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# Purification of the textile finishing effluents by the ultrafiltration technique

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#### Abstract

In the present paper, we synthesized, at first, an new permeable organic membrane based on a mixture of different percentages of polysulfone (PSU), 4,4-sulfoxylphenol S, expanded polystyrene in a solvent that is N, N-dimethylformamide (DMF), and at the end, we treated the liquid effluents from the textile finishing industry by this membrane. The latter was characterized at the first step by the hydrodynamic conditions of the ultrafiltration technique and in a second step by microscopic tools. The achieved results concerning the microscopic characterization of the synthesized membrane show that the chemical structure of the membrane is composed of three previous components (PSU/BS/PSe), but those related to wastewater treatment show that the rate of discoloration measured for the wastewater charged with indigo is the order of 87.24% and measured for the black sulphur is the order of 64.04%.

Keywords: Permeable Organic Membrane, Ultrafiltration, Hydrodynamic and Microscopic Characterization.

# 1. Introduction

In recent years, the technology of organic membranes has experienced significant progress in the fabrication of microfiltration; ultrafiltration, reverse osmosis membrane and others (Venugopal et al., 2012, p.37) have been used in various industrial processes such as the purification of industrial wastewater, desalination of sea water (Allali Hassani et al., 1990, p.699), (Plattes et al., 2007, p. 613), the recovery of acid and dehydration of organic solvents, etc. The exploitation of these membranes varies from one hand, following the industrial process monitoring and the nature of the effluent discharged by this method (Shimekit et al., 2009, p.115) and on the other hand, according to the mechanical and chemical properties of membranes used (Benavente et al., 2000, p.43), (Buzatu et al., 2012, p. 421), and (Solaymani et al., 2012, p.217). The objective of the present work in the first step is to synthesize a new organic membrane based on a mixture of the Polysulfone (PSU)/Bisphenol S (BS)/Expanded Polystyrene (PSe), and in the second step is to apply the membrane obtained in the technique of Ultrafiltration (UF) to treat effluents loaded with vat dyes (Indigo and Black sulphur) rejected in an operating processes of dyeing and finishing fiber cotton, which are intended for the manufacture of Denim factories and also to decrease relatively this type of waste.

# 2. Materials and methods

The obtained membrane was synthesized by mixing together in mass polysulfone 13.5%, polystyrene 1%, 4, 4-sulfoxyldiphenol 0.5%, N; N-dimethylformamide (DMF) 85%, including their chemical structures (Fig. 1 and Fig. 2). The prepared mixture was allowed to stir (using a magnetic stirrer) at a speed of 580 tr / min for 45 min in a normal temperature and pressure. The collodion

obtained was spread on a glass plate using a glass rod. This plate was immediately immersed in a water bath to obtain an asymmetric membrane type phase inversion (Mulder *et al.*, 1996) (Tsai *et al.*, 2006, p. 390).

After the optimization of the collodion and obtaining a semipermeable membrane, the latter was characterized respectively by the microscopic properties (Fourier Transform Infrared (FTIR), Nuclear Magnetic Resonance (NMR)) and morphological: Electron Microscopy (SEM), and then according to the hydrodynamic properties (permeability and selectivity) by the ultrafiltration technique using distilled water as a permeate water at first and then as a rejected model.

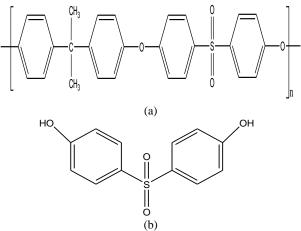
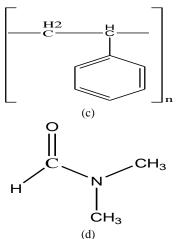
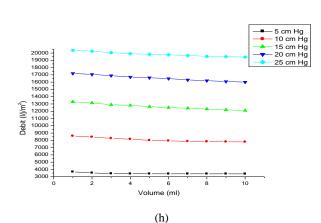


Fig. 1: Chemical structures of polysulfone (PSU) (a) and 4.4- sulfoxyldiphénol S (b).

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**Fig. 2:** Chemical structures of expanded polystyrene (c) and N, N-dimethylformamide (d).

The membrane characterized hydroynamically was applied to purify the model rejection consisting liquid effluents collected from the dyeing industry and finishing of cotton fibers (Company ICOMA Mohammedia-Morocco). These effluents are original vat dye (indigo and black sulphur) whose chemical structures are shown in Fig. 3:

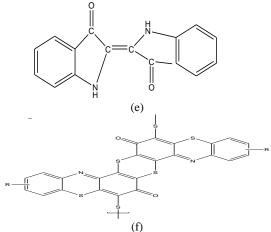


Fig. 3: Chemical structures of vat dyes (Indigo (e) and Black sulphur (f).

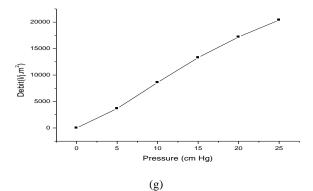
The wavelength maximum ( $\lambda$ max) of these two types of dyes was determined by spectrometer UV/Visible (JP.SELECTA.sa).

## 3. Results and discussion

In this work, the hydrodynamic and morphological characterization of the synthesized membrane is given as follows:

#### 3.1. Hydrodynamic characterization

Flow measurements in function of the pressure and the volume of distilled water are shown in Figure 4 (g, h):



**Fig. 4:** Evolution of the flow of distilled water depending on the pressure (g) and the volume of filtrate at different pressures (h).

From this figure, it is found that the filtrate flow rate gradually increases when the pressure itself is increased; this increase in speed is sufficiently proportional with the pressure operated. This partially confirms the DARCY law (Crozes *et al.*, 1997) (Fernandez *et al.*, 2011, p. 271), (Pacella *et al.*, 2011, p. 238), and also it slightly decreases depending on the volume of the filtrate followed by a steady state. This decrease may be due to compaction of the membrane under pressure that is applied in this case 20 CmHg.

#### 3.2. Measurement of selectivity

The selectivity of the membrane studied was measured as the rate of retention (%) as a function of different molecular weight dextran (Dalton) is shown schematically in Fig. 5 as follows:

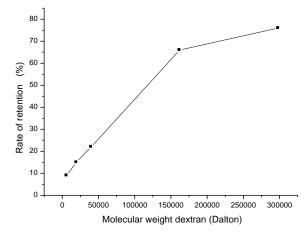


Fig. 5: Evolution of the retention of the membrane as a function of dextran molecular mass

From this figure, we notice that the retention rate of the membrane increases in a linear way when the molar masses of dextran vary to 160,000 Daltons, then it tends to stabilize after this value. This explains that the membrane is semi permeable and selective.

#### 3.3. Microscopic characterization of the membrane

The infrared spectrum of the synthesized membrane (FTIR) is shown in Fig. 6 below:

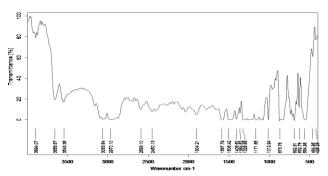


Fig. 6: Infrared spectrum of the membrane based on PSU/BS/PSe.

From this spectrum and in comparison of infrared spectra of each commodity (PSU, BS and PSe), it shows us that this figure shows all bands of the absorption belonging to those core matrices (PSU, BS and PSe), and another band of higher intensity (1689.79 cm<sup>-1</sup>) corresponding to the vibration of the amide group which is assigned to the solvent DMF.

The characterization by nuclear magnetic resonance (RMN) spectroscopy synthesized membrane is given by the spectra of Fig. 7:

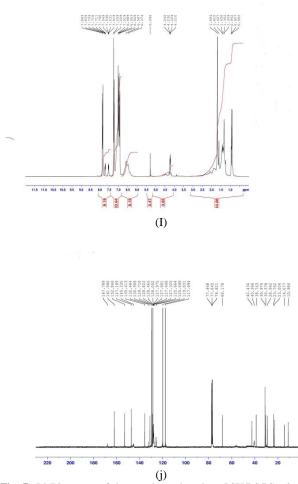
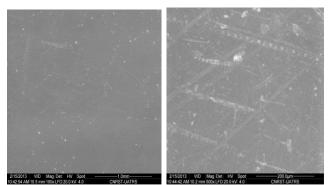


Fig. 7: RMN spectra of the membrane based on PSU/BS/PSe (i:  ${}^{1}$ H, j:  ${}^{13}$ C).

Fig. 7 corresponds to the proton RMN (i) and carbon 13 (j) confirms the structure of basic matrices separately. This shows that there is no formation of new connection due to the reaction of functional sites.

#### 3.4. Morphological characterization

The morphological characterization of the membrane was obtained by using the SEM as demonstrated in the Figures 7, 8 and 9 following the three faces:



**Fig. 7:** Photographs SEM at different magnifications of the upper face of the membrane.

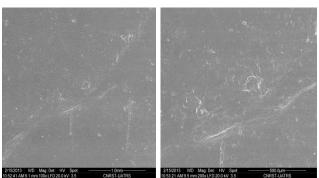


Fig. 8: Photographs SEM at different magnifications of the lower face of the membrane.

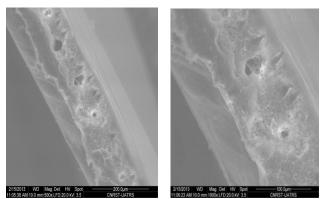


Fig. 9: Photographs SEM at different magnifications of the profile of the membrane.

From these photographs of the membrane, there is a part on the selective film that is the upper surface of a good density of fine pore diameter (Fig. 7), and secondly, the underside (Fig. 8) constituting the support of the membrane approximately has the same density with much higher porosity than those observed in the upper face diameters. This is observed by photographs taken through the section of the membrane and/or profile (Fig. 9).

# 3.5. Determination of $\lambda$ max, optical densities (OD) and rate discoloration (%) studied dyes

The measurement of the maximum wavelength and the optical densities before the treatment (O.  $D_i$ ) and after the treatment (O. $D_f$ ) of each of the effluents (black sulphur and indigo dyes) is shown in Figure 10:

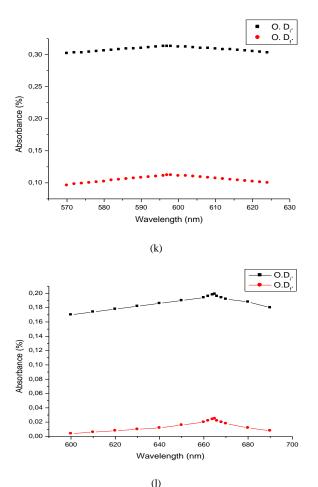


Fig. 10: Curves of absorbance of the effluent collected dyes compounds (black sulphur (k), indigo (l))

The results of the characterization of the effluent collected and the rate of discoloration (RD) are recorded in Table 1 below:

Table 1: Values of parameters characterizing studied dyes					
dyes	λmax (nm)	ph	o. d <sub>i</sub>	o. d <sub>i</sub>	rd (%)
indigo	665	8.20	0,199	0,025	87,24
black sulphur	597	7.09	0,313	0,112	64,04
oluon suipilui	271	/.0/	0,010	0,112	01,01

According to the above Table, we see that the rate of fading is in the order of 87.24% for the compounds of the effluents to indigo  $\lambda$ max equal to 665 nm and pH of 8.20, and of the order of 64.04% for the effluent loaded with black sulphur dye  $\lambda$ max equal to 597 nm and pH of 7.09.

# 4. Conclusion

Based on all theoretical and experimental studies of the proposed subject of this present article, we can conclude that the synthesized membrane is the asymmetric structure to the morphology of the different sections observed by SEM. It has mechanical properties more efficient than supported liquid membranes (MLS's) (Shuxiang *et al.*, 2006, p. 277), polymer inclusion membranes (PIM's), and grafted polymer membranes (GPM's) (Benjjar, 2013) (in view of the burst resistance (pressure up to 20 Cm Hg). It also has good hydrodynamic properties (permeability and permselectivity) obtained using the flow (Fig. 4) and the retention rate (Fig. 5). The rate of fading respectively reaches the values of 87.24%, 64.04% for indigo and black sulphur.

## Acknowledgement

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