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Study of the ultrafiltration rate of a membrane based on polysulfone modified by an epoxy resin, Octaglycidylether tetra-aniline para of methylenedianiline (OGTAPMDA), on colored waters by the indigo and the red cibacrone.

T. Lakdioui*, N. El-Aouni and A. El Harfi

[(a)]Laboratory of Agroresource, Polymer and Process Engineering (LAPPE), Team of Organic and Polymer Chemistry (TOPC), Faculty of Science, Ibn Tofail University, BP133, 14000, Kenitra, Morocco.

** Corresponding author:*
lakdiouitarik@gmail.com
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Abstract

The textile industry is one of the most chemically intensive industries on earth and it is the main polluter of drinking water. This industry generates huge amounts of complex chemical substances in the unused materials including coloring agents in the form of waste waters during the various stages of textile processing.

In this work, we prepared a membrane having good mechanical properties and improved hydrodynamics. For this task, we modified the polysulfone with synthetic epoxy resin Octaglycidylether tetra-aniline para of methylenedianiline (OGTAPMDA).

Keywords: *Ultrafiltration, polysulfone, Indigo, Red Carbon, Epoxy Resin, OGTAPMDA.*

1. Introduction

The world today is an indicator of socio-economic impacts of revolutionizing various industries. Unfortunately, the development of the industrial sector has fueled some unexpected consequences, resulting in an inevitable compromise between industrial progress and environmental degradation [1, 2]. The textile industry, for example, is one of the largest consuming fields of water, of dyes and of various treatment chemicals which are used during the various textile processing steps [3-6]. Subsequently, significant amounts of wastewater are generated, mainly consisting of dedicated or unused resources, which are not suitable for further use. These effluents are likely to cause environmental problems in case of spillage without treatment. According to the classification proposed by the Environmental Protection Agency (EPA) [7, 8], the waters of padding and/or rinsing of textile finishing issues can be divided into four main categories, namely the dispersible category , the difficult to treat, the high volume and the chemicals added to the formulation of hazardous and toxic dye bath [9, 10]. Therefore, it is necessary to develop technologies that can eliminate these pollutants from the water. Various technologies and processes have been reported and evaluated namely biological treatments [11-14], membrane processes [15-17], the adsorption technique [18-20] and the coagulation / flocculation processes [21-23]. In this work, we treated indigo and red dye cibacrone by ultrafiltration technique using a new membrane made from polysulfone (PSU) amended by octa-functional epoxy resin, Octaglycidylether tetra-aniline para of methylenedianiline (OGTAPMDA, synthesized in our laboratory) [24].

2. Materials and methods

2.1. ***Characteristics of the studied dyes***

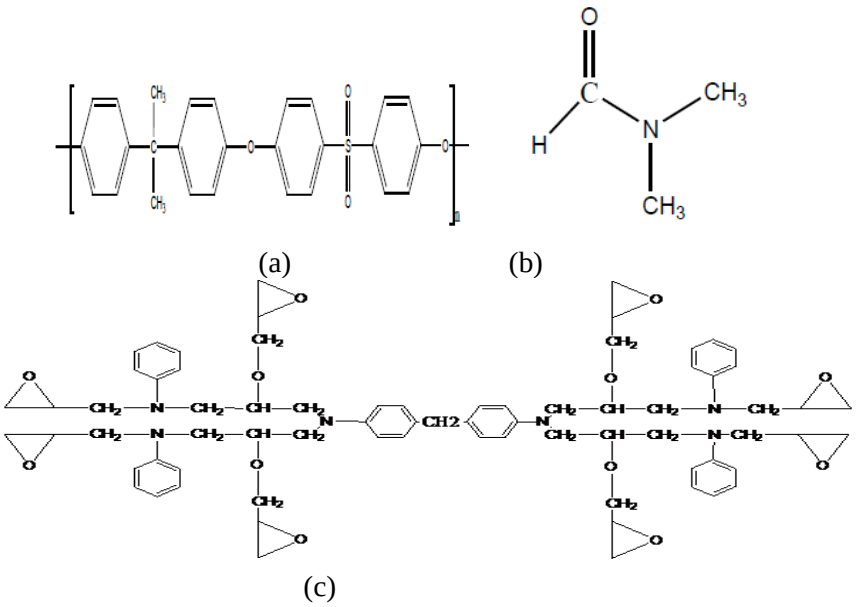
In table 1, are presented the physicochemical characteristics of the exploited dyes (indigo and red cibacrone).

Table 1: Physical and chemical characteristics of the used dyes.

	Indigo	Red cibacrone
Molar mass ($g.mol^{-1}$)	246	541
Solubility in water	insoluble	soluble
Utility	textile	textile
Vapor Pressure	low	low
λmax	660	550

2.2. ***Synthetic membrane***

The synthesized modified membrane was obtained by the inversion method of phase in a weight ratio of copolymer / solvent [(PSU / OGTAPMDA) / DMF] respectively equal to [(0.85 / 0/15) / 90%], whose chemical structures are shown in Figure 1. The prepared mixture was stirred by using a magnetic stirrer at a speed of 760 rev / min for 240 min under a normal temperature and pressure. The obtained collodion was spread on a glass plate using a glass rod. Then, the plate was immediately immersed in a water bath acting as a non-solvent for asymmetric membrane by the phase inversion. The chemical structure of the synthesized membrane was confirmed successively by Nuclear Magnetic Resonance (1H NMR) and by the FTIR spectroscopy [25,26].



(a) (b) (c)

Figure 1: Chemical structures of polysulfone (PSU) (a); N, N-dimethylformamide (DMF) (b) and epoxy resin OGTAPMDA (c).

*2.3. **Methods of analysis***

*2.3.1. **Fourier transform infrared Analysis (FTIR)***

The used IR spectrometer is a Fourier Transform Spectrometer (FTIR) BRUKER. The spectra were carried out in transmission on KBr pellets. The light beam passes through the sample to a thickness of about 2 mm. The analysis is performed between 4000 cm^{-1} and 600 cm^{-1}.

2.3.2. **Nuclear magnetic resonance (NMR)**

The ^{1}H-NMR analysis has been obtained by means of Bruker AVANCE 300 device by dissolving the product in DMSO. The chemical shifts are expressed in ppm. The letter s, d, t, q and m respectively mean singlet, doublet, triplet, quadruplet and multiplet.

*2.4. **Thickness measurement of the membrane by the micrometer***

The thickness of the membrane (E) influences the flow and the selectivity, and it can be changed by changing the characteristics of the rule used in their preparation. This size is determined by two steps L1 and L2 such as E = L2-L1, where L1 = the thickness of substrate = 2240 µm and L2 = the substrate thickness plus the membrane in µm.

*2.5. **The scanning electron microscopy (SEM)***

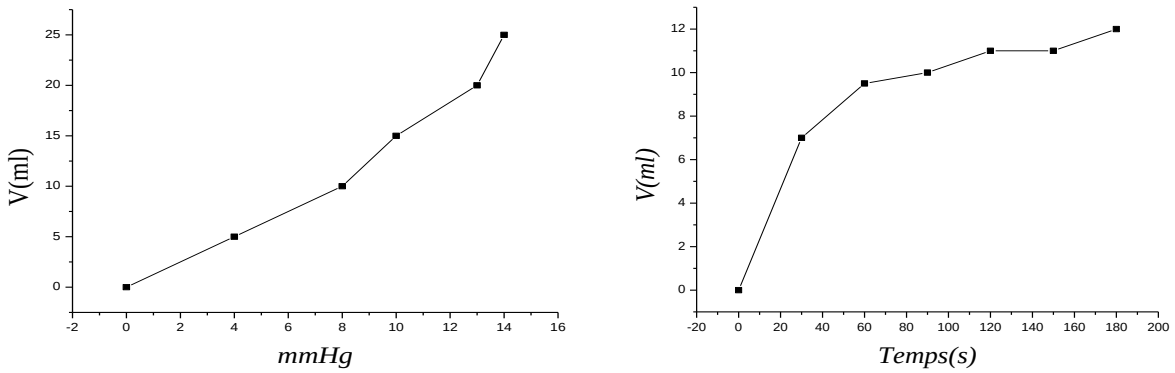
The images of ultrafiltration membranes were made with a scanning electron microscope Zeiss Supra 55 FEG VP of low voltage (2 or 3 keV). The observations of the sample on the section were made after the immersion in liquid nitrogen. The samples were all plated with a thin gold layer.

3. Results and Discussions

3.1. Permeability measurement

The permeability of the synthesized membrane depends on several parameters among which we cite the membrane structure, the pore size of the membrane, pressure, time and the type of pollutant to be removed.

Figures 2 (a) and 2 (b) respectively show the influence of the pressure and of the time on the permeability of the membrane. In figure 2 (a), we noticed that the influence of pressure on the permeability of the synthetic membrane respects the Darcy law and the starting volume increases according to the pressure. In figure 2 (b), the new membrane flux increases according to the increasing time.



(a) (b)

Figure 2: The influence of the pressure (a) and the time (b) on the membrane permeability.

*3.2. **Membrane thickness measurement***

By using the relation $E = L_2 - L_1$, we calculated and recorded in Table 2 the thickness of the membrane with the micrometer. The obtained thickness of the asymmetric membrane using the selected formulation (copolymer / solvent), enabled us for its use in the ultrafiltration process because the structure of the cross-sectional in SEM confirms the asymmetric membrane composition.

Table 2: synthesized membrane thickness

	L_1	L_2	$E = L_2 - L_1$
Membrane PSU/ OGTAPMDA/DMF	2240	2489.64	249.64

*3.3. **Structural characterization of the membrane***

*3.3.1. **Analysis by Fourier Transform Infrared (FTIR)***

The Infrared spectrum of the Fourier transform (FTIR) allowed us to prove the presence of functional groups existing in the chemical structure of the membrane. Figure 3 shows the bands of the copolymer structure (PSU / OGTAPMDA) in the membrane, obtained from samples prepared in dimethylsulfoxyde (DMSO) as a solvant.

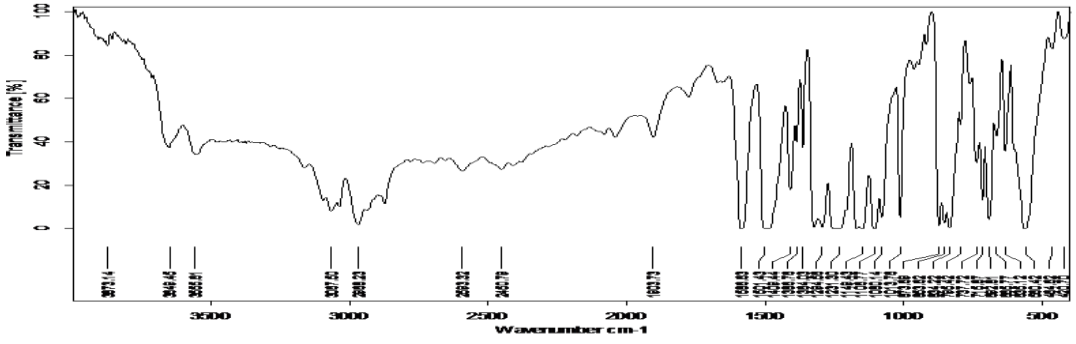


Figure 3: the specter of FTIR of the membrane.

3094-3069 υC-H ; 1579 and 1485 υC=C aromatic ; 1324 and 1299 υO=S=O ; 1245 υC-O-C ; 1162 υO=S=O ; 1152 υC-O-C saturated aliphatic; 701 et 837 substituted benzene; 628 υC-H ; 561 υO=S=O.

*3.3.2. **Analysis by nuclear magnetic resonance (NMR ^{1}H)***

The figure 4 shows the peaks of the NMR spectrum of 1H polysulfone based on the membrane modified by the OGTAPMDA resin. In this study, we used the dimethylsufolxyde (DMSO) to dissolve the analyzed samples.

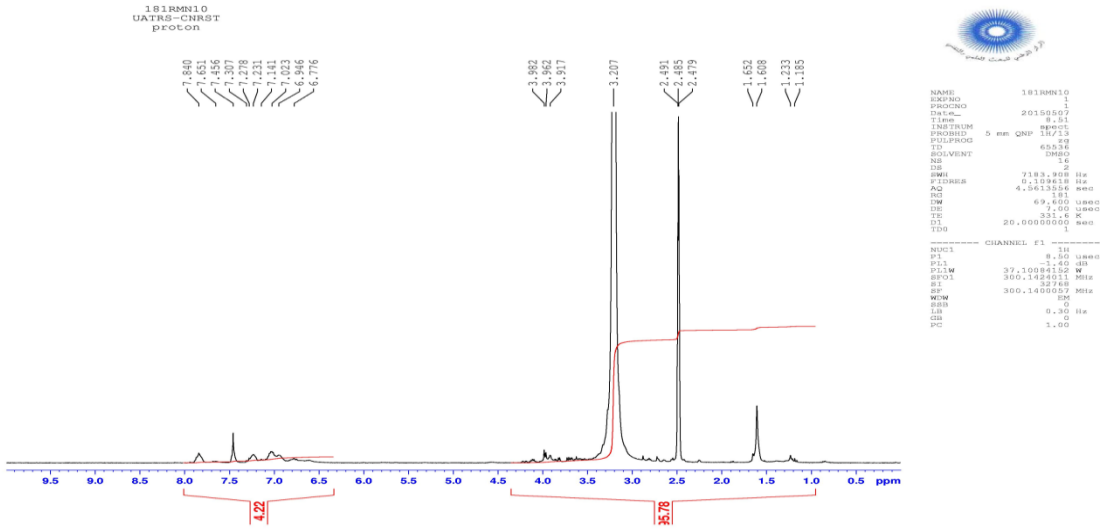
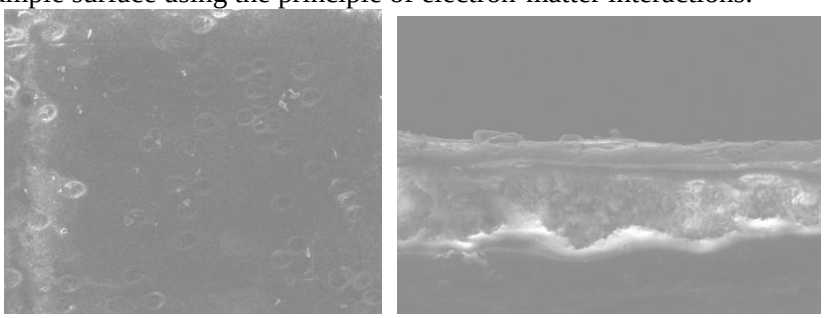


Figure 4: ^{1}H NMR spectrum of the membrane.

NMR-^{1}H : 1.7 (s, 6H, CH3), 6.84 - 7.08 (s, 4H, aromatic binds with the oxygen), 7.09 - 7.86 (s, 4H, aromatic binds with carbon), 7.8 - 7.9 (s, 4H, aromatic binds with sulfur).

*3.4. **Scanning electron microscopy:***

The scanning electron microscopy (SEM) is a technique of electron microscope which is capable of producing high resolution images of a sample surface using the principle of electron-matter interactions.



(a) (b)

Figure 5: photograph of the membrane's SEM

Figure 5 (a) shows the facial structure of the asymmetric membrane obtained by the copolymer mixture [PSU / OGTAPMDA] and Figure 5 (b) reflects the image of the cross section of the membrane. Indeed, we distinguish two stages through this image: the active phase responsible for the permeability of the membrane and the phase (b) supporting said membrane. This confirmation illustrates the structure of an asymmetric membrane whose hydrodynamic properties have clearly proven.

3.5. ***Indigo discoloration rate***

In Figure6, we have represented the rate of discoloration of Indigo and Red Cibacrone by the ultrafiltration method using the membrane PSU / OGTAPMDA.

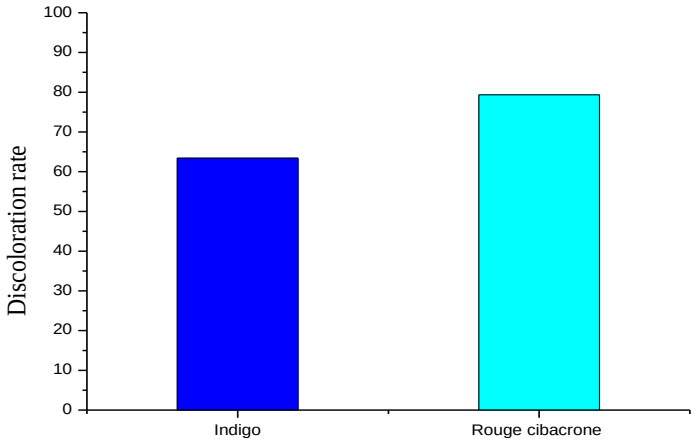


Figure 6: Rate of discoloration of indigo and red cibacrone.

From figure 6, we observed that the fading rate obtained by the ultrafiltration process is 79.34% and 64, 79% respectively for the red cibacrone and indigo.

4. Conclusion

The objective of this work was the study of the influence of synthetic epoxy resin,Octaglycidylether tetra-aniline para of methylenedianiline (OGTAPMDA) on the permeability on the one hand, and on the selectivity and mechanical property of the synthetic membrane on the other hand. In terms of the hydrodynamic properties concerning the permeability, this asymmetric membrane allowed us to have a look of the volume/pressure group that respects the Darcy law. As for the selectivity, we have obtained a retention rate vis-à-vis the red cibacrone and indigo respectively equal to 79,34% and 64, 79%. The results obtained by the SEM have confirmed the obtaining of an asymmetric membrane for its configuration (active and support).

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