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Single stage extraction and recovery of hexavalent chromium using blended TOA-TOMAC in palm oil-based diluent via supported liquid membrane process

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Abstract. Diluent is an important components in the supported liquid membrane (SLM) process. The conventional diluents used in SLM are usually flammable, volatile, and toxic. To promote a sustainable development, the palm oil was incorporated in the SLM for the removal and recovery of chromium. SLM is a three-phase system with an organic phase containing the carrier in diluent which immobilized in a membrane support and is set in between the simulated chromium and sodium hydroxide solutions that act as a feed and stripping phases respectively. Both solutions were pumped into the membrane support cell in a recycled operation for about 5 hours. To monitor the changes of the chromium ion concentration for both phases, the chromium ion concentration in the feed and stripping phases as a function of time was analysed using an atomic absorption spectrometry (AAS). Several parameters namely type of stripping agent, diluent composition and carrier concentration were investigated. Results showed that about 75 and 73% of chromium was extracted and recovered, respectively at the best conditions of using sodium hydroxide, mixture of palm oil-kerosene (50:50) and TOA-TOMAC (0.20-0.20M). Thus, palm oil is regarded as feasible as a greener diluent in SLM process for chromium ion extraction.

1. Introduction

Chromium is widely used in various types of industry owing to its good features of magnetic properties, hardness and anti-corrosion. It is also known as alloying material for steel, which is commonly used for surface coating and refractory material. Other applications of chromium are in the preservation of wood, leather tanning, synthetic manufacturing, industrial catalysts, and colour pigments for paints [1-2]. The most common oxidation state of chromium that exists in aqueous is hexavalent (Cr (VI)) and trivalent chromium (Cr (III)). However, due to the high solubility and bioavailability, hexavalent chromium is more toxic compared with the trivalent chromium due to its ability to form various anionic species in aqueous solution, which are harmful, carcinogenic and mutagenic compounds towards environment [3]. World Health Organization (WHO) and USEPA have declared chromium as one of the most toxic metal for the environment [4]. Thus, removal of chromium from the industrial effluents have become a necessary task in ensuring environmental safety.

Previously, a few conventional techniques have been employed in order to remove chromium from industrial waste and wastewater such as precipitation [5], solvent extraction [6], reverse osmosis [7], and electrodialysis [8]. Every conventional method has its own advantages and limitations. For instance, precipitation process achieves high percentage of heavy metal removal with an addition of other chemicals. However, this process generates the sludge that leads to an extra disposal cost. Meanwhile, solvent extraction involves high consumption of chemical whilst reverse osmosis is a simple operation with effective removal of contaminants but requires expensive monitoring system and high energy consumption. On the other hand, electrodialysis implies high capital and operating cost due to fouling and scaling of the membrane. However, these methods only remove without recovering the chromium from wastewater. Hence, in view of the drawbacks of the conventional methods, the liquid membrane (LM) technology has attracted much interest and appears to be an advanced technique due to its great potential in the field of separation process for various organic compounds and metallic ions [9]. The main advantages of LM technology is the removal and recovery in one single step. Therefore, the targeted solute can be simultaneously removed and recovered.

Principally, LM technology is composed of three main configurations or designs namely bulk liquid membrane (BLM), emulsion liquid membrane (ELM) and supported liquid membrane (SLM). In LM system, there are three main phases involved including feed, membrane and stripping phases. Theoritically, the mechanism of LM occurs as the targeted solute is transported from feed to stripping phases across the LM phase that acts as the barrier [10]. Mostly, the diffusion of the targeted solute is facilitated by the carrier in LM phase. In BLM, a relatively thick layer of immiscible LM contained carrier in organic diluent separate the bulk feed and stripping phases [11]. LM phase contains carrier as well as surfactant in organic diluent to form emulsion known as ELM [12]. Meanwhile, LM phase contains carrier in organic diluent and is placed in a microporous polymer membrane support known as SLM[13].

Among these configurations, SLM is one of the simplest, efficient and easy to be scaled up.The main components in SLM system are carrier, stripping agent and diluent. Carrier is chosen based on the selectivity of the targeted solute ion present in the feed phase and can either be acidic, basic and solvating. Acidic carriers such as 2-hydroxy-5-nonylacetophenone oxime (LIX841),5,8-diethyl-7-hydroxy-dodecan-6-oxime(LIX63), phosphorus derivative (di-2-ethylhexyl phosphoric acid (D2EHPA) and di-2,4,4-trimethylpentyl phosphinic acid (Cyanex 272) are generally used for extraction based on the cationic exchange mechanism [14-17]. Meanwhile, basic carriers such as trioctylamine (TOA),tridodecylamine (TDA), tri-n-octylmethylammonium chloride (TOMAC) and etc undergo metal ion extraction through anion exchange reaction [18-20]. Subsequently, the solvating or neutral carriers are basic in nature, hence extracting either neutral metal complexes or acids through the solvate formation [21-22]. To date, an application of mixed carriers is gaining attention to improve the metal ion extraction efficiency via synergistic effect. Previously, Sulaiman and Othman [23] prove that about 83% of nickel ions were successfully extracted via the mixture system of 0.08 M LIX63 and 0.02 M D2EHPA with the maximum synergistic enhancement factor, R_{max} of 29.56. This is also supported by Singh et al. [24] who reported similar observation as more than 95% of uranium (VI) were recovered in 360 minutes of SLM process using a binary mixture of 0.60 M PC88A and 0.15 M Cyanex 923 in dodecane. Subsequently, good diluent should provide good characteristics such as high solubility, low flash point [25]. So far, the petroleum based diluents have been employed for metal ion extraction in liquid membrane technology namely kerosene, n-heptane and toulene and etc [26]. Nevertheless, this type of diluents is toxic, non-renewable, non-biodegradable, flammable and volatile in nature. An introduction to the green organic diluent such as vegetable oils attracts the attention of several reseachers as a way to promote greener process in the future [27]. Acording to Jusoh et al. [28], the combination of 30/70 kerosene to palm oil also leads to high separation of succinic acid from the fermentation broth. Another observation reported by Chakrabarty et al. [29] claimed that in about 95% of mercury were extracted using coconut oil as diluent through the SLM process. Besides, Othman et al. [30] also found that almost 100% of chromium were extracted and recovered using the diluent containing kerosene-palm oil mixture with the ratio of 3:7.On the other hand, stripping agent also plays a crucial role as a

binder for the targeted solute at the membane-stripping interface. Commonly, metallic ions extracted by basic carriers seems suitable to be stripped using neutral or alkaline solutions and vice versa in order to create the chemical potential between both membrane and stripping phase [31].

In this present investigation, removal and recovery of chromium (VI) using SLM was studied. A liquid membrane formulation placed in the membrane support composed of a blended carrier of TOA and TOMAC in the mixture of kerosene and palm oil as organic diluent. Several parameters affecting the extaction and recovery performance of chromium (VI) such as the type of stripping agents, the composition of diluent and the concentration of carrier were examined.

2. Experimental

2.1. Reagent and materials
Potassium dichromate, ($K_2Cr_2O_7$), supplied by Sigma Aldrich in powdered form, was used as a source of hexavalent chromium Cr (VI) in the simulated wastewater. Trioctylamine (TOA) and Tri-n-octylmethylammonium chloride (TOMAC) as carriers were procured from Fluka. Kerosene and cooking palm oil as diluents were obtained from Merck and Mart, respectively. Meanwhile, sodium hydroxide and sulfuric acid as stripping agents were purchased from Merck. Besides, polyvinylidenfluoride (PVDF) ordered from Millipore was used as a membrane support for organic solution with an average effective pore diameter of 0.22μm, an average thickness of 125μm and porosity of 75%. All these materials were of analytical reagent grade and used directly as received from the manufacturer without further purification.

In fact, tertiary amines (TOA) and quaternary ammonium salts (TOMAC) are the most widely used ionic carriers in chromium ion extraction using SLM process owing to the high coordination ability and stability of the complex strength [32-33]. Besides, in terms of the carrier-chromium interaction perspective, more highly structured basic mixed carrier (TOA-TOMAC) is favored due to the high degree of solvation of carriers with the chromium ion in the membrane, hence enhancing the stability of the carrier-chromium complexation [34].

2.2. SLM set up and extraction
SLM rig set up is composed of membrane cell, feed and strip vessel, double head peristaltic pump, flowmeter and tubing. There are three main phase involve in SLM process which are feed, membrane and stripping phases. The feed phase contains simulated chromium (VI) solution, which was prepared by mixing an appropriate quantity of potassium dichromate in deionized water with measured pH of 2. Subsequently, organic liquid membrane was prepared by dissolving the corresponding volume of carriers in the organic diluent to obtain carrier solutions of different concentrations, ranging from 0.05 to 0.35M. The PVDF membrane support (10cm x 3.5 cm) was impregnated with the organic solution for 24 hours before leaving it to drip for a few seconds followed by placing it in the membrane cell as shown in figure 1. Meanwhile the stripping solution (sodium hydroxide) of desired concentration was prepared by dissolving appropriate weight of pellets in deionized water. About 300 mL of feed and strip solutions were added into feed and strip vessel, respectively. These solutions were pumped into the membrane cell with a recycled operation. Both aqueous feed and stripping solutions were magnetically stirred to avoid concentration polarization conditions at the membrane interfaces and in the bulk of the solutions. Ten mL sample of each feed and stripping solutions were periodically taken every 30 minutes for 5 hours to determine the changes in the chromium ion concentration using Atomic Absorption Spectrometry (AAS) with a wavelength of λ=540 nm. The new membrane support was used for each experiment. The experiment was carried out at ambient temperature (25 ± 1°C) with standard deviations of chromium ion concentrations less than ± 5%.
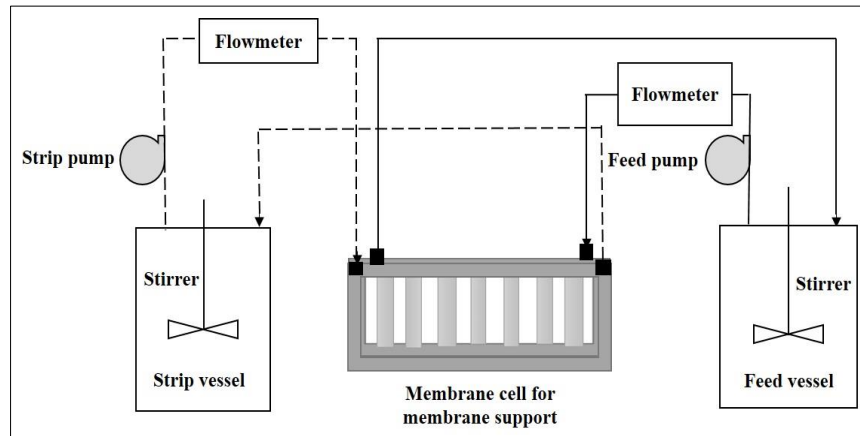
Figure 1. Schematic diagram of SLM process.

2.3 Data analysis

2.3.1. Extraction and recovery performance. The extraction and recovery performance of chromium ion through SLM process are determined using equations (1) and (2), respectively:

$$\text{Removal (\%)} = \frac{[Cr]_{f_o} - [Cr]_{f_t}}{[Cr]_{f_o}} \times 100 \tag{1}$$

$$\text{Recovery (\%)} = \frac{[Cr]_s}{[Cr]_{f_o}} \times 100 \tag{2}$$

Where $[Cr]_{f_o}$ represents the initial chromium in the feed phase; $[Cr]_{f_t}$ indicates the final concentration of chromium in the feed phase and $[Cr]_s$ denotes the concentration of chromium in the stripping phase.

2.3.2. Determination of permeability value. Membrane permeability is defined as the ability of a membrane to allow the desired solute for passing through. Permeability of the chromium ions extracted from feed to stripping phases is determined using equation (3) [35]:

$$ln\frac{c}{c_i} = -p\left(\frac{A}{V}\right)t \tag{3}$$

where c_i is the initial concentration of chromium ions in the feed phase, c is the concentration of chromium ions at a given time, p is the permeability value (cms^{-1}), A is the effective area of the membrane (cm^2), V is the volume of aqueous feed phase (cm^3) and t is the time.

2.3.3. Determination of liquid membrane loss. Liquid membrane loss, Δm is used to evaluate an instability of SLM extraction of chromium ion and can be determined using equation (4) [36]:

$$\Delta m \ (gcm^{-2}) = \frac{(m_1 - m_2) - (m_1 - m_0)}{A} \tag{4}$$

Where m_0, m_1 and m_2 are the weights of the dry, wet, and used membrane support, and A is the effective area. A weight of dry membrane is a weight membrane before used while weight wet membrane designates the weight membrane after impregnation with organic liquid membrane. Then, the weight of used membrane refers to the weight of membrane after extraction process.

3. Results and discussion

3.1. Transport mechanism of chromium (VI) ion extraction in SLM

Apparently, chromium ion is able to exist in aqueous solution as $HCrO_4^-$, CrO_4^{2-}, $HCr_2O_7^-$ and $Cr_2O_7^{2-}$ depending on the pH value of the solution and the total concentration of chromium. As the pH is lower than 0.5 or too acidic, the chromic acid ($H_2Cr_2O_7$) is predominant. Meanwhile, $Cr_2O_7^{2-}$ appears to be the main anion in an acidic aqueous phase. However, $Cr_2O_7^{2-}$ is converted into $HCrO_4^-$, in acidic aqueous solution as the concentration of chromium lies in the range lower than $(1.26 - 1.74) \times 10^{-2}$ mol/L. Beyond this critical concentration values, Cr(VI) is generally found as CrO_4^{2-} [37]. As the lowest concentration used lies in the range lower than the concentration mentioned above, $HCrO_4^-$ is the prevailing anion that existed in the chromium solution as a feed phase in this present work. The mechanism for simultaneous extraction and recovery of chromium ion using SLM process is illustrated in figure 2. In this study, both blended TOA-TOMAC and NaOH act as carrier and stripping agent, respectively.
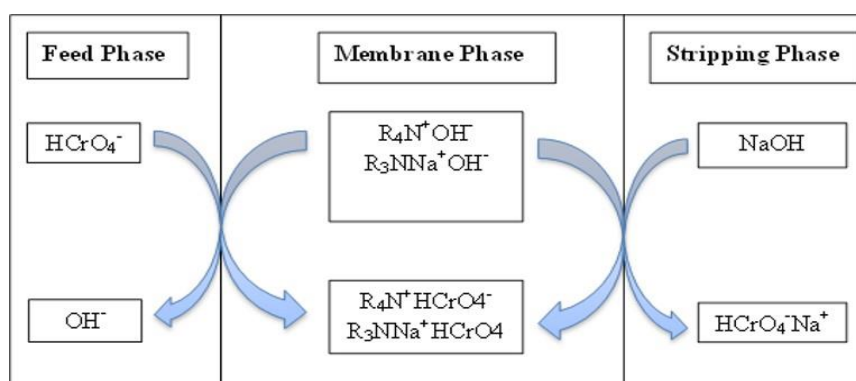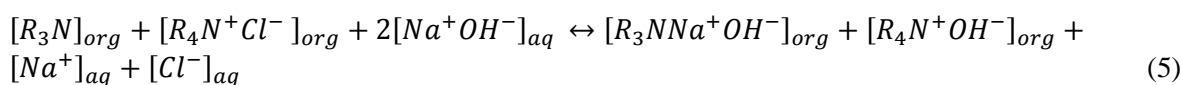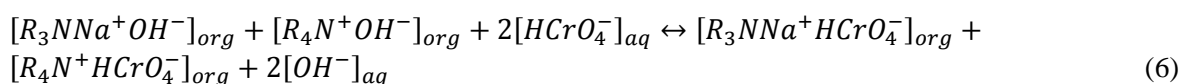

Figure 2. Transport mechanism of chromium ion extraction
via SLM process.

In SLM transport process, the diffusion of chromium ions takes place via the following steps:
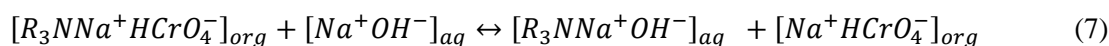a) Both basic carriers, TOA (R_3N) and TOMAC ($R_4N^+Cl^-$) in the membrane phase were protonated by the stripping agent, NaOH at the membrane-stripping interface as shown in equation (5):

$$[R_3N]_{org} + [R_4N^+Cl^-]_{org} + 2[Na^+OH^-]_{aq} \leftrightarrow [R_3NNa^+OH^-]_{org} + [R_4N^+OH^-]_{org} + [Na^+]_{aq} + [Cl^-]_{aq} \tag{5}$$
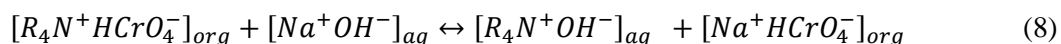
b) During the extraction, the protonated carrier molecules in the membrane phase react chemically with the hydrochromate ions at the feed-membrane interface, hence forming chromium-carrier complexes as represented in equation (6):

$$[R_3NNa^+OH^-]_{org} + [R_4N^+OH^-]_{org} + 2[HCrO_4^-]_{aq} \leftrightarrow [R_3NNa^+HCrO_4^-]_{org} + [R_4N^+HCrO_4^-]_{org} + 2[OH^-]_{aq} \tag{6}$$

c) Subsequently, these chromium-carrier complexes of $R_3NNa^+HCrO_4^-$ and $R_4N^+HCrO_4^-$ complexes diffuse across the membrane phase from feed-membrane interface to the membrane-stripping interface reversibly. At the membrane-stripping interface, the stripping reaction with NaOH take place as represented in equations (7) and (8):

$$[R_3NNa^+HCrO_4^-]_{org} + [Na^+OH^-]_{aq} \leftrightarrow [R_3NNa^+OH^-]_{aq} + [Na^+HCrO_4^-]_{org} \tag{7}$$

$$[R_4N^+HCrO_4^-]_{org} + [Na^+OH^-]_{aq} \leftrightarrow [R_4N^+OH^-]_{aq} + [Na^+HCrO_4^-]_{org} \qquad (8)$$

3.2. Effect of stripping agent type

To elucidate the effect of stripping agent type, two types of stripping agent namely sodium hydroxide (NaOH) and sulphuric acid (H_2SO_4) were employed for simultaneous extraction and recovery of chromium ion in SLM process. The extraction and recovery performance of chromium ion as a function of different types of stripping agent after 5 hours of experiment are tabulated in table 1. It can be clearly seen that NaOH showed a better performance with 75 and 73% of extraction and recovery percentage, respectively. Conversely, H_2SO_4 only provided up to 42 and 10% of extraction and recovery percentage, respectively. As both carriers used are the basic type, the alkaline and neutral stripping agent are preferable in creating the chemical potential between the membrane and stripping phases [31]. Fundamentally, both ionization energy and electron affinity become higher as we go up a column of a periodic table. Since oxygen atom (-O-) is above sulphur atom (-S-) in the periodic table, it has higher ionization energy as well as electron affinity. Therefore, it provides high tendency to rapidly break the ionic bonding with sodium ion, hence enhancing the stripping efficiency. In addition, the lowered stripping efficiency for sulfuric acid solution is probably due to the competition between sulfate and chromium ions which working in favor of forming a more stable carrier–chromium complex of larger solvation degree, allowing chromium to stay in the organic phase [34]. As a result, NaOH is employed as stripping agent throughout this study.

Table 1. Effect of stripping agent types towards extraction and recovery efficiency of chromium

Stripping agent	Extraction (%)	Recovery (%)
Sodium hydroxide (NaOH)	75	73
Sulphuric acid (H_2SO_4)	42	10

3.3. Effect of diluent composition

The feasibility of palm oil as a substitute diluent for SLM process is evaluated by studying the effect of various diluents towards the extraction and recovery performance of chromium ion as shown in figure 3. Meanwhile, the variation of the permeability values, viscosity and liquid membrane loss with respect to the different diluent composition are tabulated in table 2. According to figure 3, both liquid membrane containing 100% kerosene and mixtures of palm oil and kerosene (50:50) achieved high percentages of chromium ion extraction (73%). Likewise, the recovery efficiency also showed that the liquid membrane with 100% kerosene provided the highest recovery (81%) followed with the one containing the mixtures of palm oil and kerosene (73%) within 5 hours of experiment. Besides, the changes in the permeability values from 6.3 to 4.2 x10^{-4} cms^{-1} upon mixing with the 50% of palm oil also was observed. The low permeation of chromium ion across the membrane phase is caused by the increment of the viscosity liquid membrane from 32 to 41 cP. Theoretically, liquid membrane with high viscosity tends to retard the permeation of chromium ion across the membrane phase, thereby inhibiting a substantial amount of chromium ion being transported to the stripping phase. This is in line with Kumar et al. [37] who claimed that high viscosity solvent is able to create high resistance as well as hindering the mass transfer of solute ion in the membrane phase.

On the other hand, the liquid membrane containing 100% palm oil provided the lowest extraction (22%) and recovery (8%) performance. This is probably due to the highest viscosity of 72cP which thereby hindering the permeation of chromium ion across the membrane phase. The low permeation of chromium ion into the membrane phase is in accordance with Chakrabarty et al. [29] who indicated that the accumulation of higher amount of oil phase on the membrane surface as a consequence of high viscosity solvent seems to block the chromium ion from reacting with the carrier at the feed-membrane

interface. Hence longer time is needed for chromium ion to pass through the membrane phase. Besides, Chang *et al.* [38] also found that the non-polarity nature of vegetable palm oil tends to interact weakly with polar compounds such as chromium resulting in poor solubility of solute ion in the organic membrane phase. As referring to the liquid membrane loss analysis, liquid membrane containing palm oil suffered higher loss compared to the mixture of palm oil and kerosene as well as fully kerosene. This means the liquid membrane containing fully palm oil seems unstable and easily loss the liquid membrane into the aqueous phase.

Thus, it can be inferred that the sequence of diluent composition which significantly enhanced the extraction and recovery efficiency is as in the following order: 100% kerosene> mixture of kerosene and palm oil (50:50)> 100% palm oil. However, as a way to promote green technology, mixture of kerosene and palm oil (50:50) was employed for the next investigation.
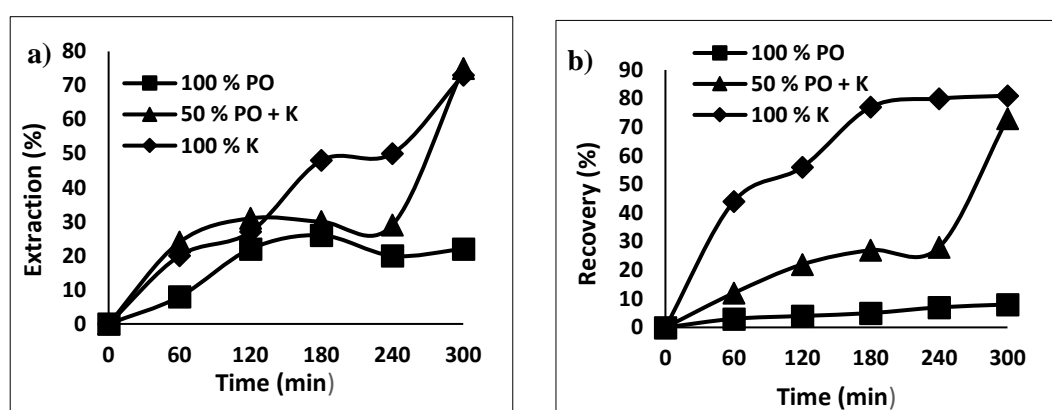

Figure 3. (a) Effect of diluent composition on chromium extraction and (b) Effect of diluent composition on chromium recovery where PO=palm oil and K=kerosene.

Table 2. Variation of permeability and liquid membrane loss with respect to the effect of diluent composition.

Diluent composition	Permeability, ρ $x10^{-4}$ (cms^{-1})	Liquid membrane loss (gcm^{-2}) $x10^{-3}$	Viscosity (cP)
100% palm oil	2.1	4.7	72
50% palm oil + 50% kerosene	4.2	1.1	41
100% kerosene	6.3	2.2	32

3.4. Effect of carrier concentration

The carrier has a profound significance in SLM process by acting as a shuttle in transporting metal ion from feed to the stripping phases through membrane phase. Theoretically, the rate of metal ion permeation increases with a rise in carrier concentration [36]. Therefore, in order to understand the effect of carrier concentrations towards extraction and recovery performance of chromium ion, an equimolar concentrations of blended TOA-TOMAC were varied from 0.05 to 0.35M as shown in figure 4. In the meantime, the variations of the permeability values, viscosity, and liquid membrane loss with respect to the different carrier concentration are tabulated in table 3. According to figure 4(a), it can be noted that the extraction percentage of chromium ion within 5 hours of extraction time showed a steady increase from 21 to 75% upon enhancing the blended TOA-TOMAC concentration from 0.05 to 0.20M, respectively. Obviously, low permeation of chromium ion in the membrane phase (2.1×10^{-4} cms^{-1}) is triggered by low carrier concentration of 0.05M. Apparently, low carrier concentration is inadequate to assist the mass transfer of chromium ion transportation in the membrane phase. This seems to be

consistent with Altin et al. [39] who found that the low quantity of carrier molecules in the membrane phase cause less transportation of targeted solute into the stripping phase. However, an increment of carrier concentration up to 0.20M actually provides high number of carrier molecules available for the formation of chromium-carrier complex at the feed-membrane interface. This behaviour led to higher permeation rate of chromium ion which was almost doubled (4.2×10^{-4} cms^{-1}) as well as improving the extraction efficiency. Beyond 0.20M, a sudden fall of extraction efficiency (26%)was observed. The reduction of permeation rate to 3.1×10^{-4} cms^{-1} can be adversely affected by the viscosity effect. It was proven by an increment trend of viscosity from 36 to 53cP as the carrier concentration ws increased from 0.05 to 0.35M, respectively. The viscous membrane phase seems to inhibit the diffusion of chromium ion across the membrane phase, which thereby leading to an extraction inefficiency. Similar result was reported by Rehman et al. [40] who indicated that according to Stokes–Einstein in equation (10), diffusity is inversely proportional to viscosity. Thus, lower viscosity has higher solute ion diffusion through membrane phase and vice versa.

$$D = \frac{KT}{6n\pi r} \qquad\qquad\qquad (10)$$

where T is the absolute temperature, K is the Boltzmann constant, r is the ionic radius of solute and n is the viscosity of the organic phase in cP.

On the other hand, figure 4(b) illustrates the recovery performance of chromium ion as a function of different carrier concentrations. Notably, recovery efficiency showed similar trend with extraction which implies a simultaneous extraction and stripping reaction during SLM process. At low concentration of 0.05M, the recovery efficiency provided a slow and steady increase up to 15% in 5 hours of experiment. A possible explanation for this situation is the insufficient number of carrier molecules present in the membrane phase, thus retards the chromium ions transportation into the stripping phase. This behaviour was also experienced by Sulaiman and Othman [3] who believed that the low carrier concentration tends to reduce the mass transfer of chromium ions, hence accumulating these complex in the feed-membrane interface without being transported into the stripping phase.
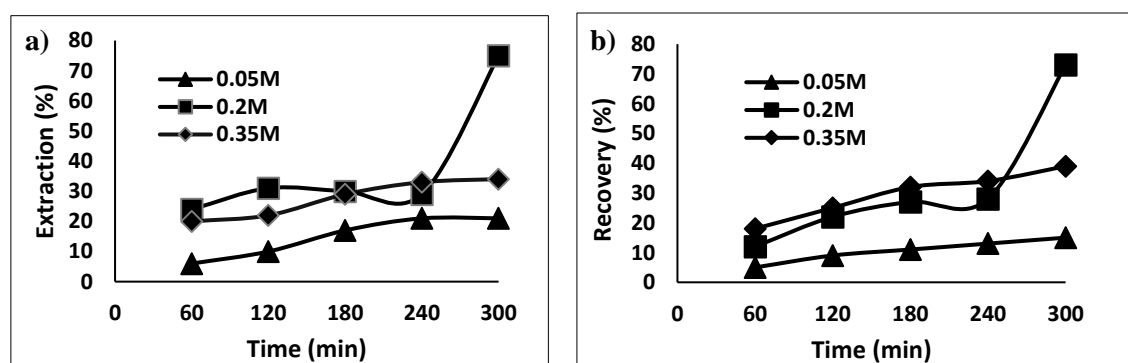

Figure 4. (a) Effect of carrier concentration on chromium extraction and (b) Effect of carrier concentration on chromium recovery where PO=palm oil and K=kerosene.

Surprisingly, further increment of carrier concentration up to 0.20M, the recovery percentage significantly increased up to 73% in 5 hours of experiment. It is due to the high number of carrier molecules which facilitates the permeation of chromium complexes in the membrane phase. Basically, the basic carriers of TOA and TOMAC containing amine group has high tendency in forming hydrogen bonding with water molecules in the aqueous phase, thus is able to create water channels through the membrane pores. This behaviour promotes direct channeling between the feed and stripping phases. For a longer experiment, the liquid membranes in the pores slowly oozes out into the aqueous phase, thus affecting the recovery efficiency.This is in accordance with Zhang et al. [35] who claimed that the

lifetime of SLM process not only relies on the porosity but also on the ability of the membranes to keep the pores free from water channels.

Meanwhile, further increment of carrier concentration to 0.35 M, resulted to steady increment of the recovery percentage up to 29% in 5 hours of extraction time. The low recovery might be due to the high viscosity membrane phase that is crowded with high number of chromium-carrier complexes without being stripped. It can also be noted that the liquid membrane loss seemed to increase in about twice $(2.1 \times 10^{-3} \text{ g/cm}^2)$. According to Huidong et al.[41], the liquid membrane is not completely insoluble in an aqueous solution and a certain degree of solubility exists at the aqueus-membrane interface. An increase in carrier concentration, increases the solubility of carrier at the aqueus-membrane interface. As a result, this phenomena reduce the interfacial tension among both phases and the aqueous solution is able to wash away the liquid membrane. Thus the best condition was obtained at 0.20 M of TOA-TOMAC with 75 and 73% of extraction and recovery, respectively

Table 3. Variation of permeability, viscosity, and liquid membrane loss as a function of the effect of carrier concentration.

[TOA-TOMAC](M)	Permeability, ρ x 10^{-4} (cms^{-1})	Viscosity (cP)	Liquid membrane loss (gcm^{-2}) x 10^{-3}
0.05 -0.05	2.1	36	1.9
0.20 -0.20	4.2	41	1.1
0.35 -0.35	3.1	53	2.1

Conclusion

Through this work, the SLM process is shown to be a promising method due to its capabilities in extracting the hazardous hexavalent chromium complex from aqueous solution. Besides, the feasibility of palm oil as a substitute green diluent in SLM process has been proven. This green process has high potential to be applied in industrial level for the treatment of wastewater containing heavy metal ions.

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