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Cellulosic Based Membranes from Synthesis to Typical Pollutants Removal

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Abstract

Cellulose acetate CA, CA / PEG, CA / SiO_2, and CA / PEG / SiO_2 based membranes were synthesized for the removal of humic acid and bovine serum albumin BSA by ultrafiltration process. The different membranes were elaborated by phase inversion using DMF as solvent and water as non-solvent. The measurements of the contact angle of these membranes show that their hydrophilic character increases by the addition of SiO_2 and polyethylene glycol PEG as hydrophilic polymer (additive). Thus, the CA/PEG / SiO_2 membrane is the most hydrophilic one with a contact angle of 42°. The characterization of the synthesized membranes using FTIR- ATR allows us to highlight new interactions due to the insertion of silica particles in the membrane as well as the possibility of hydrogen bond formation between PEG and cellulose acetate. The membrane CA / PEG / SiO_2 has the highest porosity value as well as the highest equilibrium water content. Thus, the addition of additives such as SiO_2 and the hydrophilic polyethylene glycol PEG polymer improve the hydrophilicity of the initial CA membrane. The maximum retention of humic acid by CA/SiO_2 membrane is off 92.59% for a pH of 4 and 0.5 g / L salinity. The gradual decrease in pure water fluxes of all membranes over time due to pore compaction and steady state fluxes was achieved approximately after 90 minutes of ultrafiltration process.

Keywords: SiO_2, Hybrid Membrane, Pure Water Flux, Porosity

Introduction

Drinking water is a vital source. However, if its quality is not in accordance with the OMS regulations, this water becomes the major cause of diseases for all living beings. In this case, having a quality of raw water sources in the production process of clean and healthy drinking water becomes a necessity. Although about 70% of the earth's surface is covered with water, only about 1% is available as drinking water [1]. However, due to the considerable expansion of the population, industrialization, and urbanization, water will be one of the most valuable resources in the world. The problem of its shortage is not only a problem of realization of the appropriate techniques, but also a social and educational problem [2]. Drinking water quality standards are becoming increasingly due to the presence of pollutants from industry, making them the major challenge of the 21st century [3] leading to the development of new processes that can be conventional, membrane-bound, biological, or based on ion exchange (resin) or selective adsorption [4]. Humic substances are among the main constituents of wastewater, they are organic substances of high molecular weight containing multiple functional groups: alcohol, phenol, quinone, and carboxyl. These compounds generate trihalomethanes (THM), potentially carcinogenic, as well as chlorophenols responsible for tastes and unpleasant odors. It is therefore necessary to reduce or eliminate these substances [5]. Membrane processes have been widely applied in various fields such as water treatment, food processing, pharmaceutical industry, and environmental protection [6]. Membrane technology, which refers to several processes using membranes to separate chemicals, has been recognized as the key technique for separating contaminants from polluted sources. The implementation needs special attention and expertise to achieve highly efficient, reliable, and economical industrial processes. Nanofiltration and reverse osmosis are the most widely used membrane processes for the treatment of wastewater. However, the membranes corresponding to these techniques have a very low permeability and therefore require a high transmembrane pressure, which makes them expensive processes. Ultrafiltration can operate at relatively low pressures and temperatures resulting in an excellent release rate of metals and multivalent organic elements [7]. In the last years new polymeric materials such as cellulose acetate, polyamide, and polyimide [7] are used for water treatment applications. Cellulose acetate (CA) among the hydrophilic polymers is considered as the most important organic ester of cellulose due to its most abundance and broad applicability for the synthesis of different products because of having tough, biocompatible, hydrophilicity characteristics, and moderately less expensive [8-10].

The drawback of cellulose acetate membranes is that they are susceptible to thermal and chemical stabilities depending on the environments and conditions of application [11]. CA because of its good mechanical, chemical, and thermal stability is widely used in membrane applications. Fouling is the resulting decrease in the membrane flux, either temporarily or permanently [12]. It is accepted that fouling resistance occurs when hydrophilicity is increased [13], because a more hydrophilic surface absorbs water molecules to make a layer between the membrane surface and organic molecules.

In this study, we will describe the used method for the elaboration of CA, CA / SiO_2 and CA / SiO_2 / PEG membranes by different techniques which are carried out by using each for a suitable manufacturing method.

The last part will be dedicated to the exposure of the experimental results of all the physicochemical characterizations of the elaborated samples and to valorize our work, we will make the application in ultrafiltration by eliminating the humic acid and BSA by ultrafiltration using a composite membrane CA / SiO2 / PEG.

Experimental

Material

Cellulose acetate polymer (acetyl: 29-45% content, MW=50,000 g/mol); humic acid (60-70%, dry basis) and Bovin Serum Albumin were purchased from Sigma Aldrich, France. Dimethylformamide (with 99.8% purity HPLC grade) was purchased from sigma Aldrich. France. Polyethylene glycol (MW =1000) was obtained from Merck Specialties Private Limited, Germany; SiO_2 particles with purity of 99.5% was obtained from Sigma-Aldrich Co., France. Deionized water (DI) was used throughout this experiment which was purified using Millipore system.

Membrane preparation

Herein, the CA based composite membranes are synthesized using phase inversion technique. The compositions of the polymer solutions and all the prepared membranes were detailed in Table 1. The 18wt% of pure CA membrane without PEG and SiO_2 as additives. Then the solution is stirred for 24 hours at room temperature until a homogeneous solution is obtained and the viscous solution is left to degas for 24 hours. After obtaining a homogeneous solution, it was then casted on a glass plate using a doctor's blade. The thickness of the casting membranes maintained was 0.22 ± 0.02 mm. And the temperature was maintained at 25ºC during membrane casting.

Table 1: Composition of the prepared membranes

Composition of solutions	wt % of polymers	Preparation description
S1: V_{DMF}= 20mL m_{AC}= 4.14g	18%	The cellulose acetate is poured into the DMF. Then the solution is stirred for 24 hours at room temperature until a homogeneous solution is obtained and the viscous solution is left to degas for 24 hours.
S2: V_{DMF}= 20mL m_{PEG}= 0.414g m_{AC}= 3.726g	18%	The cellulose acetate is poured into the DMF. Then the solution is stirred for 24 hours at room temperature until a homogeneous solution is obtained and the viscous solution is left to degas for 24 hours.
S3: V_{DMF}= 20mL m_{AC}= 3.933g m_{SiO2}: 0.207g	18%	SiO2 (additive) is mixed in DMF for 4 hours for complete dispersion then the solution is placed in an ultrasonic bath for 15 minutes, after which the cellulose acetate is added for 3 times separated by 30 min. Stirring is done on a plate at room temperature for 24 hours to obtain a homogeneous solution and then the solution is left for 24 hours to degas.
S4: V_{DMF}= 20mL m_{AC}= 3.519g m_{PEG}= 0.414g m_{SiO2}= 0.207g	18%	SiO2 (additive) is mixed in DMF for 4 hours for complete dispersion then the solution is placed in an ultrasonic bath for 15 minutes, after which the PEG 1000 (additive) is added to the solution and is stirred until the complete solubilization of PEG and then added cellulose acetate 3 times separated by 30min. Stirring is done on a plate at room temperature for 24 hours to obtain a homogeneous solution and then the solution is left for 24 hours to degass.

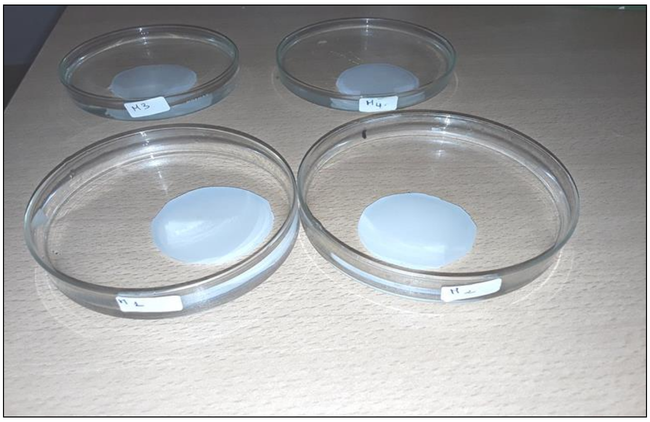


Fig 1: Photos of the prepared composite membranes

Experimental setup

An Amicon 8050 ultrafiltration cell will be used, with filtration that is carried out perpendicularly to the surface of the membrane otherwise. The applied ultrafiltration system contains the following elements:

- Filtration cell with a magnetic stirrer that contains a membrane defined with its nature and cut-off.
- A reservoir.
- A source of pressure (example: Nitrogen bottle).

In this work, an ultrafiltration cell equipped with a magnetized bar was used to provide gentle agitation that prevents the formation of a polarization layer on the surface of the membrane. The efficient membrane area for UF was 19.5 cm^2. The feed solution filled in the cell was stirred at a rate of 300 rpm using a magnetic stirrer. The entire test was carried out at 25 ± 2°C.

Characterization of CA/PEG/SiO$_2$ composite membranes

FTIR

FTIR-ATR spectra of the elaborated membranes were studied by using VERTEX 80v equipment.

Contact angle and water uptake

The contact angle characterization (CA) for the membranes was executed via optical tensiometer. The One Attention software was used to accomplish the CA measurements. When the drop falls down on the membrane surface, digital images were taken using a Digital Video Camera [14].The images are given with zoom factor of 6. Using those images, the tensiometer measured the angle between the water drop and the horizontal membrane surface. The equilibrium water content EWC of the membrane samples were determined for the assessment of bulk hydrophilicity of the membranes, by weighing the 3 × 3 cm dry and wet samples of each membrane and substituting their values in the equation (1) [15]:

$$EWC = \frac{Ww-Wd}{Ww} \times 100 \qquad (1)$$

Porosity and hydraulic resistance

The fabricated membranes were cut into 5 cm diameter circles are completely.

Washed with DI water before usage. Once the membrane fitted in the UF kit, every 1 h, the pure water flux (PWF) was measured at a transmembrane pressure (TMP) of 3 bar, then the permeate flux is measured as a function of time, more precisely at each chosen pressure the time necessary to fill 5 ml of permeate is determined.

The permeate flux obtained for each membrane designated as Jw and calculated using equation (2) [16].

$$J_W = \frac{Q}{A.\Delta t} \qquad (2)$$

The hydraulic resistance (Rm) of the fabricated membranes were calculated by plotting the applied TMP against the PWF for each membrane [17]. The percentage porosity determination of all the fabricated membranes was done by using the values of W_{dry} (dry membrane weight), W_{wet} (wet membrane weight), A_m (active membrane area), d$_w$(water density, kg m^{-3}) and L$_m$ (membrane thickness) in the equation (4) [18].

$$J_W = \frac{\Delta p}{R_m} \qquad (3)$$

$$porosity, \varepsilon = \frac{W_{wet}-W_{dry}}{d_W L_W A_m} \times 100 \qquad (4)$$

Humic acid, HA and Bovin Serum Albumin, BSA rejection

In the object to determine the percentage solute rejection of membranes, theconcentration of BSA permeates from the UF kit was measured using a double beam UVVisiblespectrophotometer (Perkin Elmer) at a wavelength of 280 nm. The experiment was repeated for the HA solution at a wavelength of 254 nm. The obtained permeate concentration (Cp) and the known feed concentration (C_f) were used in equation (5) to determine the BSA or HA rejection (%) of fabricated membranes [19].

$$\% R = \left(1 - \frac{C_P}{C_f}\right) \times 100 \qquad (5)$$

Results and discussion

FTIR of pure CA and CA/PEG/SiO$_2$ composite membranes

Spectroscopic methods play a crucial role in the characterization of polymers. The use of methods such as infrared spectroscopy is essential for studying the molecular structure of membranes. The ATR-mode IRTF spectra of the CA and CA / SiO$_2$ membrane are given in Fig 2. In the case of the cellulose acetate membrane, a peak at 3469 cm^{-1} represents an elongation vibration of the O-H bond, the peak at 2877 cm^{-1} shows an elongation vibration of the C-H bond. the one at 1739 cm^{-1} represents an intense peak characteristic of the C = O bond of the carbonyls, the peak at 1433 cm^{-1} indicates the folding of C-H followed by peaks at 1369 cm^{-1} and 1224 cm^{-1} which described the modes. Tilting and crushing of the C-H bond. The strongest peak at 1035 cm^{-1} is specific for the C-O-C bond whereas the peak at 1112 cm^{-1} and 904 cm^{-1} illustrates the presence of the saccharides. Similar results have already been reported [20, 21]. The emergence of a new peak at about 950 cm-1 in the CA / SiO2 membrane spectra could be attributed to the Si-O elongation vibration. The peak broadening at about 3200-3650 cm^{-1} is due to the establishment of a hydrogen bond in the Si-OH bond [22]. The widening and the emergence of a new peak in the CA / SiO2 membrane spectrum confirm the impregnation of the silica particles in the membrane.

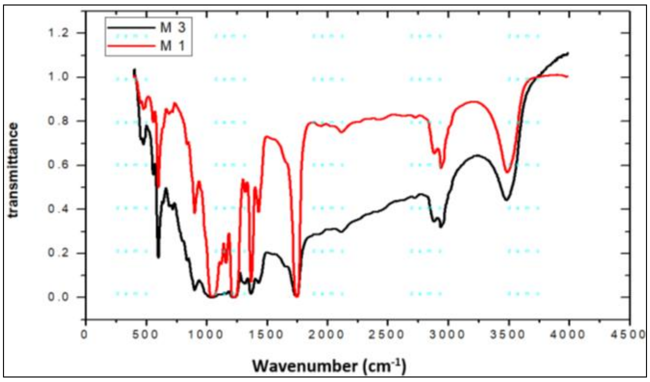


Fig 2: FTIR spectra of CA and CA/SiO$_2$ membranes

The FTIR/ATR spectra of the CA / PEG membrane shown in Fig 3 shows a broad band around 3650-3450 cm^{-1}

represents an OH elongation vibration, a band at 3000-2850 cm^{-1}corresponds to an elongation vibration of the CH bond, a strong peak at 1732 cm^{-1} relating to the vibration of the C = O bond of the carbonyls, 1369 cm^{-1} indicates the flexion of CH and 1222 cm^{-1} for the C=O group [23]. The longest peak at 1035 cm^{-1} indicates the presence of the C-O-C bond [24].

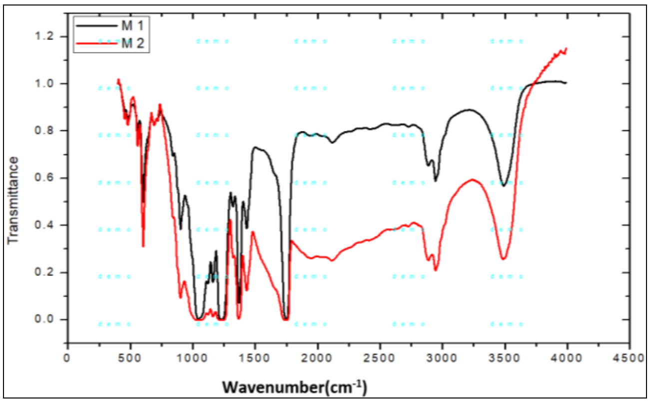
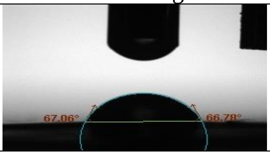
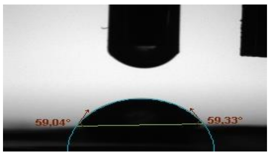
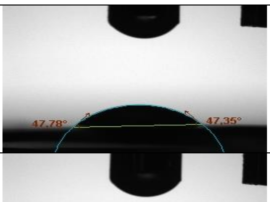
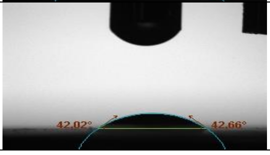


Fig 3: FTIR spectra of CA and CA/PEG membranes

Contact angle of pure and CA composite membranes

Droplets of ultrapure water were placed on the surface of each synthesized membrane, and then the contact angle between the water and the membrane was measured.

Table 2: Contact angle of elaborated membranes

Membrane	Composition	Contact angle
M1	**V $_{DMF}$= 20mL** **m$_{AC}$= 4.14g**	
M2	**V$_{DMF}$= 20mL** **m $_{PEG}$= 0.414g** **m $_{AC}$= 3.726g**	
M3	**V$_{DMF}$= 20mL** **m$_{AC}$= 3.933g** **m$_{SiO2}$: 0.207g**	
M4	**V$_{DMF}$= 20mL** **m$_{AC}$= 3.519g** **m$_{PEG}$= 0.414g** **m$_{SiO2}$= 0.207g**	

The analysis of the preceding results shows that the surfaces of the four membranes had values lower than 90 ° from where their hydrophilic character. However, the highest contact angle is that of the membrane of cellulose acetate (M1) which corresponds to the value 67 °. From these results the incorporation of PEG (1000) and SiO_2 significantly reduced the values of the contact angle having respectively the values 59 ° and 47 ° for M2 (cellulose acetate / PEG) and M3 (cellulose acetate / SiO_2). The M4 membrane having PEG and SiO_2 as additives has the minimum value (42 °) from which it corresponds to the most hydrophilic membrane. This confirms that the addition of SiO_2 and PEG has the effect of reducing the surface tension of the M1 membrane [25].

Porosity and equilibrium water content

We find that the M4 membrane (AC / PEG / SiO_2) has the highest porosity as well as the highest equilibrium water content. Thus, the addition of additives such as SiO_2 and the hydrophilic polyethylene glycol PEG polymer have the effect of improving the hydrophilicity of the initial membrane M1. Indeed, the segments of PEG in the base polymer during the immersion precipitation process can preferentially diffuse on the surface of the membrane, resulting in an improvement of the wettability on the surface of the membrane. Therefore, the contact angle is closely related to the surface energy [26].

Table 3: Porosity and equilibrium water content of studied CA composite membranes

Membrane	CA	CA/PEG	CA/SiO_2	CA/PEG/SiO_2
Thickness (μm)	150	250	200	150
Weight (g)	0.1719	0.3494	0.301	0.2297
Porosity (%)	72.1	87.9	94.6	96.33
EWC (%)	78.8	80.5	81.57	83.5

PWF and hydraulic resistance of pure and CA composite membranes

By analyzing the PWF curves of composite membranes, with increasing Transmembrane pressure, the water flux of CA and CA/PEG keep increases while at the same circumstances the PWF of CA/PEG/SiO_2 attempt the saturation (Fig 4). This is an evident that, the fluidic channels in the CA and CA/PEG not at all shrunken up to an applied pressure of 3.5 bars, which clearly confirms that SiO_2 particles have greater interaction with each other than that of CA/PEG/SiO_2. Evaluation of the hydraulic resistance of all the fabricated membranes reveals that, neat CA membrane shows the highest value, whereas the addition of $SiO_2$and PEG enhances the porosity and hydrophilicity by the development of well-defined finger like structures, makes the CA membrane less sustainable to hydraulic pressure.

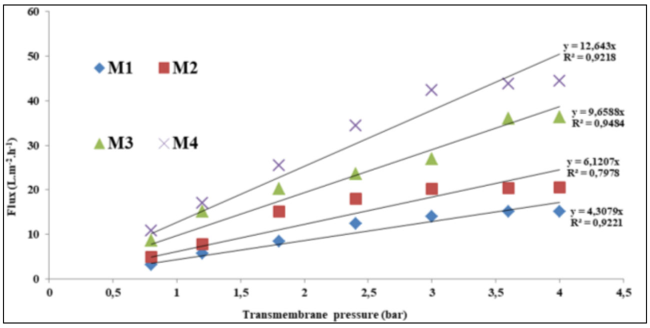


Fig 4: Pure water flux versus transmembrane pressure for the CA composite membranes

When comparing the R_m of CA/SiO_2 and CA/PEG/SiO_2 composite membranes even though there is not much Rm difference (Table 4), the former shows slightly better resistance than later due to the high mechanical strength of CA matrix.

Table 4: Permeability and hydraulic resistance of CA composite membranes

Membrane	Composition	Permeability Lp°	Hydraulic resistance Rm
M1	Cellulose acetate	4.30	291.43
M2	AC/ PEG (9/ 1)	6.120	204.76
M3	AC/ SiO2 (9.5 / 0.5)	9.658	129.75
M4	AC/PEG/SiO2 (8.5/1/0.5)	12.64	99.14

Compaction factor

The study of the membrane compaction factor is essential to study the morphological structures, in particular the pore arrangements of the membranes and the configuration of the underlayer of the membrane. Generally, membranes with high CF indicate that they are highly compacted, which further demonstrates the existence of some defective pores in the membrane underlayer structure. Thus, according to the above curves we can see the gradual decrease of the pure water flow of all the membranes in the course of time due to the compaction of the pores and finally stable state flows were reached approximately after 90 minutes of filtration process. The gradual decrease in pure water flow results can be described due to the compacting of pore walls that have reached uniform and denser structures and resulted in a decrease in pore size and flux [27].

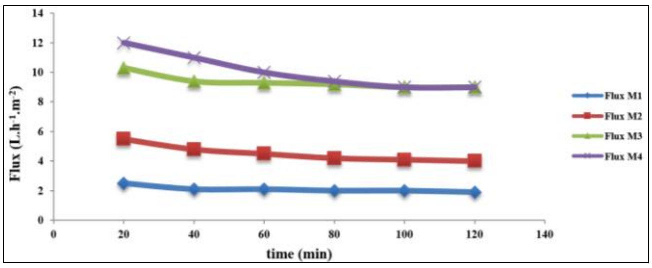


Fig 5: water flux as function of time for CA composite membranes

Humic acid removal

The humic acid was dissolved in distilled water at a concentration of 40 mg / L the solution thus prepared will be filtered with the four membranes prepared, determine for each membrane the concentration of the permeate and finally measure the retention rate. The applied pressure was 1.5 bars, the filtration volume was 20 mL. Each 10 ml of the filtrate is taken, the filtration time is determined, its absorbance and the retention rate are measured. At pH =6.7 and salinity =0 mg/L, the obtained retention rates are cited as below (Table 5).

Table 5: Percentage of retention of the different membranes M1, M2, M3, and M4 respectively

Volume (mL)	Duration of filtration(min)	Ci (mg/L)	Absorbance	$C_{permeate}$(mg/L)	$C_{retentate}$(mg/L)	Percentage of retention (%)
1^{er} 10mL	57.22	40	0.017	0.34	39.65	99.13
$2^{ème}$ 10 mL	40.56	40	0.032	0.92	39.07	97.69
1^{er} 10mL	40.35	40	0.035	1.03	38.96	97.40
$2^{ème}$ 10 mL	35.30	40	0.040	1.23	38.76	96.92
1^{er} 10mL	35.20	40	0.048	1.53	38.46	96.15
$2^{ème}$ 10 mL	31.07	40	0.03	0.84	39.15	97.88
1^{er} 10mL	34.01	40	0.046	1.46	38.53	96.34
$2^{ème}$ 10 mL	30.30	40	0.041	1.26	38.73	96.82

From the results obtained, it is found that the maximum retention was obtained with the CA membrane M1 with a retention rate equal to 99.13% and the minimum retention is obtained with M3 CA/SiO_2 with a retention rate equal to 96.15%.

pH effect

In this part we will optimize the pH, in other words we will change each time the pH value of the humic acid solution to an initial concentration equal to 40 mg / L and we will note the maximum retention that corresponds to each membrane. The values chosen are as follows: 4, 6, 8 and 10 and the applied pressure equal to 1.5bar.

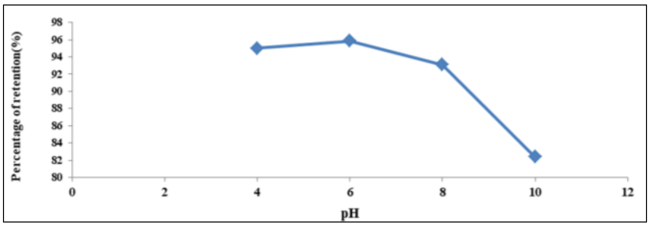


Fig 6: Percentage of retention of humic acid as function of pH for CA membrane

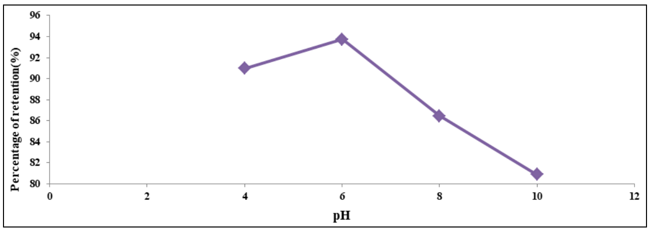


Fig 7: Percentage of retention of humic acid as function of pH for CA/PEG membrane

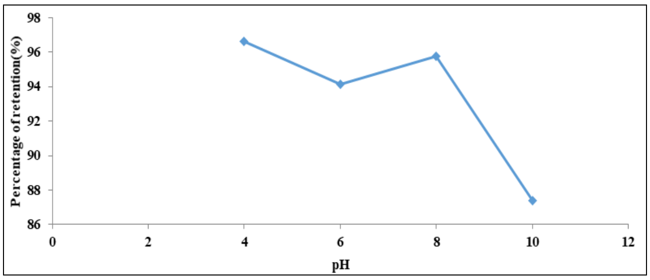


Fig 8: Percentage of retention of humic acid as function of pH for CA/SiO_2 membrane

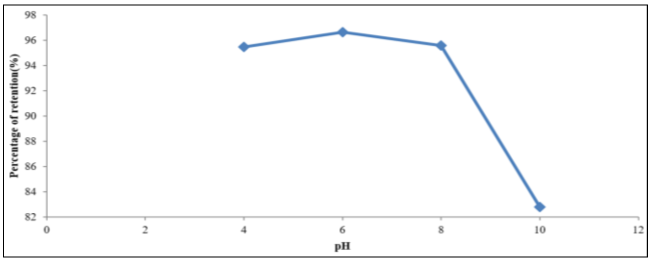


Fig 9: Percentage of retention of humic acid as function of pH for CA/PEG/SiO_2 membrane

From the previous results, it is found that the optimal retention corresponds to a pH equal to 6 for the membranes M1, M2 and M4 with retention rates equal to 95.86%, 93.75 and 96.63% respectively and at a pH equal to 4 for the M3 membrane with a retention rate of 96.63%. The minimal retention of humic acid takes place in an alkaline medium corresponding to a pH equal to 10 for all membranes. The retention rates are respectively:

M1: 82.40%
M2: 80.86%
M3: 87.40%
M4: 82.78%

The interpretation of these data leads us to confirm that the membrane M3 (cellulose acetate / SiO2) is the most resistant membrane during pH variation, moreover it has the maximum retention rate corresponding to the optimal and minimal pH(that is to say, the minimum retention) with respect to all the membranes in addition it is advantageous compared to the other membranes by its resistance in acidic medium (optimal pH equal to 4).

Salinity effect

The pH optimization of the humic acid solution for each membrane allows us to optimize its salinity. We will prepare 4 solutions of humic acid at 40 mg / L each of them corresponds to the pH which gives the optimal retention for each membrane, and each time we will add to the humic acid with a well-defined quantity of NaCl of so that the following concentrations of Sodium Chloride are obtained: 0.5g / L, 1g / L, 1.5g / L and 2g / L. we Note that the applied pressure was 1.5 bar.

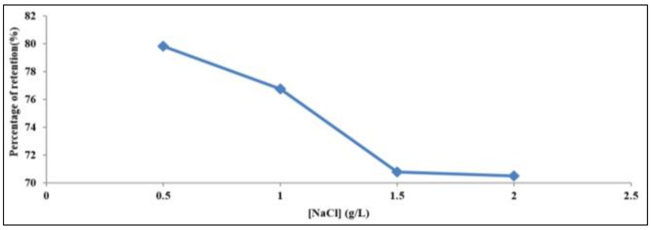


Fig 10: Percentage of retention of Humic acid as function of salinity for CA membrane

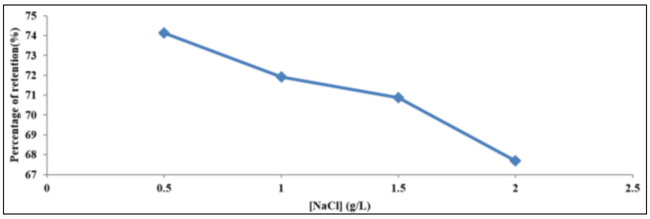


Fig 11: Percentage of retention of Humic acid as function of salinity for CA/PEG membrane

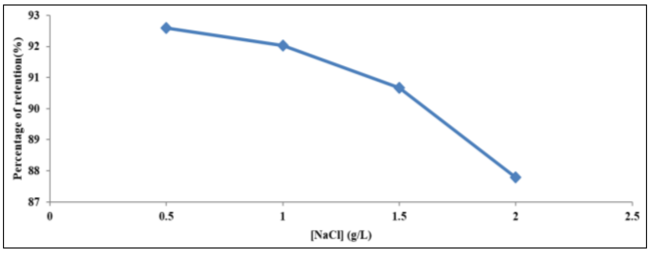


Fig 12: Percentage of retention of Humic acid as function of salinity for CA/SiO_2 membrane

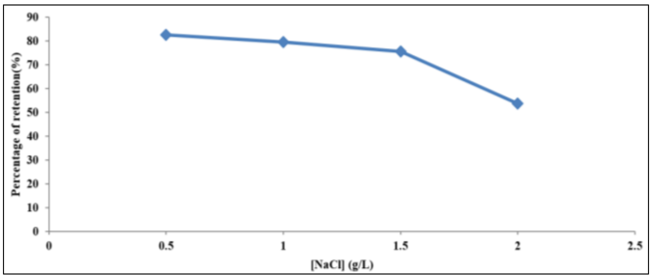


Fig 13: Percentage of retention of Humic acid as function of salinity for CA//PEG/SiO_2 membrane

From the previous data it is found that the concentration of NaCl corresponding to the optimal retention is 0.5g /L. The maximum retention rates of humic acid for each membrane developed at its optimum pH chosen are: 79.80%, 74.13%, 92.59% and 82.5% respectively for M1, M2, M3 and M4.From which it can be deduced that the maximum retention of humic acid corresponds to the membrane based on cellulose acetate and SiO_2 as additive with a value of 92.59% for a pH equal to 4 and a salinity equal to 0.5 g / L. the gradual decrease in pure water fluxes of all membranes over time due to pore compaction and steady state fluxes was achieved approximately after 90 minutes of filtration process. The gradual decrease in pure water flow results can be described due to the compacting of pore walls that have reached uniform and denser structures and resulted in a decrease in pore size and flux.

BSA removal

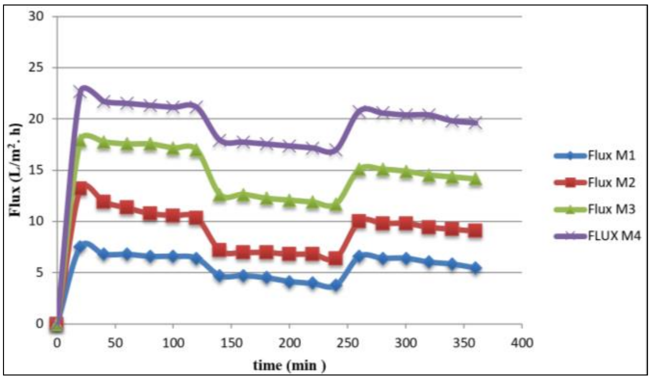


Fig 14: Water flux as function of time for the different membranes

We can observe (Fig 14) that the CA/PEG/SiO_2 membrane present the higher water flux as comparing to CA/PEG, CA/SiO_2 and CA membranes, this can be due to the addition of PEG and SiO_2 particles which can affect the hydrophilicity of the membrane and as consequence the water flux increases in that membrane.

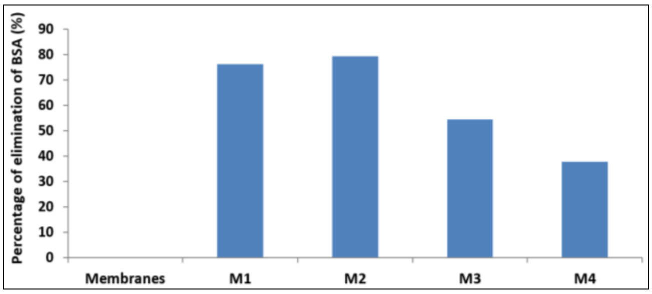


Fig 15: Performances of elimination of BSA for the different membranes

From Fig 15, we can suggest that the addition of PEG as porous agent can increase the BSA absorption in the CA/PEG membrane this can be due to possible interactions between BSA and PEG.

Conclusion

It has been proposed in this work to study the elaboration of membranes based on cellulose acetate (M1), cellulose acetate / PEG (M2), cellulose acetate / SiO_2(M3) and AC / PEG / SiO_2(M4) for application in ultrafiltration for the removal of humic acid. The different membranes were developed by phase inversion using DMF as a solvent and water as a non-solvent. The characterization of these membranes allowed us to define their structure and prove their hydrophilic character through the measurement of the contact angle. This hydrophilic character increases by the addition of SiO_2 and polyethylene glycol PEG as hydrophilic polymer (additive). Thus, we had the M4 membrane as the most hydrophilic membrane with a contact angle of 42 °. The characterization of membranes developed using Fourier transform infrared spectroscopy in attenuated total reflectance mode has allowed us to highlight new interactions due to the insertion of silica particles in the membrane as well as the possibility of bond formation. Hydrogen between PEG and cellulose acetate. The M4 membrane (AC / PEG / SiO_2) has the highest porosity as well as the highest equilibrium water content. Thus, the addition of additives such as SiO_2 and the hydrophilic polyethylene glycol PEG polymer have the effect of improving the hydrophilicity of the initial membrane M1. The maximum retention of humic acid corresponds to the membrane based on cellulose acetate and SiO_2 as additive with a value of 92.59% for a pH equal to 4 and a salinity equal to 0.5 g / L. the gradual decrease in pure water fluxes of all membranes over time due to pore compaction and steady state fluxes was achieved approximately after 90 minutes of filtration process. The gradual decrease in pure water flow results can be described due to the compacting of pore walls that have reached uniform and denser structures and resulted in a decrease in pore size and flux.

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