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# Removal of Methylene Blue Dye by Using Bulk Liquid Membrane

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**Abstract—** The goals of this work is to optimize and choose the suitable scheme, the operating factors of bulk liquid membrane (BLM) which is soybean oil-is for methylene blue (MB) elimination and rescue from aqueous resolutions. The (soybean oil)-created BLM involves of an aqueous source phase (MB), a natural film stage (soybean oil (diluent), and Tri-Octyl Amine (TOA) (carrier)) and an aqueous receiving stage (hydrochloric acid resolution (HCl)). Effects of design factors (pH of the source, and receiving stages, primary color (MB) concentration, and carrier attentiveness) of (soybean oil)-created BLM on the MB elimination and rescue from aqueous elucidations were reconnoitered and the fit factors were carefully chosen for additional revisions. Optimization of the operating factors of (soybean oil)-created BLM for extreme percentage of MB was (11 pH source and the receiving pH was 4.75 to 5.25, and 6.75 to 7.25% tri octal amine was the best proportion of the film then directed using Response Surface Methodology and the best factors were resolute. A regression model for 92 % elimination was settled and its acceptability was calculated. The trial 92 % elimination attained below the best working situations was associated with the expected one and they were originating to decide acceptably by a piece promoting $R^2$ 0.9385.

**Keywords—***BLM, soybean oil, aliquot 336, TOA, Dye*.

## 1. Introduction

Liquid membranes (LMs) are liquids that separate two aqueous phases of the source (feed) and the receiving (product) phases and are immiscible in these phases [1]These departure systems are being explored widely in many fields such as natural and mineral chemistry, wastewater treatment, chemical engineering, biomedical -manufacturing, and biotechnology. Research within these fields involve several uses of LM skill, such as exclusion of living -mixtures, recovery of heavy metals, removal of poisonous metals, gas separations, and branch of pharmaceutical compounds and fermentation goods [2]

Liquid membrane technology can be divided into three kinds which are (bulk liquid membrane (BLM), emulsion liquid membrane (ELM), and supported liquid membrane (SLM)). Currently, ELM is introduced as an alternative technique to the separation process. LM fulfils the promise of providing several attractive characteristics. Relatively low energy consumption owed to it is operated at ambient temperature and pressure [3; 4], fast extraction and high efficiency due to large available mass transfer area [5], potential for removing various toxic substances down to exact low levels, high selectivity particularly with the use of mover agents in the liquid film case.

LM process was commonly planned for developed partings for example in removing several sorts of metal ions [6, 7], organic compounds [8], and inorganic compounds [9]

Recent development in the LM process of organic compounds in the river of environmental purification of methylene blue from the water body. This is due to the increase of the methylene blue dye from industrial and textile industries.

The LM process is very unique and promising in the extraction of the targeted species due to high interfacial area for mass transfer, economical, low energy intake, immediate mining and shedding process, effective for low solute concentrations, then requisite of small amounts of conc. [10] Nevertheless, the various advantages of the LM procedure still have been limited for industrial applications owing to the emulsion drops being unstable alongside the water body. The prepared emulsion should be sufficiently stable during the dispersion for solute extraction.

Many studies were carried out to investigate the emulsion liquid membrane stability [11]Therefore, the effects of operating conditions on their moles of MB by bulk liquid membrane were investigated by using Design of experiment (DOE) which has been widely used by numerous researchers to study the connections of two or more variables. It is a gathering of numerical and mathematical techniques which are expected for emerging regression models, refining and enhancing processes. Generally it holds three steps: (I) Procedure design and trial lay out in dissimilar operating conditions, (ii) examining through model development and regression and (iii) optimization of the process. The main advantage of DOE is the claim of the abridged number of experimental tracks to assess the result of manifold bounds and their connections.

## 2. Experimental work

### 2.1 *The three-phase experiments setup*

The three-phase experiments were accepted ready in two identical volumes of four-sided-shaped vessels made with glass sheets. The two containers were separated by a glass sheet to avoid the mixing of the feed with the receiving phase as displayed in Figure. 1. The aqueous source and receive elucidations were discharged into two dissimilar vessels of half cubicles, each having an extreme volume of 50 mL. Figure.2.display a three-phase set up diagram.

**Figure 1**: Three phase experiment setup

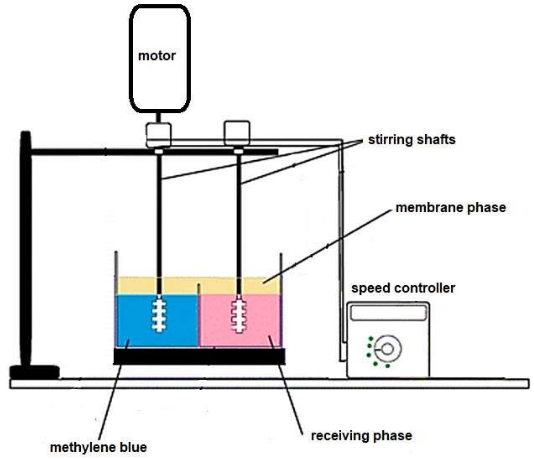



**Figure 2**: the three-phase setup diagram

Solutions in the two cubicles were mixed using perpendicular automatic agitators related to a motor with a regular speed. Trials were composed from the source and received adjacent at fixed intermissions (1 hour) through the testing.

The concert of the two-phase equilibrium circulation and three-phase carriage results were achieved in positions of % removal, calculated by Eq. (1).

% Removal= $(CC - CC\text{out}/CC) \times 100$…. (1)

Where $CC$ is an initial concentration in feed phase (ppm), $CC$ out is a concentration in strip phase after transportation (ppm) respectively.

### 2.2 *Plan of Trials*

The Plan of Trials, especially (RSM) has been practical for numerous manufacturing glitches. These trainings include approximately numerical procedures in order to examine whether those contributions have the greatest effect on the outputs? At what condition the results will content the client supplies? And how it can be enhanced. These approaches may be secondhand for both physical tough, as well as imitation trying.

#### 2.2.1 Central Composite Design

The Central Composite Design (CCD) was used to improve the performance of dye removal. It was based upon four criteria of the independent method. These important parameters are pH of feed, pH of stripping, carrier conc. Initial dye concentrations were conducted as shown in table 1 using the experimental design gained by full factorial central composite design (CCD). As shown in Table 1.

**Table 1**: experimental variables and their CCD coding

| variable | Code | | | | |
|---|---|---|---|---|---|
| | -2 | -1 | 0 | 1 | 2 |
| Feed pH | 9 | 10 | 11 | 12 | 13 |
| Stripping pH | 3 | 4 | 5 | 6 | 7 |
| Initial dye conc. | 10 | 15 | 20 | 25 | 30 |
| TOA% | 1 | 4 | 7 | 10 | 13 |

### 2.2.2 Response Surface Method (RSM)

(Response Surface) exhibiting various ladders, such as (Design of Experiment) D.O.E, conducting or numerical simulation of experiments, construction of the response surface model, and finally adequacy check. D.O.E. may be distinct as an exam or series of exams in which focused variations are practical to the contribution variables to detect and classify the details that cause significant changes in the reply. At the arithmetical imitation stage, imitations are led at circumstances strongly minded in the preceding stage. The building of the superficial perfect is approved available based on these consequences. The ideal benefits the alchemist to predict possible future results that may be achieved by varying the replication conditions between varieties of initial examination limits.

### 2.3 *Formulation of R.S.M.*

The response surface methodology (RSM) is a group of arithmetical and exact methods that are valuable for the demonstrating and examination of glitches in which a reply of interest by numerous variables and the impartial is to progress, develop and elevate the processes. Any procedure response be contingent on "n" issues can be subscribed as: $y = f(x1, x2, xn) + \epsilon$

Where:

$\epsilon$: The slip detected in the reply and this tenure is assumed to distribute usually with zero nasty and have an alteration $\sigma 2$. X is the experiment variable and y is the response. If the predictable response is meant as;

$E\,y = f(x1, x2, xn)$

As the real reply of the scheme or role is unknown, it must be assessed or aggregated. In numerous suitcases, typically moreover a "first-order model" or else a "second-order model" stands characters. For the event of liberated variables, a first-order model is apt:

$y = Bo + \beta 1x1 + \beta 2x2 + \cdots + \beta nxn + \epsilon$

This first-order typical is occasionally called as a "main effects model" meanwhile it has simply the chief effects of the issues. B is the constant of each variable. If there is a communication between the aspects it can be simply added to manner as:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \cdots + \beta_n x_n + \beta_{12} x_1 x_2 + \cdots + \beta_{1n} x_1 x_n + \cdots + \beta_{(n-1)n} x_{(n-1)} x_n + \epsilon$$

If there is slightly curving in the actual reply of the purpose, then first-order perfect is not sufficient, so that a second-order classical is usually cast-off in these luggage [3, 4]

## 3. Results and discussion

### 3.1 *Optimization of Process Parameters for MB removal by bulk liquid Membrane (BLM)*

Numerous functioning limits of (soybean oil)-based BLM, viz. pH of the source and accepting phase, carrier concentration, and initial source attention were improved for the extreme percent elimination of MB using RSM. It happened with broadcast trials to regulate which of the several functioning limits were probable to be significant in the reply surface revision. Next, an experimental plan was nominated to estimate the kindred remaining between the imperative limitations and the percent elimination of MB. Trials were then led according to the designated experimental design, showed by data analysis which includes reversion investigation, model adequacy examination and resolve of the optimal settings. The design matrix and outcomes of CCD with four constraints (pH of the source and receiving phase, mover concentration, and initial source concentration) and one response (% removal) are set in Table 2. There are overall 30 trial Arguments, which are unruffled of 16 factorial sockets, 8 star points and 6 replicates of the center point. Table (2), displays the used CCD design matrix and the estimated average percent removal of response in this work. This consists of a minimum of 30 runs to prevent the uncontrolled factors effect. All experiments were performed in one block of measurement under a homogeneous condition.

**Table 2**: Number of runs, experimental and predictable values of methylene blue

| Ru Block pH pH C D R Predicted n Feed stripping %. | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1 | Axial | 12 | 5 | 13 | 20 | 65 | 61.1 |
| 2 | Center | 12 | 5 | 7 | 20 | 92 | 86.48 |
| 3 | Fact | 12.5 | 6 | 4 | 25 | 72 | 67.68 |
| 4 | Axial | 11 | 5 | 7 | 20 | 70 | 65.8 |
| 5 | Center | 12 | 5 | 7 | 20 | 90 | 84.6 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 6 | Fact | 11.5 | 6 | 10 | 15 | 72 | 67.68 |
| 7 | Axial | 12 | 5 | 7 | 10 | 67 | 62.98 |
| 8 | Axial | 12 | 5 | 1 | 20 | 60 | 56.4 |
| 9 | Fact | 12.5 | 4 | 10 | 15 | 72 | 67.68 |
| 10 | Fact | 11.5 | 4 | 10 | 25 | 75 | 70.5 |
| 11 | Fact | 11.5 | 6 | 4 | 25 | 73 | 68.62 |
| 12 | Axial | 12 | 3 | 7 | 20 | 65 | 61.1 |
| 13 | Center | 12 | 5 | 7 | 20 | 90 | 84.6 |
| 14 | Fact | 12.5 | 6 | 10 | 15 | 72 | 67.68 |
| 15 | Center | 12 | 5 | 7 | 20 | 90 | 84.6 |
| 16 | Fact | 12.5 | 4 | 4 | 25 | 70 | 65.8 |
| 17 | Fact | 12.5 | 4 | 4 | 15 | 68 | 63.92 |
| 18 | Fact | 11.5 | 4 | 10 | 15 | 70 | 65.8 |
| 19 | Axial | 12 | 5 | 7 | 30 | 70 | 65.8 |
| 20 | Fact | 11.5 | 6 | 4 | 15 | 70 | 65.8 |
| 21 | Fact | 11.5 | 4 | 4 | 15 | 68 | 63.92 |
| 22 | Center | 12 | 5 | 7 | 20 | 90 | 84.6 |
| 23 | Axial | 12 | 7 | 7 | 20 | 65 | 61.1 |
| 24 | Fact | 12.5 | 6 | 10 | 25 | 70 | 65.8 |
| 25 | Center | 12 | 5 | 7 | 20 | 92 | 86.48 |
| 26 | Fact | 12.5 | 4 | 10 | 25 | 72 | 67.68 |
| 27 | Axial | 13 | 5 | 7 | 20 | 67 | 62.98 |
| 28 | Fact | 12.5 | 6 | 4 | 15 | 70 | 65.8 |
| 29 | Fact | 11.5 | 4 | 4 | 25 | 72 | 67.68 |
| 30 | Fact | 11.5 | 6 | 10 | 25 | 73 | 68.62 |

The readings indicate a strong match between experimental and expected results for the efficiencies of extraction and stripping, ANOVA developed quadratic model equations. Probability values ($F < 0.05$) indicate that the terms of the model are important. While, values above 0.1, indicating that the terms of the model are not significant.

### 3.2 *Three-dimensional response surface (Graphical analysis)*

The response surface method was used to analyze the three-dimensional response plot developed by bulk liquid membrane technique from aqueous solution. The graph is useful to see the surface shape of a response; hills, valleys, and ridge lines. The resulting graphics provided excellent clarity for the effects of pH feed and pH stripping, initial concentration of dye and concentration of carrier.

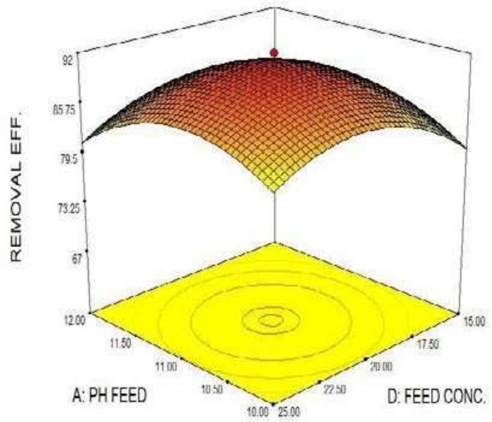
In this diagram, a piece fee of $x_1$ and $x_2$ produces a y-rate. This three-dimensional plan shows the response surface from the cross and it is called a response surface plot. Sometimes, it is less difficult to observe the response surface in two-dimensional charts. The contour plots can show contour ranks of $x_1$ and $x_2$ that have the same reply rate y. The plane response outlines plots of percent removal of MB against two of the functional factors studied when the other two bounds are detained at the center.

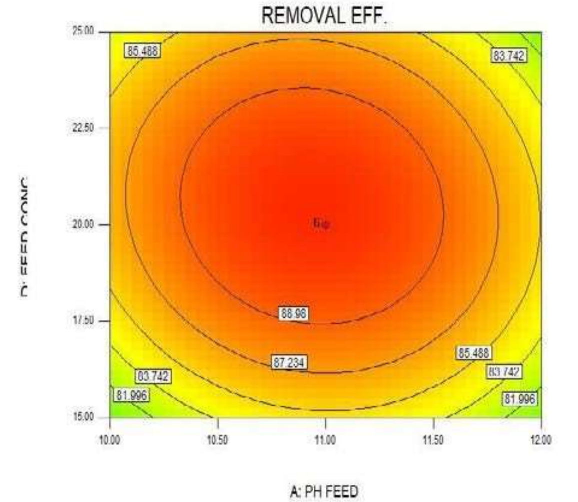
### 3.3 *Effect of pH feed and pH stripping*

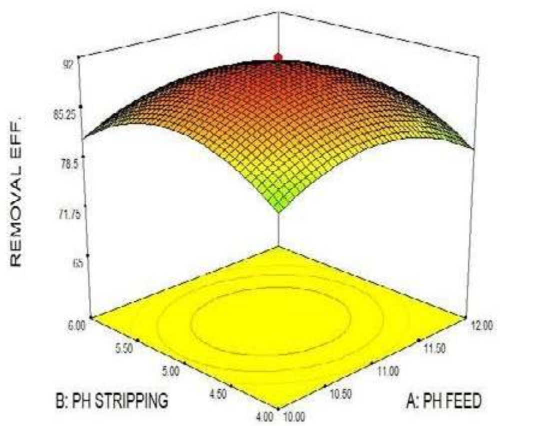
The communication of pH of source and pH of receiving on removal percent of MB dye is presented, as reply superficial and shape plot, in Figure.3. The high removal percent plateau resembles a pH of feed phase range of

10.75 to 11.25 range and pH of stripping phase range of 4.75 to 5.25 range values. When the other two parameters initial feed (MB dye) concentration, and Carrier concentration are held at midpoint.

It is noticed that the dye removal section upsurges up to pH of feed phase from 10.75 to 11.25 and then reduces. A good charge dissemination is revealed once pH of source, pH of receiving phase at this range is secondhand for the source solution making it cationic and TOA having a negative responsibility on it. Hence, MB procedures a multipart with the mover at the source/natural phases edge. Thus, a pH pitch between the source and the receiving phases is the driving force for the transport of MB to complete the liquid film. Therefore, the pH of the receiving phase is lower than pH of the source phase for the effective carrying efficiency.



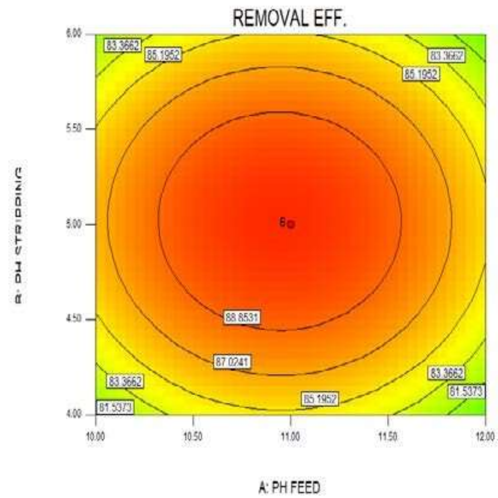




**Figure 3**: Interacting effects of pH feed and pH stripping on MB removal: surface plot; contour plot.

**Figure 4:** Effects of pH feed and dye concentration: surface plot; contour plot.

The higher removal efficiency was found about 92% at pH 10.75 to 11.25 range of feed and pH 4.75 to 5.25 range of stripping phase. Because of the acidity and alkalinity of the phases.

### 3.4 *Effect of pH feed and initial feed (MB dye) concentration*

Figure. 4 displays the consistent shape and retort surface plot for MB removal percent as a utility of pH-rate of source stage then initial feed (MB dye) concentration. It can be notice that a great-crop plateau occurs in the superficial above the pH-rate of source phase and initial feed concentration ranges between 11.75 to 20.75 ppm, respectively.

This plateau is embodied by the deepest contour. The upland's greatest matches to an MB removal % of 92. This is in conformity with the findings

It is observed that dye mining percentage augmented up to pH 10.75 to 11.25. A decent custody scattering is exposed when pH of 11 is cast-off for the source solution making it cationic and methylene blue having an adverse charge on it. Then, MB customs multifaceted with the mover at the source/nature phases edge.

The removal efficiency of feed gradually decreased with increasing the initial MB concentration up to 92%, and reached a maximum at concentration of 17.75 to 22.25 ppm. However, at a concentration of 17.75 to 22.25 ppm, MB removal efficiency extremely increased. This result is in arrangement with the whole thing presented in the works that confirm that MB concentration had a consequence on elimination adeptness of MB in the aqueous source phase. In overall, the driving force for bulk handover is classically the difference in substance budding, and component budding is straight connected to meditation pitch. Because of limits in the plan of fluid film system and decrease of the edge between platforms, MB transport through the edge is restricted. Then, the increase in MB deliberation in excess of an exact quantity in source phase causes the overload of edge between source and film phases and increase of MB grains on edge, and subsequently bargain in MB carriage.

### 3.5 *Result of pH of the source phase and Carrier concentration*

The result of pH rate of feed and Mover (TOA) concentration on MB removal efficiency, as a reply superficial and shape plot, is accessible by Figure5. It can be seen that a great-removal efficiency upland exists in the exterior over a source phase pH range of 10.75 to 11.25 and a Mover (TOA) concentration range of 6.75 to 7.25%w.

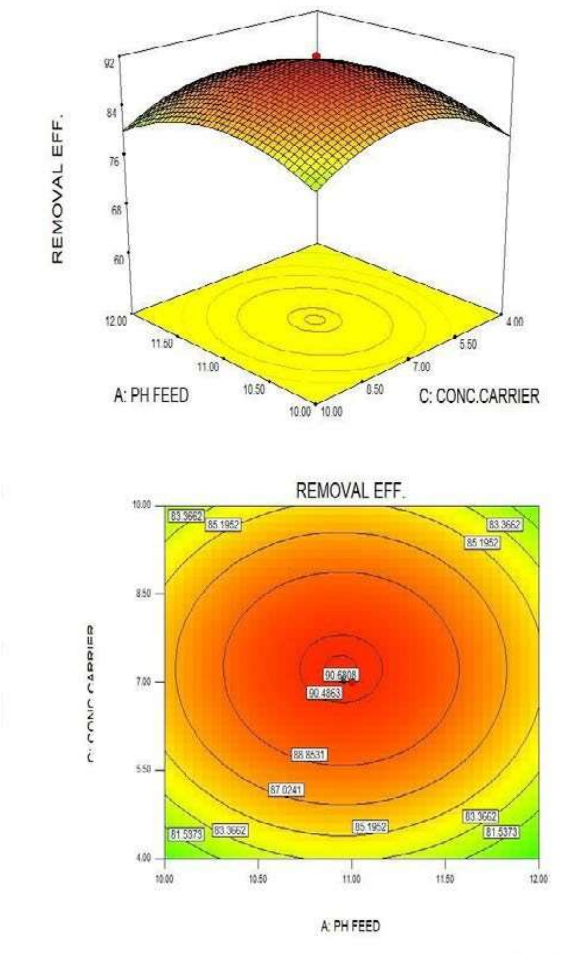



**Figure 5**: Effect of pH-value of the feed phase and Carrier concentration on dye removal: surface plot; contour plot

Figure 5, shows that the surface has a maximum dye removal efficiency. This efficiency of membrane transportation is increasing as the concentration of the carrier rises and saturates somewhere and after that, the diffusion rates are described as the viscosity of the membrane increases. Where a teeth carrier complex is formed at the feed membrane interface in the presence of a carrier as in the membrane phase which leads to a greater mass transfer rate via the interface, and thus greater separation.

### 3.6 *Effect of pH stripping and initial dye Concentration*

The three-D (response surface plot) for MB deduction is set in Figure. 8, which displays the result of pH of stripping as well as initial dye Concentration on MB deduction. It can be comprehended that MB removal displays a pure surface, it recommends that the best condition for extreme MB abstraction is well-defined and esoteric the system periphery. Communication plan concerning pH of receiving and initial dye Concentration; the interference transpired at a small flat of pH and a small flat of initial dye Concentration obviously discloses the communication among these two issues. (Response surface and contour plot) are presented by Figure.6. It can be seen that a great-removal efficiency highland occurs in the shallow above a stripping phase pH range of 4.75 to 5.25 range and an initial feed concentration range of 17.75 to 22.25 ppm.

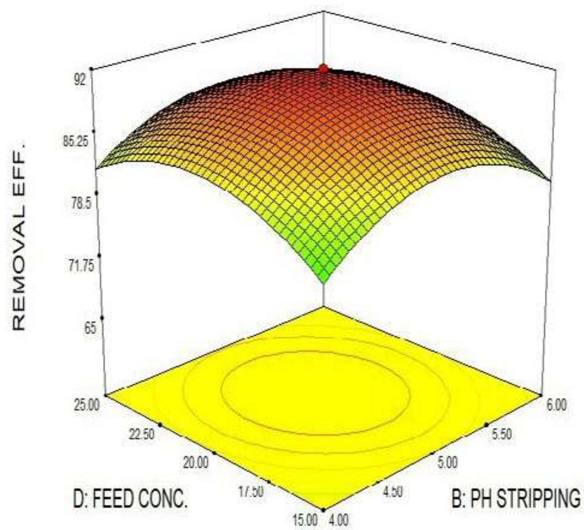



**Figure 6:** Effects of pH feed and initial feed concentration: Surface plot; contour plot

#### 3.6.1 Effect of pH stripping and Carrier concentration

Figure 7, shows removal efficiency of MB dye varied as a role of pH stripping and Mover concentration. Contour plot and response surface can be found that the surface has a high-removal efficiency plateau over the pH stripping effect at 5 and the carrier concentration is 6.75 to 7.25% MB removal was achieved 92%.

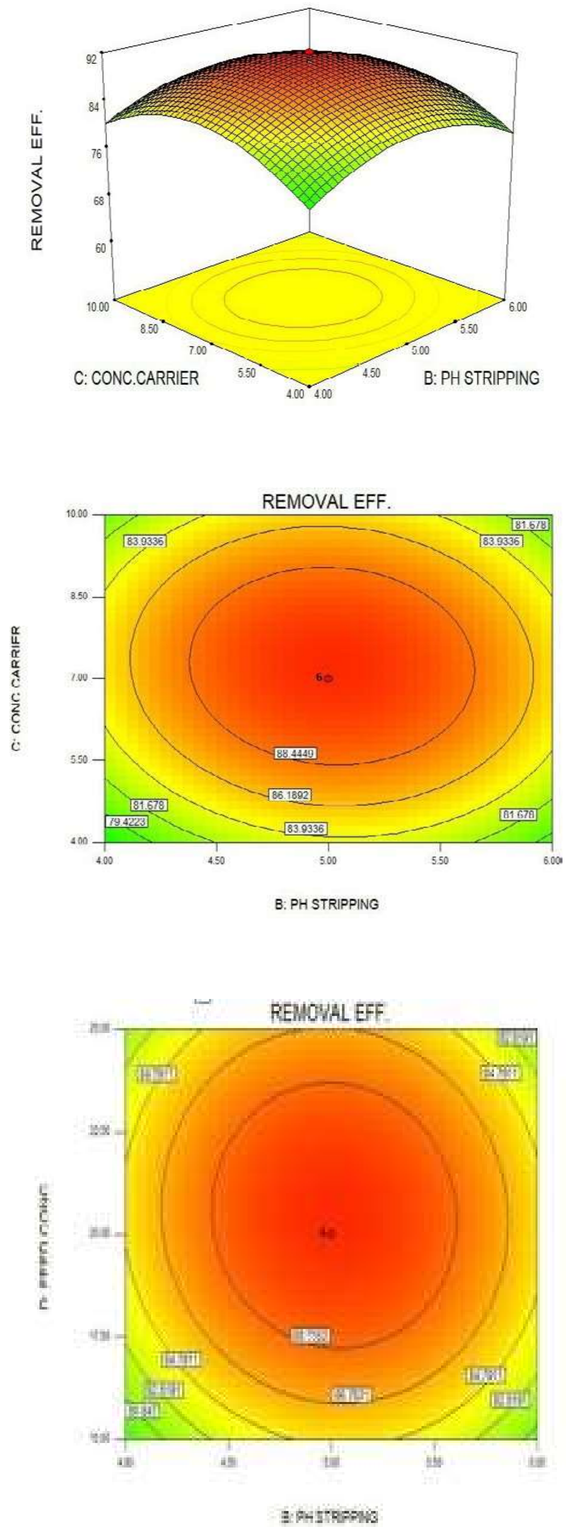



**Figure 7**: Interacting effects of pH stripping and Carrier concentration

### 3.7 *Effect of Initial Concentration and Carrier Concentration*

The effect of initial MB concentration and mover concentration on the transport efficiency for MB, as a (response surface and contour plot), is accessible by Figure.8. It can be seen that a great-removal efficiency observed and exists on the surface above a primary MB concentration array of 17.75 to 22.25 mg/l, and a carrier concentration range of 6.75 to 7.25 % (w/w). It may be due to the competition between MB at very high concentrations; therefore, the organic extracting (carrier) cannot be able to transport the MB from the aqueous source phase to receiving phases.

The grades of the effect and communication of the pH feed and stripping significance, carrier concentration, then initial feed concentration using CCD show that the maximum removal could be attained close the varieties, norms as shown on the shape plots. Figure. 8. The optimum condition values were 7%w this presented that the optimum. The fallouts obtained from three reproductions revealed that the extreme tentative deduction adeptness 92 % existed close to the expected value 17.75 to 22.25 range ppm. This result shows the authority of the classic. A surplus benefit of the (response surfaces and contour plots), chiefly the great-removal tablelands and their bulks would be the qualifications for an aviator or a manufacturing-balance part switch circle(s) to certify great-abstraction of MB on total times.

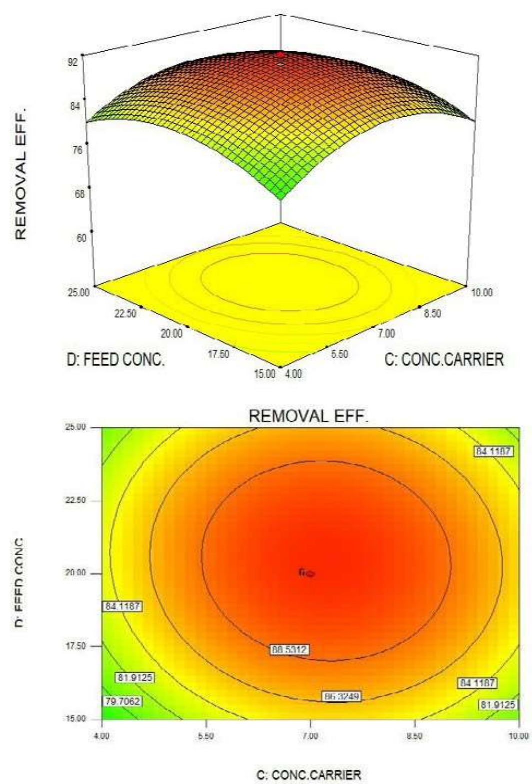



**Figure 8:** Interacting effects of Initial dye Concentration and Carrier Concentration

## 4. Conclusions

The major conclusion that can be drawn from this study are summarized below:

1-The obtained experimental results indicated that the bulk liquid membrane technique is effective for removal of MB contaminated in aqueous solutions.

It was found that the presence of carrier TOA is very important for the dye to transport .The optimum carrier concentration (w/w) of MB was 6.75 to 7.25.

The experimental results indicated that the highest MB transport occurs when the pH of the feed and strip phases are adjusted to a value of 10.75 to 11.25 and 4.75 to 5.25, respectively.

Increased MB concentration decreases the transport of metal ions through the membrane phase. As the concentration of the acid decreases, the methylene blue transferred between the aqueous solution and the membrane decreases.

The removal efficiency (92%) for MB by using soybean oil as solvent, TOA as carrier, and hydraulic acid as stripping phase at pH of the feed phase 10.75 to 11.25, pH of stripping phase 4.75 to 5.25, initial MB concentration 17.75 to 22.25 ppm, carrier concentration in the membrane phase 6.75 to 7.25 (w/w) TOA.

A simple second order quadratic model equation was developed using Design Expert software for predicting the response removal of MB, on overall experimental regions and correlating the operating parameters.

The reliability of the developed model has been ensured from the high magnitude of the correlation coefficient (R^2 (0.9385) and R^2(adj)) for MB between the experimental and model predicted values, respectively.

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# إزالة صبغة المثيلين الزرقاء من المحاليل المائية باستعمال الاغشية السائلة

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نشر في: 31 كانون الاول 2021

**الخلاصة** – يهدف هذا العمل الى اختار التصمم المناسب وتحسين معاملات التشغيل للغشاء السائل السائب فول الصوا القائم على زت لإ ازلة الميثيلين الأزرق والاسترداد من المحاليل المائة. يتكون زت فول الصوا من مرحلة تغذة مائة مصدر MB ، ومرحلة غشاء عضوي (زت فول الصوا (المخفف) ، وثلاثي أوتيل أمين( TOA) (الناقل) ومرحلة الطور المستلم (محلول حمض الهيدرولورك( HCl). تم التحقيق في آثار معاملات التصمم (درجة الحموضة في الطور المغذي ، وم ارحل الاستلام ، وتريز الصغة الأولة( MB) ، وتريز الناقل) من زت فول الص وا على إ ازلة الصغة والاسترداد من المحاليل المائة. تحسين معاملات التشغيل لـ للمنظومة المستندة إلى زت فول الصوا للحصول على أقصى نسة مئوة ) ٪( إ ازلة الصغة ان 11 درجة الحموضة تغذة وتجرد درجة الحموضة ان 5 في حين أن أفضل سرعة التحرك ان 250 دورة في الدققة،و 7٪ ناقل كان النسة المئوة المثلى للغشاء.

**الكلمات الرئيسية** – "الغشاء السائل"زيت فول الصويا"الالكوات 336"ثلاثي اوكتيل امين"المثيلين الزرقاء"