Demulsification of Remaining Waste (Water In Oil Emulsions) After Removal Of Phenol In Emulsion Liquid Membrane Process

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ABSTRACT

The aim of present work is to study the removal of phenol present in aqueous feed solution by the emulsion liquid membrane technique using kerosene as a diluent, sodium hydroxide as a stripping agent, and sorbitan monooleate (Span 80) as a surfactant. The parameters studied were: surfactant concentration, volume ratio of membrane phase to internal phase, and stirring speed. It was found that more than 98% of phenol can be removed at the conditions were surfactant concentration 2% (v/v), volume ratio of membrane phase to internal phase 5:1 and stirring speed 400 rpm. Maximum phenol extraction efficiency at 7 minutes of process time was observed. It was found that there was a good agreement between the standard kerosene and the upper layer that resulted after the demulsification of the remaining waste by applying centrifuge. Thus, it is possible to reuse this layer to prepare a new emulsion of the membrane phase.

Keywords: demulsification, phenol, emulsion liquid membrane, centrifuge, extraction time.

كسر الاستحلاب للمخلفات المتبقيه (مستحلبات الماء في النفط) بعد إزالة الفينول بعملية الغشاء السائل المستحلب

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الخلاصة

الهدف من هذا العمل هو دراسة إزالة الفينول من المحاليل المائية باستخدام تقنية الغشاء السائل المستحلب بواسطة استخدام الكيروسين كمخفف، هيدروكسيد الصوديوم كعامل انتزاع و (Span 80) كمادة منشطة للسطوح. تم دراسة العوامل وهي: تركيز المادة المنشطة للسطوح, النسبة الحجمية لمرحلة الغشاء إلى المرحلة الداخلية، و سرعة التحريك. وقد وجد أن أكثر من 98٪ من الفينول يمكن ازالته عند الظروف وهي تركيز للمادة المنشطة للسطوح 2٪ (حجم / حجم)، النسبة الحجمية لمرحلة الغشاء إلى المرحلة الداخلية 5:1 , و سرعة التحريك 400 دورة في الدقيقة. و لوحظ ان أقصى كفاءة لاستخراج الفينول في 7 دقائق من وقت العملية. و وجد أن هناك توافق جيد بين الكيروسين القياسي والطبقة العليا الناتجه بعد عملية كسر الاستحلاب للمخلفات المتبقية من خلال تطبيق جهاز الطرد المركزي. وبالتالي, فإنه من الممكن إعادة استخدام هذه الطبقة لتحضير مستحلب جديد لمرحلة الغشاء.

الكلمات الرئيسية: كسر الاستحلاب, فينول، الغشاء السائل المستحلب، الطرد المركزي، وقت الاستخلاص.

1. INTRODUCTION

As the phenol is a significant basic material in various chemical, petrochemical and pharmaceutical processes, it and all derivatives have become very common organic water contaminants resulted from these industrial processes. Even at low concentration in water, phenol is considered a toxic material and has unfavorable impacts for ecosystems. Moreover, carcinogenic compounds are chlorophenols that are formatted from disinfection and oxidation processes due to the existence of phenol in natural waters **Hasanoğlu, 2013.** Less than 1 part per billion (ppb) of phenol in surface water has set as standard by the Environmental Protection Agency (EPA) because of the toxic nature of this compound. It is necessitated and very significant challenges as well as representing a dynamic field of research to remove phenol from industrial drainage by virtue of environmental laws and regulations governing safe discharge levels are being so strict **Meda et al., 2014**.

The available treatment methods used for removing phenol from aqueous solutions are divided into two major groups: traditional and advanced methods, according to the concentration of phenol, traditional methods involve adsorption, distillation, extraction, chemical oxidation, and biodegradation. The advanced methods are classified into membrane separation processes and photo oxidation processes **Mohammadi et al., 2015**. Membrane separation processes are one of the most widely researched and fastest growing separation processes of our century because of their advantages compared to traditional processes such as liquid-liquid extraction, distillation, absorption, etc. **Han and Row, 2010.** Liquid membrane separation offers an effective powerful process for different separation processes. Compared to traditional operations, emulsion liquid membrane processes have several attractive features such as high interfacial area, simple operation, relatively low cost, high efficiency, extraction and stripping in one stage, a non-dependence on equilibrium consideration and scope of continuous operation **Ravikumar et al., 2005**. Norman Li, **Li, 1974** was the first to introduce emulsion liquid membrane to increase the interfacial area to shorten the diffusion path, its invention for the separation has shown to be an easy method for the removal of chemicals from wastewater.

There are three types of emulsions: (1) oil in water emulsion (O/W) in this type, oil is the dispersed phase and water is the dispersion medium; (2) water in oil emulsion (W/O) in this type, water is the dispersed phase and oil is the dispersion medium; (3) multiple emulsions **Manikandan et al., 2014**. Multiple emulsions can be classified in two types; water/oil/water (W/O/W) or oil/water/oil (O/W/O) emulsions. In the first type the membrane is liquid oil and in the second type the membrane is water or an aqueous solution. The (W/O/W) type is the most familiar one. Multiple emulsions are widespread for industrial, pharmacy and medical applications by virtue of the existence of liquid membrane. The multiple emulsions are a result of forming small droplets in which smaller droplets inside them, the outer droplets are dispersed in an external phase. Liquid membrane is a layer which separates the small internal droplets from the external phase which is called the immiscible phase. Consequently, multiple emulsions are also called liquid emulsion membranes or liquid surfactant membranes **Ghosh, 2011**, **Cárdenas and Castro, 2003**. For instance, the system of caustic-in-oil emulsion that is shown in **Fig.1**, can be efficiently utilized to eject small amounts of phenol from a wastewater stream. Phenol is slightly soluble in oil, and then it will permeate easily from the outside water phase across the oil membrane into the internal aqueous caustic droplets. In this process the caustic will neutralize the phenol and tied it up as sodium phenate which is insoluble in oil and then it cannot diffuse back out again **Cahn and Li, 1974**. ELM process can be divided into four stages as shown in **Fig.2** and as follows : (1) emulsification of the internal phase and the membrane phase; (2) permeation (emulsion—external phase contacting); (3) settling (separation of external phase and the emulsion phase after extraction; and (4) demulsification to recover the membrane phase **Balasubramanian, 2014**, **Fassihi and Björkegren, 2012**.

After the extraction, the membrane should be broken. The demulsification or breaking of an emulsion is one of the most significant steps for the ELM process because it is defined as the overall effective cost of the process, as the membrane phase will be recycled **Balasubramanian and Venkatesan, 2014**.

There are three stages in the demulsification process divided as follow **Pabby, 2015**: (1) globules coalescence and growth, (2) globules settling, and (3) coalescence of the large water and oil globules with their respective external phases in the centrifugal coalesce**.**

Chemical or physical treatments methods are used for demulsification as follows **Pabby, 2015**: (1) Chemical treatment methods: these treatment methods include the addition of a demulsifier. There are effective demulsifying agents for particular applications such as acetone, n-butanol and 2-propanol. Even though, these methods are effective, they change the properties and prevent reuse because additional separation steps for recycling and recovery are required; (2) Physical treatment methods: these treatments involve centrifugation, heating, sedimentation, ultrasonics, high shear, solvent dissolution, microwave, and using high-voltage electrostatic fields. ELM can be demulsified effectively by centrifugation.

2. EXPERIMENTAL WORK

2.1 Materials

(1) Phenol crystals were supplied by Loba Chemie Pvt. Ltd. (Laboratory reagents & fine chemicals).
(2) Sodium hydroxide (NaOH) was supplied by Reagent World Inc.
(3) Sorbitan monooleate (Span 80) was supplied by Wuhan Kemi-Works Chemical Co., Ltd.
(4) Kerosene was supplied by midland Iraqi refineries company.

2.2 Experimental Procedure

For ELM system preparation, (W/O) emulsion was prepared by mixing the surfactant and diluent with NaOH solution as an internal stripping agent of 2 M, for the initial experiments, a surfactant (Span 80): diluent (kerosene) ratio of 2:98 was used. This mixture was emulsified using a high speed homogenizer, operating at a rotational speed of 10000 rpm for 1 minute of emulsification time to obtain a milky white color emulsion as shown in **Fig.3**. The emulsion was dispersed in the aqueous external phase containing 300 ppm phenol with volume ratio of membrane to external phase= 0.5 and stirred by an overhead stirrer with a desired speed for 4 min to form numerous small globules of emulsion so that good dispersion of the emulsion in the waste water was maintained for mass transfer of phenol, the emulsion must be freshly prepared each time before the extraction step.

Phenol permeated into the liquid membrane and reacted with NaOH, which was the internal stripping agent to form sodium phenolate and water. The reaction is shown in the following equation:

$$C_6H_5OH + NaOH \longrightarrow C_6H_5ONa + H_2O \qquad (1)$$

Sodium phenolate cannot diffuse back into the external phase through liquid membrane due to the selectivity of the membrane. Therefore, it was not detected in the external phase. Then, the mixture is separated by 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 of settling, samples were taken from the treated water (bottom layer) and analyzed by UV spectrophotometer to determine the percentage removal of phenol. After extraction, some big emulsion droplets at the upper layer of the mixture were collected. An experiment for demulsification was carried out on the top layer of solution (waste) by applying centrifuge, then emulsion was broken up and separated within 1 hour and 8000 rpm in order to separate the phases that compose the waste (the internal aqueous phase and the membrane phase) **Fig.5**. At the end of the process, a component of membrane phase (kerosene) can be recycled. The recovered-oil phase is reused for making emulsions for the liquid membrane process and should not contain any demulsifying chemicals.

2.3 Phenol Concentration Measurement

A 2mL 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 270 nm. The concentrations of phenol were estimated from the absorbance-phenol concentration calibration curve **Fig.6**. Then, the percentage removal of phenol was determined by the following equation:

$$\text{Removal of Phenol (\%)} = \frac{(c_0-c_1)}{c_0} * 100 \qquad (2)$$

Where c_0 is the initial phenol concentration in the external phase and c_1 is the final phenol concentration in the sample after extraction.

3. RESULTS AND DISCUSSION

3.1 Effect of Process Variables on Removal of Phenol

In this work, three operating parameters, namely, surfactant concentration, volume ratio of internal phase to membrane phase (I/M) and stirring speed were studied. Volume ratio of membrane to external phase ratio= 1:2, internal agent concentration= 2 M, stirring time= 4 min, emulsification time =1 min. These conditions, as the optimal ones, were chosen based on preliminary experiments done and previous researches also the reports in other related literatures such as **Mortaheb et al., 2008**, **Ng et al., 2010**, and **Balasubramanian, 2014**

3.1.1 Effect of surfactant concentration

Surfactant concentration was shown to play a dominate role in the phenol removal and as an emulsifier for the liquid membrane also act as a protective barrier between the external phase and the internal phase, preventing the leakage of emulsion. It is very important to check the effect of surfactant concentration on the behavior of removal efficiency of phenol by ELM, The surfactant concentration has been studied in the range 1 to 5% (v/v).

Figs.7 to 9 and **Figs.10 to 12** show the effect of surfactant concentration on the behavior of phenol removal at different conditions [stirring speed (100-600) rpm and membrane to internal phase ratio (1:1–6:1) (v/v)] respectively. All these figures indicate 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 decreasing 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 5)% (v/v), increases the viscosity of the membrane phase which decreases the removal of phenol through the highly viscous membrane, this can be attributed to the fact that the increase in emulsion viscosity resulting from the increasing surfactant concentration leads to the augmentation of mass transfer resistance due to presence of excessive surfactant at the external–membrane phase interface, resulting in less transfer of phenol molecules to the internal phase. These observations are in good agreement with most investigators **Manikandan et al., 2014**, **Othman et al., 2006** and similar observations were made by **Ng et al., 2010** and **Dâas and Hamdaoui, 2010**. Therefore, Span 80 of concentration 2% (v/v) was found to be the optimum that producing maximum extraction efficiency greater than 98%, this value was fixed for all other experiments. Also it was investigated from these figures that the optimum values of stirring speed was 400 rpm and volume ratio of membrane phase to internal phase was 5:1.

3.1.2 Effect of stirring speed

Stirring speed was an important factor in the mass transfer rate of phenol through the liquid membrane. The stirring speed at which external phase and emulsion were mixed was found to have a profound effect on the extraction of phenol from the feed.

Figs.13 to 15 and **Fig.16 to 18** represent the relationship between stirring speed and the percentage removal of phenol at different conditions [surfactant concentration (1-4) (v/v)% and volume ratio of membrane to internal phase (1:1–5:1)], the stirring speed has been studied in the range 100 to 500 rpm. 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 increases the interfacial area between the external phase and the membrane phase, leading to enhanced surface area for mass transfer 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. However, this is true up to certain level of stirring beyond which an increase in the level is likely to break the emulsion droplets thereby reducing the degree of extraction. Increasing the stirring speed above (400rpm) not only decreases the extraction efficiency, but also affects the stability of emulsion. This notice is in good agreement with some researchers **Chiha et al., 2006** and **Kaghazchi et al., 2006**.

In addition, the shear induced breakage of fragile emulsion droplets near the tip of the impeller or impact on the wall of a contactor imposes upper limit on the speed of stirring. At the same time swelling is also increased due to water transport from external to internal phase. Therefore, the extraction performance would be a tradeoff between the two effects of agitation speed and swelling phenomena. These observations were in good agreement with most investigators **Othman et al., 2006** and **Dâas and Hamdaoui, 2010**. Therefore, the best value of stirring speed was found to be 400 rpm. Also, it was investigated from the above figures that the optimum values of surfactant concentration was 2%v/v and volume ratio of membrane phase to internal phase was 5:1.

3.1.3 Effect of volume ratio of membrane phase to internal phase

The volume ratio of membrane phase to internal phase has a significant effect on removal of phenol using ELM. This parameter can affect the surfactant concentration at the interface of the membrane / external phases. The results are shown in **Fig.19 to 21** and **Fig.22 to 24**. In order to form a stable and effective W/O emulsion, the volume ratio must exceed 1:1 **Luo et al., 2014**.

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. Variation of this phase ratio produced emulsions with different properties, including size, stability, and extraction capacity. Optimal phase ratio produces proper size of emulsion. The first observation was the increasing in the volume ratio of membrane phase to internal phase makes both the extraction and swelling rates strongly improved, this is due to the absolute amount of each component in the membrane phase was raised. At low volume ratio of (1:1), the volume of membrane solution is not enough to enclose the overall stripping solution (internal phase) thus producing large emulsion globule leading to low extraction efficiency. The produced emulsion tends to have thin wall therefore increasing leakage possibility.

When volume ratio of membrane phase to internal phase is increased from 2 to 4, the transport rate and extraction efficiency of phenol increase, this can be due to the fact that an increase in the membrane phase volume ratio 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. An increase in membrane to internal phase volume ratio from 4 to 5 resulted in a sharp rise in the average effect. 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. 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. This phenomenon could be due to the built-up resistance around the membrane at the high membrane to internal phase ratio. The increase in thickness of the membrane offered resistance that slowed down the phenol permeation rate.

However, higher ratio requires higher stirring speed due to the increase in viscosity of the mixture. If the speed is constant at 400 rpm then it will reduce phenol extraction due to unsuitable mixing of phases because of higher proportion of the more viscous phase. Thus, a volume ratio of membrane phase to internal phase of 5:1 was selected as the optimal volume ratio. This observation is in good agreement with most investigators **Luo et al., 2014**, **Ahmad et al., 2013**, and **Mortaheb et al., 2008**. Also, it was investigated from these figures that the optimal values of stirring speed and Span 80 concentration were 400 rpm and was 2(v/v)% respectively.

3.2 Studying the Optimal Time for Extraction

The extraction time is one of the most important parameters in emulsion liquid membrane systems, it is defined as the dispersion time of emulsion to the external phase. The effect of various extraction times (1–25 min) on the performance of phenol extraction efficiency were studied to select the optimum time for extraction of phenol and the results were shown in **Fig. 25**. Short extraction time leads to the emulsion breakage due to the large size of droplet which leads for their coalescence, so that phenol removal efficiency was low, because the time was not enough for the separation of emulsion from treated water. Also it was observed that the extraction performance of phenol increased when the extraction time increased for the first 5 min, and reached maximum extraction at 7 minutes of process time.

An increase in extraction time from 5 to 7 resulted in a sharp rise in the average effect. This can be due to the separation process was completed between emulsion and treated water. After 7 min, it was observed a slight increase in the extraction efficiency of phenol, so that 7 minutes of extraction time was taken as the optimum time, because there is no significant increases in removal of phenol so that no need to wait for longer time (longer extraction time only enhance the emulsion swelling). With further increase of the extraction time up to 15 min, the extraction efficiency of phenol decreases because some membrane droplets begin to break and therefore, the phenol remaining in the external phase. The mass transfer in ELM system occurs very fast thus, 7 minutes of extraction time was the optimum condition for this system which gave the maximum extraction of phenol. These observations are in good agreement with most investigators **Othman et al., 2006**, **Gasser et al., 2008**, and **He et al., 2015**.

3.3 Demulsification

After applying centrifuge, the upper layer was tested by using (GC) by IBN SINA FACTORY. Comparison between the standard kerosene and the upper layer was shown in **Fig.26**, indicates that there is a good agreement between them so that this layer can be reused for membrane phase.

CONCLUSIONS

1. It was demonstrated that the ELM technique was very promising in the treatment of aqueous solutions containing phenol.
2. The maximum predicted value for the percentage removal of phenol using ELM is 98.95% at the optimum parameters.
3. 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.
4. 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
5. 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 remained constant.
6. The extraction performance of phenol increased when the extraction time increased for the first 5 min, and reached maximum extraction at 7 minutes of process time. With long extraction time, the extraction efficiency of phenol decreases.
7. There was a good agreement between the standard kerosene and the upper layer obtained after centrifugation of the waste, so that this layer can be reused for membrane phase to prepare a new emulsion.

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Abbreviations

ELM	emulsion liquid membrane
GC	gas chromatography
M/I	membrane phase / internal phase
O/W	oil in water emulsion
O/W/O	oil in water in oil
rpm	rotation per minute
W/O	water in oil Emulsion
W/O/W	water in oil in water

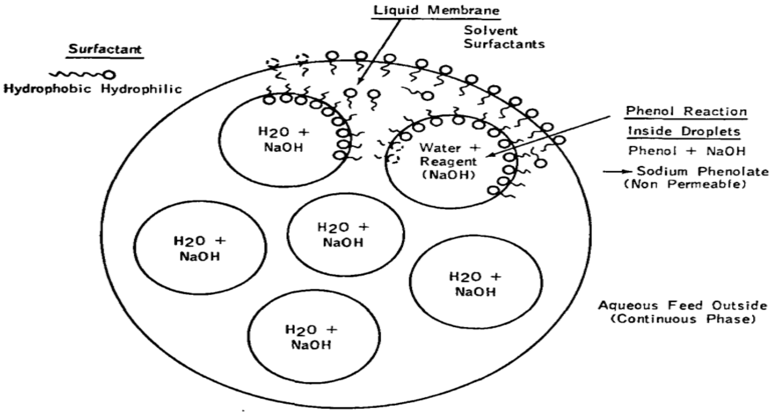


Figure 1. Schematic diagram of liquid membrane system for phenol removal **Rousseau, 2009**.

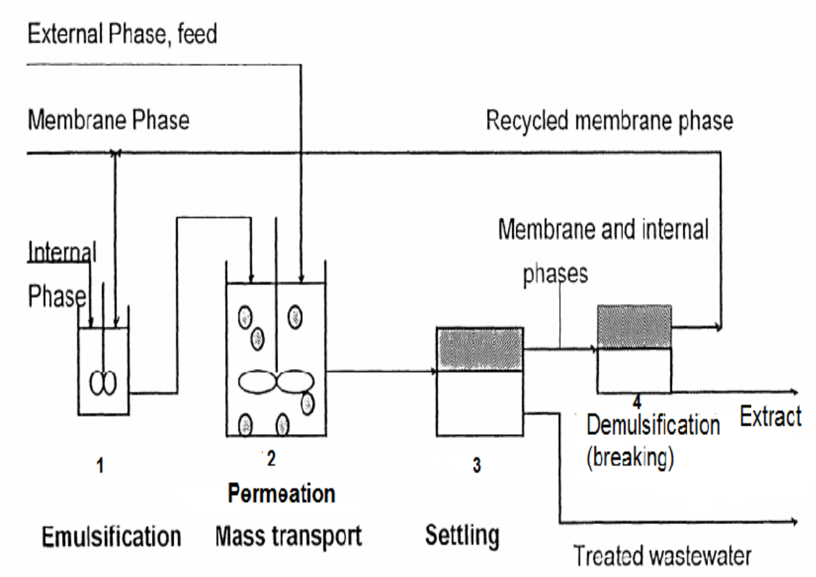


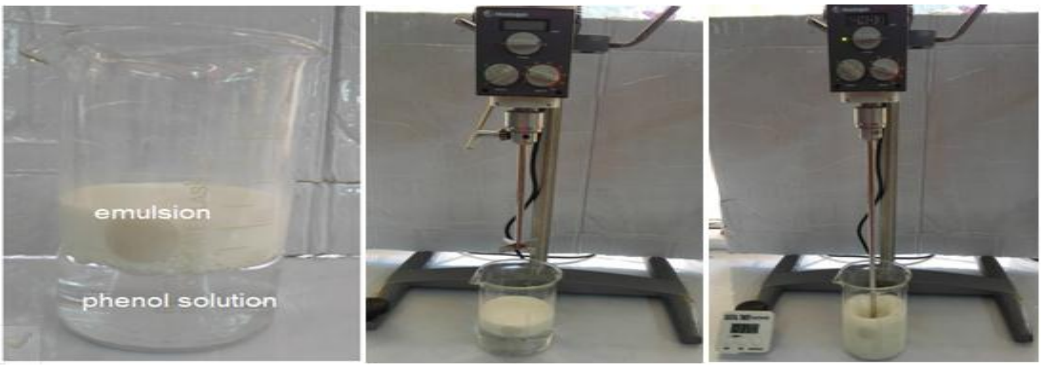
Figure 2. The operational steps in the ELM process **Fan, 1999**.

Figure 3. Milky white color liquid membrane.

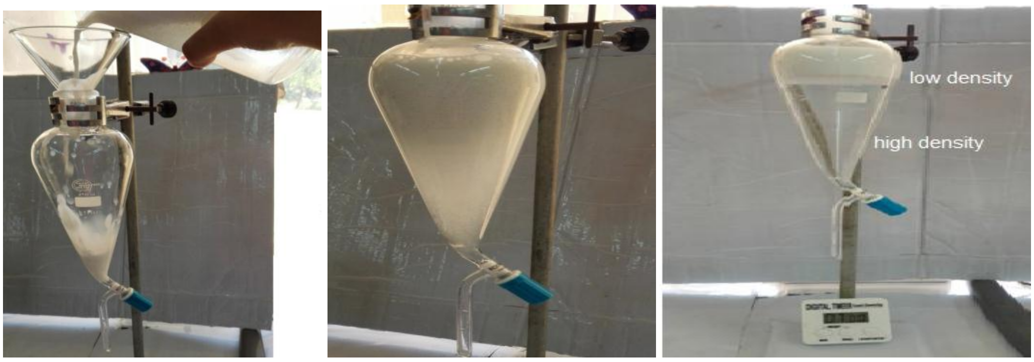
Step 1: adding membrane phase to internal phase

Step 2: mixing for 1 min to get milky white emulsion

Step 3: adding emulsion to phenol solution



Step 4: emulsion and phenol solution are separated into two layers mix do not without stirring



Step 5: adding the mixture to a separating funnel and waiting for 7 min to take the sample.

Figure 4. Experiment procedure steps (1 to 5).

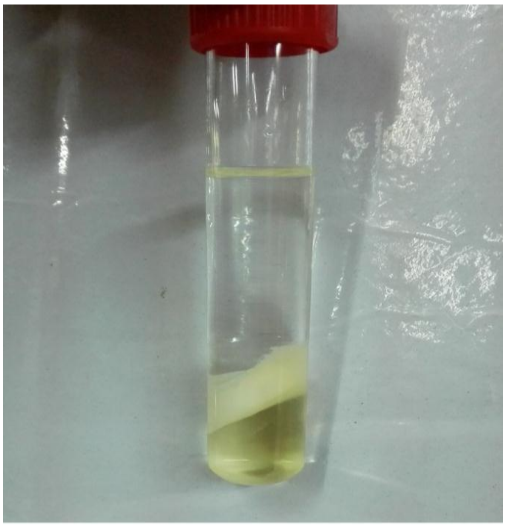


Figure 5. The separated phases after demulsification

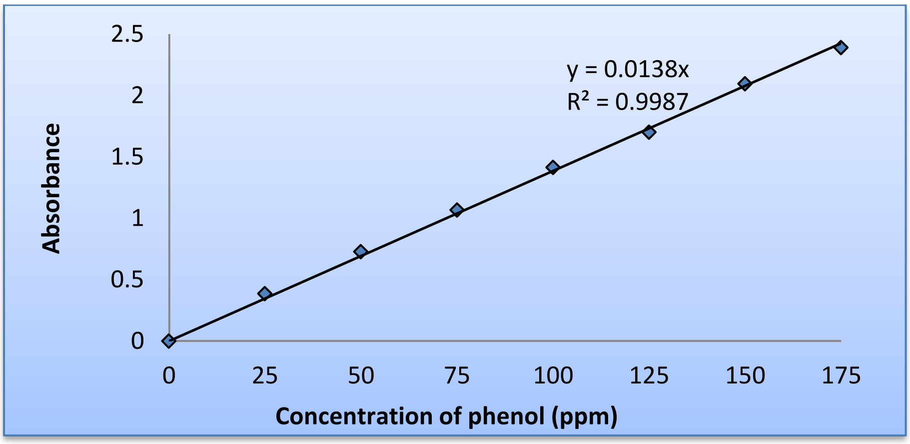


Figure 6. Calibration curve of phenol adsorption.

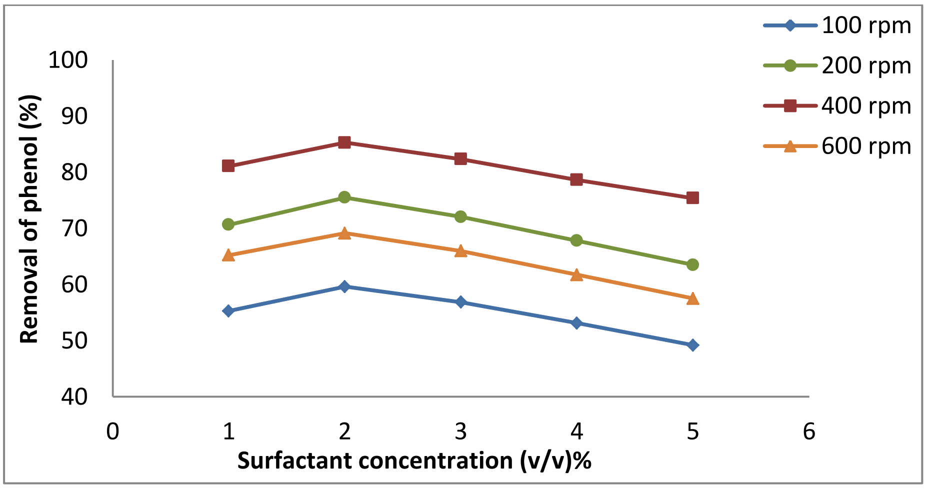


Figure 7. Effect of surfactant concentration on removal of phenol at different stirring speed, volume ratio of membrane phase to internal phase= 1:1.

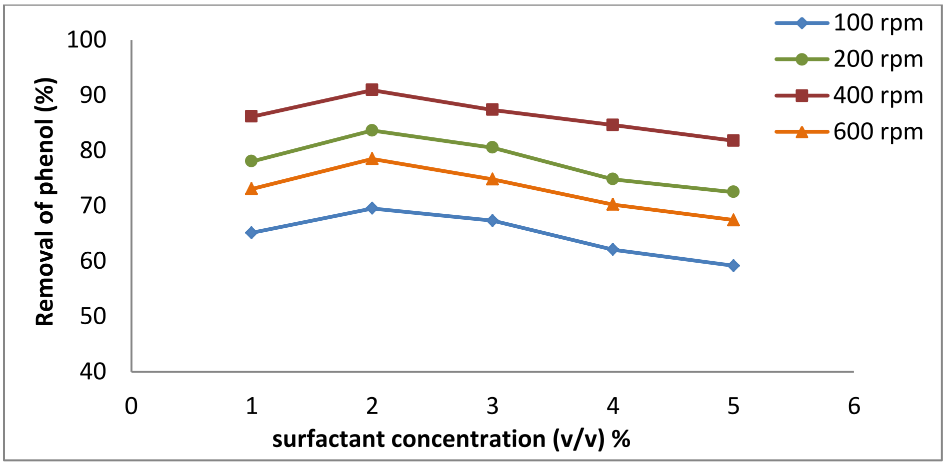


Figure 8. Effect of surfactant concentration on removal of phenol at different stirring speed, volume ratio of membrane phase to internal phase= 3:1.

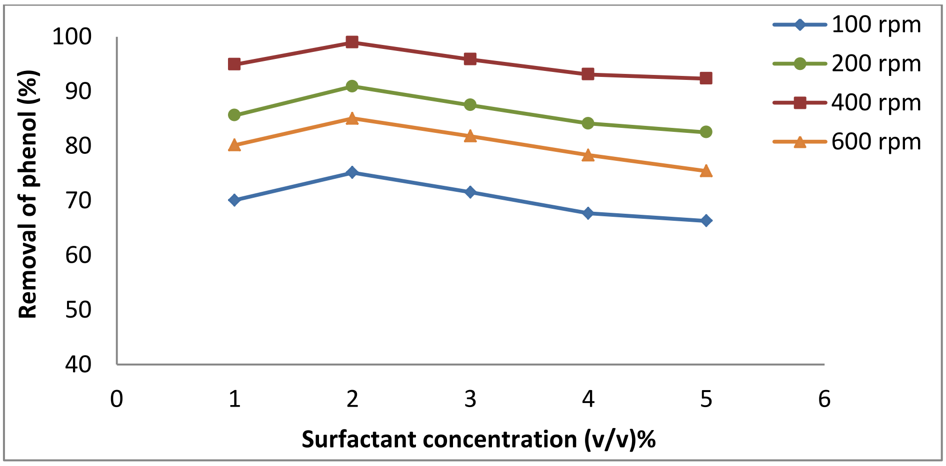


Figure 9. Effect of surfactant concentration on removal of phenol at different stirring speed, volume ratio of membrane phase to internal phase= 5:1.

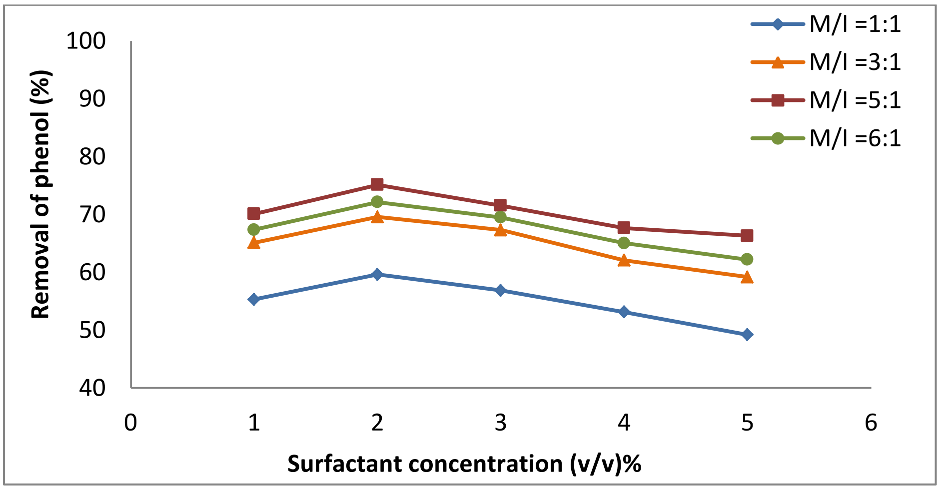


Figure 10. Effect of surfactant concentration on removal of phenol at different volume ratio of membrane phase to internal phase, stirring speed= 100 rpm.

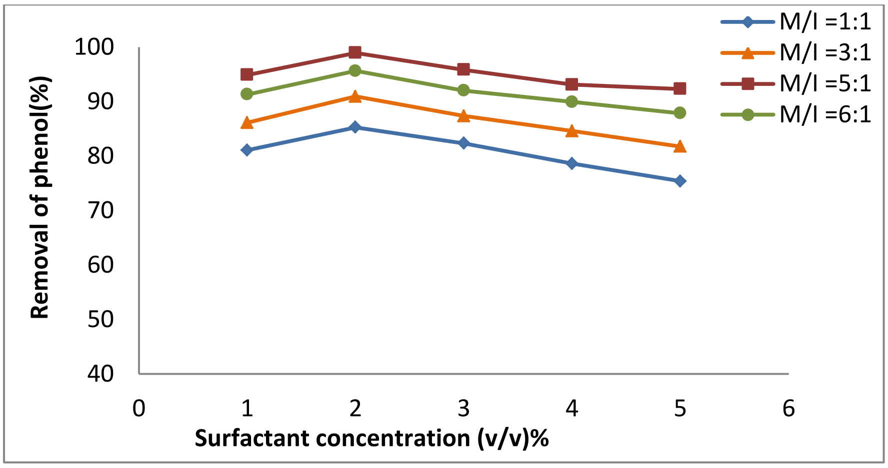


Figure 11. Effect of surfactant concentration on removal of phenol at different volume ratio of membrane phase to internal phase, stirring speed = 400 rpm.

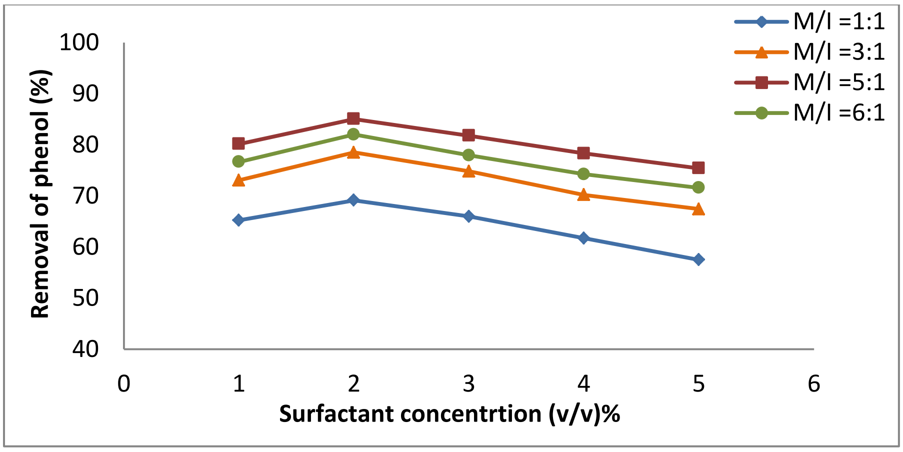


Figure 12. Effect of surfactant concentration on removal of phenol at different volume ratio of membrane phase to internal phase, stirring speed= 600 rpm.

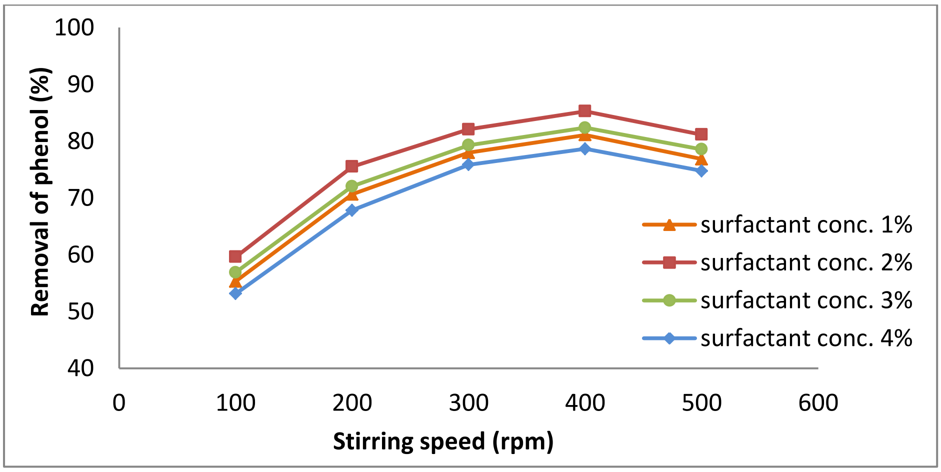


Figure 13. Effect of stirring speed on removal of phenol at different surfactant concentration, volume ratio of membrane phase to internal phase= 1:1.

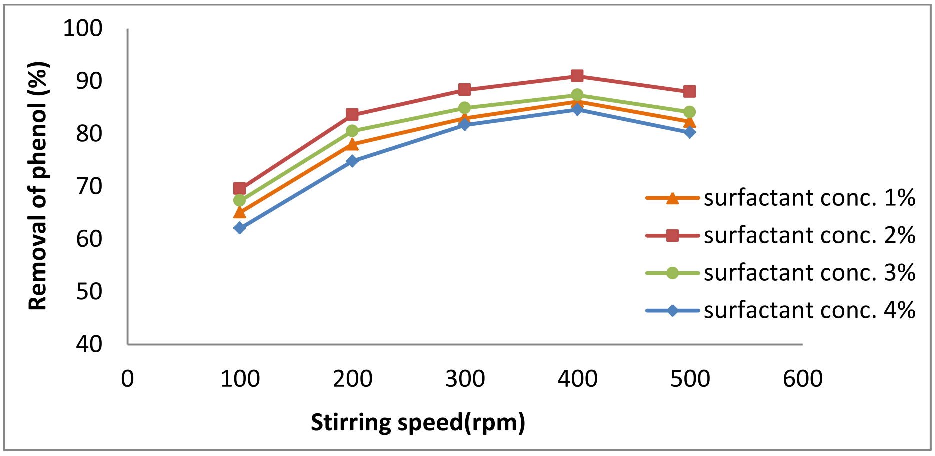


Figure 14. Effect of stirring speed on removal of phenol at different surfactant concentration, volume ratio of membrane phase to internal phase= 3:1.

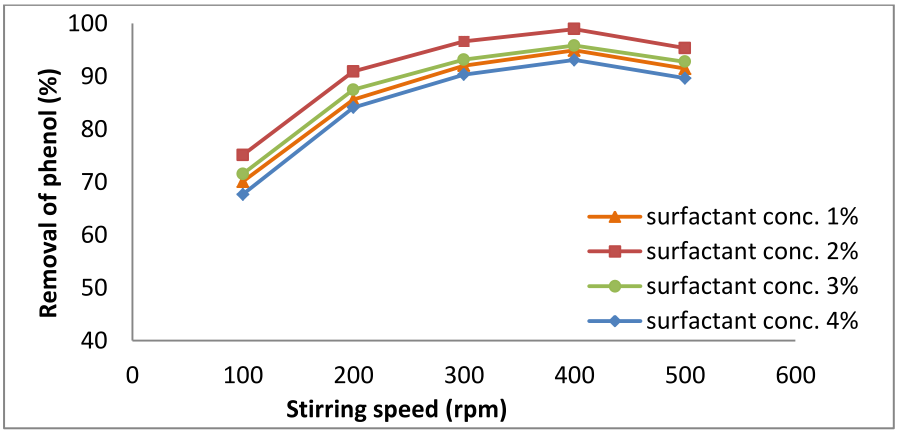


Figure 15. Effect of stirring speed on removal of phenol at different surfactant concentration, volume ratio of membrane phase to internal phase= 5:1.

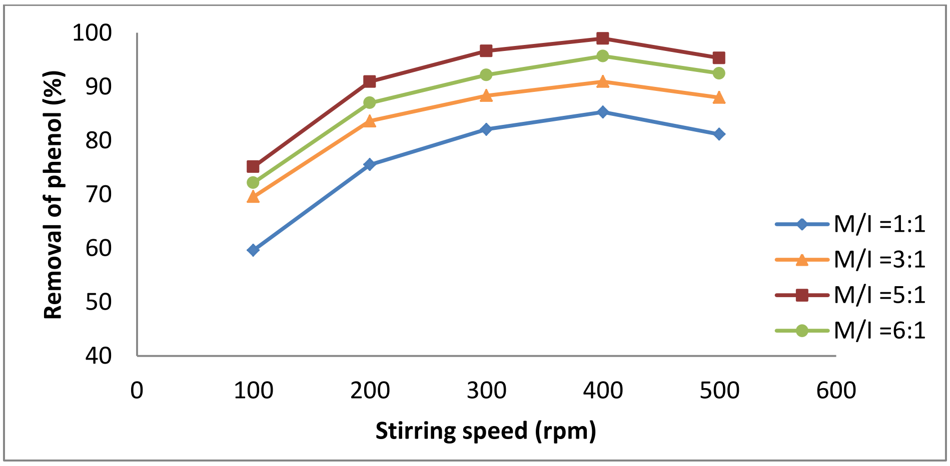


Figure 16. Effect of stirring speed on removal of phenol at different volume ratio of membrane phase to internal phase (M/I), surfactant concentration= 2%v/v.

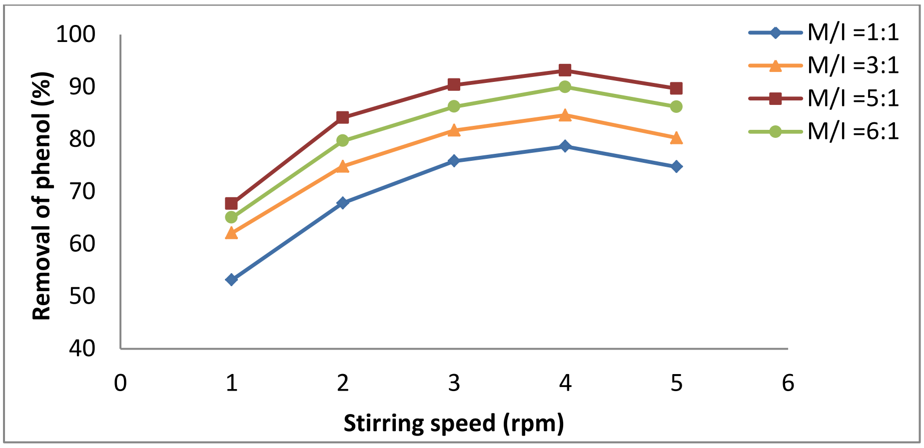


Figure 17. Effect of stirring speed on removal of phenol at different volume ratio of membrane phase to internal phase (M/I), surfactant concentration= 4%v/v.

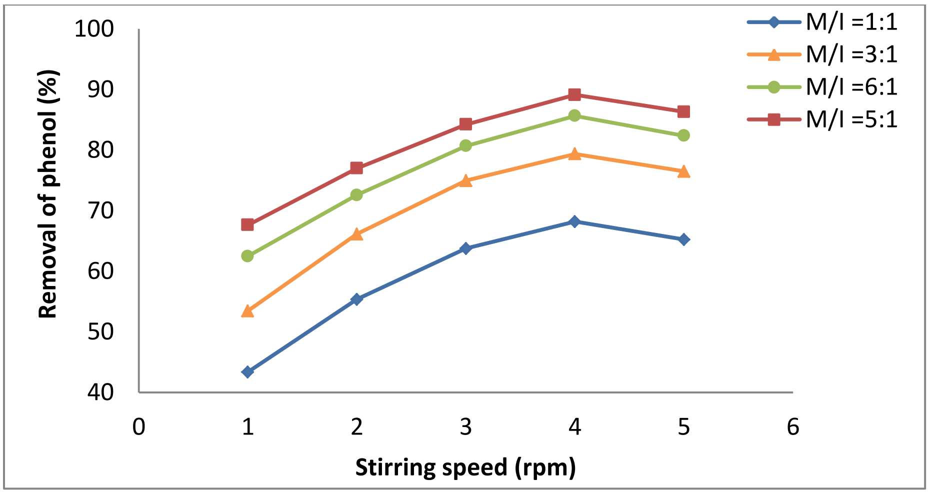


Figure 18. Effect of stirring speed on removal of phenol at different volume ratio of membrane phase to internal phase (M/I), surfactant concentration= 6%v/v.

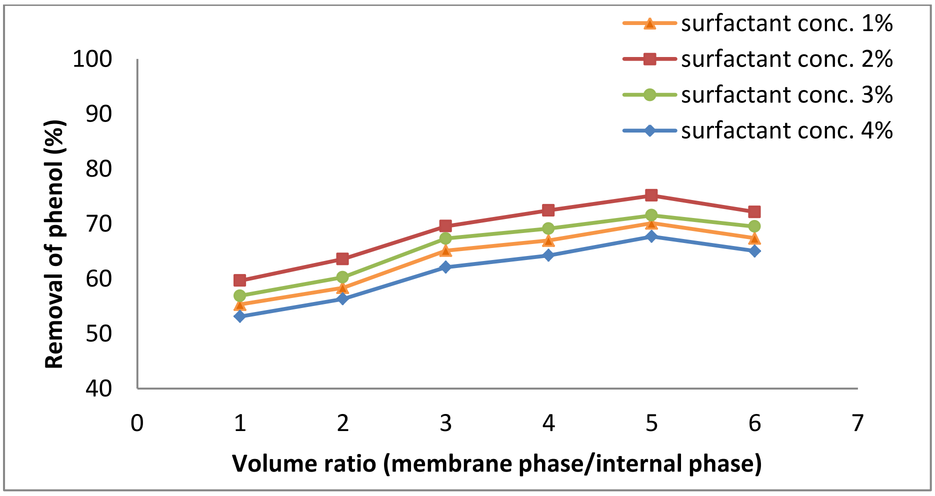


Figure 19. Effect of volume ratio of the membrane phase to internal phase on removal of phenol at different surfactant concentration, stirring speed= 100 rpm.

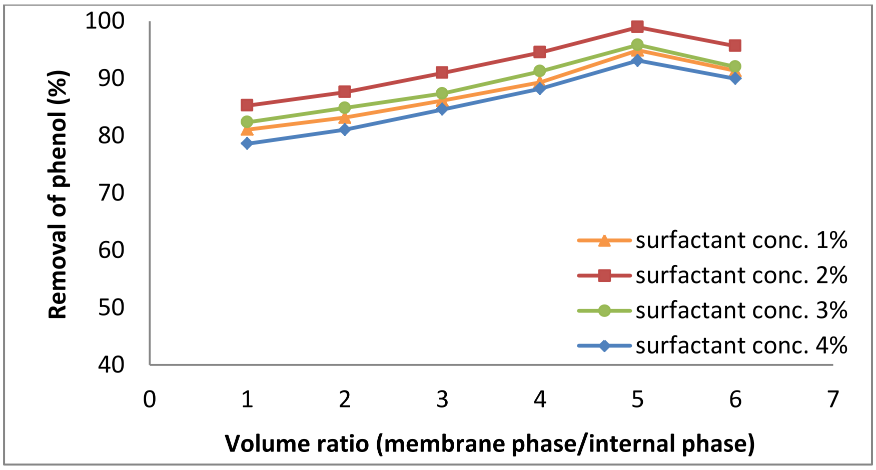


Figure 20. Effect of volume ratio of membrane phase to internal phase on removal of phenol at different surfactant concentration, stirring speed= 400 rpm.

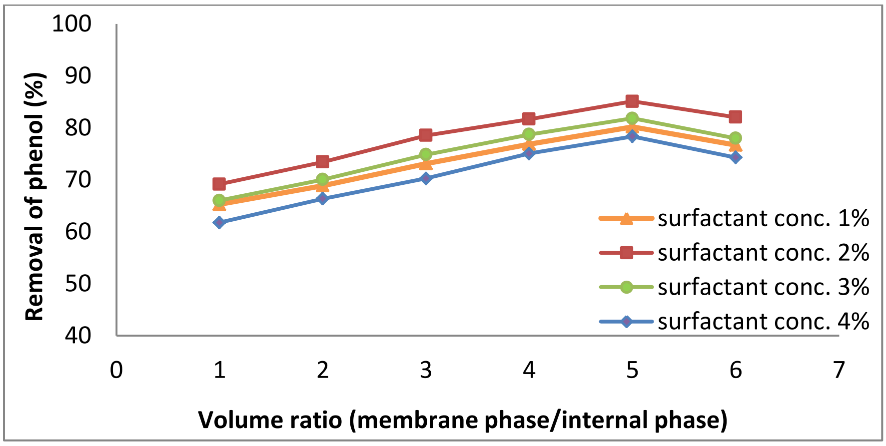


Figure 21. Effect of volume ratio of membrane phase to internal phase on removal of phenol at different surfactant concentration, stirring speed= 600 rpm.

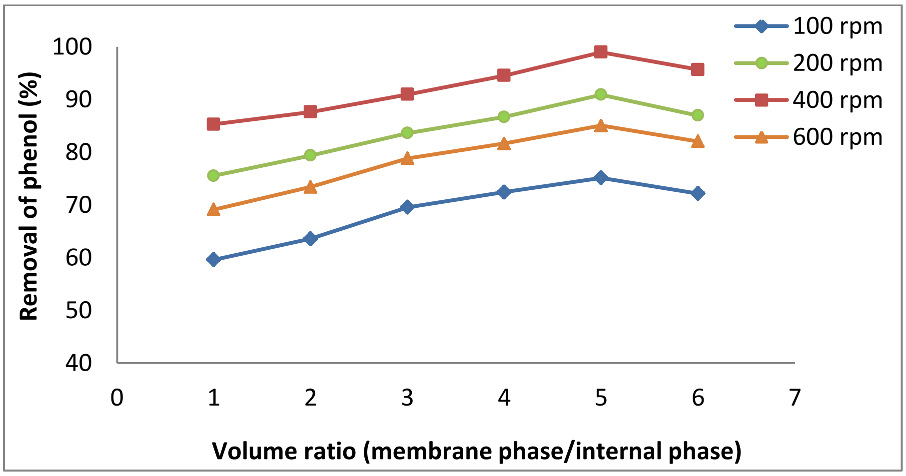


Figure 22. Effect of volume ratio of membrane phase to internal phase on removal of phenol at different stirring speed, surfactant concentration= 2%v/v.

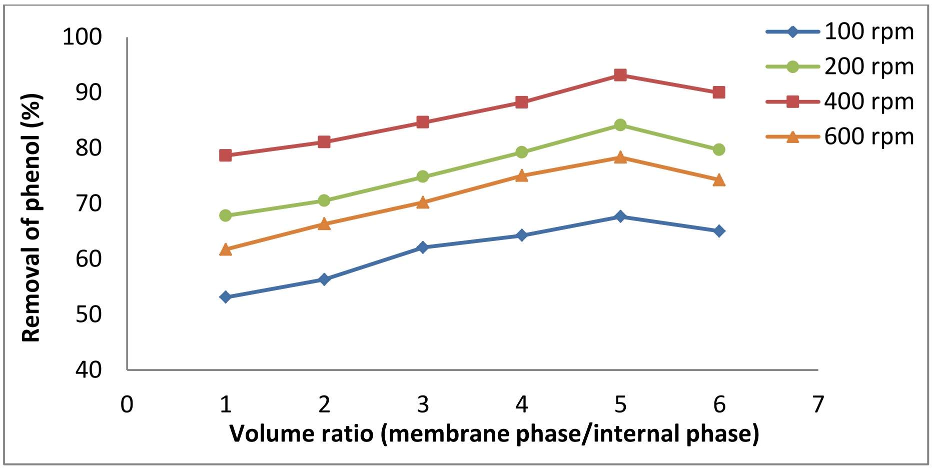


Figure 23. Effect of volume ratio of membrane phase to internal phase on removal of phenol at different stirring speed, surfactant concentration= 4%v/v.

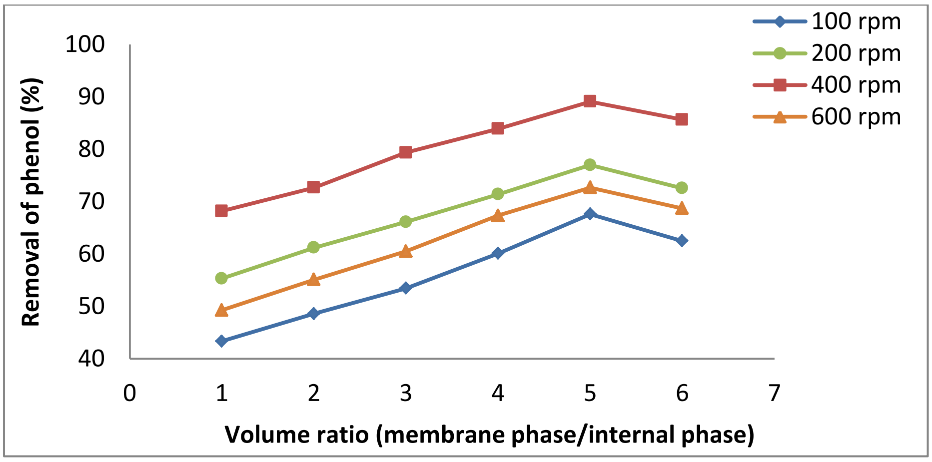


Figure 24. Effect of volume ratio of membrane phase to internal phase on removal of phenol at different stirring speed, surfactant concentration =6% v/v.

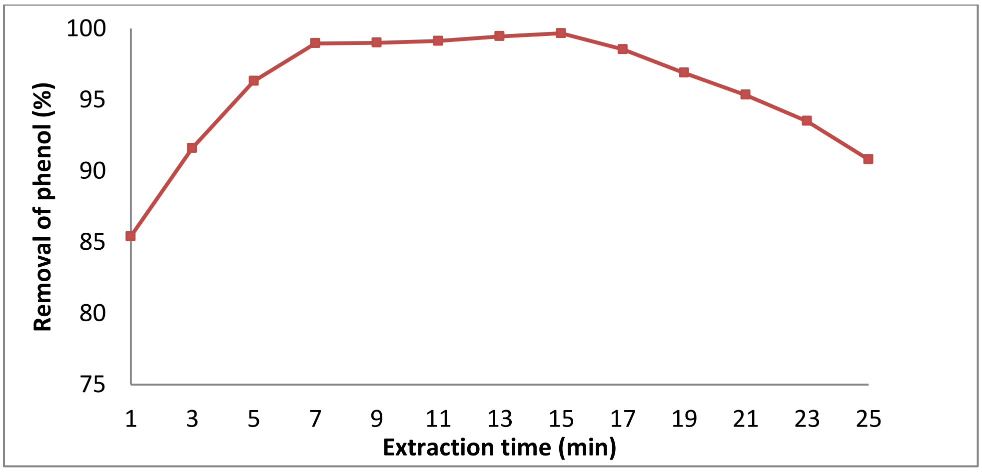


Figure 25. Effect of extraction time on the removal of phenol, experimental conditions were: ratio of membrane to external phase ratio= 1:2v/v, concentration of Span 80= 2(v/v) %, ratio of membrane to internal phase= 5:1v/v, NaOH concentration=2 M, stirring speed= 400 rpm, stirring time= 4 min and emulsification time= 1 min.

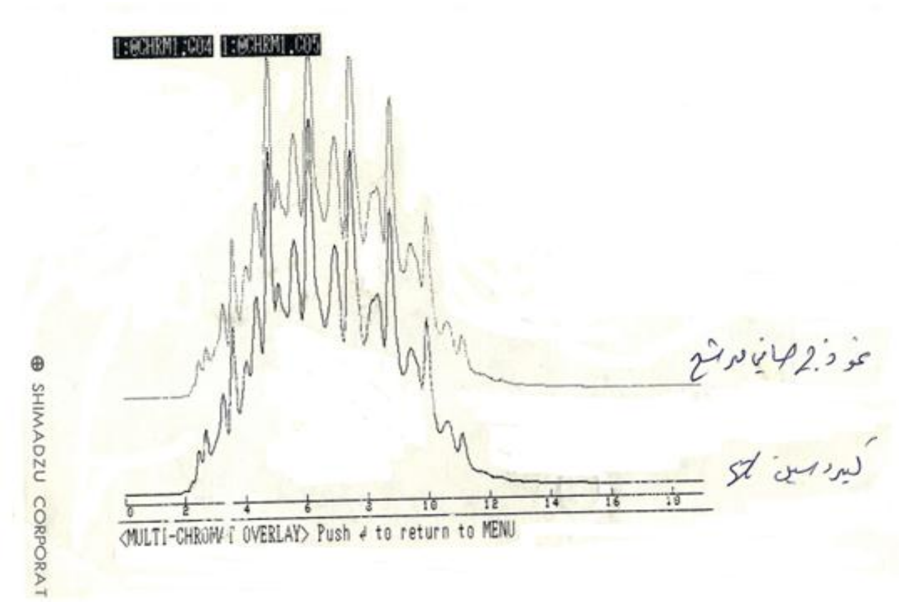


Figure 26. Comparison between the standard kerosene and the upper layer after demulsification