Removal of AY99 from an aqueous solution using an emulsified liquid membrane. Application of Plackett-Burman Design

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Abstract

Water contaminated with dyes presents serious environmental problems. An important quantity of dyes is released as industrial waste in nature. As part of the recovery and the regeneration of these complexes, the extraction of a cationic dye Acid Yellow 99 has been the subject of this work. To removal dyes from industrial wastewater, the technique of extraction by emulsion liquid membrane could provide an industrial success. The membrane used in this study consisted of SPAN80 as emulsifier and aliquat 336 as extractant. The stability of the emulsified liquid membrane has a very important role in the extraction. A study of the effects of different components of the membrane is necessary. The process parameters were studied using a statistical method of experimental Plackett-Burman design, This method allows us to study the effects of different factors simultaneously and determines which factors are most important parameters. The modeling was done by a mathematical model representing the extraction yield according to various factors. The most significant factors on the elimination of the acid yellow 99 by the emulsion liquid membrane were then studied.

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Keyword: emulsified liquid membrane, modeling, recovery, acid yellow, design of experiment (DOE).
1. Introduction

Dyes are widely used in many industries such as textile, leather, pulp, paper and plastics in order to color their products. About 700,000 tones and 10,000 different types of dyes and pigments are being produced annually across the world [1, 2]. The use of coloring species requires a separation treatment and recycling or regeneration adapted to separate the dye product of the reaction studied. For this, the technique of separation by emulsion liquid membrane (ELM) is a good opportunity [3-5]. Extraction with ELM was invented and used for the first time by Norman N.LI (1978) [6]. Several works have emerged since then, as the extraction of heavy metals [7-9] or precious metals [10], the extraction of heteropolyanion complexes [11], the extraction of chlorophenols [12], the separation of hydrocarbons [13, 14] etc. In general chemistry, the extraction of organic acids and other products can be extracted using emulsified liquid membranes [15]. In hydrometallurgy and using an ELM consists of Span80 (sorbitan mono-oleate), extraction was studied using LIX-84 and sulfuric acid, respectively as the surfactant, the extraction and the internal phase [16]. With the same emulsifier, the extraction of uranium from an aqueous solution, was carried out by the extractant TOPO (trioctylphosphine oxide) in the presence of a diluent paraffin 60% C12 alkanes [17]. In medicine, for example, hydroxyapatite Ca10(PO4)6 (OH)2 which is the main constituent of human bones and teeth is widely used as implants or prostheses layers [18,19]. To study the effect of Ca/P ratio on the texture and morphology of this product, an ELM extraction was applied using a biodegradable emulsifier and a fatty acid as extractant [20]. The ELM method was also applied in biochemistry; citric acid extraction [21], penicillin separation [22, 23], removal of cholesterol blood [24]. In Austria galvanic solutions, about 0.15m3/h are treated with ELM and Ni is extracted yielding 90%. The initial solution is then concentrated to be treated by conventional extraction [25].

Extractions of different metals have been reported and compared when using emulsified liquid membrane extraction processes in presence of different extractants, surfactants and feed solutions [26]. A good stability according to the composition of the membrane has been reported in different studies [9, 12 and 27].

The study of extraction by ELM is complicated because it depends on several factors. Mathematical models proposed by researchers often lack realism and in most cases are specific and can’t be generalized. If the transfer through the membrane is a simple diffusion, equation basis will be deduced directly from the second law of Fick and once we introduce a carrier in the membrane as an extractant, the transfer mechanism will become diffusion-reaction in place of simple diffusion [28] and the study will be necessary solving a set of equations that would require an arsenal of mathematical tools. If in addition were taken into account the rupture, the cohesion and the swelling of the membrane, the modeling will become more and more difficult. Therefore, the use of empirical models will be all indicated. Researchers often conduct a study of a phenomenon using the conventional single factor design method. They set all the parameters and vary only one. Then they fix this factor at an optimum level and vary another parameter and so on. Unfortunately this technique is not recommended in all situations particularly when the number of parameters is important or there are interactions between them. In this case the use of design of experiments is imperative. In a full factorial design the runs are carried out so that all combinations of levels (possible values of parameter) must be made. When the number of parameters increases, the number of runs becomes very high. When the runs of only one part of the complete full factorial design can give enough information, this method is called fractional factorial design. Using this experimental design we can calculate the coefficients for each parameter and express its importance in relation to the phenomenon studied. We can also calculate the coefficients associated with the interactions between parameters [29].

In this study an approach of design of experiment [30, 31] was applied to determine the parameters that influence on the extraction of AY99 from an aqueous solution using a fractional factorial design of Plackett-Burman [32, 33]. The membrane was consisting of SPAN 80 as the surfactant and aliquat 336 as the extractant. Statistical analysis of experimental results was studied and a modeling of the yield of extraction according to operating conditions was also achieved.

2. Experimental

2.1. Materials and compounds

The emulsified liquid membrane used for the extraction of acid yellow 99 consisted of Aliquat 336 as the extractant, SPAN80 (sorbitan monooleate) as the surfactant, cyclohexane as the thinner and sulfuric acid as the internal phase. The trioctylmethylammonium chloride (Aliquat 336) (Fig.1) was supplied by Sigma Aldrich. In its
ionic form, \( \text{CH}_3\text{N}[(\text{CH}_2)\text{CH}_3]_3\text{Cl} \) is an excellent carrier of solute. The sorbitan monooleate (SPAN80) (Fig.2) supplied by Sigma Aldrich is a nonionic surfactant type ester with lipophilic character (HLB = 4.3), it was used for the stability of the emulsion. Cyclohexane produced by Riedel-de Haën was used as a thinner, it was a stable product under ordinary conditions, its role was to improve mainly some physicochemical properties of the extractant and surfactant. The acid yellow 99 (Fig.3) is an anionic dye supplied by Sigma Aldrich.

The homogenizer Ultra-Turrax T8 was a mechanical agitator type RW20Junk & Kunkel, with a marine propeller; it was used to make the double emulsion W/O/W (water/oil/water). The rotating rotor at high speed sucked in the solution to be treated to the head axially and then dispersed laterally through the rejects of the rotor stator slots.

![Fig.1 : Aliquat 336](image1)

![Fig.2 : SPAN 80](image2)

![Fig.3: AY 99](image3)

2.2. Experimental procedures

Emulsions were made using the homogenizer Ultra-Turrax T8; a certain volume of \( \text{H}_2\text{SO}_4 \) as an internal phase was added to an organic phase composed of cyclohexane as thinner, Aliquat 336 as extractant and Span 80 as surfactant. The mixture was emulsified at 5000 rpm into a beaker tall form within 5 minutes. Then, using a mechanical stirrer type RW20 Kjank & Kunkel at 150rpm, this emulsified membrane was dispersed into a beaker containing 150mL of the solution to be treated (external phase containing initial concentration of AY99). The pH variation of the external phase was monitored using a pH-meter type HANA Hi 8519N.

The concentration of the residual complex AY99 at different reaction times ranging from 3 to 15 minutes was determined by up taking samples of 2mL and measuring the absorption intensity using a Jenway (6705UV/VIS) spectrophotometer. The wavelength was determined experimentally and was used in the same conditions. The samples were analyzed to determine the concentration of the residual complex AY99 from a calibration curve carried out at ordinary temperature. The extraction efficiency was calculated by the equation 1.

\[
Y(\%) = \left[1 - \left(\frac{C_{fext} \times V_{fext}}{C_{0ext} \times V_{0ext}}\right)\right] \times 100
\]

(1)
$V_{\text{ext}}$: initial volume of the external phase
$V_{\text{extf}}$: final volume of the external phase.
$C_{\text{exti}}$: initial concentration of AY99 in the external phase
$C_{\text{extf}}$: final concentration of AY99 in the external phase.
$Y_{\text{ext}}$: Extraction yield.

3. Results and discussion

3.1. Experimental results

The extraction of AY99 was conducted by varying eight factors simultaneously listed in Table 1. The minimum and maximum of level for each factor were chosen after a literature review and especially after performing preliminary tests. Table 2 summarizes the different operating conditions of extractions using an emulsified liquid membrane according to a Plackett-Burman experiments design. The experimental results of extraction yields are also presented.

Table 1: Parameters and levels

<table>
<thead>
<tr>
<th>N° run</th>
<th>Parameter</th>
<th>Unit</th>
<th>Level</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Low (-1)</td>
</tr>
<tr>
<td>1</td>
<td>SPAN 80</td>
<td>%</td>
<td>8</td>
</tr>
<tr>
<td>2</td>
<td>Aliquat336</td>
<td>%</td>
<td>3</td>
</tr>
<tr>
<td>3</td>
<td>SV</td>
<td>rpm</td>
<td>100</td>
</tr>
<tr>
<td>4</td>
<td>$[\text{H}_2\text{SO}_4]$ int.</td>
<td>Mol/L</td>
<td>0.5</td>
</tr>
<tr>
<td>5</td>
<td>O/A</td>
<td>_</td>
<td>1</td>
</tr>
<tr>
<td>6</td>
<td>Vex/Vem</td>
<td>_</td>
<td>5</td>
</tr>
<tr>
<td>7</td>
<td>$[\text{AY99}]_0$</td>
<td>ppm</td>
<td>10</td>
</tr>
<tr>
<td>8</td>
<td>$[\text{H}_2\text{SO}_4]$ ext.</td>
<td>Mol/L</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Table 2: Experimental results according to Plackett-Burman Design

<table>
<thead>
<tr>
<th>Run Order</th>
<th>SPAN80</th>
<th>Aliquat336</th>
<th>Stirring Velocity</th>
<th>$\text{H}_2\text{SO}_4$ internal</th>
<th>O/A</th>
<th>Vex/Vem</th>
<th>$[\text{AY99}]_0$</th>
<th>$\text{H}_2\text{SO}_4$ external</th>
<th>Y (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8</td>
<td>5</td>
<td>100</td>
<td>0.5</td>
<td>1</td>
<td>7</td>
<td>50</td>
<td>0.5</td>
<td>76.38</td>
</tr>
<tr>
<td>2</td>
<td>11</td>
<td>3</td>
<td>150</td>
<td>1.0</td>
<td>1</td>
<td>7</td>
<td>10</td>
<td>0.1</td>
<td>70.89</td>
</tr>
<tr>
<td>3</td>
<td>11</td>
<td>5</td>
<td>100</td>
<td>1.0</td>
<td>3</td>
<td>5</td>
<td>50</td>
<td>0.1</td>
<td>79.54</td>
</tr>
<tr>
<td>4</td>
<td>8</td>
<td>5</td>
<td>150</td>
<td>1.0</td>
<td>1</td>
<td>7</td>
<td>50</td>
<td>0.1</td>
<td>86.60</td>
</tr>
<tr>
<td>5</td>
<td>11</td>
<td>5</td>
<td>150</td>
<td>0.5</td>
<td>3</td>
<td>7</td>
<td>10</td>
<td>0.5</td>
<td>69.00</td>
</tr>
<tr>
<td>6</td>
<td>11</td>
<td>3</td>
<td>150</td>
<td>0.5</td>
<td>1</td>
<td>5</td>
<td>50</td>
<td>0.5</td>
<td>75.98</td>
</tr>
<tr>
<td>7</td>
<td>11</td>
<td>5</td>
<td>100</td>
<td>0.5</td>
<td>1</td>
<td>5</td>
<td>10</td>
<td>0.5</td>
<td>90.97</td>
</tr>
<tr>
<td>8</td>
<td>8</td>
<td>3</td>
<td>100</td>
<td>1.0</td>
<td>3</td>
<td>7</td>
<td>10</td>
<td>0.5</td>
<td>69.10</td>
</tr>
<tr>
<td>9</td>
<td>8</td>
<td>5</td>
<td>150</td>
<td>0.5</td>
<td>3</td>
<td>5</td>
<td>10</td>
<td>0.1</td>
<td>69.50</td>
</tr>
<tr>
<td>10</td>
<td>8</td>
<td>3</td>
<td>100</td>
<td>0.5</td>
<td>1</td>
<td>5</td>
<td>10</td>
<td>0.1</td>
<td>75.90</td>
</tr>
<tr>
<td>11</td>
<td>8</td>
<td>3</td>
<td>150</td>
<td>1.0</td>
<td>3</td>
<td>5</td>
<td>50</td>
<td>0.5</td>
<td>93.64</td>
</tr>
<tr>
<td>12</td>
<td>11</td>
<td>3</td>
<td>100</td>
<td>0.5</td>
<td>3</td>
<td>7</td>
<td>50</td>
<td>0.1</td>
<td>61.21</td>
</tr>
</tbody>
</table>
3.2. Pareto chart

The Pareto chart of effects is a useful field to identify the most important factors (Fig.4). It shows the estimated main plot against the horizontal effect. From Figure 4 we can see that the most important factors are the acidity of the internal phase and the proportion of the emulsion represented by the $V_{ex}/V_{em}$ ratio. The O/A ratio, the acidity of the external phase, the initial concentration of dye, the extractant and the surfactant are less important. The stirring velocity has a low effect.

![Pareto Chart of Effects for Yield as response](image)

3.3. Main effects

The main effects plot is the most useful when there are several factors (Fig.5). From level changes, we can deduce the influence of all factors. These effects may be positive or negative.

![Main Effects Plot of parameters for Y(%)](image)

From figure 5, we can see that the factors which have a positive effect on the performance of the emulsion are: the acidity of the internal and the external phases, the initial concentration of dye and the extractant. The effect of SV is low and positive. The positive effect on the response means that the increase of these factors in the chosen field implies an increase in the response. On the other hand, the factors having a negative effect on the extraction yield are: Span80, O/A ratio and Vex/Vem ratio.
The acidity of the phases is important and promotes the extraction of the dye which is itself acid. The decrease of
Vex/Vem ratio implies an increase in the volume of emulsion and promotes the extraction yield. The increase of the
quantity of Span80 promotes a decrease of the extraction yield. Indeed higher is important the concentration of
Span80, more is dense the interfacial film (thicker membrane) which prevents the dye to pass into the internal phase.
The results are in agreement with the results obtained by [7, 8 and 10], the value 8 % of Span80 seems to be better.
The results show also that when the concentration of the extractant increases in this interval, the extraction
efficiency is high; may be for more extractant which is an organic phase the viscosity of the membrane increases
and makes thicker the interfacial film. In addition, it can be said that increasing the concentration of the extractant is
unfavorable for the stability of the emulsion. These results are in agreement with those reported by [7, 10, 11, 13
and 14], this behavior is due to the properties interfacial of Aliquat336 which reduces the stability of the membrane
and supports the O/W emulsion, causes the breaking of the emulsion and the expulsion of the internal phase into the
external phase, so the extraction yield decreases. The results indicate that the increase of the stirring speed promotes
better the transfer of solute from the external phase to the organic phase (membrane), the velocity increases the
efficiency by convection. This can be explained also that the increase in stirring speed promotes the shearing of the
emulsion globules and increases the exchange surface and so the transfer (extraction). A stirring speed of 150 rpm is
chosen as the best. The volume ratio of organic phase to the internal phase (O/A) is an important parameter which
decreases the dye extraction. The internal phase must be important in volume.

3.4. Analysis of variance (ANOVA)

Analysis of variance (ANOVA) is an essential tool for determining the magnitude of an effect or a mathematical
model. The term "significant" is used in its narrow sense of statistical significance. In other words, if an effect is
significant there will be a high probability (95%, 99%, or 99.9%) that the effect is "real" [34, 35]

Table 3: Effects and coefficients of the estimated yield (coded units).

<table>
<thead>
<tr>
<th>Term</th>
<th>Effect</th>
<th>Coef</th>
<th>Coef</th>
<th>T</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>76.559</td>
<td>84.185</td>
<td></td>
<td>41.41</td>
<td>0.000</td>
</tr>
<tr>
<td>Span 80</td>
<td>-3.922</td>
<td>-1.961</td>
<td>-1.307</td>
<td>-1.06</td>
<td>0.367</td>
</tr>
<tr>
<td>Aliquat 336</td>
<td>4.212</td>
<td>2.106</td>
<td>2.106</td>
<td>1.14</td>
<td>0.337</td>
</tr>
<tr>
<td>SV</td>
<td>2.085</td>
<td>1.042</td>
<td>0.0417</td>
<td>0.56</td>
<td>0.612</td>
</tr>
<tr>
<td>[H2SO4] int.</td>
<td>10.462</td>
<td>5.231</td>
<td>20.923</td>
<td>2.83</td>
<td>0.066</td>
</tr>
<tr>
<td>O/A</td>
<td>-5.788</td>
<td>-2.894</td>
<td>-2.894</td>
<td>-1.57</td>
<td>0.215</td>
</tr>
<tr>
<td>Vex/Vem</td>
<td>-8.725</td>
<td>-4.363</td>
<td>-4.362</td>
<td>-2.36</td>
<td>0.099</td>
</tr>
<tr>
<td>[AY99]0</td>
<td>4.665</td>
<td>2.333</td>
<td>0.1167</td>
<td>1.26</td>
<td>0.296</td>
</tr>
<tr>
<td>[H2SO4] ext</td>
<td>5.238</td>
<td>2.619</td>
<td>13.096</td>
<td>1.42</td>
<td>0.252</td>
</tr>
</tbody>
</table>

The most important factors can be determined using a statistical parameter that is P value (Table 3). This value
was compared to another α value that represents the model risk. Generally α is 5% risk. The coefficients of the
parameters presented in Table 3 were calculated from the Yates’ rating [36]. The algebraic values of the coefficients
measure the average change in extraction yield when the parameters change from level (-1) to level (1).

3.5. Polynomial regression

To enable the prediction of response and system optimization, the method of experiment design on both its
design and structure allows a mathematical representation of the response (yield) according to all factors. The
regressions are represented by polynomial equations 2 and 3 corresponding to the coded and uncoded parameters
respectively.

\[
Y(\%) = 76.559 -1.961 \times \text{Span80} + 2.106 \times \text{Aliquat336} + 1.042 \times \text{SV} + 5.231 \times [\text{H}_2\text{SO}_4]\text{ int} - 2.894 \times O/A - 4.363 + \times \text{Vex/Vem} + 2.333 \times [\text{AY99}]_0 + 2.619 \times [\text{H}_2\text{SO}_4] \text{ ext} 
\]  

(2)

\[
Y(\%) = 84.185 - 1.307 \times \text{Span80} + 2.106 \times \text{Aliquat336} + 0.0417 \times \text{SV} + 20.923 \times [\text{H}_2\text{SO}_4] \text{ int} - 2.894 \times O/A - 4.362 \times \text{Vex/Vem} + 0.1167 \times [\text{AY99}]_0 + 13.096 \times [\text{H}_2\text{SO}_4] \text{ ext} 
\]  

(3)
3.6. Graphical representation of estimated responses according to experimental responses

From Figure 6 the estimated and experimental yields are fairly distributed around the regression line ($R^2 = 0.88175$). The fit is very good especially beyond 75%.

![Graphical representation of estimated responses according to experimental responses](image)

Fig. 6: Correlation between theoretical and estimated yield.

3.7. Validation of the model for high yields

Table 4: Supplement experimental results (variation of only three important parameters: SPAN80, Aliquat336 and $H_2SO_4$ external).

<table>
<thead>
<tr>
<th>N°</th>
<th>SPAN80</th>
<th>Aliquat336</th>
<th>Stirring Velocity</th>
<th>$H_2SO_4$ internal</th>
<th>O/A</th>
<th>Vex/Vem</th>
<th>External phase</th>
<th>$H_2SO_4$ external</th>
<th>Y (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12</td>
<td>3</td>
<td>150</td>
<td>0.5</td>
<td>1</td>
<td>5</td>
<td>50</td>
<td>0.75</td>
<td>92.95</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>3</td>
<td>150</td>
<td>1.0</td>
<td>1</td>
<td>5</td>
<td>50</td>
<td>0.75</td>
<td>95.56</td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td>1</td>
<td>150</td>
<td>1.0</td>
<td>1</td>
<td>5</td>
<td>50</td>
<td>0.25</td>
<td>84.79</td>
</tr>
<tr>
<td>4</td>
<td>12</td>
<td>1</td>
<td>150</td>
<td>1.0</td>
<td>1</td>
<td>5</td>
<td>50</td>
<td>0.75</td>
<td>88.71</td>
</tr>
<tr>
<td>5</td>
<td>12</td>
<td>3</td>
<td>150</td>
<td>0.5</td>
<td>1</td>
<td>5</td>
<td>50</td>
<td>0.25</td>
<td>86.48</td>
</tr>
<tr>
<td>6</td>
<td>12</td>
<td>1</td>
<td>150</td>
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<td>1</td>
<td>5</td>
<td>50</td>
<td>0.25</td>
<td>82.2</td>
</tr>
<tr>
<td>7</td>
<td>10</td>
<td>3</td>
<td>150</td>
<td>1.0</td>
<td>1</td>
<td>5</td>
<td>50</td>
<td>0.25</td>
<td>89.04</td>
</tr>
</tbody>
</table>

![Validation of the model for high yields](image)

Fig. 6: Correlation between experimental and estimated yields (A: supplement experimental results, B: basic experimental data).
In the conditions for high yields, supplement experiments had been carried out and the experimental yields (Table 4) had been compared to the yields estimated by the models developed above (Fig.7). A very good correlation with experimental yields was obtained \( A : R^2 = 0.9999 \). On the other hand and from table 2, high experimental and estimated yields were reported on the same figure (Fig.7); the correlation was also very good \( B : R^2 = 0.9887 \).

3.8. **Optimization of the model**

From main effects of the extraction yield, optimized conditions can be deduced as below:

\[
\begin{align*}
[H_2SO_4]_{\text{int}} &= 1.0 \text{ Mol/L in the internal phase} \\
[H_2SO_4]_{\text{ext}} &= 0.5 \text{ Mol/L in the external phase} \\
V_{\text{ext}} / V_{\text{em}} &= 5 \\
[A\text{Y99}]_0 &= 50 \text{ mg/L} \\
\text{Aliquat 336} &= 5 \% \\
\text{Span80} &= 8 \% \\
\text{Dilute cyclohexane} &= 84 \% \\
\text{Emulsification time} &= 5 \text{ min} \\
O/A &= 1 \\
\text{Contact time} &= 10 \text{ min} \\
\text{Stirring Velocity} &= 150 \text{ rpm}
\end{align*}
\]

Under these operator conditions, the polynomial models according to the coded and uncoded parameters give yields 99.11% and 99.12% respectively. Two experimental validations of the model were carried out and the yields obtained were 99.09% and 99.12%.

4. **Conclusion**

Using a Plackett-Burman design the extraction of AY 99 (anionic dye) was carried out varying different operator parameters simultaneously. Effects of parameters on the extraction yield were analyzed statistically and a mathematical model of the yield according to different parameters was developed. Main effects were studied and levels of all parameters correspondent to the best yield were determined. The membrane used in this study was composed of Span 80 as the surfactant, Aliquat336 as the extractant in cyclohexane as the diluents. The stability of the emulsified membrane had a very important role in the extraction. Among the eight factors studied, the acidity of the internal and external solutions, the composition and the proportion of the membrane were the most important factors for the extraction yield of AY99.

The regression was linear between the values of the estimated yields given by the established models and all experimental performances. The fit was good and the correlation constant was \( R^2 = 0.8817 \). This correlation was enhanced to \( R^2 = 0.9887 \) for higher yields and had been perfect in the best operator conditions \( R^2 = 0.9999 \).

Under optimized operator conditions deduced from main effects, a validation of the model was carried out; the extraction yields given by the polynomial models according to the coded and uncoded parameters and the extraction yields obtained experimentally, were found to be between 99.09% and 99.12%. The model was therefore adequate to represent the process.

Plackett–Burman design was a first step to determine the most important operating parameters to extract AY99 using the emulsified liquid membrane. However to enhance the efficiency of this process and in perspectives, a best optimization of these parameters by response surface methodology (RSM) can be carried out using an experimental design of second order.

**References**


