

## Phenol Removal from Aqueous Solution Using Emulsion Liquid Membrane Process: Batch Experimental Studies

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### Abstract

The present study deals with the removal of phenol from aqueous solution using the emulsion liquid membrane technique. Kerosene was used as a diluent, sorbitan monooleate (Span 80) was used as a surfactant and sodium hydroxide as the stripping agent. The effect of various parameters such as surfactant concentration in the range 1 to 6 (v/v)%, stirring speed in the range 100 to 600 rpm, volume ratio of membrane phase to internal phase in the range 1:1 to 6:1, NaOH as the internal phase concentration 0.01 to 3 M, stirring time in the range 2 to 10 min, volume ratio of membrane phase to the external phase in the range 1:1 to 1:6 and emulsification time in the range 20 to 120 second was studied. The maximum percentage removal for phenol was found to be 98.95 at the best preparation conditions were surfactant concentration 2% (v/v), stirring speed 400 rpm, volume ratio of membrane phase to internal phase 5:1, NaOH concentration 2 M, stirring time 4 min, volume ratio of membrane phase to the external phase 1:2 and emulsification time 1 minute. The increase of surfactant concentration increases the removal efficiency to a certain extent of 2% (v/v), the higher stirring speed the higher percentage of phenol removal until 400 rpm, increasing of NaOH concentration increases the removal of phenol, the removal efficiency of phenol increases with increasing volume ratio of membrane phase to internal phase up to 5:1 and decreases thereafter, the removal percentage increases with the increase of stirring time until it reaches a maximum value then starts to decrease, the increasing in the external phase volume lead to the reduction of phenol removal and the increase in emulsification time increases the phenol removal, but for long emulsification time the removal dropped.

**Key words: Phenol, Emulsion liquid membrane, Surfactant, Emulsification**

### 1. Introduction

Phenol and its derivatives are very common organic water pollutants induced by the industrial processes since phenol is an important raw material in various chemical, pharmaceutical and petrochemical processes. It has negative impacts

for ecosystems because it is toxic even at very low concentrations. Its presence in natural waters can lead further to the formation of chlorophenols during disinfection and oxidation processes, which are carcinogenic compounds [1].

Membrane separation processes are one of the most widely researched and fastest growing separation techniques of our century because of their advantages compared to traditional processes such as absorption, liquid-liquid extraction, distillation, etc. [2]. The interest in the mass transfer through membranes can be attributed to membrane processes being technically simple and having low energy consumption [3].

Emulsion liquid membrane is one of the techniques to remove and recover contaminants in wastewaters. It has been given considerable attention by researchers due to its outstanding characteristics such as simultaneous pollutants removal and materials recovery in a single unit, non-equilibrium mass transfer, high selectivity, high fluxes, reusability and low energy consumption [4]. Liquid membranes have shown potential for the removal of phenol from wastewater. They are selective permeable materials that transport certain targeted solutes. Among the different types of liquid membranes, emulsion liquid membrane (ELM) provides several advantages such as a high interfacial area for extraction, versatility, relatively low cost and a non dependence on equilibrium consideration [5].

Liquid membranes consist of three distinct phases, the feed phase, the membrane phase and the stripping phase. The feed phase, also called the external phase, is the water

containing the metal or the other species to be extracted. The stripping phase called the internal phase is where the metal will be trapped. The different phases are defined for a simultaneous extraction and stripping to occur; the separation is achieved when permeation occurs from the aqueous feed phase to the receiving stripping phase. There are three different kinds of liquid membrane: bulk liquid membrane (BLM), supported liquid membrane (SLM) and emulsion liquid membrane (ELM). Among these membranes, the double emulsion in ELM achieves the highest mass transfer area [6].

Emulsion liquid membranes are known as double emulsions system. In general, they are formed by first making an emulsion of two immiscible phases and then dispersing the emulsion in a third phase (external phase). The liquid membrane phase refers to the phase in between the encapsulated phase in the emulsion and the external phase. Usually the encapsulated phase and the external phase are miscible. But they are not miscible with the membrane phase [7]. Solute extraction is achieved by mass transfer between the two miscible phases through the membrane phase [8]. In order to form a stable and effective W/O emulsion, the volume ratio must exceed 1:1 [9].

ELM Process can be divided into four stages as shown in **Fig.** and as follows [6, 10]:

1. Emulsification of the membrane and internal phase
2. Permeation (emulsion - external phase contacting)
3. Settling (separation of the emulsion and external phase after extraction)
4. De-emulsification, to recover the membrane phase.

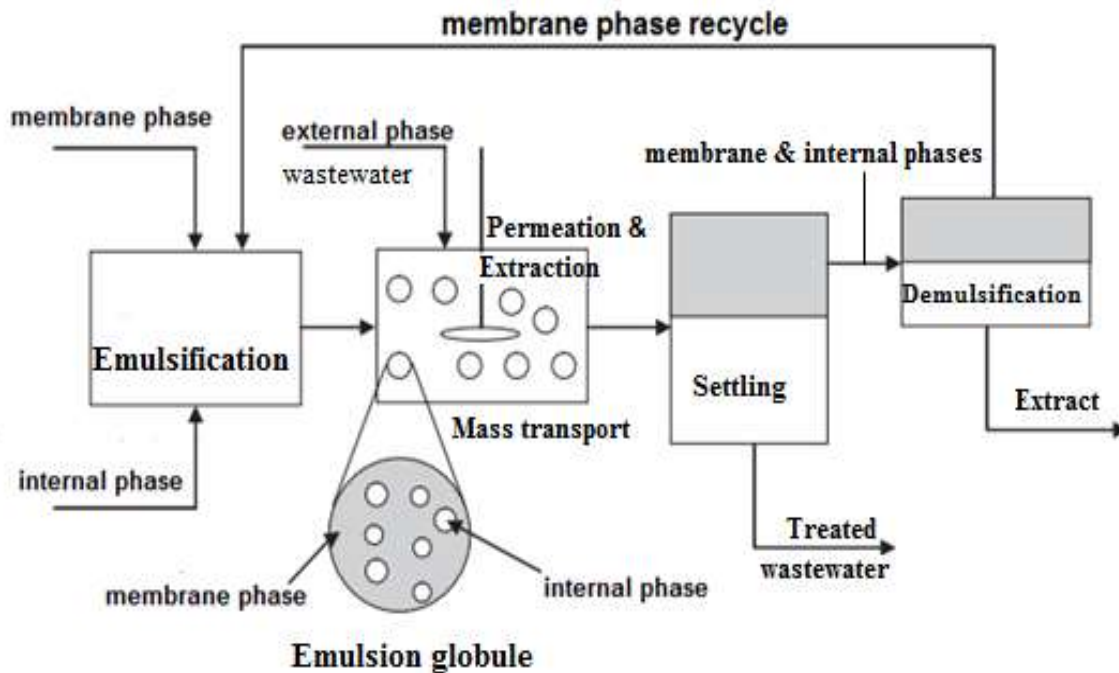


Fig. 1. The operational steps in the ELM process [11]

### 1.1 Aim of the Present Work

The main aim of this present work is to separate phenol from the aqueous solution by ELM using kerosene as a diluent, sodium hydroxide as a stripping agent and sorbitan monooleate (Span 80) as a surfactant and identify the optimal parameter values that would result in a maximum removal of phenol, several variables will be studied such as surfactant concentration, volume ratio of membrane to internal phase, stirring speed, volume ratio of membrane to external phase ratio,

internal agent concentration, stirring time and emulsification time.

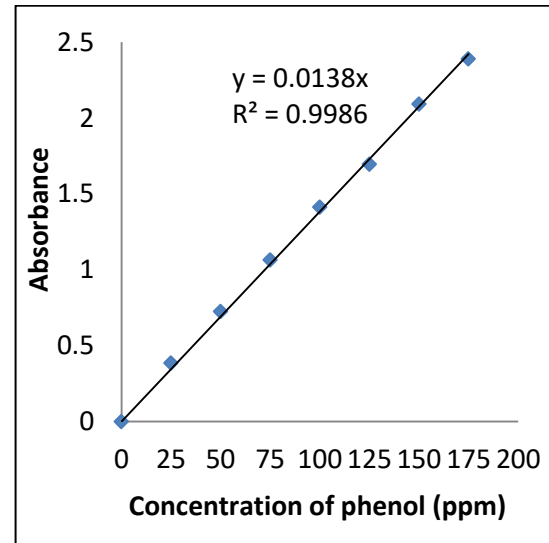
### 2. Experimental Work

1. Phenol solution of 300 ppm was prepared by dissolving phenol crystals in distilled water, molecular weight of phenol 94.111 g/mol, distinct aromatic, acrid odor, density 1.0576 g/cm<sup>3</sup>.
2. NaOH solution was prepared by the same method as phenol solution preparation, i.e. dissolving NaOH solids into distilled water.

3. Speed homogenizer was used as a ULTRA-TURRAX JANKE & KUNKEL KG for emulsion preparation, Heidolph overhead stirrer RZR 2020 Mixer System was used for the ELM extraction studies and UV-Visible Spectrophotometer (Thermo Electron Corporation Madison WI 53711 USA) was used for measuring the concentration of phenol in water. A 1mL of treated water sample was taken and analyzed by Spectrophotometer for measuring absorbance for phenol concentration. Detection of phenol can be observed at an absorbance value of 270nm. The concentrations of phenol were estimated from the absorbance-phenol concentration calibration curve. The percentage removal of phenol was then determined by equation 1. Calibration curve for absorbance - phenol concentrations **Fig.2** were prepared for checking the absorbance of phenol solution by using different known concentration samples.

$$\text{Percentage removal of phenol} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

Where  $C_i$  is the initial concentration of phenol in external phase and  $C_f$  is the final phenol concentration in the sample after extraction.



**Fig. 2. Calibration curve of phenol adsorption**

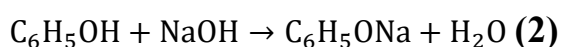
4. A batch experiment was conducted as follows: (Two beakers were used, one as the emulsifier and the other as the contactor)

(a) Emulsification: A water in oil (w/o) emulsion was made by mixing the sodium hydroxide solution at various concentration (0.01 to 3 M ) and the mixture of surfactant and kerosene in different proportions by means of high speed homogenization operating at a rotational speed of 10,000 rpm for various time (20 to 120 second) so as to obtain a milky white color liquid membrane as shown in **Fig.3**.



**Fig. 3. The obtained milky white colour liquid membrane**

(b) Phenol permeation: In the contactor a W/O/W emulsion (water/oil/water) was made by adding the W/O emulsion to the aqueous phenol solution in the contactor, the mixture was stirred by Heidolph overhead stirrer RZR 2020 with a low rotational speed (100 to 600 rpm) with varying stirring time (2 to 10 min) at a definite volume ratio of membrane to aqueous phenol solution to form numerous small globules of emulsion so that good dispersion of the emulsion in the aqueous solution was maintained for mass transfer of phenol, the emulsion must be freshly prepared each time before the extraction experiment. Phenol permeated into the liquid membrane and reacted with NaOH, which was the internal stripping agent to yield sodium phenolate and water. The reaction is:



Sodium phenolate  $C_6H_5ONa$  cannot diffuse back into the external phase

through liquid membrane due to the selectivity of the membrane. Hence, it was not detected in the external phase, which in this case, was phenol aqueous solution.

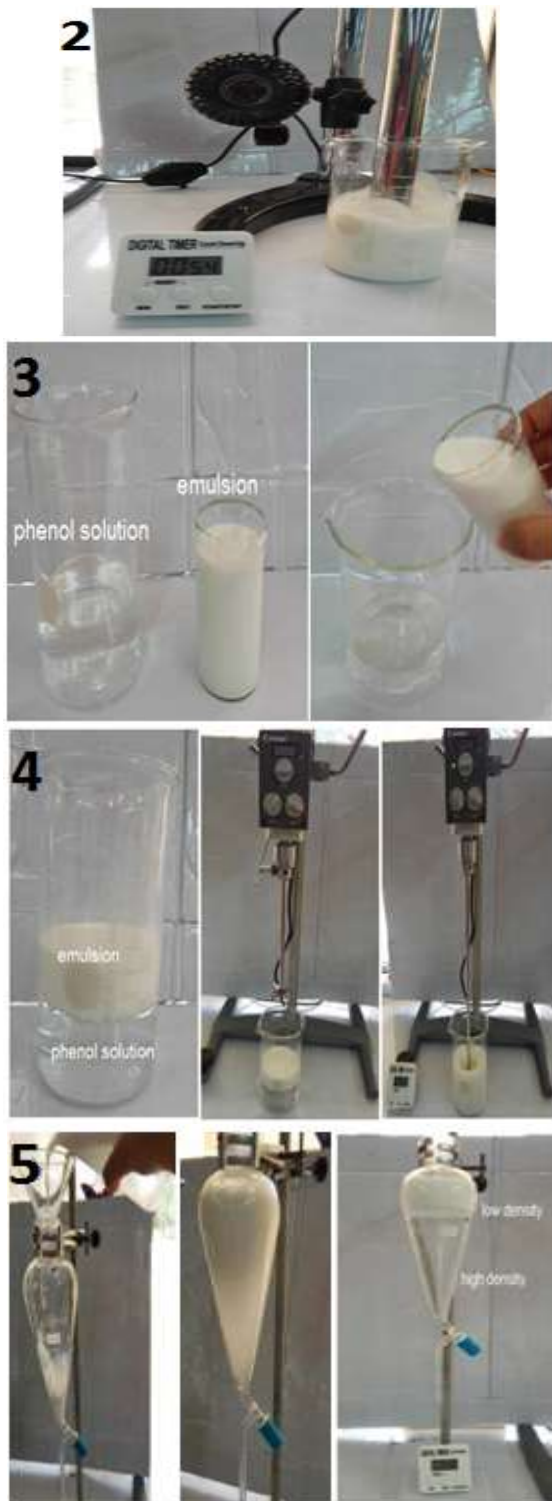
c) Settling for separation of the emulsion and external phase after extraction: after the agitation, the mixture is separated using separating funnel. As the external phase was heavier than the emulsion phase, it settled at the bottom. After the separation of the phases, the aqueous phase was carefully separated from the membrane phase, then the solution separated into two layers (the emulsion and the treated water), the steps were shown in **Fig.4** (steps 1 to 5).

After 7 minutes, samples taken from the treated water (bottom layer) and analyzed by UV spectrophotometer to determine the percentage removal of phenol.



**Fig. 4. Experiment procedure steps (1) adding membrane phase to internal phase**





**Fig. 4. Experiment procedure steps**  
**(2) mixing for 1 min to get milky white emulsion**  
**(3) adding emulsion to phenol solution**

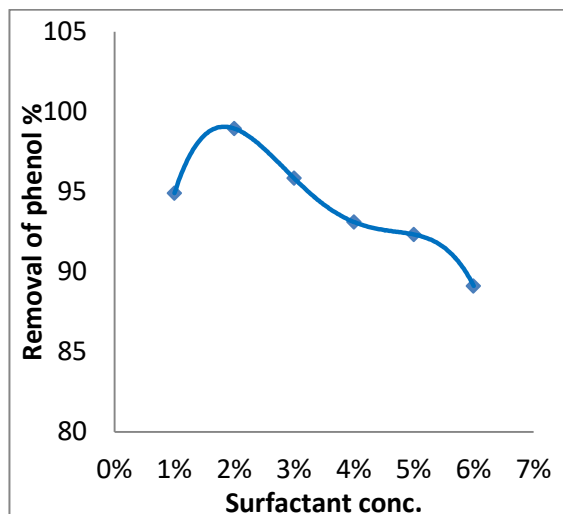
- (4) emulsion and phenol solution are separated into two layers do not mix without stirring**  
**(5) adding the mixture to a separating funnel and waiting for 7 min to take the sample.**

### 3. Results and Discussions

#### 3.1 Influence of surfactant concentration

The surfactant concentration has been studied in the range 1 to 6 (v/v) % and shown in **Fig.5**, which indicates that:

Too little concentration of surfactant 1% (v/v) makes the emulsion breaks easily so that the extraction efficiency was poor because the coverage of the membrane interface was incomplete at low surfactant concentration. The addition of more surfactant (1 to 2) % (v/v), increased the removal of phenol due to the increasing of the surface tension and results in smaller globules size of the W/O stable emulsion, which leading to a higher mass transfer area with a maximum extraction rate. Excessive amount of surfactant (3 to 6) % (v/v), increases the viscosity of the membrane phase which decreases the removal of phenol through the highly viscous membrane. These observations about the behavior are in good agreement with most investigators such as Othman et al., and Manikandan et al. [8, 12]. Therefore, Span 80 of concentration 2% (v/v) was found to be the optimum.



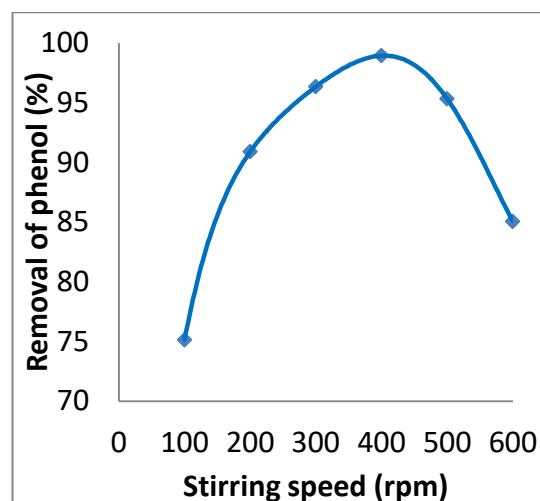
**Fig. 5. Effect of surfactant concentration on removal of phenol from aqueous solution (membrane to internal phase ratio= 5:1, membrane to external phase ratio= 1:2, NaOH concentration= 2 M, stirring speed= 400 rpm, stirring time= 4 min and emulsification time= 1 min)**

### 3.2 Influence of stirring speed

The stirring speed has been studied in the range 100 – 600 rpm as shown in Fig.6.

For lower stirring speed (100 rpm), the extraction efficiency was low because the formations of larger emulsion globules involving a decrease of the area for mass transfer. Also it was observed that higher stirring speed (over than 100 rpm) lead to the formation of smaller sized globules, which increasing the interfacial area between the external phase and the membrane phase so that the extraction efficiency increases. Further increase in the level of stirring would increase the interfacial area and the mass transfer coefficient. The area for mass transfer increases but the membrane

ruptures, spilling the internal stripping phase into the outer external phase. Increasing the stirring speed above (400 rpm) not only decreases the extraction efficiency, but also affects the stability of emulsion. These observations were in good agreement with most investigators such as Ng et al., Chiha et al., Kaghazchi et al., Dâas, A. and Hamdaoui [5, 13, 14, and 15]. The best value of stirring speed was found to be 400 rpm.



**Fig. 6. Effect of stirring speed on removal of phenol from aqueous solution (membrane to internal phase volume ratio= 5:1, membrane to external phase volume ratio= 1:2, concentration of Span 80= 2(v/v) %, NaOH concentration= 2 M, stirring time= 4 min and emulsification time= 1 min)**

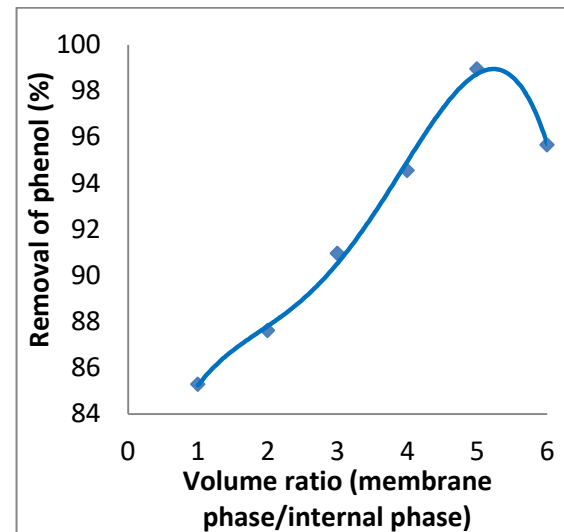
### 3.3 Influence of volume ratio of membrane phase to internal phase

The effect of the volume ratio of membrane to internal phase on the percentage removal of phenol was studied by changing the volume of

membrane phase while keeping the volume of the internal phase constant in the range from 1: 1 to 5:1 as shown in **Fig.7**. At low volume ratio of membrane phase to internal phase (1:1), the volume of membrane solution is not enough to enclose the overall stripping solution thus producing large emulsion globule leading to low extraction efficiency. When volume ratio of membrane phase to internal phase is increased from 2 to 5, the transport rate and extraction efficiency of phenol increase, this can be explained by the more stable emulsion due to the higher concentration of the surfactant at the interface of the membrane/external phases and due to the fact that an increase in the membrane phase volume fraction increases the thickness of the membrane phase and the viscosity of the emulsion phase, resulting in enhanced mass transfer and more stable emulsion droplets can be formed by an increment of the membrane phase to encapsulate the internal agent.

In addition, it was also found that increasing the volume ratio of membrane to internal phase beyond 5:1 did not enhance phenol removal because a high volume ratio of membrane phase to internal phase means that less stripping agent (NaOH) is available for phenol stripping, also because too much membrane phase produces thick emulsion wall which is not favorable for the extraction process. Thus, a

volume ratio of membrane phase to internal phase of 5:1 was selected as the best ratio. This observation is in good agreement with investigators such as Ng et al., Mortaheb et al., Ahmad et al. [5, 16 and 17].



**Fig. 7. Effect of membrane: internal phase ratio on removal of phenol from aqueous solution (membrane to external phase ratio= 1:2, concentration of Span 80= 2(v/v) %, NaOH concentration= 2 M, stirring speed= 400 rpm, stirring time= 4 min and emulsification time= 1 min)**

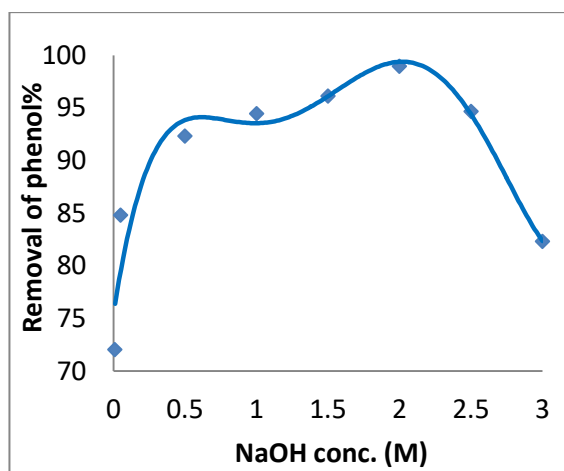
### 3.4 Influence of internal agent concentration on removal of phenol

A series of experiments were carried out in the range from 0.01 to 3 M to investigate the influence of sodium hydroxide concentration on removal efficiency of phenol in ELM as shown in **Fig.8**. It was found that increasing of NaOH concentration from 0.01 to 2 M will increase the removal efficiency because NaOH in internal phase converts phenol to sodium phenolate and traps it in the internal phase. Therefore, high



concentration of NaOH may be preferred for the extraction process.

In addition, excessive NaOH concentrations of 3M will decrease the removal efficiency, this decreasing of the removal efficiency due to the reaction of sodium hydroxide with Span 80, which results in a reduction in the properties of the surfactant by basicity in the internal phase that consequently led to a destabilization of the emulsion, also the increasing the amount of sodium hydroxide decreased the difference of densities and increased the emulsion viscosity which reflected in an increasing in the size of drops. NaOH concentration of 2 M is suitable for the efficient removal of phenol. These observations are in good agreement with investigators such as Dâas et al., and Wan et al. [15, 18].



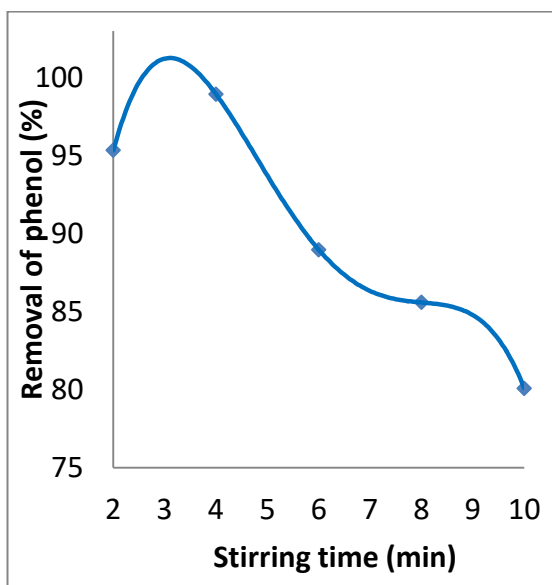
**Fig. 8.** Effect of NaOH concentration in internal phase on removal of phenol from aqueous solution (membrane to internal phase ratio= 5:1, membrane to external phase ratio= 1:2, concentration of Span 80= 2(v/v) %, stirring speed= 400 rpm, stirring time= 4 min and emulsification time= 1 min)

### 3.5 Influence of stirring time on removal of phenol

The stirring time influences the mass transfer during the extraction. It is defined as the mixing time of the three phase W/O/W emulsion system. Effect of stirring time on phenol extraction by ELM was shown in **Fig.9**. It is observed that the extraction percentage increases with the increase of stirring time between the emulsion and the external phase until it reaches a maximum value then starts to decrease.

Although 2 min gave a quite good phenol removal, it was a little bit short to give the highest removal because the contact between the external phase and the emulsion was not enough to react phenol with the internal agent and extract it into the membrane phase, thereby the concentration of phenol in the external phase remains somewhat high. An increase in the stirring time of the double emulsion increased the phenol removal by allowing the participant species a longer time to react, such that the diffusion of NaOH species through the membrane was enhanced. At longer stirring time (> 4 min), the emulsion instability results in partial membrane rupture and spillage of the trapped phenol back into the external phase at a rate exceeding the ability of emulsion to reabsorb it, this was due to the reaction of NaOH with Span 80, which results in a

partial loss of its surfactant properties, which promotes the emulsion instability. The optimum stirring time was observed about 4 min stirring with the external phase. These observations are in good agreement with most investigators such as Ismail et al., Chanukya and Rastogi [19, 20].



**Fig. 9. Effect of stirring time on removal of phenol from aqueous solution (volume ratio of membrane to internal phase = 5:1, membrane to external phase ratio= 1:2, concentration of Span 80= 2(v/v) %, NaOH concentration= 2M, stirring speed= 400 rpm and emulsification time= 1 min)**

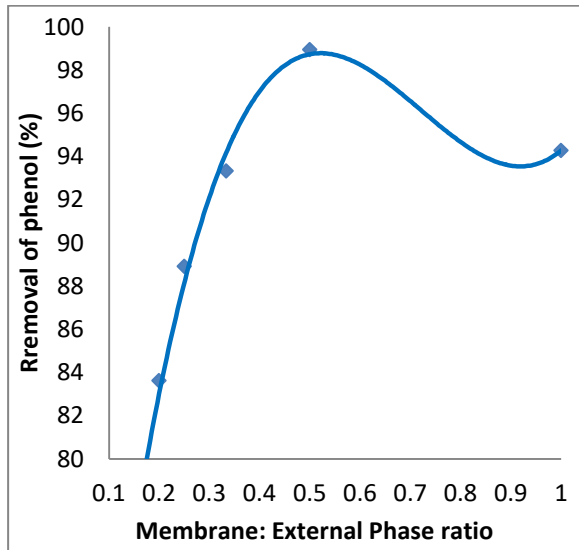
### 3.6 Influence of volume ratio of membrane to external phase on removal of phenol

The volume ratio of the membrane phase to the phenol solution affects the interfacial mass transfer across ELMs. The ratio was varied from 1:1 to 1:6 as shown in **Fig.10**.

The effect of this ratio was studied by changing the volume of external

aqueous phase while keeping the volume of the membrane phase constant. The study revealed that with the increase of the external phase volume, the membrane area per total external volume in the system was being reduced. This may lead to the reduction of phenol permeation flux into the membrane phase. The experiments proved that the percentage removal of phenol was lower at a low membrane: external phase ratio of 1:1 v/v. The explanation was that the coalescence of emulsion occurred under a high volume ratio of membrane phase to external phase due to the ineffectiveness of dispersion by stirring. The coalescence of emulsion reduces the total surface area for extraction, thus reducing the percentage removal of phenol. Also the study revealed that further increase in this ratio resulted in reduction in the percentage of phenol removal. This drop may be due to decrease in area of contact between the emulsion and external phase as the volume of external phase was increased.

Membrane to external phase volume ratio of 1:2 was chosen for further studies, as this ratio resulted in the highest percentage removal of phenol in order to ensure a good dispersion of the W/O emulsion in the external phase, this observation is in good agreement with most investigators such as Ng et al., Abbassian and Kargari, Laki et al. [5, 21 and 22].



**Fig. 10.** Effect of volume ratio of membrane to external phase on removal of phenol from aqueous solution (membrane to internal phase ratio= 5:1, concentration of Span 80= 2(v/v) %, NaOH concentration=2 M, stirring speed= 400 rpm, stirring time= 4 min and emulsification time= 1 min)

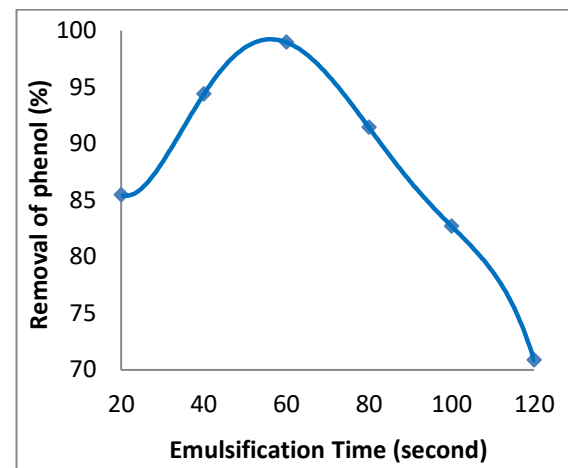
### 3.7 Influence of emulsification time on removal of phenol

The effect of emulsification time on the removal of phenol was obtained as shown in **Fig.11**.

For insufficient emulsification time (<1 min), the phenol extraction efficiency was increased slowly, because short emulsification time will cause the formation of large globules, where a less interfacial area reduced the mass transfer rate. It was also observed that the increase in emulsification time from 20 to 60 second increased phenol removal and forms a stable emulsion with a highest removal of phenol in 1 min, it is due to the reduction of the size of internal phase droplets containing NaOH and enhances the homogeneity of the external phase.

Also the long time of emulsification led to a significant increase of the leakage this was probably caused by the coalescence of the internal droplets and the breakage was increased due to high internal shearing and prolonged exposure of the emulsion to high speed.

Therefore, an emulsification time of 1 min was chosen throughout the study. This observation is in good agreement with most investigators such as Ng et al., Park and Chung, Gasseret al. [5, 23 and 24].



**Fig. 11.** Effect of emulsification time on removal of phenol from aqueous solution (membrane to internal phase volume ratio= 5:1, membrane to external phase volume ratio= 1:2, concentration of Span 80= 2(v/v) %, NaOH concentration= 2M, stirring speed= 400 rpm and stirring time = 4 min)

### 4. Conclusion

It was demonstrated that the ELM technique was very promising in the treatment of aqueous solutions containing phenol.

The increase of surfactant concentration increases the removal

efficiency to a certain extent of 2% (v/v), an excess of surfactant concentration lead to decrease percentage of phenol removal. The suitable Span 80 concentration used in liquid membrane component was 2% (v/v) that provides good emulsion stability during the ELM process. The higher stirring speed the higher percentage of phenol removal until 400 rpm and excessive speed enhance the decrease percentage of phenol removal when other conditions of the process remained constant. The internal phase reagent concentration has a great impact in the extraction efficiency of phenol. Increasing of NaOH concentration increases the removal efficiency, excessive NaOH concentrations decreases the removal efficiency. NaOH of 2 M concentration as the internal phase concentration was suitable for the efficient removal of phenol. The removal efficiency of phenol increases with increasing volume ratio of membrane phase to internal phase up to 5:1 and decreases thereafter when other conditions of the process remained constant. The extraction percentage increases with the increase of stirring time between the emulsion and the external phase until it reaches a maximum value then starts to decrease. The optimum stirring time on the phenol extraction was found about 4 min stirring with the external phase. The volume ratio of the membrane phase to the phenol solution affects the interfacial mass transfer across ELMs. It was found

that the increasing in the external phase volume lead to the reduction of phenol permeation flux into the membrane phase. The best value of membrane to external phase ratio was observed to be 1:2. The highest removal of phenol was attained at the emulsification time of 1minute. It was observed that the increase in emulsification time increases the phenol removal, but for long emulsification time the removal dropped. The maximum predicted value for the percentage removal of phenol using ELM is 98.95% and the optimum parameters were found to be: surfactant concentration 2% (v/v), internal agent concentration 2 M, emulsification time 1 min, stirring time 4 min, membrane to internal ratio 5:1, stirring speed 400 rpm and membrane to external phase ratio 1:2.

### 5. Acknowledgement

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### 6. Abbreviation

ELM	emulsion liquid membrane
rpm	rotation per minute
W/O	water in oil Emulsion
W/O/W	water in oil in water

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## إزالة الفينول من المحلول المائي عن طريق عملية الغشاء السائل المستحلب: دراسات تجريبية باستخدام النمط الدفعي

أ.م.د. نجوى صابر مجيد

منال عدنان محمد

قسم الهندسة الكيمياءوية- جامعة بغداد / العراق

### الخلاصة

تتناول هذه الدراسة إزالة الفينول من المحلول المائي باستخدام تقنية الغشاء السائل المستحلب. وقد استخدم الكيروسين كمخفف، وتم استخدام (Span 80) كمادة منشطة للسطوح و هيدروكسيد الصوديوم كعامل انتزاع. تم دراسة تأثير . تأثير معايير مختلفة مثل تركيز المادة المنشطة للسطوح في حدود 1-6 (حجم / حجم) %، سرعة التحريك في حدود 100-600 دورة في الدقيقة، والنسبة الحجمية لمرحلة الغشاء إلى المرحلة الداخلية في حدود 1 : 1 - 6 : 1 ، هيدروكسيد الصوديوم كتركيز للمرحلة الداخلية، 0.01-3 M، ووقت التحريك في حدود 2 إلى 10 دقيقة، والنسبة الحجم لمرحلة الغشاء إلى المرحلة الخارجية في حدود 1 : 1 - 6 : 1 ووقت الاستحلاب الوقت في حدود 20-120 الثانية تم دراستها. تم إيجاد أعلى نسبة لإزالة الفينول وكانت 98.95 بأفضل ظروف تحضير وهي تركيز المادة المنشطة للسطوح 2% (حجم / حجم)، سرعة التحريك 400 دورة في الدقيقة، والنسبة الحجمية لمرحلة الغشاء إلى المرحلة الداخلية 1 : 5 ، تركيز هيدروكسيد الصوديوم 2M، ووقت التحريك 4 دقائق، والنسبة الحجم لمرحلة الغشاء إلى المرحلة الخارجية 1 : 2 ووقت الاستحلاب 1 دقيقة. زيادة تركيز المادة المنشطة للسطوح تزيد من كفاءة الإزالة إلى حد معين من 2% (حجم / حجم)، وزيادة سرعة التحريك تزيد من إزالة الفينول حتى 400 دورة في الدقيقة، وزيادة تركيز هيدروكسيد الصوديوم يزيد من إزالة الفينول، وكفاءة إزالة الفينول تزداد مع زيادة النسبة الحجمية لمرحلة الغشاء إلى المرحلة الداخلية تصل إلى 5:1 وتنخفض بعد ذلك، وتزداد نسبة الإزالة مع زيادة وقت التحريك حتى يصل الحد الأقصى ثم تبدأ في الانخفاض، وزيادة المرحلة الخارجية تزيد في الحد من إزالة الفينول والزيادة في الوقت استحلاب تزيد من إزالة الفينول، ولكن عندما كان وقت الاستحلاب طويل انخفضت الإزالة.

الكلمات الرئيسية: فينول، الغشاء السائل المستحلب، المادة المنشطة للسطوح، الاستحلاب