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Process Optimization Study of Pb(II) Removal by Bulk Liquid Membrane (BLM)

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Abstract

Box-Wilson experimental design method was employed to optimized lead ions removal efficiency by bulk liquid membrane (BLM) method. The optimization procedure was primarily based on four impartial relevant parameters: pH of feed phase (4-6), pH of stripping phase (9-11), carrier concentration TBP (5-10) %, and initial metal concentration (60-120 ppm). A maximum recovery efficiency of lead ions is 83.852% was virtually done following thirty one-of-a-kind experimental runs, as exact through 2^4-Central Composite Design (CCD). The best values for the aforementioned four parameters, corresponding to the most restoration efficiency were: 5, 10, 7.5% (v/v), and 90 mg/l, respectively. The obtained experimental data had been utilized to strengthen a semi-empirical model, based on a second-degree polynomial, to predict recovery efficiency. The model was tested using ANOVA software (Design expert®) and found acceptable R-Squared were (0.9673). Yield response surface and contour plots have been created using the developed model, which revealed the presence of high-recovery plateaus whose specs will be useful in controlling pilot or industrial scale future devices to ensure economic feasibility.

Keywords: Lead ions, Xylene oil, Response Surface Methodology, Analysis of Variance.

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1- Introduction

Pollution of water by toxic pollutants is one of the most serious troubles in the treatment of industrial wastewater, which has been studied by way of many researchers for many years [1, 2]. Exposure to heavy metals has direct and serious consequences toxicity to human health and the environment [3].

Industrial wastewater always contains heavy metals like lead, nickel, cadmium etc. from anthropogenic industries, such as Petrochemicals, mining activities, batteries, pulp, paper, alloys, steel, pigment paint and fertilizer [4] [5]. Most techniques can be employed to remove toxic contaminates from synthetic wastewater such as solvent extraction, bio sorption, chemical precipitation, coagulation [5], ion-exchange, adsorption [6], Electrolysis [7], membrane filtration [8], adsorption [9, 10] etc.

These techniques have various disadvantages for example, sensitive operating conditions, high capital and operating costs, low selectivity, less efficiency, provide considerable amount of sludge, and further the disposal is a costly fair [11, 12].

Hence, more effective treatment techniques are sought after to overcome these difficulties.

Recently, separation techniques such as liquid membrane, invented by Li [2] in 1968, have been widely used for heavy metals separation from industrial wastes [13].

A few advantages of Liquid membrane such as high purification ease of operation, high selectivity high throughput, clean-up efficiency and the requirement of only small quantities of organic solvent [14] [15] [16]. Among between types of liquid membrane, bulk liquid membrane (BLM), is the most well-known and the simplest design for performing liquid membrane processes [17].

A bulk liquid membrane (BLM) consists of the feed and stripping phases separated by membrane organic, water immiscible liquid phase [16]. Researchers have studied the few parameters affecting the separation efficiency, such as effect of pH on the feed and stripping phases, organic to aqueous phase ratio, mixing time, initial concentrations, carrier concentration, salt concentration, etc. [17, 18]. Most of them studied one factor at a time. In actual, this method would possibly no longer reach the actual greatest stipulations [19].

To overcome this problem, the current work utilizes experimental design methods centered composite design (CCD) and response surface methodology (RSM) to look at of different parameters for the best lead ions extraction from synthetic wastewater.

The benefits of design experimental consist of simultaneous study of several variables, low cost, faster to implement and convenient [20].

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To our knowledge, there has not longer been any work on the use of statistical methods to optimized the procedure parameters of Pb(II) extraction through natural solvents such as xylene oil. The aim of this study is to adopt Box-Wilson design to optimize the process parameters: pH-feed phase, pH receiving phase, carrier concentration, and initial feed concentration for maximum lead ions removal efficiency by bulk liquid membrane (BLM) method. CCD design was utilized to reveal the impact and relation among technique parameters to attain the aforementioned goal.

2- Material and Methods

2.1. Material and Equipment

Lead nitrate ($Pb(NO_3)_2$) (99%, purity) (CDH Chemicals Ltd company, INDIA) was used in feed phase. Xylene oil $(CH_3)_2C_6H_4$ (98% purity) and tri-n-butyl phosphate (TBP) (99% purity) was used in membrane phase. Sodium hydroxide (NaOH) (98% purity), hydrochloric acid (HCl) (98% purity) was used to adjust pH in feed and stripping phases. A hotplate magnetic stirrer (Ika, Germany) (30-120 rpm) was used to combine the carrier and solvent organic, while a pH meters (WTW, Germany) used for pH measurement in stripping and feed phases. The concentration of Pb(II) in feed and striping phases was once decided with a flame atomic absorption spectrophotometer, AAS: (GBC, Germany).

2.2. Preparation of Aqueous and Organic Phases

Fig. **1** suggests the schemetic daigram of the BLM system used in this study. It consists of three phases, namely, feed phase, stripping phase and membrane phase. Both of the feed and striping phases are separated with a strong impermeable wall and layered on top by way of the membrane phase. An aqueous feed phase of various initial lead ions concentrations was prepared in distilled water. While, an aqueous strip phase containing distilled water. Finally, the organic membrane phases were prepared by loading tri-n-butyl phosphate (TBP) as a carrier dissolved in xylene oil. The pH of feed and stripping phases was adjusted to the desired value by adding drops of NaOH or HCl. Extraction Percentage (% E) of heavy metals was calculated according to eq (1):

$$\% \ E = \frac{C_{F^0} - C_F}{C_{F^0}} \times 100 \quad (1)$$

Where: C_{F^0} and C_F are the initial and final concentrations in the feed phase, respectively.
On the other hand, recovery percentage (% S) of Pb(II), was calculated by eq. (2):

$$\% \ S = \frac{C_S}{C_{F^0} - C_F} \times 100 \quad (2)$$

Where: Cs is the final concentration in the striping phase.

During the anelysis, the preliminary concentretions of Pb(II) in each the membrane and stripping phases were asseumed zero.

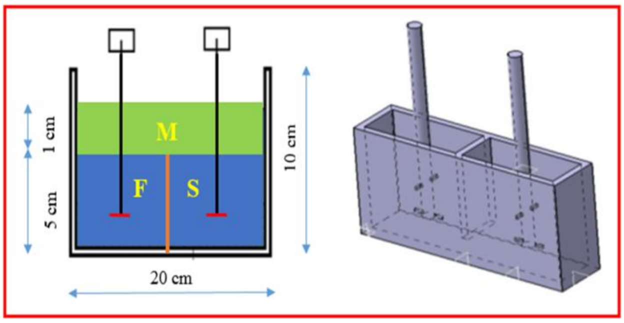

Fig. 1. Bulk liquid membrane system used in this work

3- Design of Experiment

3.1. Central Composite Design

Ever since the introduction of central composite design by Wilson and Box in 1951, it has been the most widely accepted and used experimental design for the first order and second order models [21]. There are four factors considered in this research. Central composite design (CCD) was used as a method to screen out factors that were statistically important [22]. These important parameters, namely pH feed (4-6), pH stripping (9-11), initial concentration of metal ions (60-120) and carrier concentration (TBP) (5-10), in order to determine the superior conditions for lead ions removal from synthetic wastewater. The variable was coded according to the eq (3).

$$X_i = \frac{X_i - X_0}{\Delta X_i} \quad (3)$$

Where: X_i is the coded variable, xi the real value of an independent variable, X_0 the midpoint value of the i variable range and ΔX_i he difference of the limiting two values of the i variable. The step size is the half value of the difference. The total number of design experiments is given by the eq. (4):

$$N = n_j + n_a + n_0 = 2^K + 2K + n_0 \quad (4)$$

Where: N is the total number of design experiments, n_0 is number of center points, nj is number of star points ($n_j = 2^K$) and $n\alpha$ is number of axial points ($n_a = 2K$). The factorial design have two axial points on the axis of each design variable at a distance of $a = \pm\sqrt{K}$. In this study there are 4 variables, that means ($K = 4$).

Thus, ($n_j = 2^K = 16$), ($n_a = 2(4) = 8$), $n0$ for this design is taken as 6, $a = \pm\sqrt{4} = 2$, and $N = 30$ experiments. The names and levels of the 4 independant method parameter, upon which 30 experiments of the CCD matrix have been based, are proven in Table **1** Whereas the real and coded range and level value of these variables are given in Table **2**.

Table 1. Names and levels of process factor (parameters)

Parameters	Symbol	Units	Lead	
			Low	High
pH Feed	A	….	4	6
pH Stripping	B	…..	9	11
Initial Concentration	C	mg/l	60	120
Carrier Concentration	D	%	5	10

Table 2. Real and coded ranges and levels values for independent variables

Independent Parameter	Lead				
Coded Real	Levels				
	-2	-1	0	+1	+2
pH Feed	3	4	5	6	7
pH Stripping	8	9	10	11	12
Initial Concentration	30	60	90	120	150
Carrier Concentration	2.5	5	7.5	10	12.5

3.2. Fitting a Second-Order Response Surface

When there is a curvature in the response surface the first order model is insufficient. Therefore, second order model is beneficial in approximating a portion of the true response surface with curvature. The second order model consists of all the terms in the first order model, and quadratic and cross product terms. It is generally represented as eq. (5):

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^{k} \beta_{ij} x_i x_j + \varepsilon \quad (5)$$

Where: Y is the predicted response (% lead ions removal), β_0 is the intercept (offset) term, β_i, β_{ii}, β_{ij} are the first-order, quadratic and cross-products of X_1, X_2, X_3 and X_4 on response, x_i and x_j are variables, i and j are the index numbers for a parameters, k is the number of factors and ε is the residual [23].

4- Results and Discussion

4.1. Design Matrix Used and Responses Measured

Response Surface Methods are models and designs for working with continuous treatments when finding the optimal or describing the response is the goal [24].

Table 3 shows the design matrix of CCD used in this work.

According to 2^4 – CCD, 30 trial runs were adjusted for lead ions removal in the mono-system which are composed of 16 factorial factors (Std order 1–16), 8 axial points (Std order 17–24) and 6 replicates of the center point (Std order 25–30). The table included two readings for the heavy metals removal; experimental and predicted removal.

The experimental removal come from the experimental work according to suggested conditions by design expert ®11 to independent variables.

While the predicted removal come from analysis of data by using the ANOVA. After the statistical parameters were estimated for all the readings, the fitting equation was obtained as follow (6):

$$\text{Removal of } Pb^{+2} = 81.83 + 0.69\,(A) - 0.93\,(B) + 2.22\,(C) - 3.78\,(D) + 1.06\,(AB) + 0.77\,(AC) + 0.5(AD) + 0.85\,(BC) - 1.12\,(BD) - 0.091\,(CD) - 2.08\,(A^2) - 1.49\,(B^2) - 3.28\,(C^2) - 4.84\,(D^2) \quad (6)$$

In eq. (6) the high quality coefficients show the compatible effects on the responses while the negative coefficients show their incompatible effects.

The adequacy of these models used to be assessed and they have been determined to be statistically ample from analysis of variance.

For %R model (Eq. 6), coefficient of determination (R^2) and adjusted R^2 (R^2adj) of 0.9673and 0.9368 were obtained. The lack-of-fit of these models were additionally assessed and it was determined to be significant.

Table **3** shows the design matrix of CCD used and the average response %Removal measured in this work. It consists of a complete of 30 runs which have been performed randomly to keep away from the effect of the uncontrolled factors.

All experiments have been carried out under homogeneous condition in one block of measurement. The %R was found to vary from (55.554– 83.85%).

The readings show a good matching between results experimental and predicted for lead ions. The quadratic model equations were developed by ANOVA for the extraction and stripping efficiencies.

The analysis of variance (ANOVA) showed magnitude of the Model F-test (31.72) and p-value (< 0.0001) for Pb(II), respectively, implies the model is significant fit. Furthermore, the lack of fit sum of squares was (36.24) and their R-Squared were (0.9673), respectively, which are acceptable.

Values of Probability ($F < 0.05$) indicate model terms are significant, while, values greater than 0.1, which confirm that the model terms are not significant.

Table 3. Number of runs, experimental and predictable values of lead ions

Run	Coded values				Actual values				Response	
	X1	X2	X3	X4	A: pH F	B: pH S	C: In Conc. mg/l	D: Carrier Conc. %	Experimental R. of Pb (%)	Predicted R. of Pb (%)
1	0	0	0	0	5	10	90	7.5	82.659	81.83
2	1	1	1	-1	6	11	120	5	71.528	71.731
3	0	0	0	0	5	10	90	7.5	82.659	81.83
4	-1	1	-1	-1	4	11	60	5	63.528	62.909
5	0	0	0	0	5	10	90	7.5	81.734	81.83
6	-1	-1	1	-1	4	9	120	5	67.178	67.731
7	-1	-1	-1	-1	4	9	60	5	66.983	66.349
8	-2	0	0	0	3	10	90	7.5	72.638	72.13
9	-1	1	-1	1	4	11	60	10	68.329	67.411
10	1	-1	1	1	6	9	120	10	79.502	78.149
11	1	1	-1	1	6	11	60	10	70.865	70.371
12	1	-1	-1	-1	6	9	60	5	64.331	63.069
13	0	2	0	0	5	12	90	7.5	75.185	74.01
14	0	0	0	0	5	10	90	7.5	80.047	81.83
15	0	0	2	0	5	10	150	7.5	76.462	73.15
16	0	0	0	0	5	10	90	7.5	83.852	81.83
17	-1	-1	1	1	4	9	120	10	76.22	76.349
18	0	0	0	2	5	10	90	12.5	69.931	70.03
19	1	-1	-1	1	6	9	60	10	75.465	74.051
20	0	0	-2	0	5	10	30	7.5	61.446	64.27
21	0	-2	0	0	5	8	90	7.5	77.074	77.73
22	0	0	0	0	5	10	90	7.5	80.047	81.83
23	1	1	1	1	6	11	120	10	76.749	77.869
24	-1	1	1	1	4	11	120	10	70.502	71.829
25	-1	1	1	-1	4	11	120	5	65.815	67.691
26	1	-1	1	-1	6	9	120	5	66.157	67.531
27	2	0	0	0	7	10	90	7.5	74.907	74.89
28	-1	-1	-1	1	4	9	60	10	75.465	75.331
29	0	0	0	-2	5	10	90	2.5	55.554	54.91
30	1	1	-1	-1	6	11	60	5	63.528	63.869

The "Pred R-Squared" of (0.8465) is in reasonable agreement with the "Adj R-Squared" of (0.9368), respectively. Adequate Precision measures the signal-to noise ratio. A ratio greater than 4 is desirable. The ratio of (21.265) indicates an adequate signal for lead ions, respectively. Therefore, this model can be used to navigate the design space.

4.2. Graphical Analysis

The response surface method has been used to analyze the three-dimensional response plot produced from the effect of all the process variables on pb^{+2} by bulk liquid membrane from aqueous solution. It is evident from the ANOVA table that all the individual process variables are significant in increasing the effectiveness of the percentage removal (Y).

The first purpose for response surface method is to discover the highest quality response. When, there is greater than one response then it is essential to locate the compromise ideal that does not optimize only one response [24].

When there are constraints on the design data, then the experimental plan has to meet necessities of the constraints.

The 2nd goal is to understand how the response adjustments in a given course via adjusting the design variables. In general, the response surface can be visualized graphically. The layout is helpful to see the form of a response surface; hills, valleys, and ridge lines. Hence, the characteristic f (x1, x2) can be plotted versus the levels of x_1 and x_2 as shown in Figures 2-7 [25].

The resulting graphics gave an excellent clarification for the effects of pH feed and pH stripping, initial metals concentration and carrier concentration. Fig. 2-7 illustrates the collective effect of all the independent parameters on the percentage removal of pb^{+2}. All of the values on the axis of the figures represent the real values.

a. Effect of PH Feed and PH Stripping

Fig. **2** shows the interacting effect of the feed phase pH (X1) and striping phase pH (X2) are play major role in lead ions pb^{+2} removal at 5 hr. This dimensional plan shows the response surface from the facet and it is called a response surface plot.

Sometimes, it is less difficult to view the response surface in two-dimensional graphs. The contour plots can show contour strains of x1 and x2 pairs that have the same response value y.

The high-removal efficiency plateau exists in the surface over a pH feed range of (4.9-5.3) and a pH stripping range of (9.5-10) for Pb (II) respectively. In the mildly condition, metal ions are ionized and cations pb^{+2} are available in the feed phase for subsequent complexation at feed membrane interface.

Thus, a pH gradient between the source and the receiving phases is the driving force for the transport of pb^{+2} through the liquid membrane.

Therefore, it is needed that pH of the stripping phase is higher than pH of the feed phase for the effective transport efficiency [26-27].

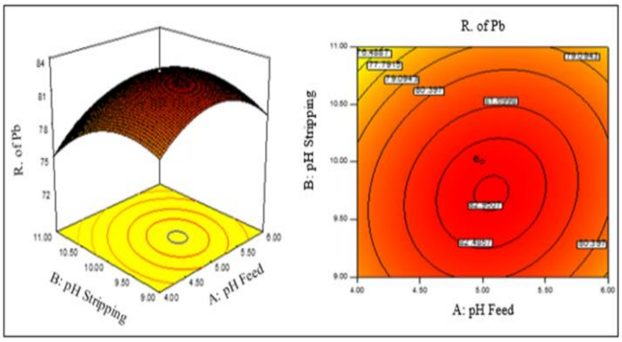

Fig. 2. Response surface plot, Contour plot of the Interacting effects of pH feed and pH stripping on lead removal

b. Effect of pH feed and Initial Metals concentration

Fig. **3** demonstrates the mutual effect of pH feed and inetial metals concentretion on the removal efficeincy for Pb (II) for the mono-system, as a response surface and contour plot.

The mass transfer rate of lead ions is increased with increasing initial feed concentration of metal ions in the aqueous phase at optimum pH and concentration.

Where, the maximum recovery efficiency appears over a pH feed range of (4.92-5.28) and Initial metals concentration range of (89.25-103.75 mg/l), respectively.

However, the percentage transport of lead ions decreases with increasing initial concentration, may be due to the driving force for the metal ions transport between the aqueous phases remains low.

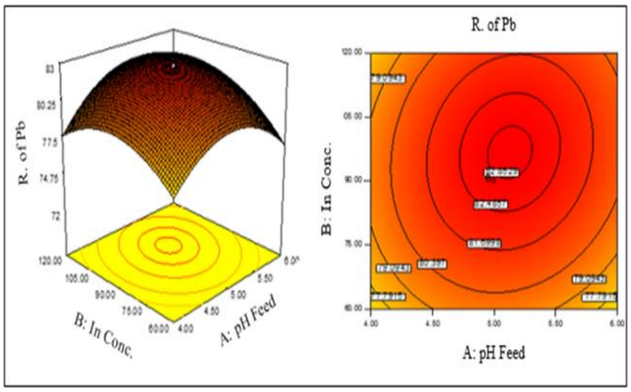

Fig. 3. Interacting effects of pH feed and initial metals concentration on lead removal: surface plot; contour plot

c. Effect of PH Feed and Carrier Concentration

Fig. **4** shows that maximum lead ions removal efficiency plateau exists in the surface for the mono-Pb (II) system, as a response surface and contour plot, over pH feed range of (5.06-5.23) and carrier concentration range of (8.38-8.85 %) (v/v), respectively. That effectiveness of membrane transport will increase as carrier concentration increases and gets saturated at some point and after that carrier concentration delays diffusion rate as viscosity of membrane increases [17]. Where, in presence of a carrier as (TBP) in the membrane phase, a metal-carrier complex (Pb-TBP complex in this case) is formed at the feed membrane interface which results in the increase of mass transfer rate through the interface and hence higher separation is achieved. On the other hand, at higher pH values there was a decrease in the transport rate of lead ions this decline can be due to a decrease in the hydrogen ion concentration with increasing pH which can cause a decline in the formation of the carrier and the metal ions complex, due to the resulting in the increase in the solubility of TBP in the aqueous feed phase main to the membrane bleeding [30, 31].

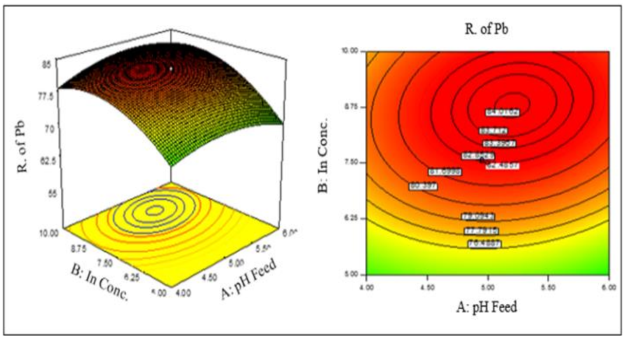

Fig. 4. Interacting effects of pH feed and Carrier concentration on lead removal: surface plot; contour plot

d. Effect of Ph Stripping And Initial Metals Concentration

In Fig. **5** the combined interaction of effect of pH stripping and initial metal concentration on lead ions removal efficiency in the mono-Pb`(II) system is shown, as a response surface and contour plot. Where, the effect of pH of the stripping phase and initial concentration on the receiving of lead ions at aqueous phase through bulk liquid membrane, was measured within the range pH stripping phase from (4 to 12) and (40 to 150ppm) for initial concentration, respectively. As shown, the high-removal efficiency plateau corresponds to effect of pH stripping range of (9.66–9.89) and initial metals concentration range of (91.25–99.47 mg/l), respectively. Observed, that membrane phase rapidly got saturated with the metal ions concentration affecting mass transfer. Therefore, the significant decrease in the stripping efficiency, located at lower and higher pH values could be related to the decreased decompiling ability of carrier between the membrane and stripping interface [32, 33].

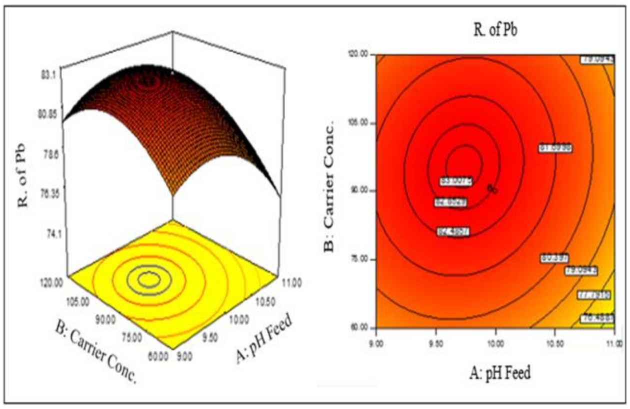

Fig. 5. Interacting effects of pH stripping and initial metals Concentration on lead removal: surface plot; contour plot

e. Effect of Ph Stripping and Carrier Concentration

It is obvious from the observed results in Fig. **6**. The effect of pH stripping and carrier concentration on the efficiency of lead ions removal for the mono-Pb (II) system, as a contour plot and response surface, is presented. It can be found that a high-removal efficiency plateau exists in the surface over effect of pH stripping range of (9.45–9.79) and a carrier concentration range of (8.09–9.17) % (v/v), respectively. The decrease of recovery efficiency at lower and higher pH values lead to due to the saturation of driving force for diffusion through xylene oil based bulk liquid membrane due to the complex formed by the TBP effect in the organic membrane at optimum carrier. It observed at lower and higher pH value could be associated to the reduced decompiling capacity of carrier between the membrane and stripping interface [28].

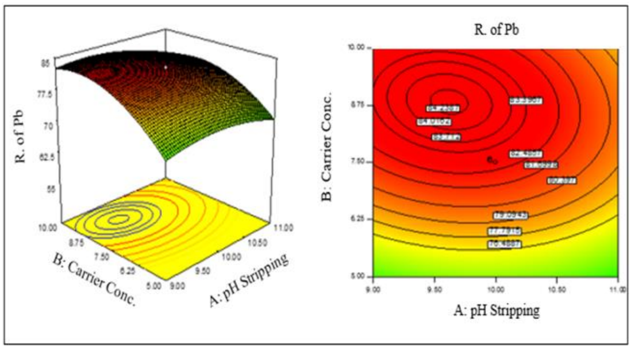

Fig. 6. Interacting effects of pH stripping and Carrier concentration on lead removal: surface plot; contour plot

f. Effect of Initial Metals Concentration and Carrier Concentration

The effect of initial metals concentration and carrier concentration on the transport efficiency for lead ions for the mono-Pb(II) system, as a response surface and contour plot, is presented by means of Fig. **7**.

It can be observed that a high-removal efficiency plateau exists in the surface over an initial metals concentration range of (91.11-99.23 mg/l) and a carrier concentration range of (8.37-8.94) % (v/v) for Pb(II), respectively. It may be due to the competition between lead ions at very high concentrations; therefore, the organic extracting (carrier) cannot able to transport the metal ions from the aqueous source phase to receiving phases [29].

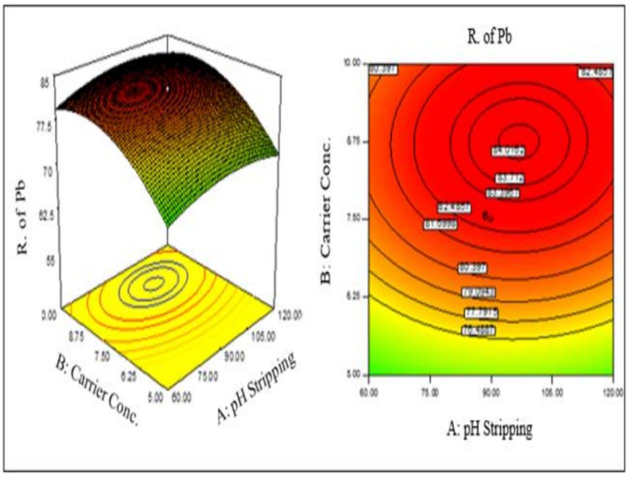

Fig. 7. Interacting effects of Initial Metals Concentration and Carrier Concentration on lead removal: surface plot; contour plot

5- Conclusion

In the present study, four variables central composite design based the response surface method (RSM) has been employed to investigate the individual as well as the combined effect of independent variables on lead removal by bulk liquid membrane from aqueous solution. The study revealed that all the parameters, i.e., pH feed and stripping phases, initial metals concentration and carrier concentration. Individually affect the BLM process significantly compared to the combined effect. With the help of design expert, the regression analysis and variables optimization were calculated to predict the significant response in the experimental domain.

A large number of experimental data at a wide range of independent variables were employed to carry out the regression analysis, statistical importance, and response surface analysis. A simple second order quadratic model equation was developed using design expert software for predicting the response (percentage removal of Pb (II)) on overall experimental regions and correlate the operating parameters and removal efficiency of Pb (II).

The coefficients of the developed model were calculated for each response, and the high acceptability of the postulated model was proven by presenting the statistical specifications of them. The reliability of the developed model has been ensured from the high magnitude of the correlation coefficient R^2 (0.9673) and R^2 (adj) (0.9368)) between the experimental and model predicted values.

The response surface was derived based on the developed model. pH feed and stripping phase was found as the most significant factor among all the process variables. The optimum condition for maximum Pb (II) removal was: feed phase pH 5, stripping phase pH 10, carrier concentration 7.5 %(v/v), and initial concentration 90 ppm. The experimental recovery efficiency 83.852% obtained under the optimum operating conditions agrees well with the predicted recovery efficiency one 81.83%.

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دراسة عملية التحسين لأزالة أيونات الرصاص بواسطة الغشاء السائل الحجمي (BLM)

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الخلاصة

تم استخدام طريقة التصميم التجريبي Wilson–Box لتحسين كفاءة أيونات الرصاص بطريقة الغشاء السائل الحجمي (BLM). استند أجراء التحسين في المقام الأول الى أربع معلمات محايدة ذات صلة وهي درجة الحموضة في مرحلة التغذية (6–4)، ودرجة الحموضة في مرحلة التعرية (11–9) وتركيز الناقل (10–5) % TBP، وتركيز المعدن الأولي (120–60). تم تحقيق أقصى استعادة من كفاءة أيونات الرصاص بنسبة 83.852% تقريبا بعد 30 تجربة فريدة من نوعها. كما هو دقيق من خلال التصميم المركب المركزي ذي 2^4. وكانت أفضل القيم للمعلمات المذكورة أعلاه، والتي تتوافق مع معظم كفاءة الاستعادة، 5، 10، 7.5 %(v/v) و90 مجم / لتر، على التوالي. وقد تم استخدام البيانات التجريبية التي تم الحصول عليها لتعزيز نموذج شبه جريبي، على أساس متعدد الحدود من الدرجة الثانية للتنبأ بكفاءة الانتعاش. تم اختبار النموذج باستخدام برنامج ANOVA Design Expert ووجد ان R–Squared مقبولة (0.9673). تم إنشاء مؤامرات لسطح استجابة الغلة ومحيطها باستخدام النموذج المتطور، والذي كشف عن وجود هضاب عالية الاسترداد تكون مواصفاتها مقيدة في التحكم في الأجهزة المستقبلية ذات النطاق التجريبي او الصناعي لضمان الجدوى الاقتصادية.

الكلمات الدالة: أيونات الرصاص ، زيت الزايلين ، منهجية استجابة سطح ، تحليل التباين.