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Response surface optimization of kraft lignin recovery from pulping wastewater through emulsion liquid membrane process

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ABSTRACT

Kraft lignin (KL) represents a key sustainable source of biomass for transformation into biofuels and high-value specialty chemicals. Excess lignin in pulping wastewater creates pollution problems, hence affecting human. Thus, the KL recovery from pulping wastewater by emulsion liquid membrane was investigated and optimized using response surface methodology in this study. The liquid membrane was prepared by dissolving carrier tricaprylylmethylammonium chloride (Aliquat 336) and hydrophobic surfactant sorbitan monooleate (Span 80) in kerosene (diluent) with sodium bicarbonate ($NaHCO_3$) as the internal stripping phase and 2-ethyl-1-hexanol as the modifier. The comparison between the experimentally optimized, and the RSM optimized values was accomplished by optimizing the following parameters: carrier and stripping agent concentration and treat ratio of emulsion to feed phase. The maximum KL recovery of 97% was obtained under the optimum condition at 0.012 M of Aliquat 336, 0.32 M of $NaHCO_3$, and 1:4.8 of treat ratio.

Keywords: Kraft lignin recovery; Pulping wastewater; Emulsion liquid membrane; Response surface methodology; Optimization

1. Introduction

The conversion of wood chips to pulp in paper manufacturing consumes a large volume of water in the process and consequently discharges effluent that contains organic and inorganic pollutants [1]. The pulping liquor generated from kraft and sulfite pulping processes composed of lignin (30–50%) along with a small percentage of sucrose. Lignin imparts brownish dark coloration to the environment, which hinders algal and aquatic plant productivity by limiting light transmittance, and eventually destroys the ecosystem. Besides, the large volume of effluent could not be recycled for other purpose.

Most of the pulping industries evaporated and burned their spent liquor to recover the cooking chemicals, and fewer attempts have been made to recover lignin. Lignin has the potential to become an inexpensive and renewable platform for the production of bio-based materials, aromatic chemicals, and clean biofuels [2,3]. Thus, an effective separation technology

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should be implemented to recover value-added lignin using pulping liquor as a source and consequently conserve the environment.

Various processes have been developed for the separation and purification of lignin from pulping liquor. The most common method used by pulp industries is the precipitation by sulfuric or hydrochloric acid, followed by filtration and washing [4]. However, this method has an inherent drawback, which is colloid formation during precipitation. This will directly complicate the filtration process, and resulting in low purity lignin [5]. Other treatment methods are ultrafiltration, microfiltration, reverse osmosis, coagulation, electrocoagulation, and ozonation [5–10]. The performance of various lignin removal methods are summarized and presented in Table 1. Its application in pulp industries is yet to be successful. Thus, many researchers are putting their effort to develop an efficient separation process for the treatment of pulping liquor.

One of the promising technologies that proposed on this study was emulsion liquid membrane (ELM). It is a versatile and useful alternative for the recovery of different compounds contained in wastewater. These approaches were invented by Li about 40 years ago [11]. The potential applications of ELM include extraction of fermentation products, hydrocarbon fraction, wastewater treatment, organic compounds elimination such as phenol and aniline, biochemical processes, metallic ions recovery, penicillin G extraction, and lignosulfonates [12–21]. ELM is a promising and valuable method that offers several advantages such as ease of operation, large mass transfer area that leads to fast extraction and stripping, capable of applying carrier-mediated transport, low energy consumption, high efficiency, and high selectivity [22].

From literatures, very few research done on recovery of lignin. Previously, Chakrabarty et al. studied ELM separation of lignosulfonates (LS) from spent liquor [21]. However, no attempt has been reported yet regarding the application of ELM process on lignin removal from kraft pulping wastewater. Kraft lignin is another type of lignin which is produced from the kraft pulping process. This process has become dominant, which produce about 85% of total world lignin production [23]. The formulation of liquid membrane on removal of kraft lignin that using Aliquat 336 as carrier, Span 80 as surfactant, and $NaHCO_3$ as stripping agent have been confirmed by Othman et al. [24].

In order to investigate the influences of different factors on ELM application, traditional approach experiments should be conducted, where one factor is changed whilst keeping the other constant. However, it demands a remarkable material expense, it is time consuming, and it does not guarantee the determination of optimal conditions [25]. Thus, response surface methodology (RSM) was implemented in this study to optimize experimental parameters, to model and also to analyze the response of interest along, as well as to reduce the number of experiments. Previously, RSM has been successfully utilized to optimize the parameters of several biotechnological processes such as solubilization, biosorption, supercritical process, and fermentation [26–28]. It has also been widely used to optimize ELM parameters in extraction of chromium, bisphenol, and DL-tryptophan [22,29,30].

However, there is not much work available on the recovery efficiency in ELM process from the literature. Therefore, a statistical experiment design was investigated to optimize the process parameters including carrier and stripping agent concentration, and also treat ratio. The objective of this study was to examine the individual and interactive effects of parameters on the recovery of lignin using ELM. A statistically optimized condition for maximum recovery of lignin was obtained, and the results were then compared with the optimized conditions from experimental studies. The success of this work will make it possible to recover lignin from pulping liquor by ELM in pulping industry.

Table 1
Performance of various lignin removal methods

Methods of recovery lignin	Performance
Acid precipitation	95–98% of lignin was extracted
Ultrafiltration using cellulose acetate membrane	Spent liquor of concentration up to 5% can be treated with a permeate flux of 37.8 Lm^{-2} h^{-1}
Microfiltration and ultrafiltration using inorganic membrane	80% removal of lignin from spent liquor was achieved with membrane of microfiltration range and 90% with ultrafiltration range
Reverse osmosis	90% of lignin was removed from integrated pulp effluent
Electrocoagulation	99% of lignin removed from black liquor
Ozonation	More than 70%of lignin removed at pH 3

2. Extraction mechanism of kraft lignin

The mechanism of carrier facilitated transport extraction and stripping process of KL by Aliquat 336 is shown in Fig. 1. Prior to the extraction reaction, Aliquat 336 transforms to the bicarbonate form in a certain ratio by contacting the membrane solution containing Aliquat 336 with stripping solution that contains $NaHCO_3$ as shown by Eq. (1).

$$AQ^{+}Cl^{-} + NaHCO_3 \Leftrightarrow AQ^{+}HCO_3{}^{-} + Cl^{-} + Na^{+} \quad (1)$$

During the extraction process, KL is transferred in its anionic form by a driving force resulting from the gradient of other anionic counter ions between the two aqueous phases. KL ion chemically reacts with the carrier to form the KL-carrier complexes, AQKL on the external interface as illustrated by Eqs. (2) and (3).

Feed-membrane side reaction:

$$AQ^{+}Cl^{-}(org) + KLNa(aq) \Leftrightarrow AQKL(org) + NaCl(aq) \quad (2)$$

$$AQ^{+}HCO_3^{-}(org) + KLNa(aq) \Leftrightarrow AQKL(org) + NaHCO_3(aq) \quad (3)$$

Then, these complexes diffuse through the liquid membrane to the internal interface. In the presence of $NaHCO_3$, the following reaction is expected to take place at the membrane phase on the stripping solution side.

Stripping-membrane side reaction:

$$AQKL(org) + NaHCO_3(aq) \Leftrightarrow KLNa(aq) + AQ^{+}HCO_3^{-}(org) \quad (4)$$

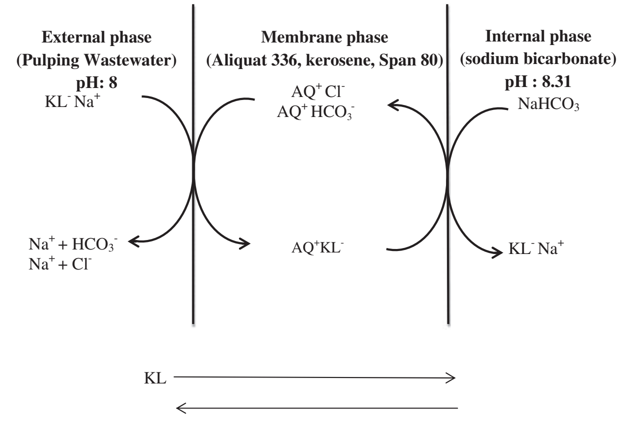

Fig. 1. Facilitated transport mechanism for KL transfer across a liquid membrane.

3. Materials and methods

3.1. Reagents and analysis

Pulping liquor consists of kraft lignin was obtained from Sabah Forest industries, Malaysia. Tricaprylylmethylammonium chloride (Aliquat 336) was used as a carrier and obtained from Acros (USA). Sorbitan monooleate (Span 80) as a nonionic surfactant and 2-ethyl-1-hexanol as a modifier were supplied by Fluka (Buchs, Switzerland). Kerosene was used as a diluent (a complex mixture of aliphatic origin and also contains aromatics about 15%w/w), which was procured from Sigma, whereas sodium bicarbonate ($NaHCO_3$) obtained from GCE Laboratory Chemical was used as an internal stripping agent. All reagents were of analytical grade.

Heidolph Silent Crusher-M Homogenizer was used for emulsion preparation, and Cole-Parmer overhead mechanical stirrer was used for the ELM extraction studies. SM 102 digital pH meter was used for measuring the pH of aqueous external phase, while the absorbance was measured using a UV–vis spectrophotometer (Jenway 7305 Spectrophotometer).

The concentration of kraft lignin in the external and internal phases after separation and demulsification was determined based on the standard curve of absorbance versus lignin concentration.

3.2. Experimental procedure

The emulsion was prepared in a 50 mL beaker by homogenizing an equivalent (5 mL) amount of organic liquid membrane (3%w/v of Span 80 and 0.01 M Aliquat 336 in kerosene) with $NaHCO_3$ solution in dropwise at 12,000 rpm emulsification speed for 5 min to obtain a milky white emulsion. The emulsion was dispersed in the feed phase of pulping liquor containing 100 ppm lignin and agitated by an overhead stirrer at 250 rpm for 10 min. After extraction, the separation of emulsion phase from the raffinate was performed through a separating funnel in response to gravity action, where the three-phase dispersion settled into the emulsion and external phase for analysis. The emulsion phase was collected for demulsification. Heat-induced demulsification approach was used to demulsify emulsion for the recovery of kraft lignin in the receiving phase and recycle the liquid membrane. It was heated under fixed temperature. After heating under 70˚C for 30 min, the mixture separated into the oil phase, the aqueous phase and the remaining emulsion phase. The recovered kraft lignin was separated and diluted for further analysis. The concentration of kraft lignin in the external and internal phases was determined using a UV–vis spectrophotometer at a wavelength of 280 nm [31]. All experiments were carried out at room temperature (25 ± 1˚C). The percentage recovery of lignin is dependent on the response determined by Eq. (5):

$$\text{Recovery of lignin (\%)} = \frac{[KL]_{\text{int}}}{2(\frac{\text{Vex}}{\text{Vem}})[KL]_0} \times 100\% \quad (5)$$

where [kraft lignin]$_0$: initial concentration of kraft lignin, [kraft lignin]$_{\text{int}}$: concentration of kraft lignin in internal phase, Vex/Vem: volume of external phase to volume of emulsion

3.3. Experimental design

The experimental design and statistical analysis were performed using Statistica 8.0 (Stat Soft). Three variables were considered as the important process parameters for the recovery of lignin from pulping wastewater including carrier concentration, stripping concentration, and treat ratio. Therefore, they were chosen as the independent variables and designated as X_1, X_2, and X_3 respectively. Each numeric factor was varied over 3 levels which were appointed as −1, 0, and +1, respectively, and are presented in Table 2. The number of the experiments was optimized using Box–Behnken design in order to verify the interaction between major process variables and their influence on the recovery process. Consequently, the number of experiments required to investigate three parameters at three levels was 15, including three replicates of the center point. Three replicates running at the center of the design were performed to determine the estimation of pure error. The percentage of recovery was the dependent response. A regression analysis was performed to estimate the response function as a second-order polynomial by Eq. (6).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^{k} \beta_{ij} X_i X_j \quad (6)$$

Table 2
Experimental range and levels of operating parameters

	Range and levels		
Variables	−1	0	+1
Carrier concentration (M), X_1	0.006	0.0105	0.015
Stripping agent concentration (M), X_2	0.05	0.2	0.35
Treat Ratio, X_3	3	6.5	10

where Y is the predicted response, β_i, β_j, and β_{ij} are the coefficients estimated from regression, which represent the linear, quadratic, and cross products of X_1, X_2, and X_3 towards response. β_0 is defined as the constant.

The equations were validated by the statistical tests known as the analysis of variance (ANOVA). The significance of all terms in the equation was determined statistically by computing the *F*-value at the probability (*P*) of 0.001, 0.01, or 0.05. Response surfaces were plotted to determine the individual and interactive effects of test variables on the percentage recovery of lignin. The optimal values of the test variables were first obtained in coded units and then converted to the uncoded units.

4. Results and discussion

4.1. Characterization of pulping effluent

The physical and chemical characterizations of pulping effluent were carried out to determine the cation and anion content, pH, viscosity, and density of the wastewater. The properties of pulping effluent are shown in Table 3. The pulping wastewater was dark brown with a characteristic of rotten egg odor.

4.2. Emulsion stability

The study of emulsion stability was carried out at different surfactant concentration, homogenizer speed, and emulsification time. According to Fig. 2(a)–(c), it was observed that the increase in surfactant concentration (up to 3% (w/v)), homogenizer speed (up to 12,000 rpm), and emulsifying time (up to 5 min) reduced the swelling percentage, which indicated that

Table 3
Waste characterization of pulping effluent

Chemical properties	Concentration (ppm)
Cations	
Na^+	1644.415
K^+	251.040
Ca^+	892.692
Anions	
Cl^-	12.395
SO_4^-	74.611
Physical properties	Value
pH	8–9
Density (g/mL)	1.02–1.60
Viscosity (cP)	1.30
Lignin (mg/L)	1,000

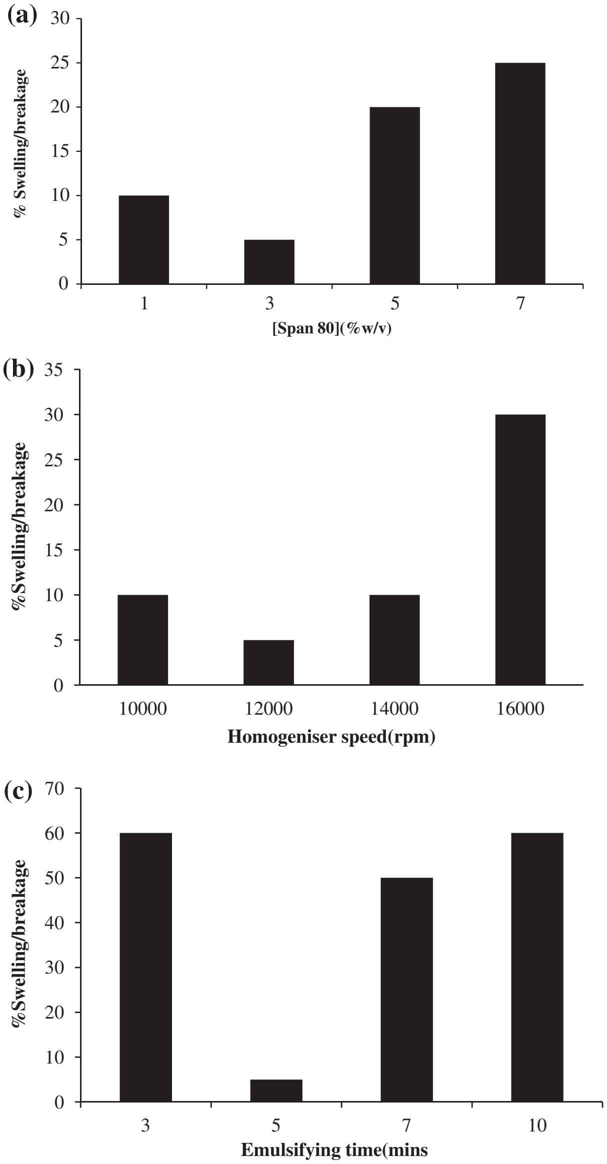

Fig. 2. (a) Effect of surfactant concentration, (b) homogenizer speed and (c) emulsifying time on emulsion swelling (Experimental conditions: [Aliquat 336]:0.01 M, [NaHCO$_3$]:0.1 M, [KL]:100 ppm, Agitation speed: 250 rpm, treat ratio:1:5, Extraction time: 10 min).

the emulsion stability increased up to the level. Further increase in those conditions reduces the stability. The results showed that a stable emulsion was formed at 3% (w/v) of surfactant concentration, 12,000 rpm of homogenizer speed and 5 min of emulsifying time.

4.3. Optimization of process parameters for the recovery of lignin

Table 4
Box–Behnken design matrix along with the experimental results and the predicted values of percentage recovery of lignin using ELM

	Coded values			% Recovery of lignin	
Runs	X_1	X_2	X_3	Experimental	Predicted
1	−1	−1	0	2.94985	0.64055
2	1	−1	0	37.02443	36.47417
3	−1	1	0	22.97481	23.52507
4	1	1	0	72.98994	75.29924
5	−1	0	−1	23.76270	23.97441
6	1	0	−1	56.70272	55.15539
7	−1	0	1	5.72763	7.27496
8	1	0	1	63.91347	63.70177
9	0	−1	−1	37.61062	39.70821
10	0	1	−1	93.28909	92.52712
11	0	−1	1	56.83382	57.59579
12	0	1	1	68.58407	66.48648
13	0	0	0	86.30210	87.13410
14	0	0	0	90.19741	87.13410
15	0	0	0	84.90281	87.13410

Note: X_1: carrier concentration; X_2: stripping agent concentration; X_3: treat ratio.

The Box–Behnken design matrix is given in Table 4 along with the experimental results and the predicted values. The obtained results were then subjected to the RSM to evaluate the relationship between the carrier concentration, X_1, stripping agent concentration, X_2, and treat ratio, X_3. Each effect was estimated independently due to the orthogonality of the design. Multiple regression analysis of the experimental data yielded the following regression equation for the recovery of lignin:

$$\begin{aligned} Y = 87.1 + 21.9019X_1 + 15.43X_2 - 2.04X_3 - 39.85{X_1}^2 \\ - 13.30{X_2}^2 - 9.76{X_3}^2 + 3.99X_1X_2 + 6.31X_1X_3 \\ - 10.98X_2X_3 \end{aligned} \tag{7}$$

The coefficient of determination ($R^2 = 0.99681$) was closer to 1, indicated 99.68% of the variability of the response, which implied that only 0.32% of the total variations were not explained by the model. The predicted values calculated from Eq. (7) were in very good agreement with the experimental values as shown in Fig. 3. The observed experimental values were distributed relatively near to the straight line and showed satisfactory correlation. Hence, the quadratic model is well suited for this experimental set up.

Table 5 shows the analysis of variance (ANOVA) model for the percentage recovery of lignin. ANOVA is required to test the significance and adequacy of the model. From the results, the F-value for lignin

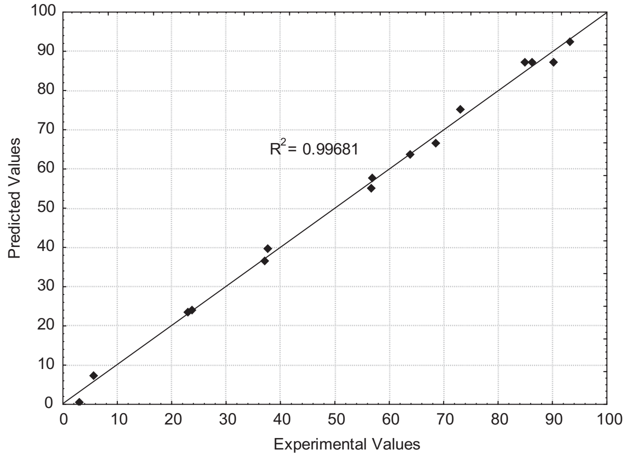

Fig. 3. Comparison of experimental and RSM predicted values for the recovery of lignin.

recovery is 173.63, higher than the tabulated F-value ($F_{0.05,\ 9,\ 5} = 4.77$) at 0.05 significant levels. It implied that the model was significant. The calculated F-value was larger than the tabulated F-value to reject the null hypothesis. The definition of null hypothesis is all the regression coefficient is zero whereby all variables did not give significant outcome to the lignin recovery. Therefore, the model is proven to be a good prediction of responses at high confidence level (95%).

The p-values are used as a tool to check the significance of each of the coefficient, which in turn, may indicate the pattern of the interaction among the variables. A p-value less than 0.05 indicate that the model terms are significant. In this case, it demonstrated that the linear model terms (X_1 and X_2) and quadratic model terms (X_1^2, X_2^2 and X_3^2) were significant coefficients. However, only X_3 was insignificant ($p > 0.05$). The significance of the parameter in the model is seen in Table 6.

4.4. *Interactive effects of carrier concentration, stripping agent concentration, and treat ratio*

The response surface plots and their corresponding contour plots of lignin recovery are presented in Figs. 4(a) and (b), 5(a) and (b), 6(a) and (b). They demonstrate the interaction of the variables and also determine the optimum level of variables for maximum response.

A noticeable elongated maxima running along respective stripping agent concentration and treat ratio axis was observed in Figs. 4(a) and 5(a). In addition, the contour plots in Figs. 5(b) and 6(b) presented an

Table 5
Analysis of variance (ANOVA) for quadratic model of recovery of lignin by ELM

Source	Sum of square	DF	Mean square	F-value	F-tabulated ($\alpha = 0.05$)
Regression	12865.44	9	1429.49	173.63	>4.77
Residual	41.17	5	8.233		
Total SS	12906.61	14			

Table 6
The significance of the parameters in the model

Source	Coefficient	Sum of square	DF	Mean square	F-value	p-value prob > F	Comments
Regression	87.1341	12865.44	9	1429.49	173.63		
X_1	21.9019	3837.56	1	3837.562	466.1172	0.000004	Significance
X_2	15.4274	1904.04	1	1904.037	231.2678	0.000022	Significance
X_3	−2.0383	33.24	1	33.236	4.0369	0.100748	
X_1^2	−39.8511	5863.78	1	5863.779	712.2252	0.000001	Significance
X_2^2	−13.2983	652.96	1	652.964	79.3102	0.000297	Significance
X_3^2	−9.7564	351.46	1	351.462	42.6892	0.001257	Significance
X_1X_2	3.9851	63.53	1	63.525	7.7159	0.039006	Significance
X_1X_3	6.3115	159.34	1	159.338	19.3535	0.007027	Significance
X_2X_3	−10.9821	482.42	1	482.422	58.5959	0.000606	Significance
Residual		41.17	5	8.233			
Total SS		12906.61	14				

Note: R-squared: 0.99681; Adjusted R-squared: 0.99107.

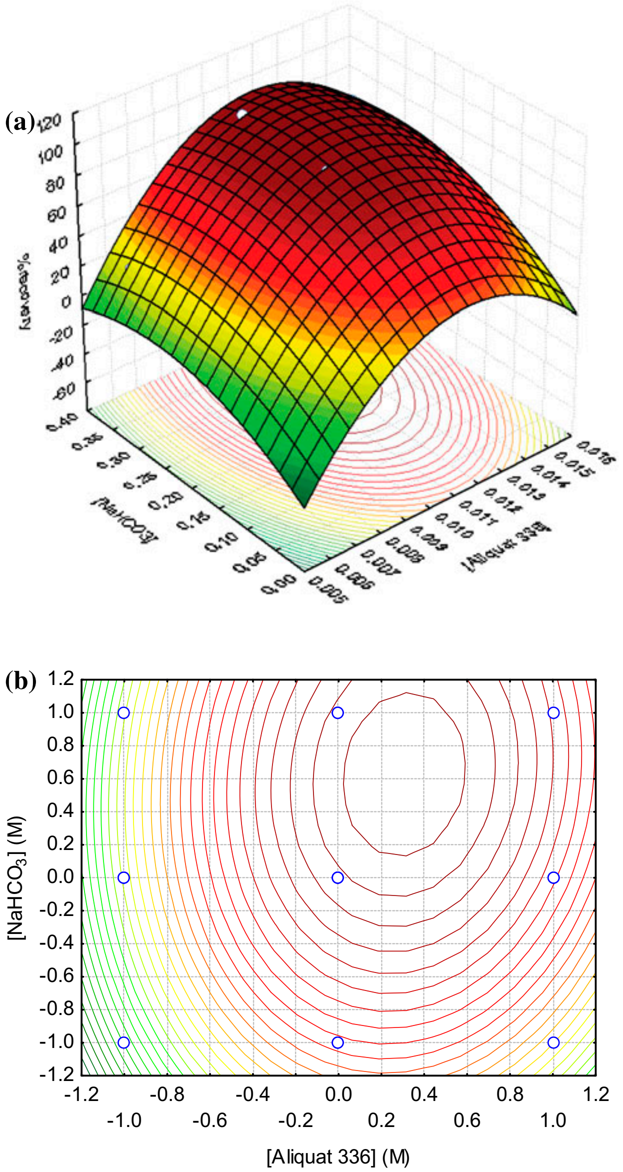

Fig. 4. (a) The 3D surface plot and (b) contour plot of interaction between carrier concentration and stripping agent concentration for lignin recovery.

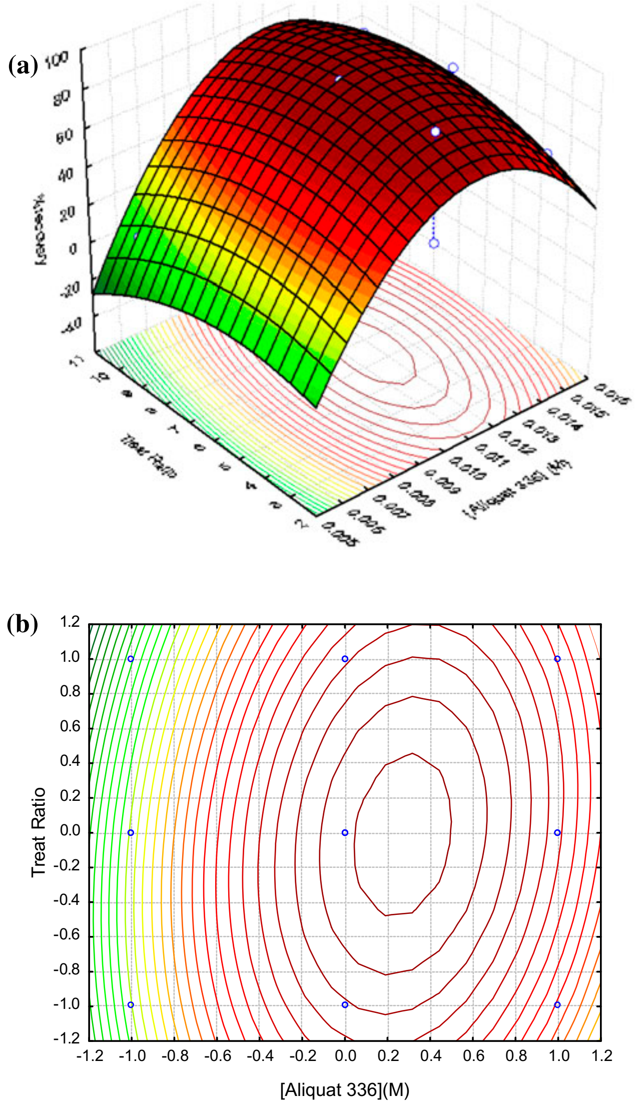

Fig. 5. (a) The 3D surface plot and (b) Contour plot of interaction between carrier concentration and treat ratio for lignin recovery.

elliptic characteristic with the long axis of the ellipse running along the stripping agent concentration and treat ratio axis. These indicated that carrier concentration was the most important factor among the three factors in lignin recovery. This was also supported by the biggest value of coefficients, which was 21.9019. It is known that the addition of Aliquat 336 significantly improved the lignin recovery when its concentration is below 0.012 M. In ELM process, carrier plays the most important role in transferring solute from the external interface to the internal interface. Sufficient carrier accelerates the migration of lignin ions towards oil phase interface and directly enhances the recovery process towards internal stripping phase. However, too much carrier increases the viscosity in organic phase, which reduces permeation efficiency. The trend of the results is similar with a previous study where the optimal Aliquat 336 concentration at the middle range achieves maximum between the lower and upper levels [30].

The interaction between carrier concentration and stripping agent concentration is demonstrated in Fig. 4(a) and (b). From the figures, the parabolic nature of contours implies the interaction between both

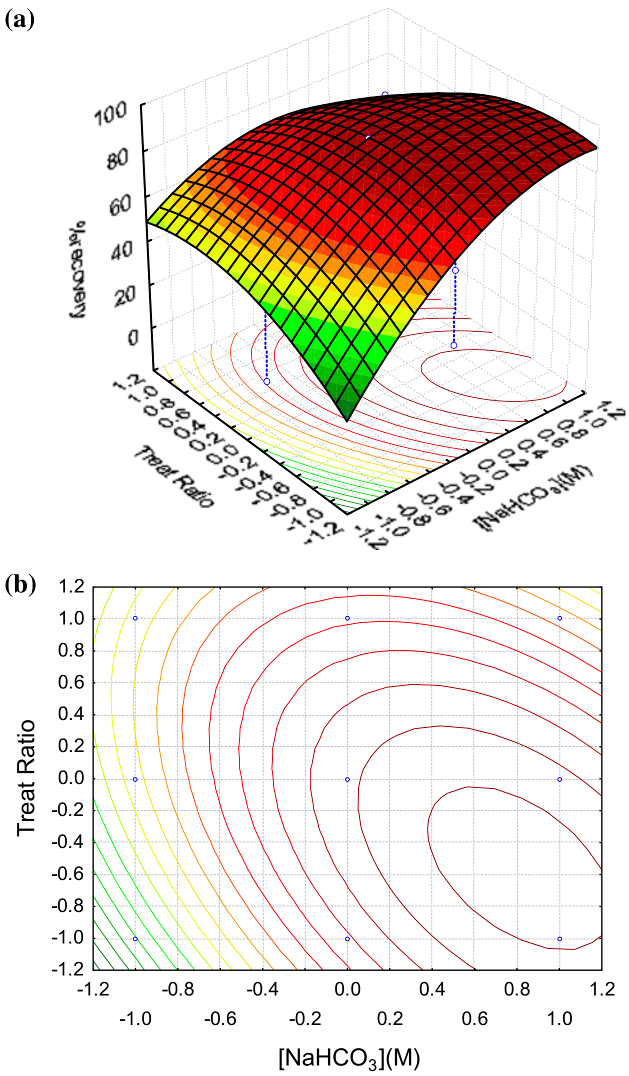

Fig. 6. (a) The 3D surface plot and (b) contour plot of interaction between treat ratio and stripping agent concentration for lignin recovery.

variables is significant. The carrier concentration and the stripping agent concentration increased the percentage recovery when their value increased from the lower range up to a certain limit. After that, the recovery efficiency declined. The interaction between the carrier concentration and stripping agent concentration can be explained by the reaction between Aliquat 336 and $NaHCO_3$, which takes place at the internal interface between membrane phase and internal phase. The increasing amount of stripping agent enhances the stripping reactions, while carrier acts as the shuttle to transfer lignin ion from the external interface to the internal interface. Therefore, these two factors involve significantly in the extraction and stripping reaction. Hence, they are highly interactive parameters in the ELM process. This result is in line with a previous research which indicated that the interaction between these parameters was significant [32].

Fig. 5(a) and (b) illustrate the interaction between treat ratio and carrier concentration. Parabolic contours signify that the interaction is quite significant. From the figure, it shows that the carrier concentration achieves maximum between the lower and upper levels. However, the increment of treat ratio results in slightly increased recovery but the recovery decreased slightly after the optimum value was achieved. The treat ratio controls the interfacial mass transfer across the liquid membrane [13]. As the volume of the external solution decreased with a fixed amount of emulsion in the feed solution, it significantly increased the emulsion phase hold up in the feed phase, which simultaneously increased the extraction and recovery capacity. This is strongly supported by Jiao et al. [29], who observed that the high treat ratio increased the amount of emulsion required for the feed phase to be treated. However, at low treat ratio (1:3), it was observed that the emulsion was dispersed poorly in the feed phase. In addition, emulsion tends to swell or break at this condition, which dilutes the concentrated lignin in the internal phase. Directly, it will result in lower recovery efficiency. Higher treat ratio indicates that more solute is to be treated by membrane phase. Thus, the increase of carrier concentration is preferred. If the treat ratio is constant at 1:5 while the carrier concentration increases further, the carrier which acts as the shuttle to carry solute will accumulate on the membrane phase. This will hinder mass transfer, thus lower the recovery efficiency [33]. Therefore, they have a good interaction for ELM process.

The interaction between treat ratio and stripping agent concentration is shown in Fig. 6(a) and (b). Parabolic contours signify that the interaction had a positive significant effect on lignin recovery. According to Fig. 6(a), the recovery percentage increased with the increase of stripping agent concentration up to the upper level. Meanwhile, there was a slightly elongated maxima and ellipse running along the treat ratio axis. This trend pointed out the higher influence of stripping agent concentration compared to treat ratio.

During the extraction process, carrier-lignin complex will form in the external interface and travel through the membrane into the internal phase. While passing through the internal phase, the lignin part of this complex will be stripped away by the stripping agent. From the figure, the recovery efficiency increased with the increase of stripping agent concentration and became plateau beyond 0.32 M. This fact

Table 7
Comparison of experimentally optimized data and RSM optimized data for lignin recovery using ELM

Parameters	Experimentally optimized (simulated kraft lignin)	RSM optimized (Real pulping wastewater containing kraft lignin)
Carrier concentration (M)	0.007	0.012
Stripping agent concentration (M)	0.1	0.32
Treat ratio	5	4.8
Recovery of lignin (%)	97.88	96.97 (predicted)

indicates that 0.32 M provides a sufficient amount for the stripping process. A stronger pH gradient was present at the higher amount of stripping agent. The pH difference between the external and internal phases is the main driving force for the transport of the complex through membrane phase. Previous researcher showed that an optimal stripping agent concentration exists, and a further increase has a negative influence on the removal efficiency [22]. Besides that, another explanation can be that $NaHCO_3$ has the tendency to react with Span 80 [34], thus modifying the properties of these components by forming other compounds that deteriorate the emulsion stability. Thus, the range of stripping agent was set in the range that could achieve stable emulsion.

4.5. Optimization response and verification test

The second polynomial model obtained in Eq. (7) in this study was utilized for response optimization using Statistica 8.0 (Stat Soft). The optimum conditions of the parameters were 0.012 M of carrier concentration, 0.32 M of stripping agent concentration, and 1:4.8 of treat ratio. Under the optimal conditions, the predicted maximum recovery percentage was 96.97%. In order to confirm the prediction, another three experimental sets were carried out under the optimum conditions. An average recovery of 96.08% was obtained. This experimental finding is in close agreement with the model prediction. This also indicates that RSM is applicable to optimize the recovery of lignin from pulping wastewater.

Table 7 shows the comparison between the experimentally optimized and the RSM optimized values. Both methods show high recovery efficiency, which are 98% and 97%, respectively. From the table, it shows that all the values are in good agreement except stripping agent concentration. The difference may be due to the additional anion in real pulping wastewater, which increases the pH of the external phase. It is very crucial to keep the pH of the external phase and internal aqueous phase in optimum to avoid the instability of the membrane and to enhance the recovery process [35]. Thus, the recovery of lignin from real pulping wastewater requires more stripping agent to recover lignin. Nevertheless, the model can be used to predict the percentage recovery of kraft lignin by ELM from real pulping wastewater.

5. Conclusion

The recovery of kraft lignin from pulping liquor using ELM is an approach that could give industrial success. A maximum predicted value of the percentage recovery of lignin was 96.97%, and the optimized parameters for ELM were 0.012 M of carrier concentration, 0.32 M of stripping agent concentration and 1:4.8 of treat ratio. Meanwhile, the statistical analysis demonstrated that all these parameters had an individual significant effect on the lignin recovery, and there were significant interactions as well. Thus, ELM technology has been successfully demonstrated to recover value-added lignin using pulping liquor as a source and simultaneously conserve the environment.

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