



Trioctylamine Functionalized Polystyrene Membrane for Anionic Dye Extraction from Aqueous Solutions.

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ABSTRACT

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In this research, innovative polymer membranes have been developed using trioctylamine (TOA), a non-ionic extractant, in combination with polystyrene (PS) as the base polymer. The resulting membranes were characterised by scanning electron microscopy (SEM) to assess their morphology, and by X-ray diffraction (XRD) to analyse their crystalline structure. They were then used to extract the anionic dye Red Burgundy Acid (RBA). The extraction efficiency was evaluated under various experimental conditions, including a pH solution between 2 and 6, an extractant concentration in the membrane ranging from 5 to 30 $\mu\text{mol}/\text{cm}^2$, an initial dye concentration between 50 and 300 mg/L, and a stirring speed ranging from 250 to 400 rpm. Under optimised conditions, a remarkable 97% extraction efficiency was obtained for the RBA dye, highlighting the performance of the PS-TOA membrane system for removing this pollutant. Additionally, the membrane had been regenerated and reused up to five times without any significant loss of efficiency.

1. INTRODUCTION

The environmental threat posed by heavy metals [1,2], pharmaceutical waste [3, 4], and dyes is a matter of great concern. Dyes, particularly those used in industries such as textiles, not only contribute to environmental degradation but also pose health hazards in drinking water [5, 6]. It is imperative to effectively manage and dispose of these contaminants to mitigate their long-term effects on both the environment and human health [7, 8].

In the last few years, the membrane separations applications for the control of pollution of dyes have been studied extensively [8, 9]. Membrane separation is an emerging separation process that is expected to eventually replace many traditional separation methods. In recent investigations, several researchers have investigated the use of membrane extraction for dyes. Muthuraman et al. [10] have reported successful dyes recovery using liquid-liquid extraction techniques. Liquid membranes have also been developed for the efficient extraction of dyes from wastewater. Some researchers have focused on the extraction of both anionic and cationic dyes from aqueous solutions through the utilization of emulsion liquid membranes [11, 12].

Liu N. et al. [13] conducted research wherein they employed anion-exchange membranes as adsorbents to eliminate anionic reactive dyes from water. And Liu C. et al. [14] employed a cation exchange membrane for the purification of water containing dye pollutants

Recently, a novel liquid membrane system, denoted to as a polymer inclusion membrane, has been formulated. The process involves casting a base polymer from an organic solution to yield a thin and stable film [15]. Polymer inclusion membranes (PIMs) preserve most of the advantages found in supported liquid membranes (SLMs) while highlighting exceptional stability and flexibility. The reduced diffusion coefficient often observed in PIMs can be easily counterbalanced by creating a significantly thinner membrane compared to its conventional SLM counterpart [16].

PIMs are typically composed of three main components: a high molecular weight polymer for mechanical strength, a plasticizer to impart elasticity, and a carrier molecule to aid in the selective transport of analytes. Among the various polymers available for preparing PIMs, poly(vinyl chloride) (PVC) and cellulose triacetate (CTA) are the most commonly employed choices due to their ability to

effectively encapsulate carrier molecules while preventing their undesired release [17,18].

In the process of preparing Polymer Inclusion Membranes (PIMs), various carriers are utilized, among which TOA is a colorless oily liquid, characterized by a boiling point of about 365.812 °C. This extractant is used for the extraction of organic molecules such as carboxylic acid [19,20], for the separation of copper(II), cobalt(II) and nickel(II) ions from chloride acidic aqueous solution through polymer inclusion membrane [21] for the extraction of maleic acid and phthalic acid by supercritical carbon dioxide [22] and extraction of Uranium extraction from sulfuric acid by impregnated and activated carbon [23].

The aim of this study is to gain a better understanding of the behaviour of polystyrene-based polymer membranes activated by trioctylamine in the selective extraction of an anionic dye, Red Burgundy Acid (RBA). The main objective is to develop an effective extraction method by taking advantage of the complementary properties of polystyrene and trioctylamine, used as an extractant that promotes transfer of the dye from the aqueous phase to the membrane. To this end, various parameters influencing the extraction efficiency will be optimised, such as the pH of the solution, the concentration of the dye, the content of extracting agent in the membrane and the operating conditions, with the aim of improving the overall performance of the PS-TOA system in the retention of RBA.

2. EXPERIMENTAL

2.1. Reagent

Polystyrene (PS) and trioctyl mine (TOA) were obtained from Sigma-Aldrich. Chloroform ($CHCl_3$), sulfuric acid (H_2SO_4 ; purity 99.99%) and sodium hydroxyde $NaOH$ were acquired from Fluka. Red burgundy acid (acid violet 90) was purchased from Biochem Chemopharma. The structure of this dye is given in Figure 1. Ultra-pure distilled water was utilized to solubilize a variety of reagents in the formulation of the aqueous phases.

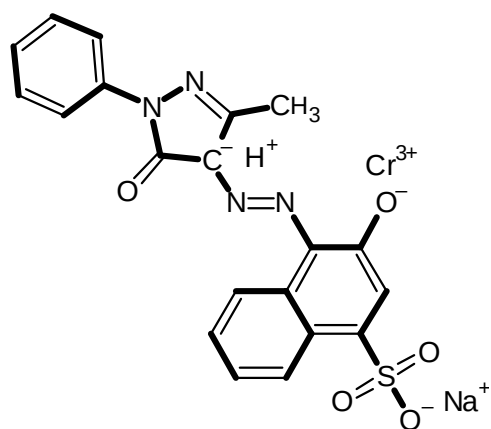


Figure. 1 The structure of Red burgundy acid (RBA) (Acid Violet 90).

2. 2. Polymer membrane preparation

The PS-TOA matrix was developed using the same method as explained by Aitali et al. [24]. At first, 0.2 grams of PS (polystyrene) were dissolved in 10 milliliters of $CHCl_3$ (chloroform) at room temperature. After vigorous stirring, 2mL of TOA were added and the solution was agitated for 30 minutes to obtain an homogenous mixture. The solvent of this blended solution was then allowed to gently evaporate at room temperature in a flat-bottom vessel with a diameter of 9.0 centimeters that was loosely covered. The evaporation process was conducted overnight. Subsequently, a small amount of water was added to facilitate the detachment of the film from the glass support.

2.3. Characterizing membrane structures

SEM images of the membrane polymer and the RBA dye were captured using a HITACHI S4500 microscope, which boasts a resolution capability of 1.5 nm. In addition, X-ray diffraction (XRD) analyses were conducted using an Expert Pro Panalytical diffractometer.

2.4. Polymer membrane extraction

The solid-liquid extraction experiments were carried out in a batch reactor at controlled temperature. To investigate the influence of various parameters on the solid-liquid extraction efficiency, the following factors were considered: pH solution (2-6) (adjusted using dilute H_2SO_4 or $NaOH$ (0.1M)), extractant concentration in the membrane (5-30 $\mu\text{mol}/\text{cm}^2$), initial RBA concentration (50-300 mg/L), and stirring speed (250-400 rpm). Each experiment was carried out using a membrane with a mass of approximately 0.05 grams. After each extraction test, the RBA concentration was determined by measuring the absorbance using a Shimadzu UV-1280 UV-visible spectrophotometer at maximum wavelengths of $\lambda_{RBA} = 525$ nm. The following equation was used to calculate the extraction efficiency (E).

$$E(\%) = \frac{[RBA]_{aq0} - [RBA]_{aq}}{[RBA]_{aq0}} \times 100 \quad (1)$$

$[RBA]_{aq0}$: the initial concentration of the dye in the aqueous phase (mg/L).

$[RBA]_{aq}$: the concentration of the dye in the aqueous phase after extraction (mg/L).

3. RESULTS AND DISCUSSION

3.1. Polymer membrane characterisation

The SEM images presented in Figure 2 (a, b, c) provide interesting details about the textures of the RBA dye, PS-TOA, and PS-TOA-RBA. The SEM image of the RBA dye (Fig 2a) reveals a complex surface morphology, characterised by well-defined crystalline structures and the presence of agglomerations of particles of varying sizes, suggesting a non-uniform organization of the dye. However, the

observation of the PS-TOA membrane SEM image (Fig 2b) revealed no apparent porosity, suggesting a relatively dense and compact structure. This lack of visible porosity could influence transport performance across the membrane, in particular by limiting passive diffusion, and indicates that the transfer mechanism probably relies on specific interactions between the components of the system.

Comparative analysis of SEM images of PS-TOA (Fig b) and PS-TOA-RBA membrane (Fig c) reveals morphological changes after extraction of the RBA dye, suggesting changes in surface texture and the possible presence of dye residues. These variations are important for assessing the impact of the extraction process on the physical properties of the membrane, particularly with regard to homogeneity and surface roughness.

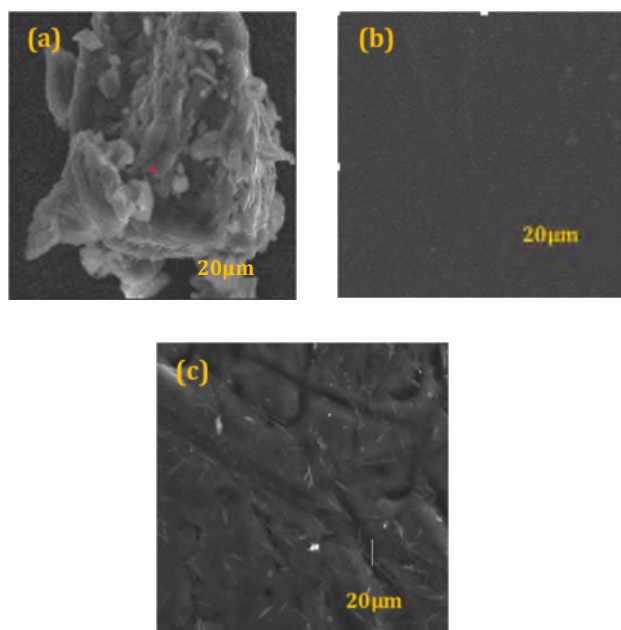


Figure. 2 SEM micrographs of RBA dye and polymer membranes before and after extraction: (a)RBA dye, (b) PS-TOA and (c) PS-TOA-RBA.

The X-ray diffraction data for membranes PS-TOA before and after RBA dye extraction were obtained in 2θ of 5-60° range.

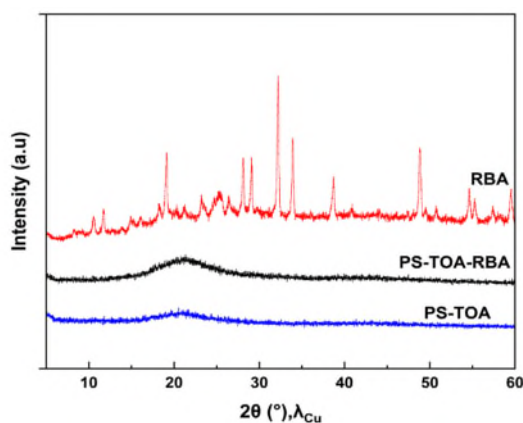


Figure. 3 XRD spectra of RBA dye and polymer membranes before and after the extraction of the dye.

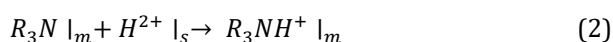
The diffractogram of PS-TOA shows a broad peak at $2\theta = 21^\circ$, characteristic of amorphous materials. This amorphity is typical of polystyrene, the membrane's base material, and indicates the absence of long-range crystalline order. However, PS-TOA-RBA membrane diffractogram shows a slight modification compared to that of pristine PS-TOA, notably a moderate increase in intensity of peak at $2\theta = 21^\circ$. This suggests effective incorporation of RBA into the membrane, although its overall structure remains predominantly amorphous.

In contrast, the spectrum of the pure RBA dye reveals numerous sharp, intense peaks, reflecting a highly crystalline structure. The absence of these peaks in the PS-TOA-RBA diffractogram indicates that the dye, once integrated into the polymer matrix, loses its initial crystalline organization. This observation can be attributed to a molecular dispersion of the dye in the membrane or to a strong interaction with the polymer, disrupting its crystalline stacking.

3.2. Solid- Liquid extraction

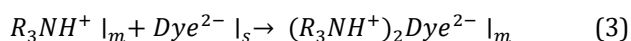
3.2.1. Mechanism of acid dye extraction by PS-TOA

In the solution phase, TOA (R_3N) was protonated using sulfuric acid (H_2SO_4), forming ammonium salt ions (R_3NH^+). Hydrogen ions (H^+) were generated through the dissociation of H_2SO_4 , and subsequently protonated the non-ionic tertiary amine, facilitating the extractant's involvement in the extraction process, than behave as a basic extractant. The protonation reaction is depicted in Eq. (2).



With: m = membrane phase, s= solution phase.

The interaction between the anionic dye ions and the protonated extractant takes place at the interface between the solution and the membrane, resulting in the formation of a complex. Equation (3) provides an eventual and a visual representation of this complexation reaction.



3.2.2. Effect of the salt solution

In water, the membrane PS-TOA was not able to extract RBA. It is well-known that the presence of salts can vary widely in concentration depending on the source and quality of the water

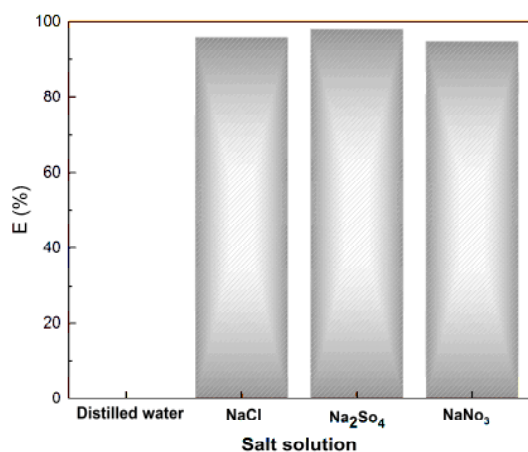


Figure 4. Effect of the salt solution onto the extraction efficiency of the RBA.

To investigate the impact of salt on RBA extraction, extraction solutions containing different salts were prepared and tested. The salts used included sodium chloride, sodium sulfate, and sodium nitrate. The results show that the extraction efficiency was highest with sodium sulfate (97.79%). The varying outcomes obtained with these three salts are presented in Figure 4.

3.2.3. Effect of the extractant concentration

The concentration of the extractant within the membrane was systematically varied across a range spanning from 5 to 30 $\mu\text{mol}/\text{cm}^2$, as illustrated in Figure 5. It was observed that the percentage of extraction exhibited a direct correlation with the TOA concentration up to approximately 15 $\mu\text{mol}/\text{cm}^2$. Beyond this threshold, further increases in carrier concentration did not yield significant improvements in extraction efficiency, indicating a saturation effect in the extraction process. This finding underscores the critical importance of optimizing the carrier concentration to achieve maximum extraction efficiency for RBA. In contrast to the result observed when extracting this dye with Aliquat 336 which requires a higher concentration (20 $\mu\text{mol}/\text{cm}^2$) [20],

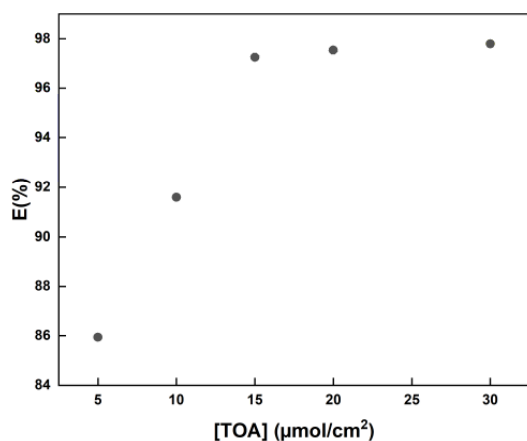


Figure 5. Effect of carrier concentration on extraction efficiency. Conditions: time = 6 hours, stirring speed = 350 rpm, pH = 3 and dye concentration = 100 mg/L.

3.2.4. Effect of the pH in aqueous solution

The pH of the aqueous solution is an important parameter that controls the dye extraction process. To study the effect of pH on the extraction percentage of dye from the aqueous solution, pH was varied between 2 and 9, while keeping the carrier concentration in the membrane matrix and the dye concentration in the aqueous phase constant. Figure 6 presents the relationship between extraction efficiency and pH. The results indicate that extraction with TOA is more efficient and remains consistent in the acidic pH range (from 2 to 5). This underscores the significance of pH control in optimizing the dye extraction process. Under acidic conditions, the N atom of TOA takes protons to form cationic quaternary ammonium groups, which then interact with the negatively charged groups of the dye. Consequently, as the acidity level rises, the quaternary ammonium groups and the extraction rate both increase. Conversely, at lower pH levels, the dye predominantly exists as undissociated molecules, promoting the formation of a complex between TOA and the dye. In contrast, when the aqueous phase has a high pH, the dye primarily remains unbound, resulting in a decrease in the extraction rate [25].

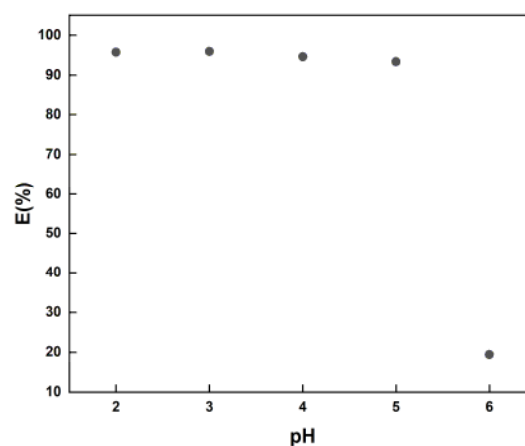


Figure 6. Effect of aqueous solution pH on the extraction efficiency. Conditions: time = 6 hours, stirring speed = 350 rpm, [extractant] = 20 $\mu\text{mol}/\text{cm}^2$ and the concentration = 100 mg/L.

3.2.5. Effect of initial dye concentration

To assess the retention capability of a 4cm² membrane sample, an investigation into the impact of dye concentration was carried out. Dye concentration was systematically varied within the optimal conditions, covering a range from 50 to 300 ppm. Figure 7 illustrates the findings, showing that the extraction of RBA remains consistently efficient within the range of 50 to 200 ppm, with an extraction efficiency of approximately 98%. However, beyond the 200 ppm mark, an observable decrease in extraction efficiency is evident as dye concentration increases. This decline in efficiency is likely attributed to membrane saturation and a reduction in the effective membrane area. Similar behavior has been

observed in previous studies focused on the removal of acidic dye by the matrix CTA-Aliquat336 [20].

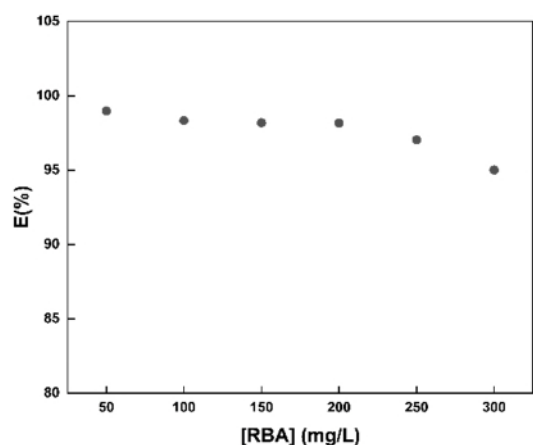


Figure 7. Effect of dye concentration on extraction efficiency. Conditions: time = 6 hours, pH = 3, stirring speed = 350 rpm and [extractant] = 20 $\mu\text{mol}/\text{cm}^2$.

3.2.6. Effect of Stirring Speed

Figure 8 depicts the effect of stirring speed on the RBA extraction by the PS-TOA matrix. The results show that increasing the speed increases extraction efficiency, with the greatest reached at 350 rpm. The decrease in the *ecovery efficiency* is caused by turbulence generated by stirring at speeds greater than 350 rpm. The same result was found in our previous study of dye acid extraction using a CTA-Aliquat336 membrane [20].

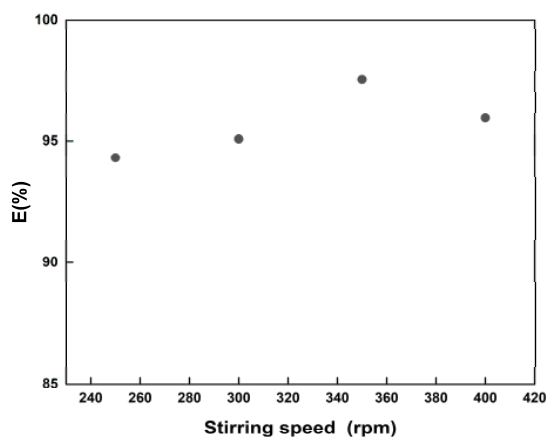


Figure 8. Effect of stirring speed on extraction efficiency. Conditions: time = 6 hours, pH = 3, stirring speed = 350rpm, [Extractant] = 20 $\mu\text{mol}/\text{cm}^2$, and dye concentration= 100 mg/L.

4. Regeneration and reusability of PS-TOA membrane

The saturated membranes must be regenerated so that they can be reused in another treatment cycle. As the extraction of the RBA dye was carried out within the acidic pH range [2–5], a 1 M NaOH solution was used to regenerate the PS-TOA membrane. The regeneration experiments were conducted over a 2-hour stirring period, resulting in a recovery rate of approximately 93% over four regeneration cycles as seen in Figure 9.

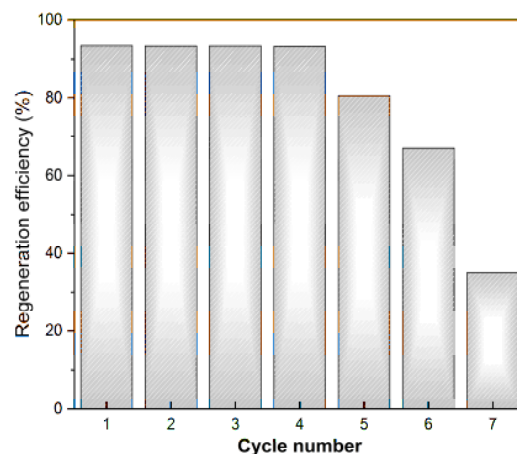


Figure 9. Efficiency removal of VG dye by PS-OA regenerated with NaOH

These finding demonstrate the good reusability and stability of the PS-TOA elaborated membrane, making it suitable for dyes wastewater treatment. However, from the fifth regeneration cycle onwards, efficiency decreases by around 10% compared with the previous cycle, and then continues to decrease gradually until it reaches around 60% by the seventh cycle. This suggests that when sodium hydroxide desorbs the RBA molecules, it can also lead to progressive degradation of the PS-TOA membrane structure.

5. CONCLUSION

The incorporation of trioctylamine (TOA) into polystyrene membranes has demonstrated its exceptional efficacy as an extractant for Red Bordeaux Acid (RBA). Under carefully optimized conditions, which encompass factors such as ionic strength, pH of the aqueous phase, the quantity of extractant present in the PS membrane, initial RBA concentration, and stirring speed, a remarkable 98% of RBA was successfully extracted and recovered from the aqueous phase. Notably, the highest extraction yield was observed within the acidic pH range, specifically between pH 2 and 5. X-ray diffraction confirmed amorphous characteristics in both PS-TOA and PS-TOA-RBA, with a significant peak shift in the latter, signifying successful RBA incorporation. Additionally, SEM images highlighted post-extraction variations in surface texture and porosity, emphasizing the extraction process's impact on membrane properties. The PS-TOA matrix was found to be an excellent extractant for removing RBA from aqueous solutions and can be regenerated and reused up to four times. This study suggested that this matrix could potentially be applied to extract other dyes and pigment.

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