# Performance Characterization of Cellulose Acetate and Poly(vinylpyrrolidone) Blend Membranes

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**ABSTRACT:** Hydrophilic ultrafiltration membranes have been prepared by blending cellulose acetate (CA) as a matrix polymer with increasing concentrations of poly(vinylpyrrolidone) (PVP) using $N,N'$-dimethylformamide as the solvent. It is observed that the presence of PVP beyond 50 wt % in the casting solution did not form membranes. Prepared membranes have been subjected to ultrafiltration characterizations such as compaction, pure water flux, water content, and membrane hydraulic resistance. The results indicate significant changes in the characteristics upon the addition of PVP, which may lead to improved performance. The porosity, pore size, and molecular weight cut-off of the membranes also increase as the concentration of PVP increases. It is estimated that the pore radius of the CA/PVP membranes increases from 30 to 63 Å, when the concentration of PVP increased from 0 to 50 wt %. This is in agreement with the results obtained from scanning electron microscopic studies. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 104: 3042–3049, 2007

**Key words:** membrane; compatibility; blending; additive; phase separation

## INTRODUCTION

Polymeric materials can be modified to improve the properties either by blending or by copolymerization with another polymer. Melt blending and solution blending are the two types of polymer blending.[1] Membrane technology currently employs a wide range of polymeric materials, and continual development of new membrane materials is crucial to sustain and expand the growing interest in the technology.[2,3] In recent years, the solution blending of polymer materials assumes an attractive technique to prepare various membranes of desired properties. The capability of an asymmetric membrane to reject or admit a species is determined by the morphological structure and properties of the membrane. Accordingly, it is necessary to prepare asymmetric membranes with desired properties to achieve the required performance.

Cellulose acetate (CA)-based polymers are always preferred over other membrane materials because of 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 nontoxicity.[4–7] Hence, in this investigation, attention is focused on CA-based membranes. Poly(vinylpyrrolidone) (PVP) is one of the good polymeric additives widely used in the preparation of phase inversion membranes in view of the fact that PVP is compatible with many membrane materials and is quite well soluble in many solvents as well as nonsolvent and water.[8–10] The effects of PVP additive on membrane formation have been thoroughly studied for polymers like polysulfone, polyethersulfone, polyimide, etc.[11–13] However, studies on the effect of PVP as a blend polymer on membrane formation with CA are very limited.

The effect of additive on the structure and properties of polymeric membrane is strongly dependent on a number of factors such as the type of solvent and nonsolvent used in membrane preparation, molecular weight of the additive and concentration of the additive.[14–17] In an earlier study,[18] polyacrylonitrile (PAN) has been blended with high molecular weight PVP using $N,N'$-dimethylformamide (DMF) as the solvent. The results of this study reveal that the use of high molecular weight PVP provides lower flux membranes, indicating that PVP was not completely leached out from the membrane and the residual PVP entrapped during fabrication leading to swelling and subsequent blocking of the pores during the ultrafiltration. However, a low molecular weight PVP easily leaches out from the membrane and forms uniform small pores in the membrane

**TABLE I**
**The Composition, Water Content, and Membrane Hydraulic Resistance of Cellulose Acetate and PVP Blend Membranes**

| Polymer (17.5 wt %) | | Solvent (wt %) | Water content (%) | $R_m$ (kPa $\cdot$ l$^{-1}$ $\cdot$ m$^2$ $\cdot$ h) |
| --- | --- | --- | --- | --- |
| CA (%) | PVP (%) | | | |
| 100 | 0 | 82.5 | 81.8 | 84.54 |
| 95 | 5 | 82.5 | 83.2 | 37.98 |
| 90 | 10 | 82.5 | 84.6 | 24.96 |
| 85 | 15 | 82.5 | 85.3 | 16.53 |
| 80 | 20 | 82.5 | 86.5 | 12.96 |
| 75 | 25 | 82.5 | 87.6 | 11.55 |
| 70 | 30 | 82.5 | 88.6 | 10.30 |
| 65 | 35 | 82.5 | 89.2 | 09.18 |
| 60 | 40 | 82.5 | 90.5 | 08.94 |
| 55 | 45 | 82.5 | 91.6 | 07.29 |
| 50 | 50 | 82.5 | 92.7 | 05.22 |

thereby leading to high permeation flux with good solute rejection.[19] Hence, in the present investigation, a low molecular weight PVP ($M_w$ = 40 kDa) was chosen to blend with CA using $N,N'$-dimethylformamide (DMF) as the solvent.

A previous study by Sivakumar et al.[20] reported the preparation of CA/PU blend membranes with 0–7.5 wt % PVP as additive. They found enhancement in the performance of CA/PU blend membranes on increasing the concentrations of PVP. However, they have not carried out detailed studies by varying the additive concentration to the maximum possible extent, beyond which the polymeric blends may be incompatible or the membrane formation will be poor. Hence, in this investigation, an attempt has been made to prepare membranes in all compatible compositions of CA/PVP. To the best of our knowledge, such a study has not been attempted earlier with CA and PVP polymers. Prepared ultrafiltration membranes were characterized for compaction, pure water flux, membrane hydraulic resistance, and water content. The surface properties such as molecular weight cut-off, average pore size, porosity, and number of pores were estimated using aqueous solutions of proteins of different molecular weight as feed. The cross-sectional morphologies of the prepared membranes were also studied by scanning electron microscope (SEM).

## MATERIALS AND METHODS

### Materials

Commercial grade cellulose acetate (CA) was procured from Mysore Acetate and Chemical, Mysore, India. The CA was recrystallized from acetone and then dried in a vacuum oven at 70°C for 24 h prior to use. Poly(vinylpyrrolidone) (PVP)was procured from SRL, India. $N,N'$-dimethylformamide (DMF) and sodium lauryl sulfate (SLS) were obtained from Qualigens Fine Chemicals, Glaxo India, India, which were of analytical grade. DMF was sieved through molecular sieves (Type-4 Å) to remove moisture and stored in dry condition prior to use. Acetone (AR) of analytical grade procured from SRL, India was used as such. Disodium hydrogen orthophosphate anhydrous and monosodium dihydrogen orthophosphate heptahydrate were procured from CDH Chemicals, India. Proteins viz., bovine serum albumin ($M_w$ = 69 kDa), pepsin ($M_w$ = 35 kDa), and trypsin ($M_w$ = 20 kDa) were purchased from SRL, India. Deionized and distilled water was used throughout the study.

### Formulation of polymer casting solution

The polymer casting solutions based on CA and PVP polymers at a total polymer concentration of 17.5 wt % were prepared by dissolving them in DMF under constant mechanical stirring in a two-necked round bottom flask for 3 h at 40–45°C. A series of such polymer solutions were prepared by varying the composition of CA and PVP with an increment of 5 wt % of PVP, as shown in Table I. The homogeneous polymer solutions were allowed to stand for 1 h before casting to eliminate air bubbles.

### Preparation of membranes

Membranes were prepared from the deaerated polymer solutions following the standard method of phase inversion technique. The polymer solution was first cast on a smooth glass plate with the help of a doctor blade. The thickness of the membrane was maintained at 0.22 ± 0.02 mm with the help of an oil sheet rolled at both the ends of the blade as followed in earlier studies.[20,21] During casting, the relative humidity (RH) of the casting chamber was maintained at 50% and the temperature was kept at (20 ± 2)°C. Prior to casting, a gelation bath of 2 L consisting of 2.5% (v/v) DMF and 0.2 wt % SLS in

distilled water (non solvent) was prepared and the bath was ice-cooled to (18 ± 2)°C.[22] After 30 min of solvent evaporation in the casting chamber, the glass plate along with the polymer film was immersed in the gelation bath. After an hour of gelation, the membrane was removed from the gelation bath and thoroughly washed with distilled water to remove the residual solvent and surfactant from the membrane. The membrane sheet was subsequently stored in distilled water containing 0.1% of formalin solution to prevent microbial growth. Similar casting and gelation conditions were maintained for all the membranes.

## Characterization of membranes

All the permeation experiments were carried out in a batch type, dead end stirred ultrafiltration cell (UF cell-S76-400-Model, Spectrum, USA) with a diameter of 76 mm, fitted with Teflon-coated magnetic paddle.

### Compaction

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 UF cell and pressurized with distilled water at a transmembrane pressure of 414 kPa. The pure water flux was measured every hour 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 following equation:[20]

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

where $J_w$ = pure water flux (L m$^{-2}$ h$^{-1}$); $Q$ = quantity of pure water permeated (L); $A$ = membrane area (m$^2$); $\Delta T$ = sampling time (h).

### Pure water flux

The pure water flux is a measure of hydrophilicity of a membrane, and from the knowledge of pure water flux, we can predict the hydrophilicity of the 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 eq. (1).

### Water content

Membrane samples were cut into desired size and soaked in water for 24 h and weighed immediately

after blotting the free surface water. These wet membranes were dried for 12 h at (100 ± 5)°C and the dry weights were determined. From the dry and wet weights of the samples, the percent water contents were calculated using the equation given elsewhere.[23]

### Membrane hydraulic resistance ($R_m$)

To determine the membrane hydraulic resistance ($R_m$), the pure water flux of the membranes was measured at different transmembrane pressures ($\Delta P$) viz., 69, 138, 207, 276, and 345 kPa. The variation of pure water flux was plotted as a function of transmembrane 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:[20]

$$R_m = \frac{\Delta P}{J_w} \qquad (2)$$

### Molecular weight cut-off

Molecular weight cut-off (MWCO) is an attribute of pore size of the membranes and is related to the rejection of a spherical solute of given molecular weight. The molecular weight has a linear relationship with the pore size of the membrane.[3] In general, the MWCO of the membrane is determined by identifying an inert solute of lowest molecular weight that has a solute rejection of 80–100% in steady state UF experiments.[15] Thus, the proteins of different molecular weights such as bovine serum albumin (69 kDa), pepsin (35 kDa), and trypsin (20 kDa) were taken for rejection studies of the membranes. Aqueous solutions of bovine serum albumin (BSA), pepsin, and trypsin were prepared at a concentration of 1000 mg/L by dissolving the proteins (0.1 wt %) individually in phosphate buffer (0.5$M$, pH 7.2). The UF cell was filled with protein solution and pressurized at a constant pressure of 345 kPa and stirred throughout the experiments to minimize fouling. During ultrafiltration, the permeate solutions of corresponding membranes were collected over a period of time in a graduated tube and were analyzed for the concentration of protein by UV–visible spectrophotometer (Shimadzu, Model UV-160A) at 280 nm. From the feed and permeate concentrations, the percentage rejection was calculated using the equation reported in previous literatures.[3,23]

### Pore statistics

The average pore size, surface porosity, and number of pores per unit membrane surface area were determined by the ultrafiltration of protein solutions of

different molecular weights. The molecular weight of the solute that has a solute rejection (SR) above 80% was used to evaluate the average pore size, $\bar{R}$, of the membranes by the following equation:[15,24]

$$\bar{R} = 100\left(\frac{\alpha}{\%\text{SR}}\right) \tag{3}$$

where $\bar{R}$ is the average pore size (radius) of the membrane (Å) and $\alpha$ is the average solute radius (Å). The average solute radii, also known as the Stoke radii, were obtained from the plot of solute molecular weight versus solute radius in aqueous solution, which was developed by Sarbolouki.[24]

The surface porosity, $\varepsilon$, of the membrane was calculated by the orifice model given below, assuming that only the skin layer of the membrane is effective in separation.[25]

$$\varepsilon = \frac{3\pi\mu J_w}{\Delta P \bar{R}} \tag{4}$$

where $\mu$ 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$^{-1}$), $\bar{R}$ is the average pore radius in (Å), and $\Delta P$ is the transmembrane pressure in (Pa).

From the values of $\varepsilon$ and $\bar{R}$, the number of pores per unit area, $n$, can be calculated from the following expression:[15]

$$n = \frac{\varepsilon}{\pi \bar{R}^2} \tag{5}$$

Morphological studies

The membranes were cut into small pieces and mopped with filter paper. These pieces were immersed in liquid nitrogen for 20–30 s and were frozen. The frozen bits of membranes were broken and kept in a desiccator. These membrane samples were used for SEM studies. The membrane samples were mounted on studs and gold-sputtered to provide electrical conductivity to very thin layer of the polymeric membrane.[26] The cross sections of the membranes were viewed using JEOL JSM-840A scanning electron microscope.

## RESULTS AND DISCUSSION

### Membrane preparation

The membranes were prepared from different compositions of polymer solutions by the phase inversion technique. During the preparation of membranes, it was found that the polymer solutions prepared at a PVP concentration more than 50 wt % did not form membrane. This is because of two factors: (i) the total membrane-forming polymer (i.e., CA) concentration in the casting solution was re-

duced below 50 wt %, in other words the polymer-rich phase became very weak in the membrane formation, which in turn lead to the inhibition of formation of membranes; (ii) the presence of PVP in very high concentrations increased the segmental gap among the CA units and when the cast film was immersed in the gelation bath, PVP was leached out from the membrane-forming system (polymer-rich phase) and consequently the polymer chain lost its integrity.[27] Thus, membranes were prepared with CA/PVP in the compositions 100/0, 95/05, 90/10, 85/15, 80/20, 75/25, 70/30, 65/35, 60/40, 55/45, 50/50 wt % for further study.

### Characterization of the blend membranes

Membrane compaction

The membranes were compacted at a transmembrane pressure of 414 kPa. The effect of compaction on PWF of membrane is shown in Figure 1 for different compositions of CA/PVP. It is observed that the flux, which is high initially, reduces on compaction and attains a steady state after 6–7 h for all the membranes. This decline in flux is apparently due to the result of a creep process in which the skin grows in thickness by amalgamation with the substrate or sublayer immediately beneath it.[4] The flux of pure CA membrane (CA 100 wt %) has decreased from an initial flux of 7.79 L m$^{-2}$ h$^{-1}$ to a steady state flux of 4.36 L m$^{-2}$ h$^{-1}$. However, membranes prepared with increasing PVP concentrations initially had a
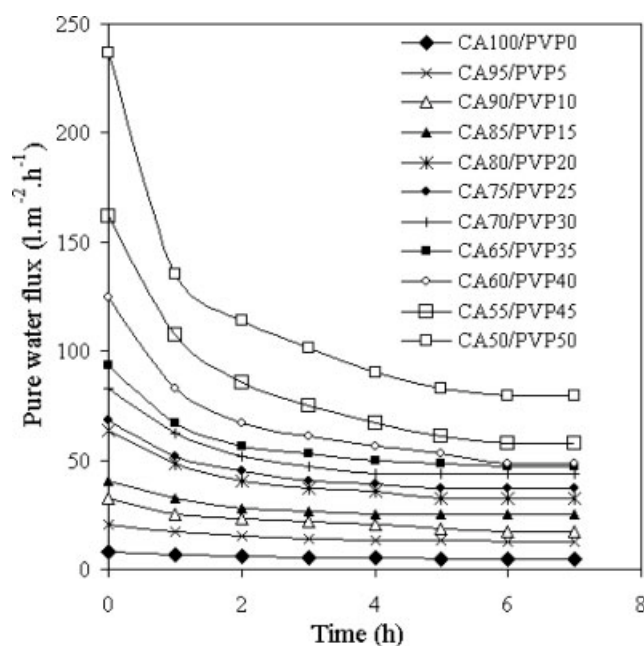


**Figure 1** Effect of pure water flux on increasing compaction time at 414 kPa for various CA/PVP blend membranes.
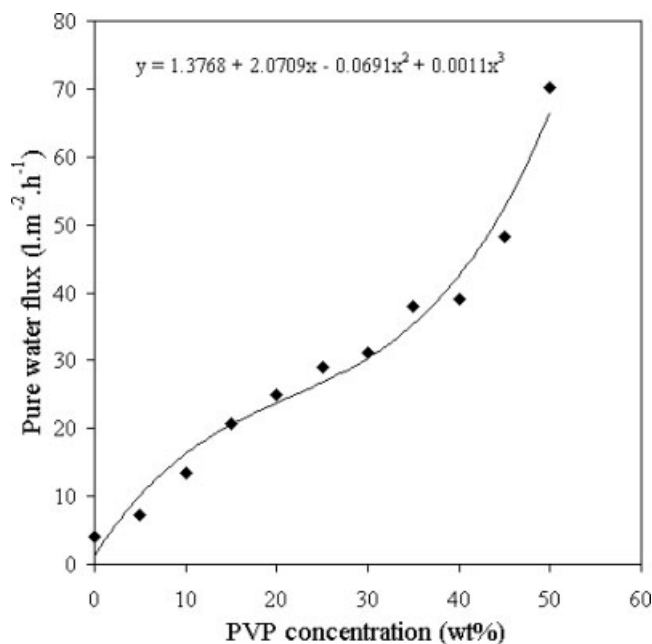
**Figure 2** Change in pure water flux on increasing PVP concentration after compaction at 345 kPa.

very high flux but it declined drastically to a lower value within a short period of time. Difference between the initial and final flux values was very high, especially for 50/50 wt % CA/PVP membrane, which had initial flux of 237 L m$^{-2}$ h$^{-1}$ and steady state flux of 79 L m$^{-2}$ h$^{-1}$ (Fig. 1). This shows that the compaction effects are more pronounced for the membranes prepared with high concentration of PVP in the casting solution. This is because of two factors such as the presence of big macrovoids and hence less interconnectivity of polymer matrix in the substrate, as it is evident from the SEM images shown in Figure 4 and lower thickness of the polymer walls surrounding the pores in the substrate.
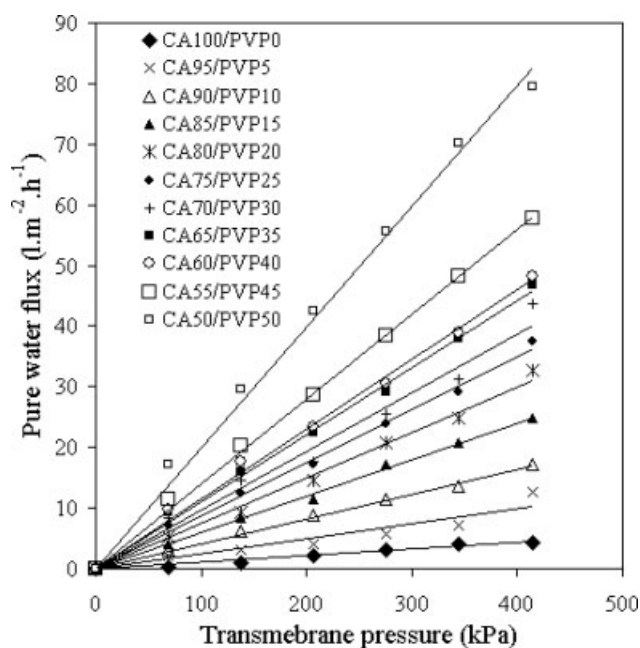
Pure water flux

After compaction, all the membranes were subjected to pure water flux at a pressure of 345 kPa. Figure 2 shows the extent of pure water flux on increasing PVP concentration. The values of pure water flux have been fitted using polynomial expression to the order of three. It is seen that the increase in the concentration of PVP results in a slow and steady increase of pure water flux up to 45 wt % PVP. This is due the fact that PVP leads predominantly to swelling rather than leaching out from the membrane-forming system. Consequently, the flow path in the membrane was reduced and hence the increase in the flux was not steep.[20] However, there is a steep increase in PWF when the concentration of PVP

increased from 45 to 50 wt %. Consequently, the PWF increases from 48 to 70 L m$^{-2}$ h$^{-1}$. This is because as the concentration of PVP increases, the hydrophilicity of the membrane increases.[28] It would be possible to find the PWF theoretically using the regression equation in Figure 2 for the PVP weight percentages not studied in this work by substituting the required PVP weight percentage. This will be helpful especially from 45 to 50 wt %, where there is a steep increase in PWF.

Water content

The water content of the membranes prepared using different concentrations of PVP in the casting solution is given in Table I. The water content for the pure cellulose acetate membrane is 82%. It is seen that the water content of the membranes increases with the increase in the PVP concentration in the casting solution. For the membrane prepared with 50 wt % PVP in the casting solution, the water content is found to be 93%. 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 PVP in the casting solution may be attributed to the formation of macrovoids by leaching out of PVP from the membrane-forming system during gelation, which becomes the domain of water molecules.[21]



**Figure 3** Effect of pure water flux on transmembrane pressure for various CA/PVP blend membranes.
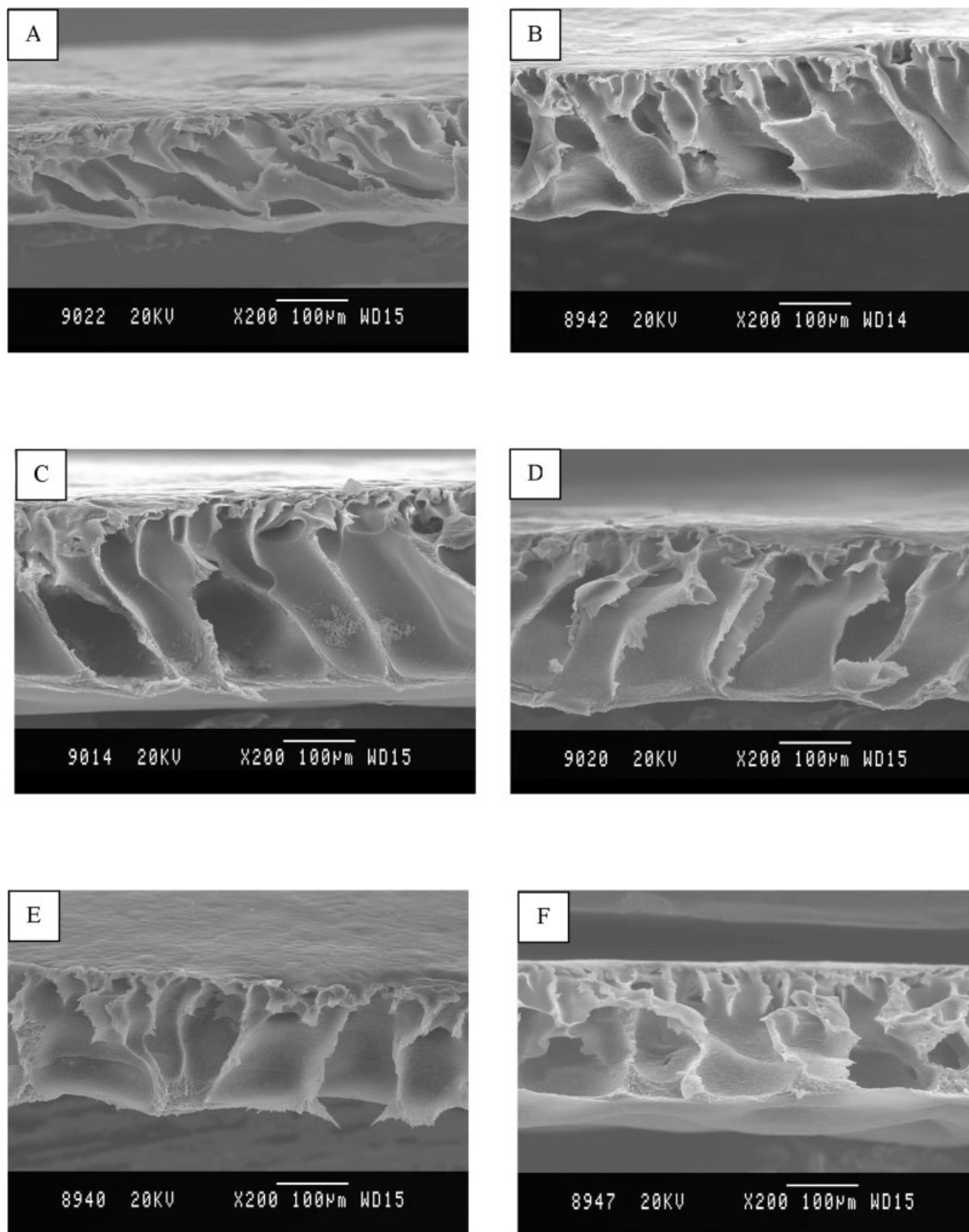
**Figure 4** Scanning electron microscopic pictures showing the cross section of the membranes: (A) CA100/PVP0, (B) CA90/PVP10, (C) CA80/PVP20, (D) CA70/PVP30, (E) CA60/PVP40, and (F) CA50/PVP50.

## TABLE II
### Protein Rejection, Molecular Weight Cut-Off, Average Pore Radius, Surface Porosity, and Number of Pores of Cellulose Acetate and PVP Blend Membranes

| Polymer (17.5 wt %) | | | | | | |
|---|---|---|---|---|---|---|
| CA (%) | PVP (%) | %SR | MWCO (kDa) | $R$ (Å) | $\varepsilon$ ($10^5$) | $n$ ($10^{-11}$) |
| 100 | 0 | 86.0 | 20 | 29.6 | 1.06 | 3.86 |
| 90 | 10 | 84.3 | 35 | 37.6 | 2.72 | 6.12 |
| 80 | 20 | 80.1 | 35 | 39.6 | 4.77 | 9.69 |
| 70 | 30 | 87.2 | 69 | 47.5 | 4.98 | 7.04 |
| 60 | 40 | 80.9 | 69 | 51.2 | 5.77 | 7.02 |
| 50 | 50 | 66.2 | >69 | 62.5 | 8.51 | 6.93 |

### Membrane hydraulic resistance ($R_m$)

The effect of concentration of PVP in the casting solution on pure water flux at different transmembrane pressures viz., 69, 138, 207, 276, and 345 kPa is shown in Figure 3. The plot depicts a linear relationship between the pure water flux and the transmembrane pressure for all the membranes.[15] Membrane hydraulic resistances of the membranes were calculated from the inverse of slopes of the corresponding flux versus pressure lines and are shown in Table I. It is evident from these values that as the concentration of the PVP in the blend system increases, the membrane hydraulic resistance decreases. This can be explained by the fact that the increase in the additive leads to the formation of pores, which in turn increases the flux thereby leading to the decrease in membrane resistance. It can be further explained by the simple fluid flow equation for laminar flow, Hagen–Poiseuille equation.[29] According to this equation, the membrane hydraulic resistance or the pressure drop divided by the flux is inversely proportional to the porosity and pore diameter of the membrane or the permeability. Thus, as the pore size and number of pores increases, the permeability increases and hence the resistance decreases.

### Molecular weight cut-off

The molecular weight cut-offs (MWCOs) of CA/PVP membranes determined individually based on the percentage rejection of proteins are shown in Table II. It can be observed that the pure CA membrane (100/0%) has a lowest MWCO value of 20 kDa, as the membrane has yielded 86% rejection for trypsin (20 kDa). It is apparent that the MWCO increases as the concentration of PVP in the casting solution increased. At 50 wt % PVP content, the MWCO is found to be greater than 69 kDa, since it is calculated based on solute rejection of less than 80%. This increase in MWCO value indicates, in principle, an increase in the pore size or porosity of the membranes, which is discussed in detail in the following Pore Statistics section.

### Pore statistics

The pore size, porosity, and number of pores of the membranes determined from the protein rejection studies are shown in Table II. It is evident from these results that the membrane prepared in the absence of additive has relatively smaller pore size and porosity as well as number of pores. Addition of 10 wt % PVP 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 additions of PVP 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 $9.69 \times 10^{-11}$ for a PVP content of 20 wt %, while the porosity and pore size increased to a maximum of $8.51 \times 10^5$ and 62.53 Å, respectively, for a PVP content of 50 wt %. Although bigger pore size will favor high permeate flux, 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 PVP concentration of 20 wt % 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. Cross-sectional views of the prepared membranes are shown in Figure 4. In general, the cross section of the membranes can be distinguished into three layers viz., the top layer, the sublayer, and in between these two, the intermediate layer.[30] From

Figure 4(A,B), it is clearly seen that the pore size as well as the structure has significantly enhanced by adding 10 wt % PVP in the casting solution. Figure 4(B) 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 PVP in the casting solution is increased to 20 wt %, the intermediate layer has significantly grown in thickness and the pore size in the sublayer has also increased [Fig. 4(C)]. However, as the concentration of PVP further increases from 20 to 50 wt %, the thickness of intermediate layer reduces progressively and the formation of macrovoids increases in the sublayer as shown in Figure 4(D–F). 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, especially in Figure 4(E–F). This should result in increased permeability for the membranes, especially those prepared using 40 and 50 wt % PVP. The results from this study support this reasoning.

## CONCLUSIONS

In this work, cellulose acetate as a matrix polymer was blended with poly(vinylpyrrolidone) ranging from 0 to 50 wt % using DMF as the solvent to prepare modified ultrafiltration membranes. It was found that polymer solutions having PVP concentration more than 50 wt % did not form membrane. The characterization of prepared membranes illustrates that the pure water flux and water content were increased, while the membrane hydraulic resistance was decreased, as the concentration of PVP in the casting solution is increased. The MWCO and pore statistics results obtained from protein rejection studies demonstrate that the MWCO, pore radius, and porosity show significant increase as the concentration of PVP 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 PVP in the casting solution increased. The 50/50 wt % CA/PVP blend membrane shows highest pure water flux, water content, and porosity compared to other membranes, which demonstrates the hydrophilic nature of PVP polymer. The results of this study will be useful

to design appropriate membranes for the required performance in industrial applications.

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