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Preparation and Performance of Polysulfone-Cellulose Acetate Blend Ultrafiltration Membrane

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In the development of high performance polymeric membranes, it is essential to design the molecular and morphological characteristics for specific applications. Polysulfone and cellulose acetate of blend membranes with various concentration of polymer pore former, PEG600 were prepared by phase inversion technique and used for ultrafiltration. Polymer blend composition, additive concentration, and casting conditions were optimized. The blend membranes were characterized in terms of compaction, pure water flux, water content, hydraulic resistance and separation of dextran studies. Surface morphology of the embranes was analyzed using scanning electron microscopy at different magnifications. Further, the characterized membranes were attempted for treatment of distillery effluents after secondary treatment and the results are discussed in detail.

Keywords ultrafiltration, polysulfone, cellulose acetate, polyethylene glycol, color removal, blend membrane

Introduction

Ultrafiltration membranes are highly suitable for separation of suspended materials, colloidal particles and dissolved solutes with molecular mass of at least two orders magnitude higher than the solvent (usually) water. The polymeric membranes have the following potentially important properties: porosity and pore size distribution (influences flux, separation and fouling); hydrophilic-hydrophobic character (influences separation and fouling); chemical compatibility (influences separation and cleaning); and ease of fabrication and cost (influences economics and applications) (1). These characteristics make them suitable in various fields of application ranging from separation problems in laboratory to separation processes in the chemical, food and pharmaceutical industries (2, 3).

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The membranes of single polymer component such as cellulose acetate, polysulfone and polyamide etc., have certain drawbacks at lower resistance towards fouling, chemical and separation efficiency. In order to overcome these deficiencies, membranes made from multiple components are gaining importance. Polymer blending is a proven tool to obtain new types of membrane materials with a wide diversity of properties with economic advantages (4). The use of this technique in preparing UF membranes offers the possibility of controlling the structure and porosity of the membrane to some extent by simple variations of the preparative parameters.

The previous studies of single polymer membranes such as cellulose acetate (5), polysulfone (6) for the separation of protein and metal ions by ultrafiltration have revealed their limitation due to the lower flux stemming from their rigidity. In contrast, the blend membranes of CA-polyurethane (7), CA-polyethersulfone (8), showed better performance than cellulose acetate for the separation/flux of protein and metal ions. Malaisamy et al. (9) prepared CA-sulfonated polysulfone based blend UF membranes and estimated pore size using dextran separation based on molecular weight cutoff.

As polysulfone has very excellent properties (3) such as resistance towards pH, solvent and temperature, it was proposed for blending with the hydrophilic polymer cellulose acetate. The hydrophilic/hydrophobic balance, together with other properties of the membrane system, can be altered if the membrane is prepared through a multi-component polymer blend. We have already reported (10, 11) about cellulose acetate/ polysulfone blend membranes in order to improve the separation properties of cellulose acetate to polysulfone and the performance of blend membranes are here reported.

In order to prepare the blend membranes with different pore size (12, 13), PEG600 was found to be suitable for this study (8, 14). The effect of various concentrations of this macromolecular additive on the function of flux behavior was studied. It has been found that a variety of morphologically different membranes can be prepared by changing the composition and concentration of polymer, solvent, and additive. The blend membrane thus prepared were subject to various characterizations such as effect of compaction on pure water flux (PWF), water content, membrane resistance (Rm), and molecular weight cut off (MWCO). The determination of morphological characteristics plays an important role in evaluating the performance of membranes (15, 16).

Further, the membranes were utilized in a preliminary study of removal of color from distillery effluent. The disposal of a large quantity of spent wash from distilleries on to surrounding agricultural lands and its present popular methods pose a lot of pollution problems due to the lack of copious supply of water. The presence of color in the effluent pollutes the water resources and prevents the penetration of light which in turn reduces the photosynthetic activities.

Experimental

Materials

Polysulfone (PSf), Udel-P 3500 supplied by Amoco Chemical Inc. US, was used without any modification (Mw 77 kDa). Commercial grade MYCELL cellulose diacetate CDA5770 (Mw 15 kDa,acetyl content 39.99 wt%) was procured from Mysore Acetate and Chemicals Limited, India and was used after recrystallization from acetone. Polyethylene glycol (Mw 600 Da) was procured from Merck Limited, India and used as a nonsolvent swelling agent. Analar grade N, N-Dimethyl formamide (DMF) from SD Fine Chemicals, India, was distilled in vacuum, treated through molecular sieves for removing moisture and stored in dry form. Other solvents of analar grade such as acetone and methanol from SD Fine Chemicals, India were used. Sodium lauryl sulphate (SLS) of AR grade, obtained from SD Fine Chemicals, India was used as surfactant. Dextran supplied by Sigma Chemical Co., St. Louis, US was used for MWCO studies.

Membrane Preparation

Binary solutions were prepared in DMF in the presence of the additive PEG by the addition of a known quantity of polysulfone and cellulose acetate under continuous stirring for 4 h at $95 + 5^{\circ}$ C. These solutions were used as a casting solution for preparation of the membrane by a phase inversion technique after storing at room temperature. The thickness of the membrane was maintained at 0.22 ± 0.02 mm. After casting, the solvent present in the cast film was allowed to evaporate for 30 s with $19 \pm 1\%$ relative humidity followed by immersion in a gelation bath containing 2 wt% DMF/water and 0.5 wt% surfactant (SLS) kept at 10°C. Membrane sheets were stored under a distilled water/formaldehyde mixture. The prepared membranes were cut into the necessary size (dia = 76 mm). These were then pressurized before use for the ultrafiltration experiments at 345 kPa using a UF kit supplied by SPECTRUM (Los Angeles, US). For the retention studies, 0.1% solutions of dextran with different molecular weights e.g., 12 kDa, 32 kDa, 77 kDa and 167 kDa were used. The concentration of dextran was quantitatively determined (17) using UV Spectrophotometer at $\lambda_{max} = 485$ nm. For the color removal in the distillery effluents, % of color intensity was measured using a UV Spectrophotometer at $\lambda_{\text{max}} = 680$ nm for feed and permeate.

Characterization of Membranes

The membranes were pressurized at 414 kPa for 5 h to get a steady state pure water flux. The compacted membranes were then subjected to further characterization. During compaction, the pure water flux was measured at every hour. The flux generally declined sharply in the initial hours, and leveled off at 3–4 h, and these membranes were used in the subsequent UF experiments at 345 kPa (18). The pure water flux (Jw) is determined using the following Equation 1 (18, 19). The initial water flux was taken about 30 min after the pressurization in the test cell, at 414 kPa.

$$J_W = \frac{Q}{\Delta t \cdot A} \tag{1}$$

Where Q is the quantity of permeate collected (L); A is the membrane area (m^2) ; Δt is the sampling time (h).

The water content of the membrane was determined as reported earlier (5). The membrane was soaked in water for 24 h, followed by mopping with blotting paper, then the membranes were weighed. These wet samples were then placed in a drier at 75° C and weighed after 48 h to get the dry weight. From the above values, the percent water content was derived as below (Equation 2):

%Water content =
$$\frac{(\text{wet sample weight} - \text{dry sample weight})}{\text{wet sample weight}} \times 100$$
 (2)

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The hydraulic resistance of the membrane was evaluated from the slope of the water flux (Jw) vs. transmembrane pressure difference (Δ_P) using the following Equation 3 (5, 17, 20):

$$R_m = \frac{\Delta_{\rm P}}{J_w} \tag{3}$$

The percent solute rejection (SR) of Dextran was calculated from the following Equation 4 (5, 20):

$$\% SR = \left[1 - \frac{Cp}{Cf}\right] \times 100 \tag{4}$$

The top surface of the PSf/CA blend membranes were studied using Scanning Electron Microscopy (LEICA STEREOSCAN S440, Philips, UK). Scanning Electron micrographs were taken at various magnifications for the top surface view of the polymeric membranes.

The solutions of macromolecules having sharp molecular weight distributions (14, 21) are widely used for the purpose of determining molecular weight cut off of Ultrafiltration membranes. The molecular weight cutoff of the membrane was determined by identifying an inert solute, which has the lowest molecular weight and has solute rejection (SR) of 80–100% in steady state UF experiments (21). Thus, dextran with different molecular weights e.g., 12, 42, 71, and 167 kDa were chosen and its percent separation through the blend membranes were analyzed (17) using UV-spectrophotometer at $\lambda_{max} = 485$ nm.

Results and Discussion

In this investigation, polysulfone was blended with cellulose acetate, along with various composition of PEG600 additive in a solution medium. The hydrophilic polymeric additive PEG600 was introduced from 2.5 wt% to 12.5 wt% into the casting solution.

The blend membranes, which are membranes with asymmetric pore structure, were prepared in a phase inversion technique. The compositions of cellulose acetate in blend solution varied from 0 to 15 wt% of the total polymer (15 wt%) concentration in the casting solution. An increase in cellulose acetate composition in the casting solution beyond 15 wt% resulted in phase separation. So, it was finally restricted to 15 wt% of cellulose acetate. It was chosen as the maximum possible blend composition (85/15% of PSf/CA). The blend membranes were first compacted with pure water at 414 kPa and then characterized by pure water flux (PWF), water content and hydraulic resistance at 345 kPa. The MWCO of membranes were determined by dextran solutions of various molecular mass. In order to apply these membranes for effluent treatment, a preliminary attempt on the color removal from distillery effluents (22) were studied and the results are discussed.

Compaction Studies, Pure Water Flux and Water Content

In this investigation, the PSf/CA blend (85/15 wt%) membranes were prepared with various concentrations of PEG600. To avoid any irreversible change during the operation, each membrane sample was pressurized at 414 kPa for 5 h. It was observed from Figure 1 that, an increase in PEG600 content from 0 to 12.5 wt\%, increases



Figure 1. Effect of compaction time on PWF of PSf/CA (85/15% composition) blend membrane with different concentrations of PEG600.

the pure water flux from $10 \text{ Lm}^{-2} \text{ h}^{-1}$ to $80.5 \text{ Lm}^{-2} \text{ h}^{-1}$. It was also observed that, during compaction there was a gradual decrease in pure water flux over the initial few hours for all membranes except 12.5 wt% of PEG600 content. The pure water flux initially decreases notably for about 0 to 2 h, and then remains almost constant. It may be due to the fact that big pores are situated in the membrane at higher concentration of PEG600. However, the pure water flux was steadily decreased for the content of PEG600 from 10.0 wt% to a lower concentration in the blend membrane. The pure water flux remains almost constant after 3 h due to compaction of membrane pores for all the cases. However, the pure water flux is not reduced much, to 0 wt% PEG content in CA/PSf blend membrane.

The pure water flux of compacted membranes was determined by measuring the time required for the permeation of a known volume of water at 345 kPa transmembrane pressure. The influence of different concentrations of PEG600 in PSf/CA (85/15 wt%) blend membrane on pure water flux and water content were shown in Table 1. The PSf/CA blend membrane with 0 wt% of PEG had a lower flux of 9.8 Lm⁻² h⁻¹. An increase in the PEG600 content in the PSf/CA blend composition from 2.5 to 12.5 wt% increases the flux from 24.4 to 74.0 Lm⁻² h⁻¹. The addition of PEG600 in the blend not only increases the hydrophilic behavior but also the macrophase separation of the blend membranes.

The relative higher flux with increasing PEG content in the blend may be due to the increase in network pore size of membranes. Network pores were formed during the addition of pore former, in the phase inversion technique, in addition to the enhancement of aggregate pore size due to segmental gaps between polymeric chains. Similar results were reported for the incorporation of PVP as an additive in the case of pure polysulfone membranes (23).

The blend membranes prepared at definite proportions of PSf/CA (85/15% composition) were washed out thoroughly with distilled water before being subjected to

(15 wt%) blend membranes				
Polymer	PEG, wt%	Pure water flux ^{<i>a</i>} $(Lm^{-2}h^{-1})$	Water content (wt%)	$(kPa L^{-1} m^2 h)$
PSf/CA:85/15	0 2.5 5.0 7.5 10.5 12.5	9.8 24.4 29.9 30.8 35.3 74.0	78.0 80.3 80.5 80.7 81.0 81.5	31.0 13.7 10.6 8.3 7.6 3.7

 Table 1

 Composition and characteristics of PSf/CA (85/15% composition) (15 wt%) blend membranes

^{*a*}@ 345 kPa transmembrane pressure.

the percentage of water content evaluation. It is evident from Table 1 that water content increases with an increase in the PEG600 concentration from 0 to 12.5 wt% of casting solution in PSf/CA blend membranes. The leachability of PEG600 is enhanced and this favors the formation of a larger number of bigger pores on the blend membrane. This leads to a higher water content of 81.5% in membranes with 12.5 wt% PEG, as compared to membranes without PEG (78.0%). The availability of pores in blend membrane could be less in the absence of PEG, which in turn explains less water content and less pure water flux (Table 1).

Membrane Hydraulic Resistance

Membrane hydraulic resistance is an important parameter in pressure-driven membrane processes. In order to determine the membrane resistance, all the PSf/CA blend membranes prepared with or without pore former PEG600, after compaction, were subjected to different transmembrane pressures (TMP) under steady-state flux conditions. Thus, PWF at various transmembrane pressures between 69 and 345 kPa were determined by measuring the time required for permeation of a known volume of water. The plot of pressure vs. pure water flux gives a linear relationship (24) and the inverse slope is the membrane hydraulic resistance. As operating pressure increases, the driving force for the permeation of water also increases and hence. an increase in pure water flux is observed. Figure 2 illustrates the PWF of membranes with different additive compositions at various transmembrane pressures. It is evident from the figure that an increase in TMP increases PWF at a linear rate. The hydraulic resistance (Rm) of the membrane was evaluated from Equation 3. From Table 1, the blend membrane (85:15 wt%, PSf/CA) showed a higher membrane hydraulic resistance of 31.0 kPa $L^{-1}m^{2}h$ in the absence of PEG600. Furthermore, the Rm is decreased with increasing composition of PEG600 from 0 to 12.5 wt%. This behavior of a decrease in Rm with an increase in additive concentration may be due to the formation of macro voids on the membrane surface due to leaching out of PEG600 during gelation. Because of the increase in PEG concentration, the diffusion rate of PEG600 out of cast solution into a gelation bath is faster due to the higher concentration gradient. This can be supported from a previous study in which an increase in PVP content favors an increase of flux, as in the CA/PSf blend membrane.



Figure 2. Different transmembrane pressures of PSf/CA (85/15% composition) blend membrane with different concentrations of PEG600.

Morphological Studies

The top surfaces of polysulfone, cellulose acetate, blend membrane (PSf/CA, 85/15% composition) and in the presence of pore former PEG600 was analyzed by scanning electron microscopy (SEM) at different magnifications and the results are depicted in Figure 3. It is observed from Figure 3(c) that in the absence of an additive, the pores are very small. Even the pores are distributed randomly in a PSf/CA blend membrane. It can be seen that two type matrix pore voids are observed in the PSf/CA blend membrane due to partial compatibility. The top matrix pores are from polysulfone and inside pores from cellulose acetate, which is respective of the blend composition (85/ 15 wt%). As the PEG600 was increased from 0 to 2.5 wt%, the significant surface changes were observed when compared to pure polymeric membranes (25) and the pore size increased as is evident in Figure 3(d). The shape and structure of the pores are related to the concentration of additive in the casting solution. The distribution of pores are almost even in PSf/CA (85:15) blend membranes with pore former PEG600. Hence, PEG600 chain exists as a random coil, which swells to accommodate solvent molecules. A swollen linear polymer molecule in the solution occupies a space which is generally quite spherical with a larger radius and it will form larger voids (pores) (Figure 3(d)). The average pore size of ultrafiltration membrane (2) is from $1 \sim 100$ nm. The observations made by SEM are in good correlation with pure water flux, water content, membrane resistance, and MWCO of corresponding pure membranes and PSf/CA blend membranes.

Molecular Weight Cutoff (MWCO)

The molecular weight cutoff is a key parameter for membranes and are determined using inert, stable molecules having various molecular weights. When the molecules have a molecular weight which is larger than the molecular weight cutoff (MWCO), it will not



Figure 3. SEM micrographs of the top surfaces of a) PSf (100% composition) (\times 2500); b) CA (100% composition) (\times 2000); c) PSf/CA (85/15% composition) blend (\times 2000); d) PSf/CA (85/15% composition) (\times 2500) with PEG600 (2.5 wt%).

pass through the membrane. Generally, the cut off characteristics can be assessed by linear polymers. Further, the separation of solutes by a membrane depends not only on the dimensions of the solute molecules, but also on its shape, flexibility and interactions with the membrane surface. Here, the MWCO of the membranes were determined using different reference solutions of dextran. MWCO also depends on the structure of the diffusing species and its three dimensional structure (26).

The determination of exact MWCO was not carried out because of the unavailability of higher dextran (more than 167 kDa) molecular weight. Figure 4 illustrates the % rejection of dextran solutions of different molecular weights. The MWCO of a particular membrane corresponds to the molecular weight of the solute having a solute rejection (21) beyond 80%. But, in the present investigation, it was observed that the rejection was 66.3% for the dextran solution of 167 kDa. Observations indicate that the pore sizes of the membranes are higher and hence, determination of MWCO using higher molecular weight dextran will be studied.

Color Removal from Distillery Effluent

Several membrane processes have been studied for molasses purification. Ultrafiltration membranes of 20 kDa MWCO, were used to remove 85% color, 95% turbidity and 60% gums form diluted molasses (17).

Distillery effluent is treated with PSf/CA blend membranes using a UF kit at 345 kPa. The present investigation shows that the MWCO of the membranes used were beyond 167 kDa. Figure 5 illustrates the color removal efficiency and the effluent permeated



Figure 4. Rejection of Dextran using PSf/CA (85/15% composition) blend membrane with various concentration of PEG600.

flux of the blend membranes (PSf/CA: 85/15). It was observed from the graph that, flux of the distillery effluent is higher while increasing the PEG concentration and, on the other hand, changes in the color removal efficiency is not as significant. Attempts for color removal from distillery effluents using the blend membranes indicate the removal efficiency of 34.2% for membranes without the presence of PEG. The blend membranes for color removal do not have a single pore size, but rather have a distribution of pore



Figure 5. Color removal and flux of distillery effluent in PSf/CA (85/15% composition) blend membrane with different concentrations of PEG600.

sizes. Rejection capabilities depend on the distribution of pore sizes. Here, the two matrix pores are formed from blend membranes of polysulfone and cellulose acetate (Figure 3(c)). Further work on reduced pore size blend membranes, for higher separation of color, turbidity, and gums from diluted molasses will be carried out.

Conclusions

Ultrafiltration blend membranes based on PSf-CA were optimized in the absence and presence of PEG600. The suitable compatibility was found to be 15/85 wt% PSf/CA and this composition is selected for this study. Phase separation occurred beyond 15 wt% of cellulose acetate in the blend. An increase in PEG content in the blends resulted in increased pure water flux and water content. The different molecular weights of Dextran were used to carry out the investigation of MWCO of PSf-CA blend membrane. The pore size increases with increasing concentration of PEG. Furthermore, the color removal of distillery effluent tested with PSf-CA blend membranes was found to be higher with lower concentration of PEG content and other low hand flux. Hence, the modification in the casting conditions and polymer compositions would help in improving the membrane performance.

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