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Factorial screening of platinum removal using flat sheet supported liquid membrane

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ABSTRACT

Excessive platinum (Pt) discharged into the environment from industrial wastewater must be effectively treated since it contaminates the environment and causes substantial environmental problems and severe health risks to humans. Flat sheet supported liquid membrane (FSSLM) was used to remove Pt from an aqueous solution using liquid membrane formulated from Aliquat 336 carrier in kerosene. Fractional factorial design (FFD) was applied to screen five FSSLM operation parameters, including the Aliquat 336 carrier concentration (factor A), Pt feed phase concentration (factor B), sodium perchlorate stripping phase concentration (factor C), feed flow rate (factor D) and stripping phase flow rate (factor E). The percentage of Pt removal from an aqueous solution was the response in the FFD. The feed flow rate (factor D) was the most significant main factor to the FSSLM performance, with 25.02% of the contribution. The interaction between factor AC and factor BE induced the most significant effect with 26.95% and 12.14% of the contribution, respectively. The highest removal percentage for Pt via the FSSLM process was 98.7% using 0.5 M Aliquat 336 concentration, 5 mg/L feed concentration, 0.5 M stripping concentration, 100 mL/min feed flow rate, and 50 mL/min stripping flow rate.

Keywords: Factorial screening; Supported liquid membrane; Liquid membrane; Platinum removal

1. Introduction

The growth of new technologies in this period of globalization has increased the amount of industrial effluent released into the environment. Pt group metals (PGMs) are pollutants widely discovered in urban wastewater that have a highly significant negative influence on the environment and human health [1]. The PGMs include six rare metals: Pt, palladium, rhodium, ruthenium, iridium, and osmium [2]. However, Pt has special characteristics that play a critical role in a wide range of industrial and medical applications [3]. The direct consumption or exposure of the complex halide Pt salts can cause health effects such as hearing damage, DNA mutation, allergic skin, and cancer [4,5]. Furthermore, the Pt alteration in the environment has a significant impact on soil and water quality, especially on the macro- and microbiological activity [6]. Therefore, the

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leakage of metals into the environment and drainage needs to be minimized urgently.

Various methods for separating heavy metals from wastewater have been studied, such as chemical leaching and bioleaching. The chemical leaching produces high heavy metal extraction yields, but it requires a large volume of acid to be effective. It also necessitates a lot of water for rinsing [7]. Bioleaching is a simple and environmentally friendly method of removing a high amount of heavy metals from sewage sludge without any secondary pollutant emission [7]. However, there is a high possibility of producing toxic chemicals from this process [8]. Apart from that, the complicated selective Pt precipitation method is required a large amount of chemicals and manpower [9]. Moreover, the process also has concerns with slow kinetics, solid-liquid separation, and environmental impact due hazardous chemicals used [9].

Multi-walled carbon nanotubes have been used in the Pt adsorption, resulting in a high Pt adsorption of 48.25 mg/g from the Pt aged aqueous solution [10]. However, the adsorbents only possess a limited capacity for the solute on the solid surface and promote limitation in the adsorption process [11]. Another method for Pt extraction is via liquid-liquid extraction by using the solution made of a carrier such as trialkyl phosphine oxide and a diluent such as kerosene [12]. However, the extracted Pt in liquid-liquid extraction must undergo further separation or stripping to recover the extracted Pt and carrier for reuse. Another problem with liquid-liquid extraction is using a massive amount of solvent, leading to highly polluted or toxic waste and causing environmental pollution [13]. Alternatively, LM is the most attractive process because extraction and back extraction occur continuously in a single operation devise and minimum amount of chemical involves.

The supported liquid membrane (SLM) is a membrane-based system with many advantages over existing metal removal technologies. Minimal solvent and energy requirements, low operating costs, and efficient removal performance are among the advantages of the SLM [14]. SLM offers efficient simultaneous extraction and stripping processes in a single operation device with low energy and solvent consumption [9]. A porous membrane is impregnated with a carrier that can interact specifically with the targeted solute in the SLM. The carrier is dissolved in an organic diluent to form a liquid membrane phase. The microporous membrane provides mechanical strength for the SLM [15,16], while the organic LM is responsible for interacting with the targeted metal during the SLM.

Numerous studies were used flat sheet supported liquid membrane (FSSLM) to remove heavy metals from the wastewater. Zinc(II) was removed from the waste discharge liquor of galvanizing zinc plant using microporous polypropylene coated with Celgard 2400 as the membrane support [17]. 91% of nickel ions were removed from the electroplating wastewater using palm oil as a diluent in the flat sheet polyvinylidene fluoride (PVDF) membrane [18]. The PVDF membrane support also effectively removed 95% mercury from its aqueous solution [19]. Hence, SLM is ideal for removing heavy metals such as Pt due to lower solvent requirements and high extraction performance [20].

The main problem of the LM is its instability and poor permeability, which reduces metal removal performance [14]. The instability occurs when the LM leaks into the aqueous phase from the pores of the membrane support, resulting in carrier loss [21]. Carrier loss can also occur at high carrier concentrations due to the decreasing carrier interfacial within the SLM phase [22]. LM stability is also affected by the concentration of the feed solution. When the feed concentration increase, the pressure at the feed side also increases. This considerable pressure difference between the feed and stripping phase makes LM leaching out from the membrane support possible [23]. The pressure on the LM phase in the SLM can also be affected by feed and stripping flow rates. If the flow rate exceeds the optimum flow rate, it might result in high turbulence flow and LM leakage [24].

Typically, screening experiments performed by a one-factor-at-a-time (OFAT) process were used to obtain stable and optimum conditions for the SLM process. One factor is modified during OFAT, while all other independent variables remain constant. However, this method is costly and time-consuming due to excessive expenses and multiple experiments. A few more designs can be used to screen out the factors, including full factorial design and fractional factorial design (FFD). Full factorial design is suggested to be used in screening for the number of factors below four. Above four factors, it involves many runs and is inefficient. Therefore, a two-level FFD was used in this study to screen five FSSLM parameters on the removal of Pt, including the concentration of Aliquat 336 in kerosene, Pt concentration in the feed phase, $NaClO_4$ concentration in the stripping phase, the flow rate of feed, and the flow rate of stripping phase. To the best of our knowledge, Pt removal using customized hybrid PES-graphene membrane support has never been investigated before for FSSLM.

2. Experimental

2.1. Materials and methods

The dope solution was prepared using polyethersulfone (PES) from Amoco Chemicals, dimethylacetamide (DMAC), and polyethylene glycol 200 (PEG 200) from Sigma-Aldrich, and graphene powder, which was kindly donated by the Low Dimensional Materials Research Centre, University of Malaya, Malaysia. Tap water at 40°C was used to solidify the cast film. The N-methyl-N,N,N-trioctylammonium chloride (Aliquat 336), kerosene, and $NaClO_4$ from Sigma-Aldrich and were used as the carrier, diluent and stripping agent, respectively. Chloroplatinic acid (Sigma-Aldrich) was used as Pt feed phase in the FSSLM process.

2.2. Flat sheet membrane preparation

The homogenous dope solution was prepared using 30 g PES, 85 g PEG 200, and 85 g DMAC with additional 0.03 g graphene. The membrane was fabricated through the vapour inducing phase separation method as described in the previous publication [25]. The flat sheet membrane was cut in 11 cm × 5 cm size and was impregnated with a LM made of 0.1 M Aliquat 336 in kerosene for 24 h. The supported membrane was then attached to the FSSLM system, as shown in Fig. 1.

2.3. Set up of FSSLM system

200 mL feed solution (pH 2.05) containing 5 mg/L of Pt in hydrochloric acid (H_2PtCl_6) and 200 mL stripping solution containing 0.1 M sodium perchlorate was prepared. The solutions are circulated in a counter-current flow by two peristaltic pumps into the chambers in the membrane cell. The flow rate of the solutions was set at the targeted value. The concentration of Pt from the feed phase at 0 h and 6 h were analyzed to calculate the Pt removal percentage.

2.4. Platinum removal percentage

The concentrations of Pt samples from the feed phase were determined using the Inductive Coupled Plasma Mass Spectrometry (ICP-MS) (Agilent 7500a) technique. The samples were diluted appropriately with 2 vol.% nitric acid (Fisher Scientific) solution to ensure their concentration was below the detection limit of the calibration curve. The percentage of Pt removed from the feed phase was calculated by using Eq. (1):

$$\text{Removal\%} = \frac{C_{f1} - C_{f2}}{C_{f1}} \times 100\% \qquad (1)$$

where C_{f1} and C_{f2} are depict the Pt concentrations in the feed phase at 0 and 6 h, respectively.

2.5. Screening by using fractional factorial design

A two-level FFD was used to screen FSSLM operation parameters using Version 7.1.6, 2008 of Stat-Ease Design-Expert software (Minneapolis, MN, USA). Five factors were screened: the Aliquat 336 concentration in kerosene, Pt concentration at the feed phase, $NaClO_4$ concentration at the stripping phase, and the flow rate of both phases. Table 1 shows the low and high values of the factors set in the Design-Expert. Based on these values, 16 experiments were generated by Design-Expert at different conditions.

3. Result and discussion

3.1. Design of experiments and data analysis

The results of the FSSLM experiment are shown in Table 2. Based on these collected data, the software generated the mathematical model, as given in Eq. (2), in terms of coded factors:

$$\begin{aligned} Y = {} & 91.44 + 0.69A + 0.91B - 1.87C + 3.96D - 0.52E \\ & + 2.21AB + 4.11AC - 1.08BC - 2.76BE + 2.34CD \\ & - 1.56CE - 1.31DE \end{aligned} \qquad (2)$$

where Y is response as the removal of Pt, and the letters A, B, C, D, and E represent an interaction between the individual factors where A is Aliquat 336 carrier concentration, B is Pt feed concentration, C is $NaClO_4$ stripping concentration, D is feed flow rate, and E is stripping flow rate.

The relationships between variables were demonstrated using half-normal plots and Pareto charts, as shown in Figs. 2 and 3, respectively. The half normal used to verify the adequacy of generated mathematical models to the experiment. Based on Fig. 2 the data points on this plot are significantly nearer to the straight line. This indicates that the error terms are evenly distributed, and no large variance deviation occurs. Moreover, it strongly supports the adequacy of the least square fit.

The Pareto chart of this experimental design is shown in Fig. 3, which illustrates the influence of important factors on Pt removal. The factors' significance and interactions were determined by employing the "Bonferroni limit" and "*t*-value limit" tests. The bar which extends beyond the "Bonferroni limit" was very certainly significant, whereas the bar that

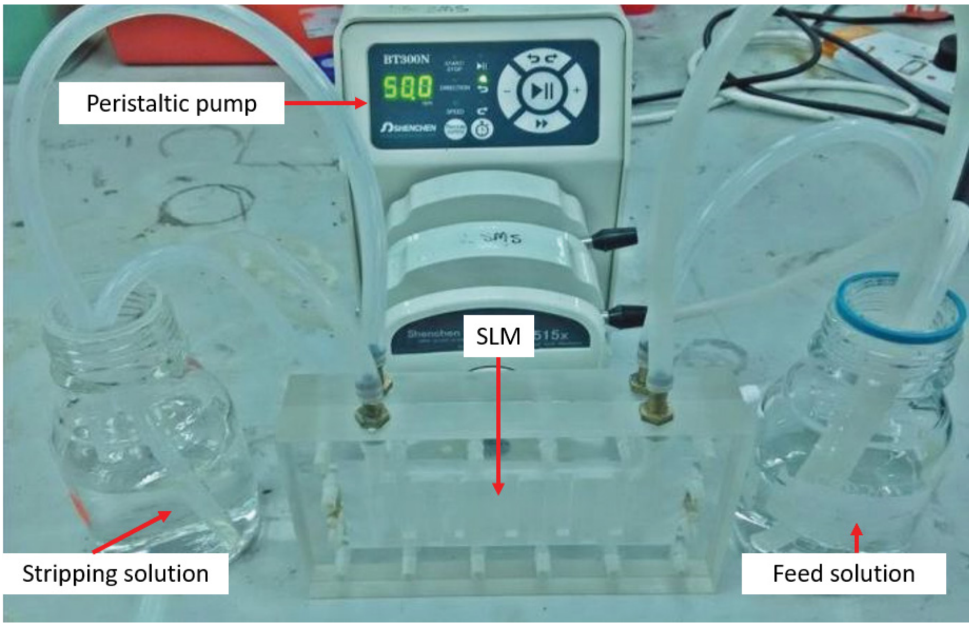

Fig. 1. FSSLM system setup.

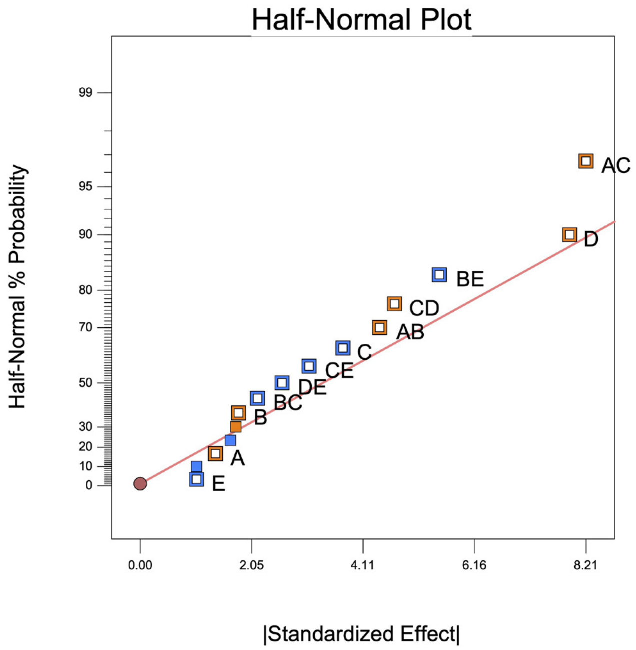

Fig. 2. Half-normal plot.

extends beyond the "*t*-value limit" was perhaps significant [26]. There are no main factor bars and interaction effects bars beyond the "Bonferroni limit". While the bars of factors AC, D, and BE were extended beyond the "*t*-value limit", thus the factors are considered likely significant. Furthermore, the factors CD, AB, C, CE, DE, BC, B, A, and E have insignificant bars since they are placed below the "*t*-value limit". And it was considered insignificant. Although main factors A, B, C, and E are not really significant, interactions of AC and BE are significant thus included in this model.

In order to test the significance of the developed model, analysis of variance (ANOVA) was performed, and the result is presented in Table 3. Based on the ANOVA table, the p-value for this model was 0.0499, which is less than 0.05, indicating that the model term was significant. The main factor (D) and interaction factors (AC and BE) are statistically significant since their p-value is less than 0.05. But the other factors and interactions are considered insignificant as the p-value is more than 0.05. The model F-value of 8.76 indicates that it is statistically significant. Moreover, the probability of a model F-value occurring due to noise is only 4.99%.

Table 4 shows the value of R-squared, Adj. R-squared and Pred. R-squared. Adj. R-squared is the modified version of R^2 that has been adjusted for the number of predictors in the model. It measured the amount of variation about the mean explained by the model. Pred. R-squared is used to reveal the experiment data for the response fitted well with the model's predicted value or not. Based on Table 4, the regression model has a high coefficient of determination R-squared of 0.9722. It means that the model only unexplained 2.78% of the variations in the Pt removal. The accuracy of this model can be verified by determining the R-squared. The closer the value of R-squared to one, the better the model is [27]. The value of Adj. R-squared of 0.8612 described that the independent variables account for 86.12% of the overall variance in Pt removal %. Only approximately 13.88% variation cannot be described using this model.

3.2. Main factor effect analysis

Table 5 shows that main factor D was significant and had the highest contribution of the main factors, at 25.02%. The other factors A, B, C, and E, were considered less significant. Hence, the feed flow rate was the most important parameter on the Pt removal. When the feed flow rate was increased, the percentages of Pt removal were increased. This can be explained due to the high flow rate was capable of reducing the thickness of the boundary layer between feed and membrane without any leakage of the LM and breakage of the membrane support [28]. Consequently, more Pt can be easily diffused at the membrane interface. As a result, it enhanced the complexion between Aliquat 336 and Pt to form complexes. Thus, the percentage of removal of Pt increased.

3.3. Interaction between factors

The interactions between AC and BE were significant and showed the highest percentage of contribution. The other

Table 1
Experimental ranges used in the factorial design

Study type		Factorial	
Initial design		2 level factorial	
Runs		16	
Symbol	Independent variables	Low actual (–1)	High actual (+1)
A	Aliquat 336 carrier concentration, M	0.1	0.5
B	Pt feed concentration, mg/L	5	20
C	$NaClO_4$ stripping concentration, M	0.1	0.5
D	Feed flow rate, mL/min	50	100
E	Stripping flow rate, mL/min	50	100
Response	Name		
Y1	Removal of Pt, %		

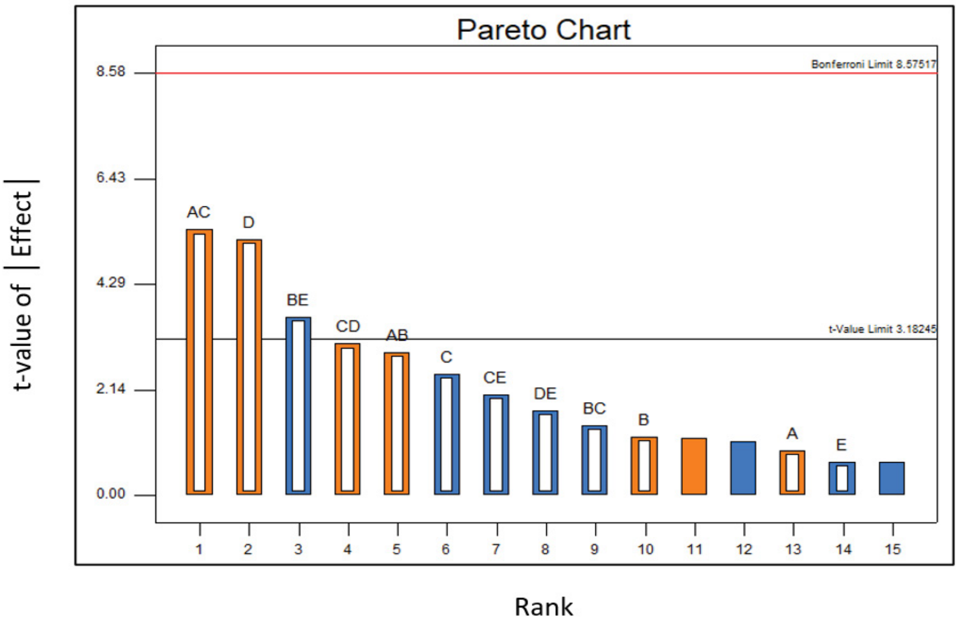

Fig. 3. Pareto chart.

factors AB, AD, AE, BC, BD, CD, CE, and DE, were considered insignificant.

3.3.1. Interaction effect of carrier concentration and stripping concentration

The interaction between carrier concentration and stripping concentration (AC) factors showed a positive effect and contributed to 26.95% of the developed model. Fig. 4 depicts the interaction graph between factors AC. As shown in Fig. 4, when carrier concentration was increased, the removal of Pt was increased. This is because higher available carriers are reacting with Pt at the interface of feed solution [27]. It enhanced the complexion between carrier and Pt to form complexes. Thus, increases the removal of Pt. At the same time, when the stripping concentration was higher, the space availability for Na^+ at the stripping phase increased [28]. As a result, it rapidly interacted with the Pt-amine complex and easily extracted the Pt. This prevented the accumulation of Pt complexes in the liquid membrane. Consequently, more Pt can be easily diffused at the membrane interface. Thus, increase the extraction of Pt.

Table 2
FFD for Pt removal as developed by Design-Expert

Run	Coded values of the independent factors					Y (Removal of Pt, %)
	A	B	C	D	E	
1	–1	–1	–1	–1	+1	98.2
2	+1	–1	+1	+1	–1	98.7
3	–1	–1	–1	+1	–1	98.3
4	+1	+1	–1	–1	–1	77.8
5	–1	–1	+1	+1	+1	93.3
6	+1	+1	–1	+1	–1	96.5
7	+1	–1	+1	–1	+1	88.6
8	+1	+1	+1	+1	+1	96.1
9	+1	+1	+1	–1	–1	94.1
10	–1	+1	+1	–1	+1	72
11	–1	–1	+1	–1	–1	78.4
12	+1	+1	–1	–1	+1	94.3
13	–1	+1	–1	–1	–1	96.5
14	–1	+1	+1	+1	–1	95.4
15	–1	+1	–1	+1	+1	93.9
16	+1	–1	–1	+1	+1	91

There was a noticeable decrement in the Pt removal when carrier concentration was increased at a lower stripping concentration of 0.1 M. Naturally, if the carrier concentration increases, the viscosity of the LM solution also increases [14]. The accumulation of the Pt-carrier complex in the LM phase further enhanced the viscosity of the LM phase. The viscous LM caused the transportation of the complexes through the LM to become more complicated and slow [29]. The function of the stripping agent is to re-extract the Pt from the Pt-carrier complex to the stripping phase and prevent the accumulation of the complex for further removal cycle. 0.1 M $NaClO_4$ contains a lower amount of Na^+ and lower space availability of Na^+ at the stripping phase than the 0.5 M $NaClO_4$. The higher water molecules at 0.1 M $NaClO_4$ interrupted the interaction between the Na^+ and Pt in the LM phase and reduced the Pt removal in the FSSLM. During the FSSLM run, white precipitate appeared in the feed solution due to the failure of the stripping phase to re-extract all the Pt from the LM phase. This induced difficulty of the Pt to diffuse at the

Table 3
Analysis of variance model

Factor	Degree of freedom	Sum of squares	Mean of squares	*F*-value	*p*-value
Model	12	973.31	81.11	8.76	0.0499
A	1	7.7	7.7	0.83	0.4291
B	1	13.14	13.14	1.42	0.3193
C	1	55.88	55.88	6.03	0.0912
D	1	250.43	250.43	27.04	0.0138
E	1	4.31	4.31	0.46	0.5443
AB	1	77.88	77.88	8.41	0.0625
AC	1	269.78	269.78	29.13	0.0125
BC	1	18.71	18.71	2.02	0.2504
BE	1	121.55	121.55	13.12	0.0362
CD	1	87.89	87.89	9.49	0.0541
CE	1	38.75	38.75	4.18	0.1334
DE	1	27.3	27.3	2.95	0.1845

Table 4
Value of *R*-squared, Adj. *R*-squared and Pred. *R*-squared

R-squared	0.9722
Adj. *R*-squared	0.8612
Pred. *R*-squared	0.2105

membrane interface. Thus, a decrease in Pt removal was observed.

However, the high amount of Na^+ at the concentrated stripping phase of 0.5 M easily interacted with the complexes and re-extracted a high amount of Pt from the LM phase. The accumulation of the Pt-carrier complex in the LM is minimized. Therefore, the Pt removal increased as the carrier concentration increased at the higher stripping concentration of 0.5 M. On the other hand, the large amount of Na^+ in 0.5 M $NaClO_4$ only interacted with a small number of complexes formed by 0.1 M of the carrier. The excess Na^+ in the stripping phase accumulated on the membrane and generated a boundary layer. It interrupted the transportation of the solute to the stripping phase. As a result, the Pt removal was lower at 0.5 M $NaClO_4$ than at 0.1 M $NaClO_4$ at 0.1 M Aliquat 336, as shown in Fig. 4.

3.3.2. Interaction effect of feed concentration and stripping flow rate

The interaction between factors feed concentration and stripping flow rate (BE) contributed to 12.14% of the proposed model and gave a negative effect to the Pt removal. The interaction graph between factors BE is shown in

Table 5
Contribution of each factor and their interaction in percentage

Symbol	Factor	Contribution (%)
A	Carrier concentration	0.77
B	Feed concentration	1.31
C	Stripping concentration	5.58
D	Feed flow rate	25.02
E	Stripping flow rate	0.43
AB	Carrier concentration and feed concentration	7.78
AC	Carrier concentration and stripping concentration	26.95
AD	Carrier concentration and feed flow rate	0.43
AE	Carrier concentration and stripping flow rate	1.24
BC	Feed concentration and stripping concentration	1.87
BD	Feed concentration and feed flow rate	1.10
BE	Feed concentration and stripping flow rate	12.14
CD	Stripping concentration and feed flow rate	8.78
CE	Stripping concentration and stripping flow rate	3.87
DE	Feed flow rate and stripping flow rate	2.73

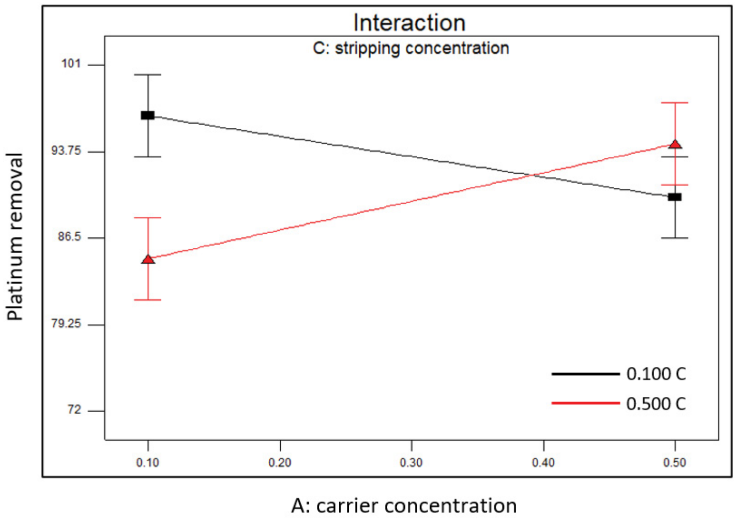

Fig. 4. Interaction graph between factors AC.

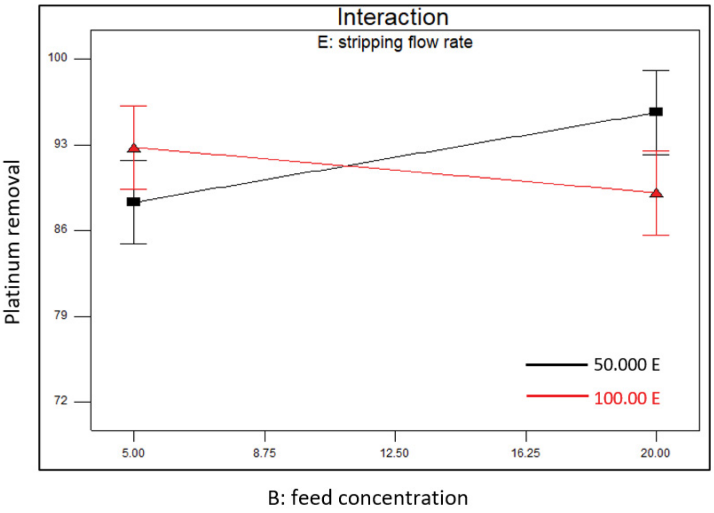

Fig. 5. Interaction graph between factors BE.

Fig. 5. The results show that when feed concentration was increased, the percentages of removal were decreased. As the feed concentration increases, much Pt accumulates at the feed-liquid membrane interface, causing fouling [27]. Fouling obstructs the movement of complex species across the liquid membrane phase, thus reducing the percentage of Pt removal [27]. It was discovered that the membrane support broke down throughout the experiment at a high stripping flow rate. This phenomenon has a negative impact on the Pt removal process, resulting in a drop in the percentage of Pt extracted.

There was an increment in the Pt removal at a low stripping flow rate when feed concentration increased. The large space was available for the Pt at the highly concentrated feed phase induced rapid interaction and removal of Pt by the carrier. Furthermore, the low flow rate enhanced the contact time of stripping agents with the complexes in the LM phase [30]. As a result, the back extraction of the Pt was improved. It indirectly enhanced the Pt removal as well.

4. Conclusion

The FFD method was effectively used to screen the important factors involved in the FSSLM process for Pt removal. Common statistical tools based on analysis of variance (ANOVA) and *F*-test were used to define the most important process variables in the extraction of Pt by the FSSLM system. The use of FFD allowed for the identification of the most significant parameters under tested conditions. The main effects, which were feed flow rate (D), played an important role with the highest contribution of 25.02%, and interaction effects of carrier concentration with stripping concentration (AC) and feed concentration with stripping flow rate (BE) were the most significant with 26.95% and 12.14% contribution, respectively.

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Conflict of interest

The authors declare that they have no conflicts of interest.

Credit author statement

Siti Masyitah Mokhtar: Investigation, Writing - Original Draft; Vikneswary Rajendaren: Investigation, Writing - Original Draft; Syed Mohd Saufi: Supervision, Conceptualization, Writing - Review & Editing; Norasikin Othman: Writing - Review & Editing; Raja Norimie Raja Sulaiman: Writing - Review & Editing.

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