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Preparation and Characterization of Cellulose Acetate and PEG Ultrafiltation Membrane for Dextran and Sugar Cane Juice Purification

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Cellulose acetate membranes have been prepared by varying from 15 to 20 wt. % with increment of 1 wt. % using N,N′- dimethyl formamide as the solvent. The 2.5 wt. % of pore forming agent, polyethylene glycol 600 (PEG 600) was also added. Prepared membranes have been subjected to ultrafiltration characterizations such as compaction, pure water flux, water content, membrane hydraulic resistance and morphological studies. The porosity and pore size of the membranes also increased as the addition of PEG. The effects of cellulose acetate content and additive PEG in casting solution, pressure on concentration of clarified juice are studied. Good correlations have been found between the dextran flux and sugar solution reduction due to adsorption and for bound dextran on the cellulose acetate membranes with respect to time. The 15 wt. % of cellulose acetate and 2.5 wt. % of PEG in same cellulose acetate achieved high flux and higher retention for dextran and sugar solution.

Keywords: Ultrafiltration, Dextran retention, Sugar solution concentration, Cellulose acetae, Membrane modification.

INTRODUCTION

Purification of sugarcane juice by membrane filtration promises a significant improvement in the sugar quality and yield[1]. Membranes vary in their make up from the relatively crude structure of a screen to extremely fine configurations in the order of thickness of a molecular layer membrane[2] may be either passive or reactive depending on the membranes ability to alter the chemical nature of the permeating species[3]. It is useful to pharmaceutical, sugar and food industries[4] since the membrane type is easy to exchange[5] and have measured the permeability and osmotic permeability for cellulose acetate membranes of varying water contents.

Utrafiltration is an established process for the clarification of fermentation broths and for cell harvesting. Membrane is a slowly but steadily evolving technology and may well become the separation technology of the future. Fouling studies for sugarcane juice clarification by membranes were performed using only ultrafiltration and ultrafiltration coupled with coagulation experiments for decolourization of raw brown sugar obtained from Indian Sugar Industry. Using a feed solution of 50° brix and 50 % reduction in colour was obtained. The ultrafiltration process provided juice with higher purity and lower colour. The sugar mills adjusted the pH of the juice on the neutral alkaline side so as to avoid the inversion of sucrose. By adjusting the pH, it was found that around pH 8 gave the best results. Kishihara and Fujii[6] carried out the ultrafiltration process for the recovery of sugar by crystallization. Sugar crystallization is a principal process in sugar manufacturing and sugar recovery through crystallization and quality of the sugar greatly depend on the kind and the quantity of impurities contained in the sugar solution.

Cellulose acetate based polymers are always preferred over other membrane materials due to the advantages such as excellent pore forming properties, high salt rejection, moderate flux, relatively low fouling of the membrane, easy manufacturing, renewable source of raw material, cost effectiveness and non-toxicity[7-10]. Hence in this investigation, attention is focused on cellulose acetate based membranes. Polyethylene glycol is one of the good polymeric additives widely used in the preparation of phase inversion membranes in view of the fact that PEG is compatible with many membrane materials and is quite well soluble in many solvents as well as non-solvent, water[11-13]. To a large extent, our current understanding of the mechanisms governing this permselectivity of dextran is compared to that of a reference solute. As test macromolecules tritiated, electrically neutral dextrans (selected because dextrans are known to be biologically and chemically inert) of high specific activity were employed, with molecular size determined by standard quantitative gel chromatographic techniques. In case of ultrafiltration membranes, durability of the membranes is attributed to the membrane material.

Considering dextran as polysaccharide, it turned out that dextran does play also a major role in real feed streams, for example in sugar beet or sugar cane processing[14,15]. Furthermore, dextrans are extensively used for the characterization of the rejection of ultrafiltration membranes including the nominal cut-off[16]. However, specific studies on membrane fouling by dextran had not been clearly conducted yet. Therefore the selection of the membrane material is the most important aspect in the design of the highly resistant ultrafiltration membranes.

With this view, the present investigation has been formulated for preparation of cellulose acetate membranes, characterization of the modified membranes for pure water flux, compaction, water content, and hydraulic resistance and to study the effects of polymer composition and concentration of the pore former (PEG) on the performance of the membranes in terms of concentration of sucrose.

EXPERIMENTAL

Polymers: Commercial grade MYCEL cellulose acetate 5770 (acetyl content 39.99 wt. %) was procured from Mysore Acetate and Chemicals Company Limited, India, after recrystallization from acetone. The glass transition temperature T_g and molecular weight of the recrystallized polymer are 219 °C and 115 kDa, respectively. Dextran of molecular weights 19, 42, 77 and 150 kDa were procured from Sigma-Aldrich Company, USA and stored at suitable temperature before use. N,N'-Dimethyl formamide from SD fine Chemicals, India. Sodium lauryl sulphate of AR grade was obtained from SD Fine Chemicals, India and used as a surfactant. The sugarcane juice is obtained from Vellore co-operative sugar mills. The juice is then subjected to clarification by adding lime. Then, the clear supernatent liquid is separated and used for analysis.

Dope solution preparation: Cellulose acetate membranes were prepared by dissolving in DMF in different compositions (Table-1) in presence and absence of additive, pore former, PEG 600, under constant mechanical stirring in a three necked round bottom flask for 3 h at 40 °C. The homogeneous solution was allowed to stand for 1 h in air tight condition to get rid off the air bubbles..

Membrane formation: Casting environment *viz*. relative humidity and temperature were standardized for the preparation of membranes with better physical properties such as homogeneity, thickness and smoothness. The relative humidity was maintained between 48-50 % and the temperature was kept at 23 ± 3 °C for all the casting experiments. Prior to casting, gelation bath of 2 L consisting of 2.5 % (v/v) DMF (solvent) and 0.2 wt. % surfactant, sodium lauryl sulphate (SLS), in distilled water was prepared and kept at 20 °C. The membranes were cast using a casting blade (polished stainless steel bar) on a glass plate as follows. The casting solution was spread on the glass plate and a desired thickness was maintained by adjusting the height of the casting blade, by fixing oil sheet paper at both ends of the casting blade. Thus the respective solutions of cellulose acetate were poured over the glass plate at specified casting conditions mentioned earlier and were uniformly cast by a casting blade at constant casting time. Solvent present in the casting solution was allowed to evaporate for 30 s, followed by gentle immersion of the membrane along with the glass plate in the gelation bath of known composition. After 0.5 h of gelation ,the membranes were removed from the gelation bath and washed thoroughly with distilled water to remove all DMF and surfactant from the membrane. The membranes were subsequently stored in distilled water containing 0.1 % of formalin solution to prevent microbial attack.

Ultrafiltration setup: Ultrafiltration experiments were carried out in a batch type cell fitted with a magnetic stirrer. The cell was placed on a magnetic stirrer. The membrane used is made up of cellulose acetate with and without additive in the form of a flat sheet.

Membrane characterization: Thickness of the membrane was measured by a micrometer (Mityuto, Japan) at various parts of the membrane. It is around 0.22 ± 0.02 mm. The prepared membrane were cut into the necessary size for the use in the ultrafiltration cell. The membranes were initially pressurized with distilled water at 414 kPa for 5 h.

Compaction: In order to conduct the experiments at steady state conditions, the prepared membranes have to be compacted at an elevated pressure than the pressure that is to be maintained in the ultrafiltration study, until a constant flux is reached. Thus the membranes were loaded in the stirred ultrafiltration cell and pressurized with distilled water at a transmembrane pressure of 414 kPa. The pure water flux was measured after every hour in order to monitor the compaction behavior. The membranes were compacted until a steady flux was observed. Pure water flux was calculated over measured time intervals using the following equation[17].

$$J_w = \frac{Q}{A \cdot \Delta T} \quad (1)$$

where, J_w = Pure water flux, l m^{-2} h^{-1}; Q = Quantity of pure water permeated, l; A = Membrane area, m^2; ΔT = Sampling time, h.

TABLE-1
MEMBRANE CASTING COMPOSITION, PWF, WATER CONTENT AND PORE ARCHITECTURE OF CELLULOSE ACETATE MEMBRANES WITHOUT PEG 600

Type	Cellulose acetate (%)	Solvent (DMF) (mL)	PWF at 345 kPa (l/m^2 h)	Water Content (%)	R_m (kPa l^{-1} m^2 h)	$\bar{R}$ # (Å)	N± (× 10^{12})	ε* × 10^5 (%)
CA1	15	85	22.0	87	3.9	64	9.98	8.43
CA2	16	84	18.7	85	4.3	59	9.43	7.23
CA3	17	83	16.3	81	6.3	51	9.10	5.83
CA4	18	82	11.5	79	8.3	45	8.31	3.98
CA5	19	81	6.0	75	11.5	38	6.54	3.01
CA6	20	80	3.5	71	20.6	30	5.32	2.31

#Average pore radius, ±number of pores per 1 m^2 area, *surface porosity

Pure water flux: Pure water flux is a measure of permeability of a membrane and from the knowledge of pure water flux we can predict the pore formation in a given membrane. Thus, the compacted membranes were subjected to pure water permeation studies and the fluxes were measured under steady state flow at a transmembrane pressure of 345 kPa using eqn. 1[17].

Water content: Membrane samples were cut into desired size and soaked in distilled water isothermally for 2 h and weighed immediately after blotting the free surface water. These wet membranes were dried in a vacuum oven for 6 h at 80 ± 2 °C and the dry weights were weighed. From the dry and wet weights of the samples, the water uptake was calculated using the following equation[18].

$$\text{Water content} = \frac{(W_{wet} - W_{dry})}{W_{dry}} \times 100 \quad (2)$$

Membrane hydraulic resistance: To determine the membrane hydraulic resistance (R_m), the pure water flux of the membranes was measured at different transmembrane pressures (ΔP) *viz*., 69, 138, 207, 276 and 345 kPa. The variation of pure water flux was plotted as a function of pressure for all the prepared membranes. The hydraulic resistances of the membranes (R_m) were determined from the inverse of slopes using the following equation[19].

$$R_m = \frac{\Delta P}{J_w} \quad (3)$$

Morphological studies: The membranes were cut into small pieces and mopped with filter paper. These pieces were immersed in liquid nitrogen for 20-30s and frozen. The frozen bits of membranes were broken and kept in a desiccator. These membrane samples were used for scanning electron microscopy (SEM) studies. The membrane samples were mounted on studs and gold-sputtered to provide electrical conductivity to very thin layer of the membranes[20]. Cross-sections of the membranes were viewed using Jeol JSM-840A scanning electron microscope.

Purification dextran: Rejection of dextran can be calculated with the following equations[20].

$$\text{Dextran rejection (\%)} = \left[1 - \left(\frac{C_p}{C_f}\right)\right] \times 100 \quad (4)$$

where, C_p = concentration of permeate; C_f = concentration of feed.

Pore statistics: The average pore radius ($\overline{R}$), surface porosity or porosity percentage and number of pores of cellulose acetate membranes were determined by ultrafiltration of dextran of different molecular weights. The analysis of dextran was performed[15,21] with an UV spectrophotometer at λ_{max} = 485 nm.

Average solute radii, known as stoke radii, can be evaluated according to the procedure developed by Sarbolouki[21]. From the values of SR % (the average solute radius), was derived from the Sarbolouki equation, which is constant for each molecular weight of dextran.

$$\overline{\alpha} = \frac{0.096M^{0.59} + 0.128M^{0.5}}{2} \quad (5)$$

Average pore radius ($\overline{R}$) can be calculated using the following equation.

$$\overline{R} = 100\left(\frac{\overline{\alpha}}{\%SR}\right) \quad (6)$$

The surface porosity (ε), of the membrane was calculated by the orifice model given below assuming that only the skin layer of the membrane is effective in separation[21].

$$\varepsilon = \frac{3\pi\mu J_w}{\Delta P \overline{R}} \times 100 \quad (7)$$

where μ is the viscosity of the permeate water in (Pa.s), J_w is the pure water flux of the membrane in ($m^3/m^2.s$), $\overline{R}$ is the average pore radius in (Å) and ΔP is the transmembrane pressure in (Pa).

From the values of ε and $\overline{R}$ the number of pores (n), per unit area (m^2) can be calculated from the following expression[21].

$$n = \frac{\varepsilon}{\pi \overline{R}^2} \quad (8)$$

Purification of sugarcane juice: Sugarcane juice is obtained from Vellore co-operative sugar mills, Ammundi. It is then subjected to clarification by adding lime. The clear super latent liquid is taken without any disturbance and collected in a 1000 mL measuring jar. The concentration of the total sugars in the feed and permeate streams were determined by collecting samples and analyzing through pol measurement[22]. It may also be noted that on a dry basis, the total concentration of sucrose and dextrose exceed 85 % of the total dissolved solids . The raw juice is treated with milk of lime under constant stirring to raise the pH from around 5 to 8. The liming was carried out at room temperature (about 30 °C). Experiments revealed that 2.3 g of lime was required to raise the pH to 8. The treated juice was then kept undisturbed for around 2 h to facilitate the settling of solids. The clear juice is then taken without any suspended solids and analyzed for brix by using a refractometer and for pol by using a polarimeter. The sugar juice was passed through the cellulose acetate membranes at a pressure of 276 kPa and permeate was analyzed using brix and the pol readings. To make membrane process viable, it is desirable to retain maximum amount of sucrose.Therefore, it is essential to estimate the retention of solids (total dissolved solids) and sugars by measuring the brix and pol of the feed and permeate solutions. The observed retention for total dissolved solids ($R°_{TDS}$) and sugars ($R°_s$) were calculated by following equation[21].

$$R^O_{TDS} = 1 - \left(\frac{C_p}{C_f}\right) \quad (9)$$

$$R^O_S = 1 - \left(\frac{C_{pol\,p}}{C_{pol\,f}}\right) \quad (10)$$

$$\text{Purity} = \frac{\text{Pol}}{\text{Brix}} \times 100 \quad (11)$$

$$\%\ \text{Pol} = \frac{2 \times 26 \times \text{Pol reading}}{99.913 \times \text{Sp.gr}} \quad (12)$$

RESULTS AND DISCUSSION

Effect of cellulose acetate and PEG composition on pure water flux: Cellulose acetate membranes were prepared with different compositions of cellulose acetate and PEG 600 (Tables 1 and 2) and subjected to hydraulic compaction at 414 kPa TMP. The compaction was carried out for 5 h in ultrafiltration stirred cell to attain steady-state flux and the flux of pure water was measured at every 1 h interval. The 20 % of cellulose acetate membrane was found to have very low flux at beginning of compaction itself and upon further compaction, a negligible amount of water flux was observed. However, 15 % cellulose acetate membrane was subjected to compaction at 414 kPa TMP (transmembrane pressures), showed (Fig. 1) an initial flux of 217.5 L m^{-2} h^{-1}. Upon compaction, the walls of the pores become closer, denser and uniform which results in the reduction of pore size and consequently the flux[23]. The increase in flux upon decrease composition cellulose acetate may be due to the reduced polymer rich phase and formation of in homogeneity and second phase separation, which results in the formation of voids between the polymer components[24]. Additives used in membrane preparations in general are hydrophilic and hygroscopic in nature. Such additives are chosen to improve the pore statistics and morphology of the membranes. Thus, polyethylene glycol 600 was added to the cellulose acetate casting solutions of all compositions, the 2.5 wt. % was compatible extent of total cellulose acetate composition (Fig. 2). The polymeric additive was also expected to offer enhancement in pure water flux without loss of the rejection efficiency of the membranes. Hence, in this investigation, the cellulose acetate membranes with 2.5 % additive concentrations were subjected to compaction.

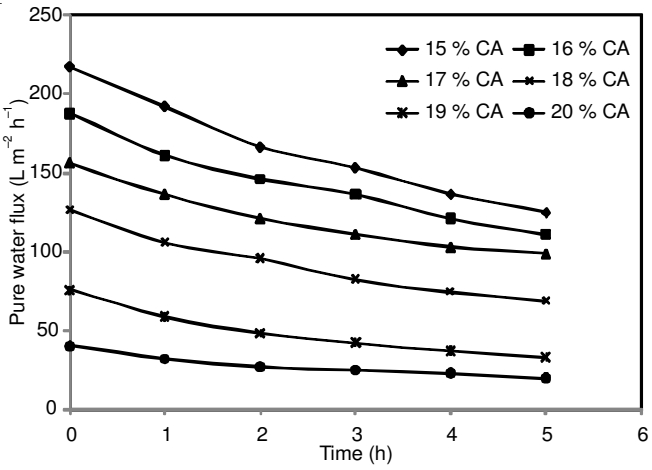

Fig. 1. Effect of pure water flux on time of cellulose acetate (CA) membranes without PEG 600

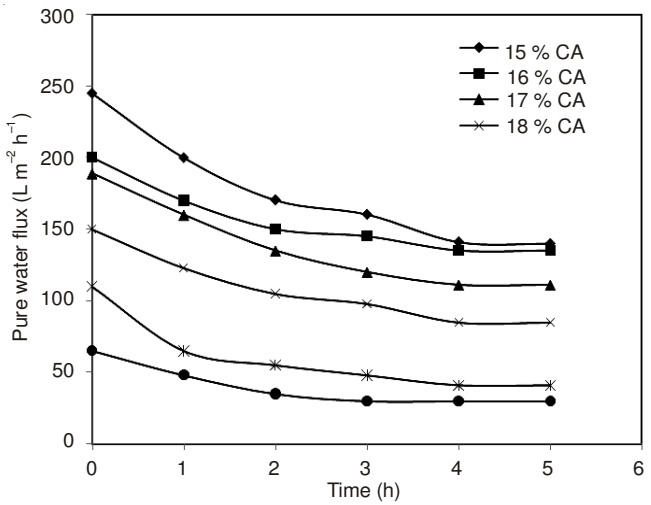

Fig. 2. Effect of pure water flux on time of cellulose acetate (CA) membranes in presence of PEG 600

Effect of cellulose acetate and PEG composition in transmembrane pressure: Variation of pressure or the intermediate pressure ranges are important conditions to be studied for ultrafiltration operations. The membranes were subjected to different transmembrane pressures from 69 to 414 kPa and the corresponding pure water fluxes were measured. The plot of pressure *versus* pure water flux gives a linear relationship and the inverse of slope is the membrane hydraulic resistance.

From Fig. 3, it is evident that as TMP was increased the PWF also increased cellulose acetate composition membranes prepared in absence of pore former PEG 600. Table-1 shows the membrane hydraulic resistance of cellulose acetate membranes with 15 to 20 % compositions. As cellulose acetate composition was increased from 15 to 20 %, the hydraulic resistance has decreased from 20.9 to 3.9 kPa L m^{-2} h^{-1}. The decrease in membrane resistance may be due to the reducing content of cellulose acetate and increasing amount of DMF in casting solution, which forms a porous gap in cellulose acetate polymer matrix, which can be directly related to the reduction in resistance toward hydraulic pressure. Further, in the absence of PEG, the hydraulic resistance was found to be lower than that for the all cellulose acetate membranes. The linear relation between pressure and flux is depicted in Fig. 4.

Effect of cellulose acetate and PEG composition on water content: Water content of the membranes prepared using different concentrations of cellulose acetate with 2.5 % PEG in casting solution is given in Table-1. The water content for the 20 % of cellulose acetate membrane is 71 %. The same

TABLE-2
MEMBRANE CASTING COMPOSITION, PWF, WATER CONTENT AND PORE ARCHITECTURE OF CA MEMBRANES WITH PEG 600

Type	CA (%)	PEG 600 wt. (%)	Solvent (DMF) (mL)	PWF at 345 kPa (L/m^2h)	Water Content (%)	R_m (kPa L^{-1} m^2 h)	$\overline{R}$ # (Å)	$N^{\pm}$ (× 10^{12})	ε* × 10^{-5} (%)
CA-P1	15	2.5	82.5	25	91	3.2	83	11.45	11.69
CA-P2	16	2.5	81.5	21	88	3.9	69	10.21	9.18
CA-P3	17	2.5	80.5	17.9	84	5.4	61	9.78	8.56
CA-P4	18	2.5	79.5	12.5	81	7.2	57	9.01	6.14
CA-P5	19	2.5	78.5	7.6	80	9.5	49	8.12	5.53
CA-P6	20	2.5	77.5	5.5	76	18.4	41	6.32	4.01

#Average pore radius, ±number of pores per 1 m^2 area, *surface porosity; CA = Cellulose acetate

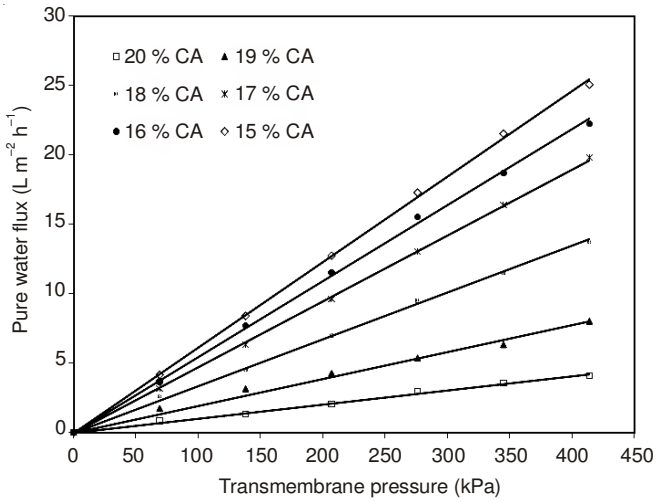

Fig. 3. Effect of TMP on pure water flux of cellulose acetate (CA) membranes without PEG 600

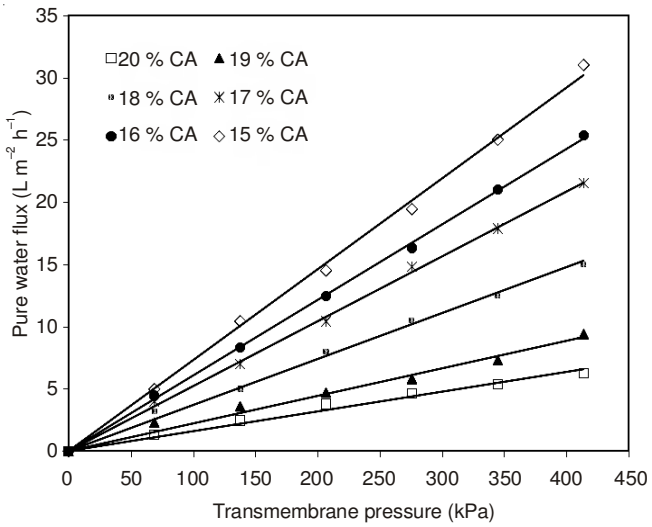

Fig. 4. Effect of TMP on pure water flux of cellulose acetate (CA) membranes in presence of PEG 600

20 % of cellulose acetate membrane with 2.5 % PEG in the casting solution, the water content is found to be 76 %. It is observed that the water content of the membranes increases with the decrease in cellulose acetate composition (Table-1) and addition of 2.5 % of PEG (Table-2) in the casting solution. The water content actually represents the fraction of water molecules occupied in the pores of the membrane. An increase in the water content indicates that the membrane has become more porous. The increasing water content with the increase in the concentration of PEG in the casting solution may be attributed to the formation of macrovoids by leaching out of PEG from the membrane forming system during gelation, which becomes the domain of water molecules[25].

Effect of cellulose acetate and PEG composition on pore architecture of membranes: Pore size, porosity and number of pores of the membranes determined from the dextran rejection studies are shown in Tables 1 and 2. It is evident from these results that the membrane prepared from higher concentration cellulose acetate and in absence of PEG has relatively smaller pore size and porosity as well as number of pores. The reduction of cellulose acetate by increasing solvent (DMF) had higher pore size and porosity as well as number of pores. Addition of 2.5 % PEG into the casting solution induced the formation of more number of pores in the skin with relatively bigger pore size. Increase in the pore size and number of pores, in principle, would lead to the increase in the permeate flux of the membrane. This is in agreement with the results obtained in this study. Further, reduction of cellulose acetate resulted in the increase of the pore size and porosity, while the number of pores does not follow a uniform trend. It should be noted that the number of pores has increased to a highest value of 9.98×10^{12} for a cellulose acetate content of 15 %, while the porosity and pore size increased to a maximum of 11.45×10^{12} and 83 Å, respectively for a 2.5 % of PEG content in 15 % of cellulose acetate . Although bigger pore size will favor high permeate flux of sugar solution, the solute rejection will drastically fall. The membrane best suited for any application would be the one that has more number of pores with smaller size[19]. Because smaller size of pores will favor better retention and higher number of pores will favor high permeate flux. Hence, it seems that a 2.5 % PEG concentration of 15 % cellulose acetate in the casting solution would provide high permeate flux with high solute retention.

Morphological studies: To develop high performance polymeric membranes, it is essential to design the molecular and morphological structure of the membrane for their specific applications[21]. Scanning electron microscope is a powerful tool to investigate the structure of asymmetric membranes. The top surface and cross sectional views of the cellulose acetate (CA) and CA/PEG membranes are shown in Fig. 5. From Fig. 5, 19 wt. % cellulose acetate membrane without PEG is showing homogenous dense top layer. The cross section of the membranes can be distinguished in to three layers *viz*., the top layer, the sublayer and in between these two, the intermediate layer[26]. From Fig. 5, it is clearly seen that the pore size as well as the structure has significantly enhanced by adding 15 wt. % cellulose acetate. From Fig. 5 (15 % cellulose acetate with 2.5 PEG 600) shows a dense skin layer at the top with small pore size, immediately beneath it, a thin intermediate layer with relatively medium pore size and underneath of it, a thick sublayer having relatively bigger pores of fingerlike structure. As the concentration of cellulose acetate in the casting solution is decreased to 15 wt. %, the intermediate layer has significantly grown in thickness and the pore size in the sublayer has also increased. However, as the concentration of PED at 2.5 wt. % added, the thickness of intermediate layer reduces progressively and the formation of macro voids increases in the sublayer. Further, it is also seen that the interconnectivity of polymer matrix is reduced and the thickness of the walls surrounding the pores is also decreasing. This should result in increased permeability for the membranes, especially prepared using 2.5 wt. % of PEG. The results from this study support this reasoning.

Purification dextran solution: Dextran passes through the membrane because they become deformed by the shear in the pores and have a small cross section normal to the direction of flow. The globular proteins cannot deform and always have a relatively large cross section normal to the direction of flow. The retention of dextran in batch cells stirred at an increased markedly with increasing cellulose acetate composition (Figs. 6-9). It is

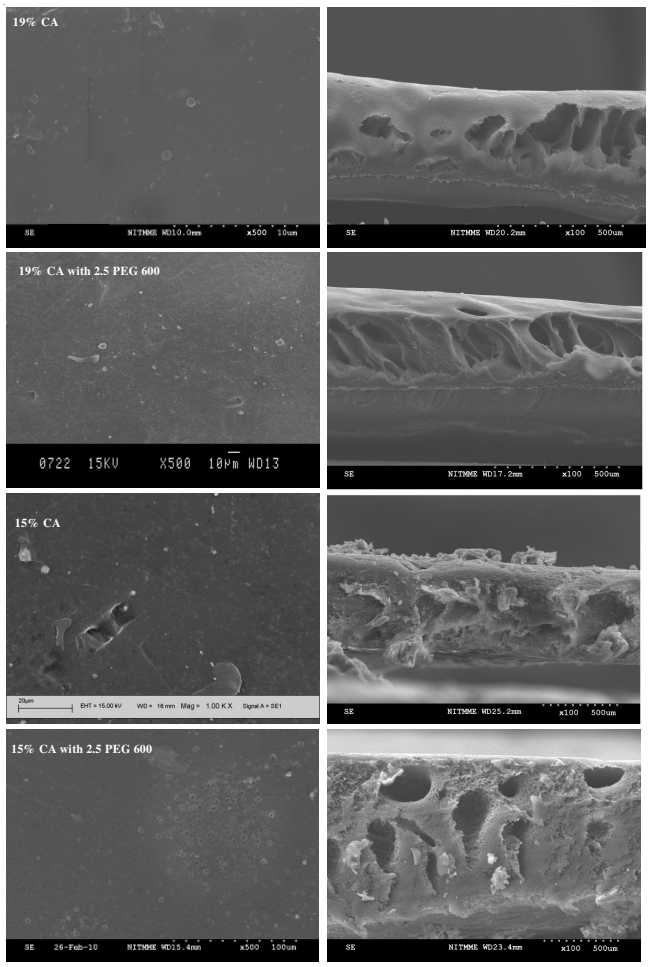

Fig. 5. SEM image of cellulose acetate and CA/PEG 600 membarnes with various compositions

believed that this effect is the result of two processes. At low pressures the flux increases with pressure and consequently the amount of shear deformation increases as the pressure increases. Further, the retention therefore falls with increased cellulose acetate composition. At higher pressures the gel layer formed at the membrane surface makes the flux independent of pressure. In this region increased pressure may compress the gel, resulting in a higher concentration of macromolecules at the membrane surface. This also lowers the flux as the pressure increases. Fig. 7 shows that the permeation of dextran of the cellulose acetate membranes decreases when the composition is enhanced (15 to 20 %). The separation performance for a particular solute increases with a decrease in composition, which means cellulose acetate membranes becomes apparently thinner or lesser with decrease in composition. Once the permeation of dextran reaches a steady state, it shows that some solute formed gel layer concentration due to concentration polarization[27]. This result is consistent with the observed morphology (Fig. 5). This is also due to the fact that molecular weight of solutes is more sensitive to surface morphology. The permeation and rejection values had a change in magnitude when PEG was added into the casting solutions of cellulose acetate blend membranes (Fig. 8). Thus, upon 2.5 addition of additive, all cellulose acetate membrane composition showed an increase in value of permeate flux. However, when the concentration was increased up to 20 % of cellulose acetate,

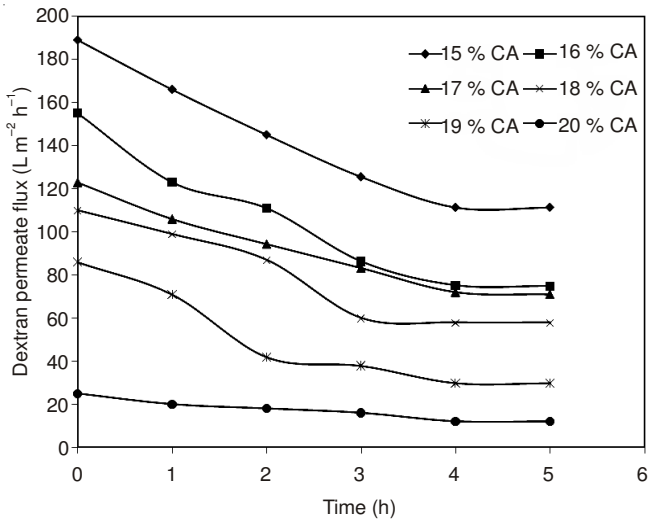

Fig. 6. Effect of dextran flux on time of cellulose acetate (CA) membranes without PEG 600

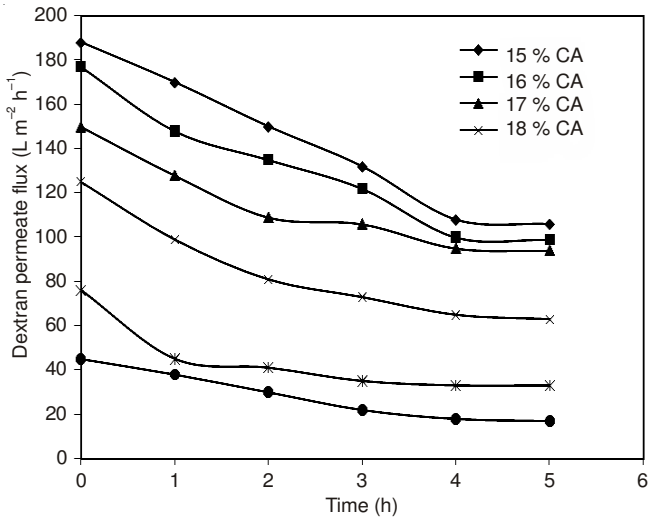

Fig. 7. Effect of dextran permeate flux on time of cellulose acetate (CA) membranes in presence of PEG 600

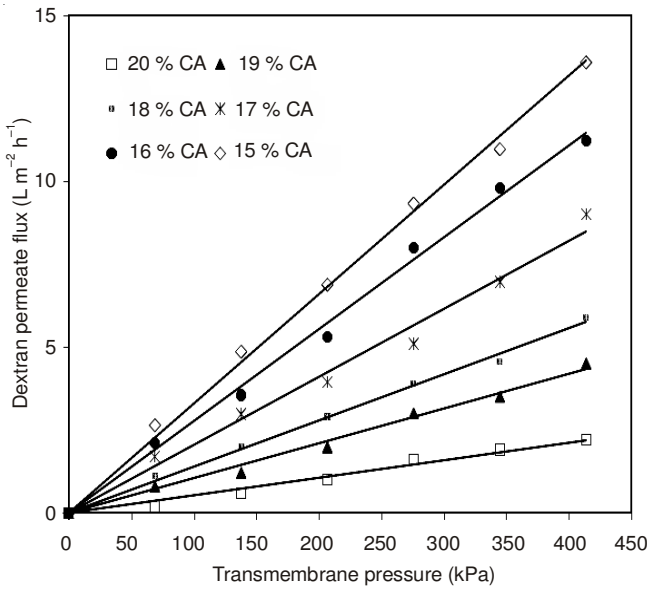

Fig. 8. Effect of TMP on dextran flux of cellulose acetate (CA) membranes without PEG 600

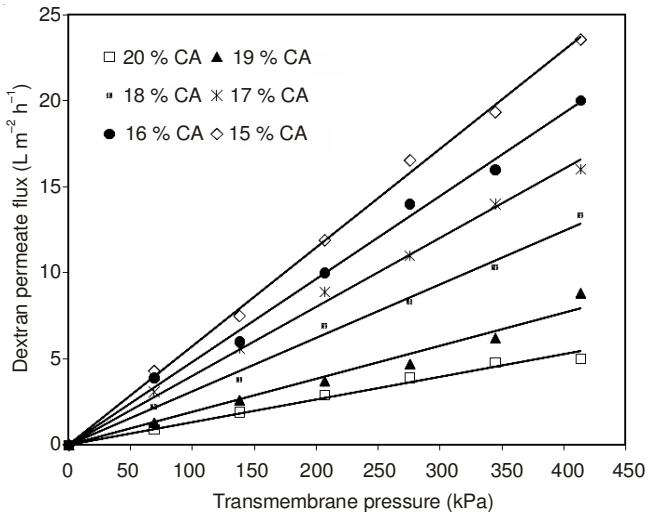

Fig. 9. Effect of TMP on dextran flux of cellulose acetate (CA) membranes in presence of PEG 600

there was appreciable change in the value (5.17 L m^{-2} h^{-1}), since the percentage separation of dextran 42 kDa itself fell in the least needed 80 % separation.

Concentration of sugarcane juice: Retention of sugar should be as less as possible and that of other solids should be high enough so that the permeate flux is rich in sugars only. It is clear from Table-3, the membranes cellulose acetate without PEG retains maximum and cellulose acetate with PEG retains minimum sugars. In between the membranes, cellulose acetate-1 and cellulose acetate-P1, the membrane cellulose acetate-P2 retains minimum sugar. Similar trends for the retention of total dissolved solids can also be observed from Tables 3 and 4. Different samples of sugarcane juice were taken, analyzed for brix and pol and ultrafiltration was carried out for cellulose acetate and CA/PEG membranes. The juice has brix content between 10 -15 %. The juice was passed through the membrane at a constant pressure of 414 kPa. The permeate flux was analyzed for brix and pol. The performance of the membrane to retain the solids was also measured. The retention of sucrose and solids were calculated for all the membranes at 414 kPa. Since the solids also play a key role during evaporation, retention of solids were also measured. After the analysis, the ultrafiltration stirred cell was dismantled and the membrane was washed thoroughly using distilled water and replaced. For 15 % cellulose acetate without PEG, the retention of solids was 12% and sucrose was 7.13 % at 414 kPa. However, cellulose acetate membrane with an additive of PEG of 2.5 wt. %, the retention of solids was 16.5 % and retention of sucrose was 14.36 at 414 kPa. This was because of the increase in the size of the pores due to addition of PEG-600 in casting solution during the preparation of cellulose acetate membranes[28]. But there was a better flux due to larger pore size in the membranes (Figs. 10-13).

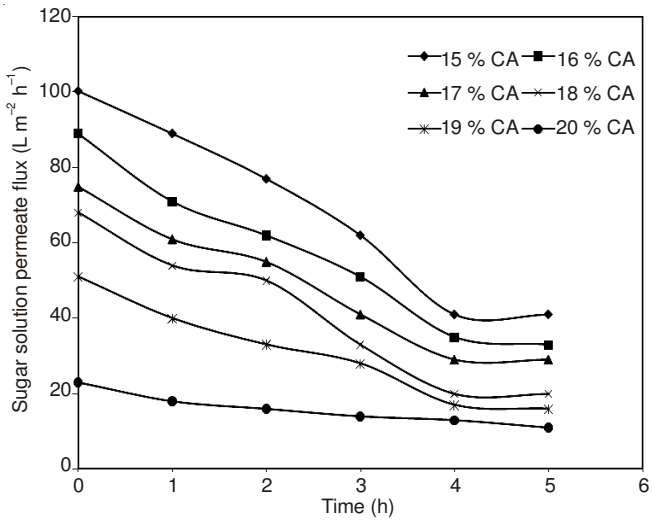

Fig. 10. Effect of sugar solution flux on time of cellulose acetate (CA) membranes without PEG 600

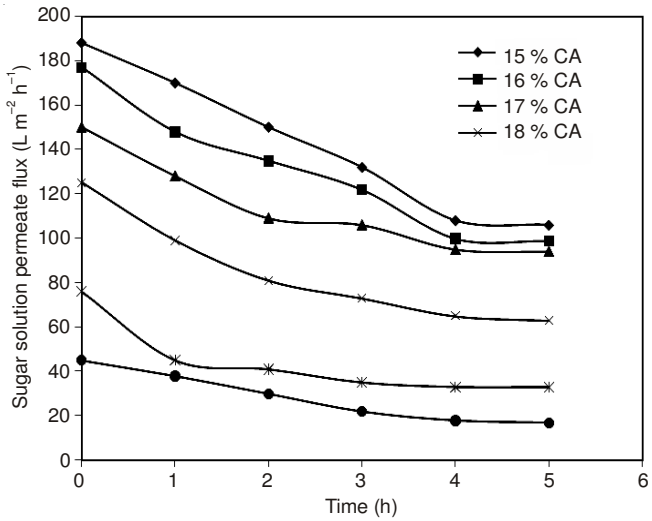

Fig. 11. Effect of sugar solution permeate flux on time of cellulose acetate (CA) membranes in presence of PEG 600

In order to increase the retention, the wt. % of cellulose acetate was increased from 16 to 20 wt. % and the same experiments were carried out with and without additive. It was found that there was a better retention of sucrose and solids compared to that of 15 wt. % cellulose acetate. It was found

TABLE-3
DEXTRAN AND SUGAR REJECTION OF CELLULOSE ACETATE MEMBRANES WITHOUT PEG 600

Type	Feed of sugar solution				Permeate of sugar solution						Retention of Dextran
	Brix (%)	Pol	Pol (%)	Purity (%)	Brix (%)	Pol	Pol (%)	Purity (%)	Retention of solids (%)	Retention of Sucrose (%)	
CA-P1	14.5	46	11.3	78.27	12	33.2	9.72	81	16.5	14.36	60
CA-P2	14.5	43	10.6	73.3	11.5	35.6	8.74	76	20.68	17.84	65
CA-P3	14.5	46	11.3	78.27	11.5	36.1	9.24	80.34	21.37	18.5	71
CA-P4	14.5	43	10.6	73.3	10.5	34.1	8.40	80	26.2	21.03	76
CA-P5	14.5	46	11.3	78.27	10.5	33.2	8.5	80.95	28.96	25.11	85
CA-P6	14.5	43	10.6	73.3	9.0	30.4	7.44	82.6	35.8	30.0	87

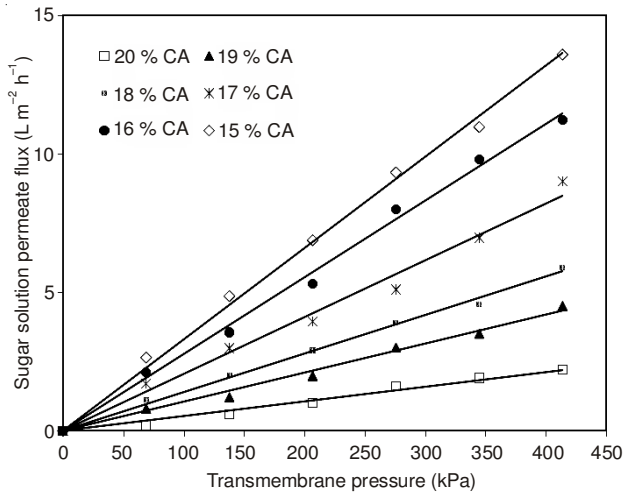

Fig. 12. Effect of TMP on sugar solution flux of cellulose acetate (CA) membranes without PEG 600

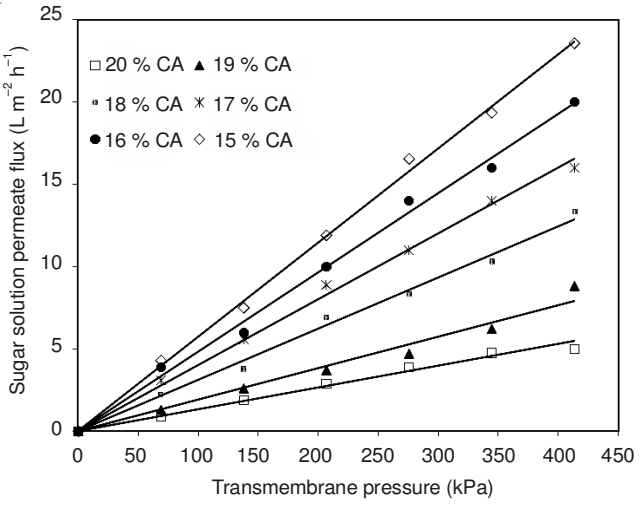

Fig. 13. Effect of TMP on sugar solution flux of cellulose acetate (CA) membranes in presence of PEG 600

that in Tables 3 and 4, the retention of sucrose increases with pressure was clearly seen. To further enhance the retention, the PEG 600 was added to all cellulose acetate membranes. The results show with the increase in pressure the retention increases, as the solids form cluster and agglomerate due to pressure[29]. From the Tables 3 and 4, it is evident that the membrane cellulose acetate-P6 provides the highest recovery of sugar. Therefore, among the 12 membranes studied herein, cellulose acetate-P6 may be the suitable choice for the treatment of the limed cane juice. In the context of present study, retention of sugars as well as that of total dissolved solids is important issues. The general trend can be observed that retention of solutes increases with pressure for a fixed feed concentration. At higher pressure, the polarization layer becomes more compact. This reduces the permeability of this layer and consequently, the solutes are retained more and therefore, the retention of total solids and sugars increase with the operating pressure.

Conclusion

In this work, cellulose acetate as a matrix polymer was prepared with varying composition and 2.5 wt. % addition of PEG 600 using DMF for ultrafiltration membranes. It was found that cellulose acetate polymer solutions having 20 wt. % had lower performance compared to 15 wt. % of cellulose acetate membranes. Further, the performance increased when 2.5 wt. PEG added in all cellulose acetate membranes. The characterization of prepared membranes illustrates that the pure water flux and water content were increased while the membrane hydraulic resistance was decreased, as 2.5 wt. % of PEG in the casting solution is added. The pore architecture results obtained from dextran rejection studies demonstrate that pore radius and porosity show significant increase as the concentration of cellulose acetate in the casting solution increased. The scanning electron microscopic study shows a continuous increase in the pore size in the sublayer and subsequent decrease in the thickness of intermediate layer, as the concentration of PEG in the casting solution increased. The 15 wt. % of cellulose acetate and 2.5 wt. % of PEG in same cellulose acetate achieved reasonably high flux, low sugars retention and higher retention of other dissolved solids. Membrane cellulose acetate-P1 provided better performance in terms of recovery of sugars. The experimental evidence of gradual decline of permeate flux of limed cane juice over time indicates a sluggish growth of polarized layer over the membrane surface. This work may be useful for adequate description of flux decline of a complex industrial feed solution like limed cane juice. Furthermore, it is clear that the adsorption was influenced by dextran concentration and time of exposure and the main process seemed to be the binding of dextran on the surface in the cellulose acetate amd CA/PEG membranes pores contributed to the overall effects. The rejected dextran accumulates at the membrane surface, which lowers the flux at 5 h of ultrafiltration. By adding a PEG to a cellulose acetate polymer solution, forms pores on the surface of membranes which normally do not effect a separation. The dextran gel layer may be overcome by lowering the concentration or the pressure. The results of this study will be useful to design appropriate membranes for the required performance in industrial applications.

TABLE-4
DEXTRAN AND SUGAR REJECTION OF CELLULOSE ACETATE MEMBRANES WITH PEG 600

Type	Feed of sugar solution				Permeate of sugar solution						Retention of Dextran
	Brix (%)	Pol	Pol (%)	Purity (%)	Brix (%)	Pol	Pol (%)	Purity (%)	Retention of solids (%)	Retention of Sucrose (%)	
CA-1	13	40.2	9.9	76.15	11.5	37	9.2	80	12	7.13	73
CA-2	15	41	10.1	66.7	13	37.2	9.16	70.53	13.0	9.3	77
CA-3	15	41	10.1	66.7	12.5	36.5	8.93	71.42	17.0	12.0	81
CA-4	12.5	41	10.2	81.92	10.5	36.2	9.01	85.80	16.2	12.11	86
CA-5	12.5	41	10.2	81.92	10.5	35.1	8.77	83.5	21.0	14.35	88
CA-6	12.5	40.3	9.97	79.76	8.5	29.3	7.33	86.23	32.0	14.4	92

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