

Blend membranes of sodium alginate and hydroxyethylcellulose for pervaporation-based enrichment of *t*-butyl alcohol

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Abstract

Blend membranes of sodium alginate (SA) and hydroxyethylcellulose (HEC) were synthesized and ionically crosslinked with phosphoric acid for the separation of *t*-butanol/water mixtures. The membranes were characterized by FT IR to confirm crosslinking and assess the intermolecular interactions. Thermal stability and crystallinity were determined from TGA and XRD studies. Sorption studies and porosity measurements were carried out to evaluate the extent of interaction and degree of swelling of the membranes, in pure liquids as well as binary mixtures. The crosslinked polymer was found to have good potential for breaking the azeotrope of 88 wt% *t*-butanol by giving high selectivity of 3237 and substantial water flux of $0.2 \text{ kg m}^{-2} \text{ h}^{-1}$. The pervaporation performance was evaluated by varying experimental parameters such as feed composition, membrane thickness and permeate pressure and found to be promising for *t*-butanol dehydration.

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Keywords: Pervaporation; Sodium alginate/hydroxyethylcellulose blend; *t*-Butanol/water mixture; Membrane characterization

1. Introduction

Unlike pressure driven membrane processes that use porous barriers for separation, pervaporation is a membrane-based process useful for the separation of aqueous–organic mixtures and organic–organic having an azeotropic point besides similar physical and chemical properties (Aminabhavi et al., 1994; Urgami, Matsuda, Okuno, & Miyata, 1994). The application of pervaporation (PV) as means of achieving the dehydration of solvents has been receiving a substantial amount of attention from the chemical, petrochemical and pharmaceutical industries. In addition, recent technology improvements have led to the commercial viability of several membrane materials, which contain hydrophilic groups in the polymer structure, that preferentially absorb and transport water, at high flux and selectivity (Noble & Stern, 1995). However, the presence of hydrophilic groups sometimes swells the membrane significantly leading to its plasticization action, which results in low selectivity (Huang, 1991). Hence, modification of the polymer,

in order to retain the hydrophilic groups without substantial loss in the stability is the key to prepare potentially useful pervaporation membranes.

Among the hydrophilic membranes, sodium alginate (SA), which is one of the polysaccharides extracted from seaweed, is found to have an excellent performance as a membrane material for pervaporation-based dehydration of organic solvents (Shi, Wang, & Chen, 1996; Urugami & Saito, 1989). It has a linear chain structure of β -D-mannuronic acid (M) and α -L-guluronic acid (G) residues arranged in block wise fashion (Fischer & Dorfel, 1955; Haug, Larsen, & Smolder, 1966). Presence of α -L-guluronic acid in various ratios and molecular weight alters physico-chemical properties of the polymer (Moe, Draget, Break, & Smidsrod, 1995).

Hydroxyethylcellulose (HEC), a non-ionic carbohydrate polymer is water-soluble cellulose ether, which is compatible with a wide range of other water-soluble polymers (Chanachai et al., 2000; Nicholson & Merritt, 1985; Savge & Ethers, 1971). HEC is a commercially useful polymer that finds applications as a thickener in latex paints and paper finishes. However, reports on usage of this membrane in pervaporation applications are few (Naidu, Krishna Rao, & Aminabhavi, in press; Naidu, Sairam, Kothapalli, Raju, & Aminabhavi, 2005).

Tertiary butanol (*t*-butanol) is commonly used as a solvent in chemical and pharmaceutical synthesis. It is an important

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raw material in the production of synthetic perfumes, artificial musk and denatured alcohols. Like many other alcohols, *t*-butanol is also known to form an azeotropic mixture with water, a characteristic that creates difficulties in its recovery by conventional distillation (Kirk-Othmer, 1984). The present investigation attempts to test the applicability of SA/HEC blends crosslink in a novel fashion with phosphoric acid, for dehydration of *t*-butanol. The work also explores the effect of varying water concentration in the binary feed mixture on membrane flux and selectivity. Characterization by FTIR analysis, wide angle X-ray diffraction (WAXD), TGA, tensile strength measurements and sorption studies have been used to explain PV results.

2. Experimental

2.1. Materials

HEC, having a viscosity average molecular weight of 150,000, was purchased from PIDILITE Industries Ltd, Mumbai, India. Sodium alginate used for blending with HEC was obtained from Aldrich chemical Co. had an average molecular weight of 500,000. The degree of deacetylation was found to be 84%. *t*-Butanol, isopropanol and H_3PO_4 were purchased from Loba Chemicals, Mumbai. Dematerialized water (conductivity = 0.02 S cm^{-1}), which was used for the preparation of feed solution, was generated in the laboratory itself.

2.2. Preparation of membranes

Membranes were prepared by solution casting and solvent evaporation technique. Three weight percent solution of SA and 1 wt% of HEC in aqueous medium were prepared individually and mixed in the ratio of 3:1, respectively. The blend solution was then stirred for a period of 1/2 h for homogeneity and kept aside for 1 h to obtain a bubble free solution, which was cast on to a clean glass plate and evaporated to dryness in atmosphere at room temperature to obtain dense non-porous membrane. The so-formed membrane was crosslinked for 3 h in an isopropanol–water bath (90/10 vol%) containing 3.5 vol% of H_3PO_4 . After removing the membrane from the crosslinking bath, it was washed with water repeatedly and dried in an oven at 80°C to eliminate the presence of residual acid, if any. The membrane thickness as measured by a micrometer screw gauge was in the range of 35–40 μm .

2.3. Pervaporation procedure

Experiments were carried out on a 100 ml batch level with an indigenously constructed pervaporation manifold operated at a vacuum as low as 0.05 mmHg in the permeate line. The membrane area in the pervaporation cell assembly (Fig. 1) was approximately 20 cm^2 . The experimental procedure is described in detail elsewhere (Sridhar, Ravindra, & Khan, 2000). The *t*-butanol aqueous feed was stirred vigorously with

the speed of 160 rpm during experiments to minimize concentration polarization. Permeate was condensed and collected in a liquid nitrogen cold trap for a duration of 6–8 h. Each experiment was commenced only after PV equilibrium was reached (12 h) in order to ensure sufficient absorption of the feed into the surface of the membrane and minimize errors in evaluating flux. Tests were carried out at room temperature ($30 \pm 2^\circ\text{C}$) and repeated twice using fresh feed solution to check for reproducibility. The collected permeate was weighed after allowing it to attain room temperature in a Sartorius electronic balance (accuracy: 10^{-4} g) to determine the flux and then analyzed by gas chromatography to evaluate the membrane selectivity as follows.

Flux, J , of a given species, say faster permeating component, i of a binary mixture comprising of i (water) and j (*t*-butanol) is given by:

$$J_i = \frac{W_i}{At}$$

where W_i represents the mass of water in permeate (kg), A is membrane area (m^2) and t represents the evaluation time (h). In the present study, even though membranes of different thickness were fabricated, flux was normalized and reported for the constant thickness of 10 μm . Membrane selectivity, α is the ratio of permeability coefficient of water and *t*-butanol, which was calculated from the respective concentrations in feed and permeate sides using the equation:

$$\alpha = \frac{y(1-x)}{x(1-y)}$$

Here, y is permeate mass content of water (%) and x is its feed mass content.

Pervaporation separation index (PSI) is a measure of the separation capability of a membrane and is expressed as a product of selectivity and flux (Huang & Yeom, 1990).

$$\text{PSI} = J\alpha$$

2.4. Analytical procedure

The feed and permeate samples were analyzed using a Nucon Gas Chromatograph (Model 5765) installed with thermal conductivity detector (TCD) and packed column of 10% DEGS on 80/100 Supelcoport of 1/8" ID and 2 m length. The oven temperature was maintained at 70°C (isothermal) while the injector and detector temperatures were maintained at 150°C each. The sample injection size was 1 μl and pure hydrogen was used as a carrier gas at a pressure of 1 kg cm^{-2} . The GC response was calibrated for this particular column and conditions with known compositions of *t*-butanol/water mixtures and the calibration factors were fed into the software to obtain correct analysis for unknown samples.

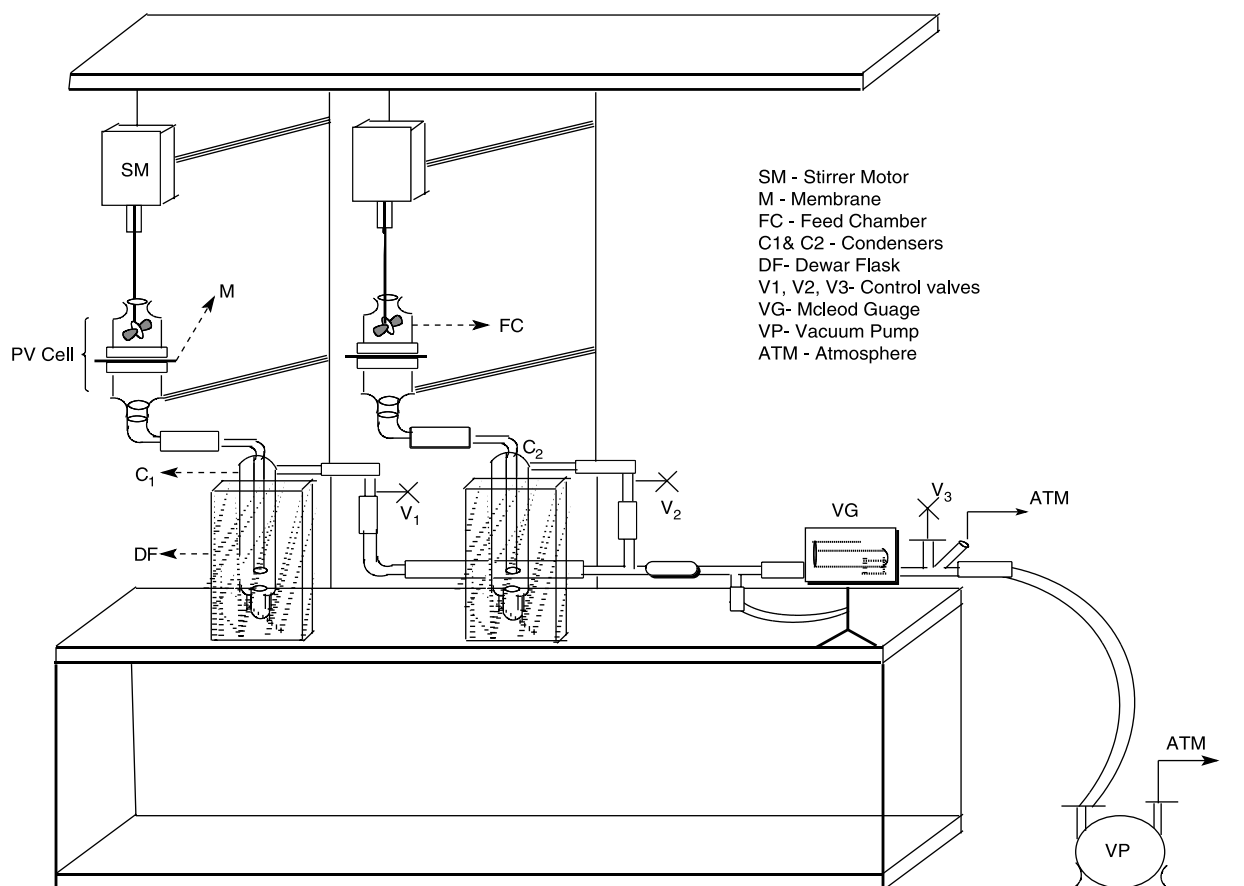


Fig. 1. Schematic of laboratory vacuum pervaporation set-up.

2.5. Membrane characterization

2.5.1. FTIR studies

FT IR spectra of the plain SA, HEC, uncrosslinked SA/HEC and phosphoric acid crosslinked SA/HEC (P-SA/HEC) blends were scanned in the range between 4000 and 400 cm^{-1} on a Nicolet-740, Perkin–Elmer-283B FTIR spectrophotometer by KBr pellet method.

2.5.2. XRD analysis

A Siemens D 5000 powder X-ray diffractometer was used to study the solid-state morphology of P-SA/HEC in powdered form. X-rays of 2 Å wavelengths were generated by a CuK source. The angle of diffraction was varied from 2 to 60° to identify the changes in the crystal structure and intermolecular distances between the inter-segmental chains after cross-linking. The d -spacing value can be calculated from the Braggs equation given below

$$2d \sin \theta = \lambda$$

where θ , angle of contact/diffraction; λ , wavelength = 2 Å; d , effective d -spacing.

Since, the angle θ is known the space between the clusters d can thus be identified.

2.5.3. Thermal gravimetric analysis (TGA)

Thermal stability of the polymer films was examined, using Seiko 220TG/DTA analyzer from 25 to 700 °C at a heating rate of 10 °C min^{-1} with continuous flushing using pure nitrogen gas at 200 ml min^{-1} . The samples were subjected to TGA both before and after phosphorylation to determine the thermal stability and decomposition characteristics.

2.6. Sorption characteristics

In order to evaluate membrane-liquid affinities, weighed samples of circular pieces of the polymer films (3 cm diameter) were soaked in *t*-butanol/water mixtures of various concentrations. The films were taken out after different soaking periods and quickly weighed after carefully wiping out excess liquid to estimate the amount absorbed at the particular time t . The film was then quickly placed back in the solvent. The process was repeated until the films attained steady state as indicated by constant weight after a certain period of soaking time. Degree of swelling was calculated as:

$$DS = \frac{M_s}{M_d}$$

where M_s , mass of the swollen polymer in grams; M_d , mass of the dry polymer in grams.

The percentage sorption was calculated using the equation:

$$\text{Sorption (\%)} = \frac{M_s - M_d}{M_d} \times 100.$$

2.7. Determination of the ion exchange capacity and degree of substitution

The ion exchange capacity (IEC) indicates the number of milliequivalents of ions in 1 g of the dry polymer. The degree of substitution (DS) indicates the average number of acid groups present in the polymer. To determine the degree of substitution by acid groups, the phosphorylated membranes and unphosphorylated specimens of similar weight were soaked in 50 ml of 0.01 N sodium hydroxide solution for 12 h at ambient temperature. Then, 10 ml of the solution was titrated with 0.01 N sulfuric acid. The sample was regenerated with 1 M hydrochloric acid, washed free of acid with water and dried to a constant weight. The IEC was calculated according to.

$$\text{IEC} = \frac{B - P \times 0.01 \times 5}{m}$$

where IEC is the ion exchange capacity (mequiv./g), B , the amount of sulfuric acid used to neutralize blind sample soaked in NaOH (ml), P , the amount of sulfuric acid used to neutralize the sulfonated membrane soaked in NaOH (ml), 0.01, the normality of the sulfuric acid, '5', the factor corresponding to the ratio of the amount of NaOH taken to dissolve the polymer to the amount used for titration, and m is the sample mass (g).

3. Results and discussion

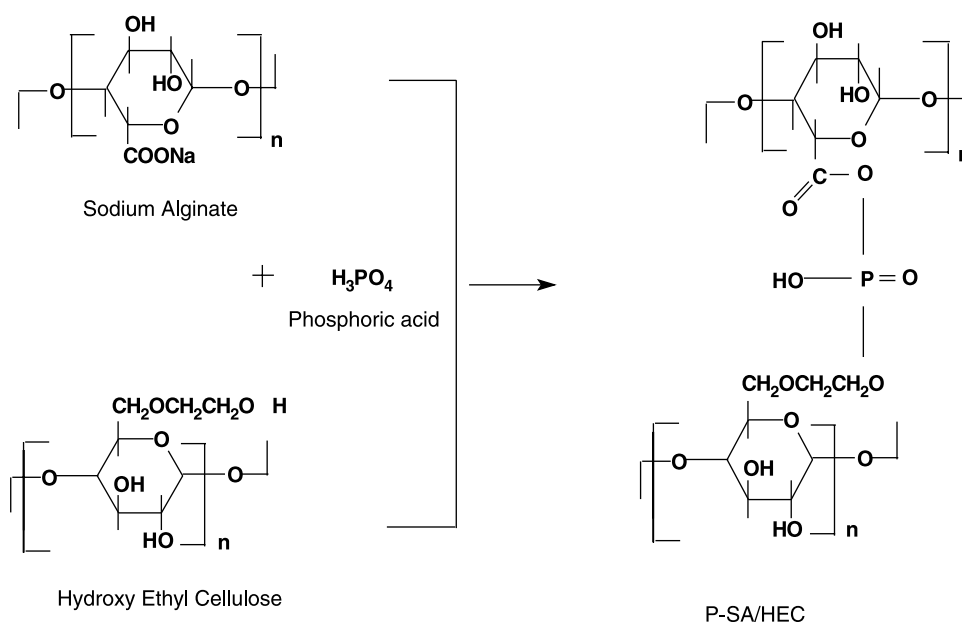
Scheme 1 represents the structures of the polymers used in the study and also represents the structure of SA/HEC blend

crosslinked with phosphoric acid. The hydroxyl groups present in H_3PO_4 , react with the acetate group of SA and hydroxyl group of HEC resulting in the formation of a covalent bond. This can also be confirmed by FTIR. It was noticed that both the homopolymers and the SA/HEC blends were optically clear to the naked eye. No separation into two layers or any precipitation was noticed when allowed to stand for one month at room temperature. Amount of residual hydroxyl and acetate groups in blend membranes after crosslinking was estimated from IEC studies. It was found that the uncrosslinked SA/HEC blend had an IEC of 3 mequiv./g, whereas phosphoric acid crosslinked polymer exhibited an IEC of 2 mequiv./g.

The IEC, which is equivalent to the total number of acetate and hydroxyl groups present in the membrane, decreased upon crosslinking because some hydroxyl groups and sodium acetate groups were consumed during the reaction (Yeom & Lee, 1998). The IEC results show that almost 50% of hydroxyl groups present in the unmodified SA/HEC have now formed crosslinks with phosphoric acid. A model of the possible interaction is shown in Scheme 1.

3.1. Effect of blending ratio

The homopolymers viz., SA and HEC were mixed in various ratios to obtain blend membranes. It was noted that an increase in the HEC content in the blend renders the membrane brittle as evidenced by the membrane stability test. The stability of the membrane was assessed by bending the film. The membrane is considered stable if its mechanical strength is restored after bending it, i.e. it does not break upon bending. The mechanical weakness, introduced by the addition of HEC as a component has a profound effect as its content in the blend increases. Hence, various blending ratios of sodium alginate to hydroxyethylcellulose were studied among which only one



Scheme 1. Structural representation of sodium alginate–hydroxyethylcellulose blend membrane crosslinked with phosphoric acid.

mechanically stable blend was considered for pervaporation studies.

3.2. Membrane characterization

3.2.1. Fourier transform infrared spectroscopy

Fig. 1 shows the FTIR spectra of the homopolymers, i.e. SA (Fig. 2(a)) and HEC (Fig. 2(b)) in comparison with their uncrosslinked (Fig. 2(c)) and crosslinked blends (Fig. 2(d)). The FTIR spectra of SA shows the prominent peak of C=O stretching of the carboxylic group at 1651 and 1637 cm^{-1} . The characteristic O–H deformation peak in HEC can be noted at 626 cm^{-1} . All the membranes synthesized (Fig. 2(a)–(d)) showed characteristic peaks appearing in the range 3200 – 3550 cm^{-1} corresponding to O–H stretching vibrations.

The spectra of the crosslinked blend showed a shift in the C=O peak from 1637 cm^{-1} in the uncrosslinked blend to 1738 cm^{-1} for the modified film. This shift may be attributed to the formation of –OC–O–P– bond due to the interaction of hydroxyl group of phosphoric acid with acetate group of sodium alginate. It can also be noted that the P=O remains intact and does not participate in the reaction. This can be confirmed by the presence of a peak at 960 cm^{-1} which corresponds to P=O. The formation of a new peak (Fig. 2(d)) at 1260 cm^{-1} corresponds to the interaction of hydroxyl group of phosphoric acid with hydroxyl group present in the linear chain of HEC resulting in the formation of –P–O–C– bond. It can be concluded that phosphoric acid acts as a crosslinking agent to both SA and HEC. The model structure proposed in Scheme 1 is well in accordance with the FTIR spectra.

3.2.2. X-ray diffraction

X-ray diffraction studies not only indicate the nature of the compounds but also enable in identifying the space between the clusters of the polymer chains. It is a well-known fact that on crosslinking the polymer chains are in close compaction with

one another and thus a reduction in the cluster space may be encountered. The effective d -spacing (d) gives an indication of cluster space existing in the polymer before and after crosslinking.

The X-ray diffractograms of crosslinked and uncrosslinked SA/HEC blend membranes are shown in Fig. 3(a) and (b). The XRD spectra of the blend showed the amorphous nature with broad peaks at $2\theta=10$ and 20° . This indicates the presence of higher free volumes in the blend, which might result in a reduction in selectivity but enhanced fluxes.

The diffraction peaks are observed at $2\theta=7$ – 9° (crystal 1) and $2\theta=20^\circ$ (crystal 2) for hydroxyethylcellulose and at $2\theta=14^\circ$ (Guang, Lina, Tao, & Wei, 2000) for alginate. Of the two peaks observed for HEC, the crystal 1 is responsible for the separation, since it corresponds to the hydroxyl functional group. It would thus be interesting to know if these groups might have further undergone significant changes after crosslinking. The reduction in effective d -spacing from 6.4 \AA ($2\theta=20^\circ$) for uncrosslinked blend to 6.3 \AA for the crosslinked one, suggests the occurrence of reaction between the carboxylic groups of SA with hydroxyl groups of phosphoric acid. On the other hand, the reduction in effective d -spacing from 9.5 \AA ($2\theta=9^\circ$) for the uncrosslinked blend (Fig. 3(a)) to 9.3 \AA in the crosslinked blend membrane (Fig. 3(b)) indicates the shrinkage in cell size or inter-segmental spacing occurring due to crosslinking between hydroxyl group of HEC and phosphoric acid, which would improve the selective permeation of the membrane.

3.2.3. TGA studies

The TGA curves of the crosslinked and unmodified blend are shown in Fig. 4. The TGA curve of SA/HEC blend (Fig. 4(a)) shows one weight loss stage occurring at 220 – 260°C followed by a final decomposition at 260°C . The weight loss stage can be attributed to the decomposition of

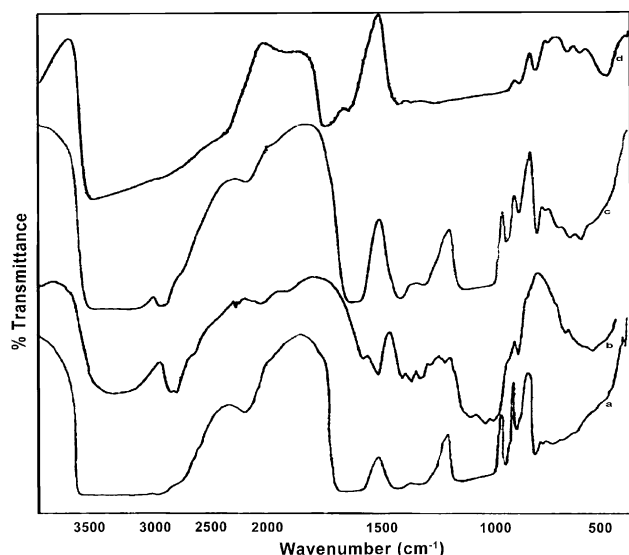


Fig. 2. FTIR spectra of (a) SA, (b) HEC, (c) uncrosslinked SA/HEC blend, and (d) crosslinked SA/HEC blend.

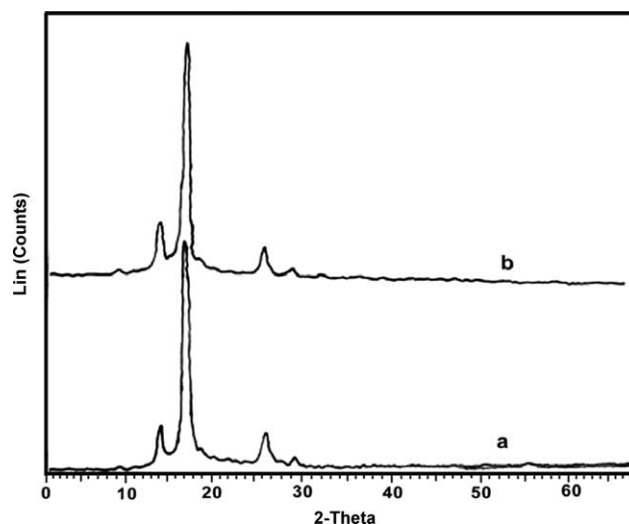


Fig. 3. X-RD diffractograms of (a) SA/HEC uncrosslinked blend and (b) crosslinked SA/HEC blend.

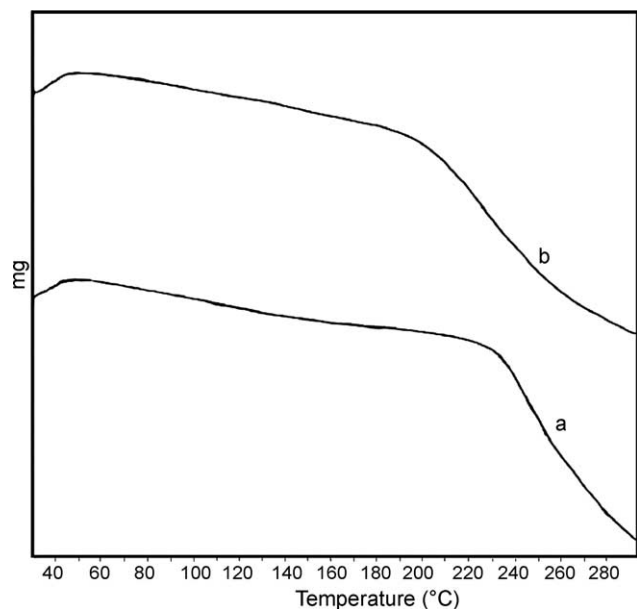


Fig. 4. TGA curves for (a) crosslinked SA/HEC blend and (b) crosslinked SA/HEC blend.

main chain in the blend. The T_m of the uncrosslinked blend was found to be 250 °C.

The TGA curve of the P-SA/HEC blend (Fig. 4(b)) also exhibited one weight loss stage at 200–250 °C followed by a final decomposition at 250 °C. The T_m of the crosslinked blend reduced to 230 °C upon crosslinking. It is worth mentioning that there is negligible change in the T_m before and after crosslinking, which is indicative of the high thermal stability of the crosslinked blend.

3.3. Pervaporation studies

3.3.1. Effect of feed composition

The variation of pervaporation performance over a wide range of feed mixtures at 30 °C was investigated using crosslinked blend membranes. The pervaporation performance of P-SA/HEC membrane was investigated for varying feed compositions comprising of 5–40 wt% water keeping other operating parameters such as permeate pressure and membrane thickness constant. For this study, the membrane thickness and permeate pressure were maintained at 60 μm and 0.7 mmHg, respectively. Expectedly, a rise in the feed concentration of water produced an increase in the water flux from 0.2 to 0.5 $\text{kg m}^{-2} \text{h}^{-1}$ 10 μm (Fig. 5). Mass transport through the hydrophilic blend membrane occurs by solution diffusion mechanism (Osada & Nakagawa, 1992). In addition, the sorption data for binary feed mixtures presented in Table 1 also show a high degree of sorption for the crosslinked membrane in pure water (66%) but relatively negligible sorption (0.001%) in pure *t*-butanol. The preferential affinity of the membrane towards water causes swelling which allows rapid permeation of feed molecules.

The degree of swelling correspondingly rises with an increase in the feed water concentration resulting in enhanced

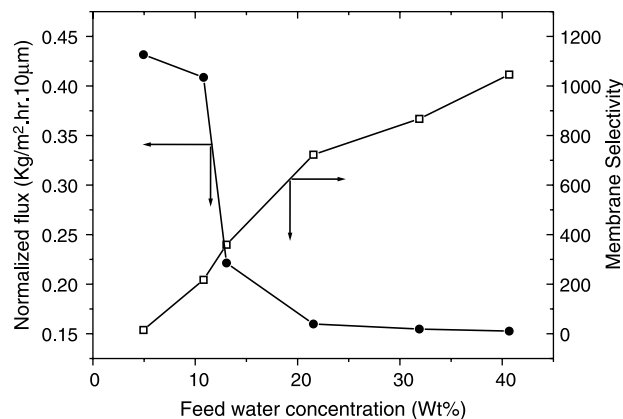


Fig. 5. Effect of feed water composition on PV performance of SA/HEC blends (membrane thickness 60 μm and permeate pressure at 0.7 mmHg).

flux. However, increased swelling has a negative impact on membrane selectivity since the swollen and plasticized upstream membrane layer allows some *t*-butanol molecules also to escape into the permeate side along with water. Therefore, the permeate water concentration was found to reduce from 99.3 to 88% indicating a drop in selectivity from 1127 to as low as 10 over the same feed concentration range of 5–40% water as reported in Fig. 5. However, it is worth mentioning that the membrane showed promising results for dehydrating feeds having 5–25% water. Moreover, the azeotropic composition of (88 wt% *t*-butanol) was easily broken by pervaporation and about 99.3% water.

3.3.2. Effect of membrane thickness

The effect of varying membrane thickness on separation performance was studied at constant feed composition (azeotropic) and permeate pressure (0.7 mmHg) by synthesizing membranes of thickness ranging from 40 to 115 μm . The variation in selectivity with membrane thickness is a bit too high. Generally, selectivity is constant or increases marginally with rising thickness. With an increase in the membrane thickness, a gradual reduction in the flux from 0.3 to 0.2 $\text{kg m}^{-2} \text{h}^{-1}$ can be clearly evidenced from Fig. 6. Though the availability of polar groups enhances with an increase in the thickness, flux decreases since diffusion becomes increasingly retarded as the feed molecules have to travel a greater distance to reach the permeate side. The permeate concentration of water varied from 93.3 to 99.6 wt% which meant that the selectivity increased from 93 to 3237.

Table 1
Effect of feed concentration on degree of swelling, percentage sorption and PSI values

S. no.	Feed water concentration (wt%)	Degree of swelling	% of Sorption	PSI
1	5.0	1.1	9.6	273
2	10.8	1.2	17.7	211
3	13.0	1.3	24.0	68
4	21.5	1.5	46.1	13
5	31.8	1.6	52.0	7
6	40.6	1.7	65.5	4

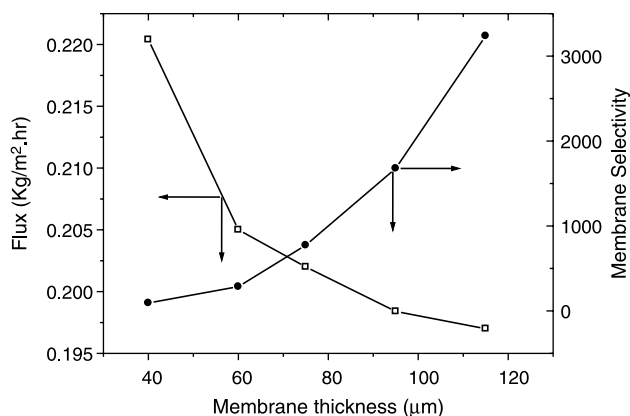


Fig. 6. Effect of membrane thickness on PV performance (azeotropic feed composition 12 wt% of water and permeate pressure at 0.7 mmHg).

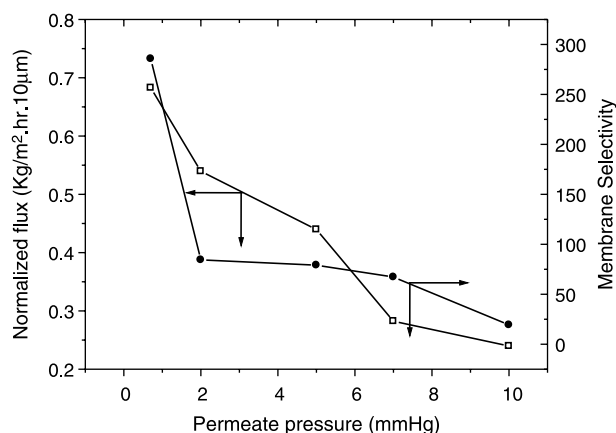


Fig. 7. Effect of permeate pressure on PV performance (azeotropic feed composition 12 wt% of water and membrane thickness 60 μm).

In pervaporation process, the upstream layer of the membrane is swollen and plasticized due to absorption of feed liquid and allows unrestricted transport of feed components. In contrast, the downstream layer is virtually dry due to continuous evacuation in the permeate side, and therefore, this layer

forms the restrictive barrier which allows only interacting and smaller sized molecules such as water to pass through. It is expected that the thickness of the dry layer would increase with an increase in the overall membrane thickness resulting in improved selectivity as observed in the present case.

3.3.3. Effect of permeate pressure

The permeate pressure was varied from 0.7 to 10 mmHg to study the separation characteristics at a constant membrane thickness of 60 μm and azeotropic feed composition. At lower pressures (high vacuum), the influence of the driving force on the diffusing molecules in the membrane is high, and will result in the components being swept out immediately from the permeate side resulting in high mass transfer rates. Fig. 7 shows that the membrane exhibits considerable lowering of flux from 0.7 to 0.2 kg m⁻² h⁻¹ · 10 μm as well as a reduction in selectivity from 286 to 20 with an increase in permeate pressure from 0.7 to 10 mmHg. Under high vacuum conditions (lower pressures), diffusion through the membrane is the rate determining step of the pervaporation process and the diffusing water molecules experience larger driving force, which enhances the desorption rate at the downstream side. Lower vacuums reduce the driving force, thus slowing the desorption of molecules. In such cases, the relative volatilities of the two components of the mixture govern the separation factor of the membrane. *t*-Butanol being more volatile (BP 83 °C) than water permeates competitively with the latter thus lowering membrane selectivity.

4. Comparison of present work with literature

Pervaporation performance of the P-SA/HEC membranes is compared with the literature data and is reported in Table 2. From the table, it can be noted that the membranes synthesized in the study yielded higher fluxes than that of other researchers work but showed comparable selectivity. However, it can be said that a marginally good selectivity combined with high fluxes render the covalently crosslinked blend suitable for dehydration of *t*-butanol/water mixtures. Furthermore, the ease

Table 2

Comparison of flux and selectivity of PVA-SA blend membrane with values reported in literature

S. no.	Membrane	Feed composition		Selectivity	Normalized flux (kg m ⁻² h ⁻¹ 10 μm)	Reference
		Water (%)	<i>t</i> -Butanol (%)			
1	Chitosan	4.3	94.7	2658	0.21	Smitha, Sridhar, Murthy, & Satyajai Mayor (in press)
2 ^a	T-PDMS	3.2	96.8	12	0.63	Stefan, Denis, Trong, Pierra, and Philippe (1998)
3 ^a	IP-PDMS	3.2	96.8	12	0.39	Stefan et al. (1998)
4 ^a	T-PDMS-T-BFH	3.2	96.8	11	0.43	Stefan et al. (1998)
5	PVA/PVS blend	3	97	3200	0.31	Ali, Lutz, and Bernd (2002)
6	PVA/PSS blend	5	95	17,900	0.21	Takegami, Yamada, and Tsujii (1992)
7	NaA zeolite	10	90	16,107	1.5	Tatiana, Emma, Giuseppe, and Luisa (2002)
8	P-SA/HEC blend	13	87	3237	2.3	Present work

PVA/PVS, poly(vinyl alcohol)/poly(vinyl sulfate); PDMS, poly dimethyl siloxane; IP, isophorone diisocyanate; BFH, 2,2-3,3-4,4-5,5-oct-fluoro-hexane-1,6-diol; PVA/PSS, poly(vinyl alcohol)/poly(styrene sulfonic acid); P-SA/HEC, phosphorelated sodium alginate/hydroxyethylcellulose.

^a Siloxane-urea block copolymers.

in fabrication of these membranes associated with low cost render them more attractive for pervaporation of aqueous alcohols.

5. Conclusions

In this study, the blend membrane of sodium alginate and hydroxyethylcellulose were prepared and crosslinked with phosphoric acid for pervaporation-based dehydration of *t*-butanol/water mixture. The number of groups crosslinked in SA/HEC blend polymer was identified from the IEC studies. Characterization of the crosslinked membranes by FTIR and XRD confirmed the crosslinking reaction. The membranes showed adequate thermal stability to withstand the PV experimental conditions. FTIR spectroscopy confirmed the predicted interaction between the SA/HEC blend and the crosslinker. With increasing feed water compositions, the membrane performance exhibited a reduction in selectivity and an improvement in flux due to increased swelling. Expectedly, with increasing membrane thickness, selectivity improved, but flux decreased. Higher permeate pressure caused a reduction in both flux and selectivity. Pervaporation could be combined with distillation in an integrated process wherein the former overcomes the azeotropic barrier of *t*-butanol (88%) after which the latter is applied to achieve final purity.

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