 4-b) shows that RE ≥75% exists within a limited region bounded by initial concentration ranging from 5 ppm to 6 ppm and pH from 4.5 to 5.5). Hence, applying optimisation techniques would lead to the optimum value within the limits established.

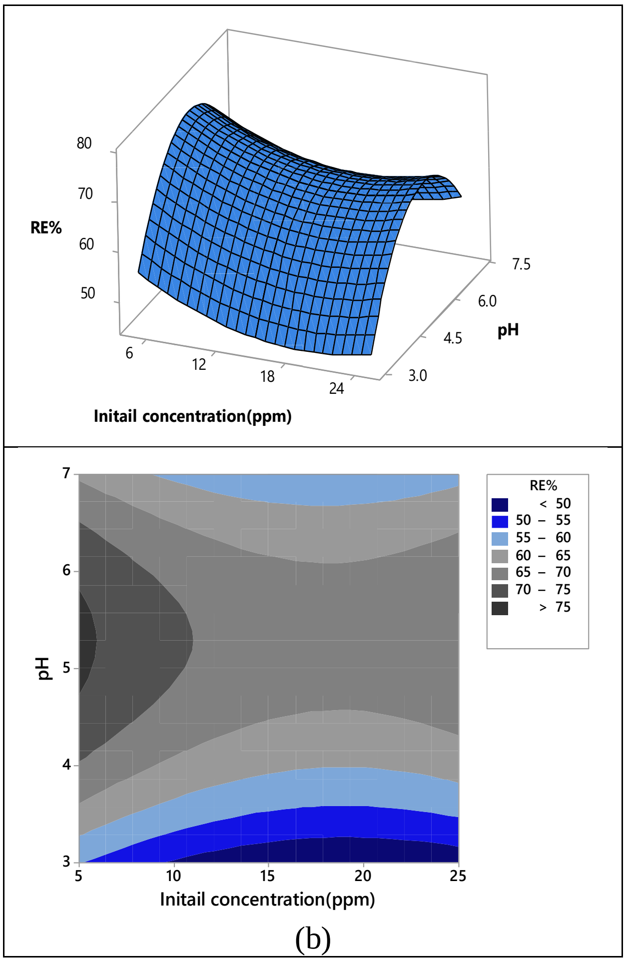


Fig. 4. Effect of pH and initial concentration on copper removal efficiency: a) surface plot, b) contour plot

3.3 Optimisation and confirmation test

Optimisation is a crucial step used to determine the optimum conditions that yield high removal with low energy consumption in electrochemical systems. During optimisation of any system, many factors should be considered to maximise desirability function (DF) and reach the intended outcome [28]. Options for optimisation were none, within the limit or maximise and minimize. With DF=1.0, the RE objective was set to 'maximum'. After optimisation, the outcomes are displayed in Table 4. A confirmative run was carried out to verify the optimal conditions (Table 5). A RE of 80% was attained after electrolysis for 6 hours, starting with a copper concentration of 5 ppm and pH of 5.18 at 60 V. Consequently, BBD is helpful and efficient in determining the impacts of process variables and increasing the removal efficiency to an expected level.

Table 4,
Process parameter optimisation for copper removal

Response	**Goal**	**Lower**	**Target**		**Upper**	**Weight**	**Importance**
RE (%)	***maximum***	**40.98**	**Maximum**		**80**	**1**	**1**
Solution: **Parameters**			**Results**				
E (Volt)	**pH**	**Cu^{2+} (ppm)**	**RE (%) Fit**	**D_F**	**SE Fit**	**95% CI**	**95% PI**
60	5.18182	5	81.1334	1	1.62	(76.96, 85.31)	(74.77, 87.50)

Table 5,
Confirmative run

Run	**E (Volt)**	**pH**	**Time (hr)**	**Cu^{2+} (ppm)**		**RE (%)**
				Initial	**Final**	
1	60	5.18	6	5	1.6	80

The concentration of copper decreased to 0.05 ppm by refreshing the membrane with a new solvent and carrier.

4. Conclusions

EME was successful in removing copper from an aqueous solution at high efficiency. The studied parameters were applied voltage, pH and copper concentration. RSM was used to determine the interactions among process parameters. Results revealed that the squared interaction had the most contribution to RE (70.5%), followed by the linear term while two-way interaction had no significant effect. pH had the greatest influence on removal. The optimum conditions were an applied voltage of 60, pH of 5.18 and an initial copper concentration of 5 ppm. The removal efficiency of 80% was achieved during 6 hours of operation when the copper concentration was reduced to 1.6 ppm. This work confirms the vital role of EME for removing heavy metals.

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الاستخلاص الكهروكيميائي باستخدام الغشاء السائل المدعوم لإزالة النحاس من مياه الصرف الاصطناعية: التحسين من خلال منهجية سطح الاستجابة

نور راغب كاظم*، حسين فليح مجيد ، علي حسين عبار***، حسن العزاوي******

*قسم هندسة البيئة، كلية الهندسة، جامعة بغداد، بغداد، العراق**،**
*قسم الهندسة الكيميائية الاحيائية، كلية الهندسة الخوارزمي، جامعة بغداد، بغداد، العراق****
*جامعة لنكولن، ماليزيا*****
*البريد الالكتروني:noor.jabbar2011d@coeng.uobaghdad.edu.iq
**البريد الالكتروني:Hussain-altemmimi@coeng.uobaghdad.edu.iq
***البريد الالكتروني:ali.abbar@kecbu.uobaghdad.edu.iq
****البريد الالكتروني:hazzawie2@yahoo.com

المستخلص

في هذه الدراسة، تمت دراسة إزالة النحاس من محلول مائي باستخدام استخلاص الغشاء الكهربائي (EME) كنهج جديد تم فيه استخدام الخلية الكهروكيميائية ذات الغرفتين. تتكون الخلية من قطبين كهربائيين (كاثود الفولاذ المقاوم للصدأ وأنود الجرافيت) فضلاً عن غشاء سائل صلب (SLM). يتكون SLM من غشاء بولي بروبيلين داعم مشرب بـ 1-أوكتانول كمذيب عضوي وفوسفات مكرر (2-إيثيل هكسيل) (DEHP) كحامل. وتمت دراسة تأثير معلمات العملية مثل الجهد المطبق، ودرجة الحموضة، وتركيز النحاس في إزالة النحاس. وتم تطبيق منهجية سطح الاستجابة (RSM) لإيجاد الظروف الأمثل لهذه المعلمات وكذلك التفاعلات فيما بينها. أظهرت النتائج أن الرقم الهيدروجيني له تأثير كبير في إزالة النحاس يليه الجهد المطبق. يمتلك مصطلح التفاعل التربيعي في نموذج RSM أكبر مشاركة بنسبة 70.5٪ يليه المصطلح الخطي الذي يؤكد وجود تفاعل كبير بين المتغيرات. وكانت الظروف الأمثل هي الجهد الكهربي المطبق 60 فولتاً، ودرجة الحموضة 5.18، وتركيز النحاس الأولي 5 جزء في المليون إذ تم تحقيق كفاءة إزالة قدرها 80% خلال 6 ساعات من التشغيل. توضح نتائج هذا العمل نشاط استخراج الغشاء الكهربائي كوسيلة فعالة لإزالة المعادن الثقيلة التي توفر رؤى قيمة للتطبيق المستقبلي لهذه الطريقة في العمليات البيئية ومعالجة المياه الأخرى.
