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Research Article

Separation of 1,4-Dioxane/Water Mixtures Using Zeolite Incorporated Membranes via Pervaporation: Comparison with Glutaraldehyde Crosslinked Membranes

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ABSTRACT

Novel dense pervaporation (PV) membranes were prepared by incorporating ZSM-5 nanosized zeolite particles in sodium alginate membranes and crosslinked with glutaraldehyde. The membranes were analyzed by way of Ion Exchange Capacity (IEC) to verify the cross-linking. Further, X-ray diffraction (XRD) and Scanning electron microscope (SEM) studies were used to observe the crystalline nature and surface morphology of the membranes respectively. Sorption studies were carried out to evaluate the extent of interaction and degree of swelling of the membranes in pure, as well as binary mixtures of water and 1,4-dioxane. The effect of experimental factors, such as concentration of feed solutions, and membrane thickness on both membrane performances was evaluated and compared. The membranes were found to have good potential for breaking the aqueous azeotrope of 82 % 1,4-dioxane concentration, but the crosslinked membranes show better PV performance than that of uncrosslinked membranes.

Keywords: ZSM-5 nanosized zeolites; Sodium alginate; Glutaraldehyde crosslinking; Pervaporation; 1,4-dioxane/water azeotrope; Sorption studies.

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1. INTRODUCTION

Nowadays, pervaporation (PV) is considered the most promising membrane technology for the separation of close boiling liquids, azeotropic aqueous–organic mixtures, organic–organic solutions[1,2], and thermally sensitive organic compounds[3], which cannot be separated by a traditional distillation process. PV has gained considerable interest because of its safe, eco-friendly process, low cost, and high selectivity[4]. PV is an efficient technique for the separation of azeotropic mixtures because the separation mechanism in PV is not based on the relative volatility of the components, but on the difference between the sorption and diffusion properties of feed components, as well as on membrane properties[5,6]. Therefore, the membrane is the key factor in the PV process to achieve high separation performance[7].

Sodium alginate (SA), a widely used biopolymer, was chosen for this study due to its excellent film-forming ability, hydrophilic nature, separation selectivity towards the water, and good resistance to organic solvents[8-10]. Unfortunately, pristine SA membranes exhibit significant swelling in higher water concentrations of feed solutions, which results in a remarkable decline with time in membrane selectivity as well as mechanical strength due to the presence of high numbers of carboxyl and hydroxyl groups[11,12], and large volume between the chains[13]. Many researchers have put great efforts to improve the physicochemical and transport properties, and membrane performance of the SA membranes by blending with other polymers, crosslinking the membranes, and adding the appropriate filler particles[12]. In the previous studies, modification of SA is attempted by loading different inorganic fillers such as zeolites, mesoporous materials, clays, and alumino-phosphates[14-18], which have shown a pronounced increase in membrane performance due to their unique structural characteristics and hydrophilicity, resulting in appreciable separation performance and permeation flux. Zeolite-filled membranes are proven to be better than pristine (unfilled) membranes due to their uniform molecular size pores that provide improved transport rates due to molecular sieving effects and better chemical stability than pristine polymers[19-21]. To explore further in this area, we have chosen the ZSM-5 zeolite, a versatile molecular sieve available from the zeolite family.

1,4-Dioxane is generally used in the chemical, petrochemical, and pharmaceutical industries[22]. It is highly miscible with water in all proportions and forms an azeotrope with 18 wt.% concentration of water[23]. Due to its close boiling point (101°C) to that of water, the widely used conventional distillation process cannot be separated 1,4-Dioxane and water mixtures. Hence, PV has emerged as one of the most promising and economical separation techniques that can be used as an alternative to distillation[24]. In earlier research, we have developed glutaraldehyde crosslinked blend membranes comprising sodium alginate, poly(vinyl alcohol), and polyethyleneimine for the PV separation of aqueous-organic mixtures[25,26]. In continuation of our ongoing efforts, we now report on PV separation characteristics of

1,4-dioxane/water mixtures using the newly developed ZSM-5 nanosized zeolite incorporated SA (ZSA) membranes and glutaraldehyde crosslinked ZSA (GZSA) membranes. The membranes are characterized by XRD and SEM. The effects of experimental parameters such as feed composition, and membrane thickness on normalized flux and selectivity are determined for both membranes and compared. Uncrosslinked and crosslinked membranes are subjected to sorption studies to evaluate the extent of interaction and degree of swelling in 1,4-dioxane/water mixtures.

2. MATERIALS AND METHODS

2.1. Materials

Sodium alginate (average molecular weight of 500,000) and zsm-5 nanosized zeolite powder were purchased from Aldrich Chemical Co., USA. Reagent grade 1,4-dioxane of purity 99.9% and glutaraldehyde were purchased from S.D. Fine Chemicals, Mumbai, India. Oxalic acid, isopropanol, hydrochloric acid, sulfuric acid, and sodium hydroxide were purchased from Loba Chemicals, Mumbai, India. The water of conductivity <0.02 S/cm, used throughout the experiment work, was generated in the laboratory by distilling the deionized water twice in a quartz distillation plant.

2.2. Membranes Preparation and Crosslinking

ZSA membranes were prepared via solution casting and solvent evaporation techniques[27,28]. 3 wt.% of SA was dissolved in 75 mL of 3% oxalic acid solution under constant stirring. Then, 0.3 wt.% of ZSM-5 nanosized zeolite powder was weighed separately and dispersed in 25 mL of water, sonicated for 30 min, and added to the above prepared SA solution with further stirring for 3 hrs. The zeolite-filled solution was poured on a glass plate to cast the membranes. The resultant membrane (ZSA) was removed from the glass plate, followed by cross-linking with 5 vol.% of glutaraldehyde in isopropanol–water (90/10 vol.%) bath containing 1 vol.% of hydrochloric acid as a catalyst. After crosslinking for 4 hrs, the membranes (GZSA) were stored in distilled water for 2 hrs to remove traces of unreacted glutaraldehyde in the membrane, if any, to prevent further crosslinking[29]. Different thicknesses of the membranes were prepared by varying volumes of the zeolite-filled solution. The thickness of each membrane was measured by a micrometer screw gauge at different locations and the average values of these were taken as the thickness of the membrane.

2.3. Membrane characterization

A Seifert X-ray diffractometer was used to study the solid-state morphology of SA, ZSA, and GZSA membranes in powdered form. X-rays of 1.546°A wavelength were generated by a CuKα source at 40 kV and 20 mA. The angle of diffraction varied from 5 to 60° to identify the change in the crystal structure and intermolecular distances between the intersegmental chains after cross-linking.

The morphology of the ZSM-5 zeolite particles, SA, ZSA, and GZSA membranes was observed using Jeol SEM attached with an x-ray energy analyzer model JSM-840A.

2.4. Sorption Studies

To determine membrane-liquid affinities, known-weight circular pieces of polymer films (3 cm dia.) were soaked in deionized water and 1,4-dioxane as well as mixtures of different concentrations. Sorption was one of the control steps in PV separation in the solution-diffusion models. The swollen samples 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 a steady state as indicated by constant weight after a certain period of soaking time. The degree of swelling (DS) was calculated as,

$$DS = \frac{Ms}{Md} \qquad (1)$$

The sorption represents the fraction of the extracted liquid mixture by the membrane. The percentage sorption was calculated using equation 2:

$$\%\,Sorption = \frac{Ms-Md}{Md} * 100 \qquad (2)$$

Where M_s was the mass of the swollen polymer in grams, and M_d was the mass of the dry polymer in grams.

2.5. Determination of the Ion Exchange Capacity

IEC studies of the membranes were estimated to determine the effect of crosslinking. The IEC indicates the number of groups present before and after crosslinking, which gives an idea about the extent of crosslinking[27,30,31]. Thus, IEC gives the number of milliequivalents of ions in 1 g of dry polymer. To determine the IEC, ZSA, and GZSA specimens of similar weights were soaked in 50 mL of 0.01N NaOH solution for 12 hrs at ambient temperature. Then, 10 mL of the sample solution was titrated against 0.01N H_2SO_4. The membrane was regenerated with 1M HCl, washed with water until the washings are free from acid, and dried to a constant weight. The IEC was calculated according to the equation,

$$IEC = \frac{(B-P)*0.01*5}{m} \qquad (3)$$

Where B was the volume of H_2SO_4 used to neutralize the blank sample soaked in NaOH (mL), P was the volume of H_2SO_4 used to neutralize the membrane soaked in NaOH (mL), 0.01 was the normality of the H_2SO_4, and '5' was the factor corresponding to the ratio of the amount of NaOH taken to dissolve the polymer to the amount used for titration, and m was the sample mass (g).

2.6. Pervaporation and Analytical Procedure

2.6.1. Influence of operating conditions

PV experiments were carried out in a 100 mL cell operated at a vacuum as low as 0.5 mmHg in the permeate line (Figure 1). The effective membrane area in contact with the feed solution was 19.6 cm^2. The experimental procedure described in the earlier literature[26,27,30,31]. The feed solution consisting of 1,4-dioxane and water was stirred vigorously at a speed of 150 rpm during experiments to minimize concentration polarization. Permeate was condensed and collected in a liquid nitrogen cold trap for 6 hrs. Tests were carried out at room temperature (30±2°C) and repeated twice using fresh feed solution to check for reproducibility. The collected permeate was weighed in a Sartorius electronic balance with an accuracy of 10^{-4} g to determine the flux. Analysis of permeate composition was carried out by measuring the refractive index of the mixture with an accuracy of ±0.001 units, using an Abbe Refractometer (Advance Research Instruments Company, New Delhi, India). A calibration plot of refractive index versus percent composition of 1,4-dioxane and water was obtained with the known quantities of mixture components.

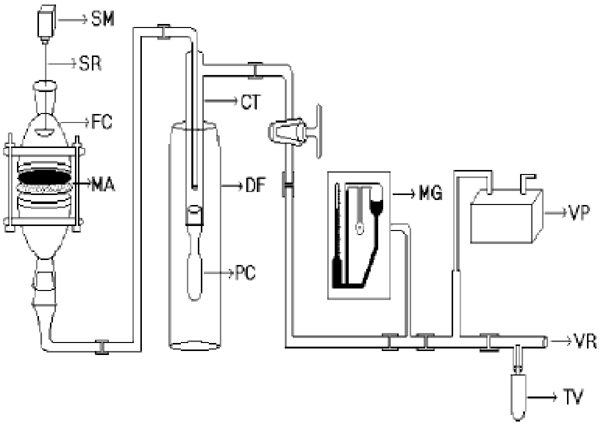


Figure 1: Block diagram of pervaporation set-up. SM: stirring motor, SR: stirring rod, FC: feed chamber, MA: membrane assembly, CT: condenser trap, DF: Dewar flask, PC: permeate collector, MG: McLeod gauge, VP: vacuum pump, VR: vacuum release, TV: Teflon valve.

2.6.2. Flux and Selectivity

The flux *J* of a given species says faster permeating component *i* of a binary liquid mixture comprising of *i* (water) and *j* (1,4-dioxane) is given by:

$$Ji = \frac{Wi}{A * t} \qquad (4)$$

Where *Wi* represents the mass of water in permeate (kg), *A* is the membrane area (m^2) and *t* represents the experiment time (hr). PV flux on a commercial level is generally reported for a membrane of 10 µm thickness. The observed flux for a membrane of any given thickness is converted to flux for 10 µm by multiplication of the corresponding factor assuming a linear relationship between thickness and flux.

The membrane selectivity (α) is the ratio of permeable coefficients of water and 1,4-dioxane, and can be calculated from their respective concentrations in feed (x) and permeate (y) as given below:

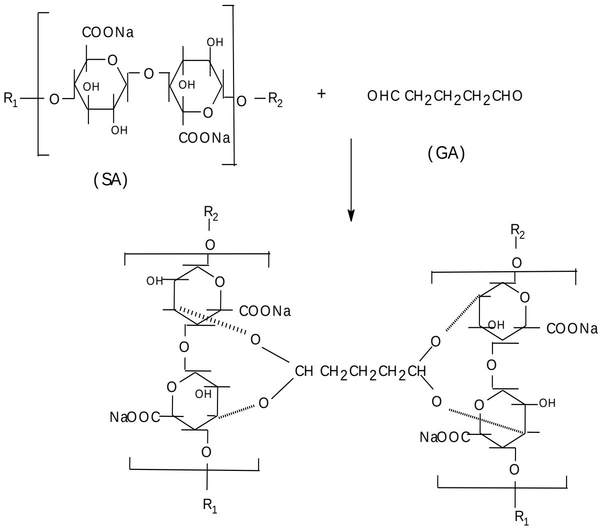
$$\alpha = \frac{y(1-x)}{x(1-y)} \qquad (5)$$

The pervaporation separation index (PSI), which is a measure of the separation capability of a membrane, is expressed as a product of selectivity (α) and flux (*J*),

$$PSI = J * \alpha \qquad (6)$$

3. RESULTS AND DISCUSSION

Hydrogen bonding interactions take place between ZSM-5 nanosized zeolite particles and SA in the matrix of SA/ZSM-5. After crosslinking with glutaraldehyde, the –CHO groups present in it react with the –OH groups of SA due to the formation of ether linkage by eliminating water[32]. A model of the possible interactions of SA and glutaraldehyde is shown in Scheme 1 and these interactions are confirmed in our earlier study[25]. It is noticed that the ZSA and GZSA membranes are optically clear to the naked eye. No separation/splitting into two layers or precipitation was noticed when allowed to stand for one month at ambient temperature.



Scheme 1. Structural representation of crosslinked reaction of sodium alginate (SA) with glutaraldehyde (GA).

3.1. Membrane Characterization

3.1.1. XRD Studies

The microstructures of SA, ZSA, and GZSA membranes are studied using XRD shown in Figure 2(a–c). In this study, dried samples of 50 µm thickness of membranes are used. From Figure 2a, the prominent peak at 13.6° represents the amorphous nature of pristine SA. In the microstructures of ZSA, sharp peaks at 6°, 16°, and 25° represent the crystalline nature of the ZSM-5 zeolite particles, which are incorporated in the membrane (Figure 2b). However, similar peaks with lesser intensity also appeared in GZSA membranes (Figure 2c) due to the crosslink of glutaraldehyde. It indicates the crystallinity nature of ZSM-5 zeolite particles decreases, and the GZSA membrane shows an amorphous nature.

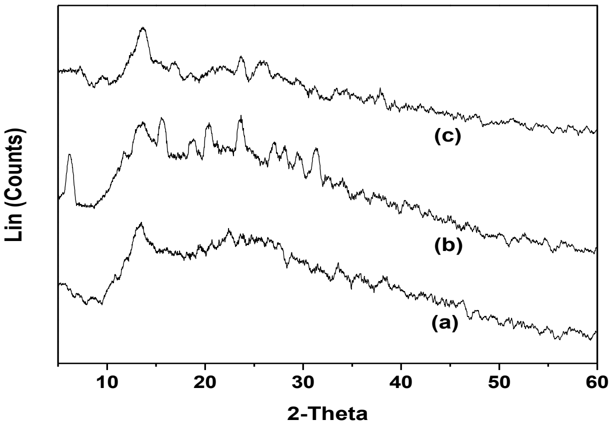


Figure 2: X-ray diffractograms of (a) SA, (b) ZSA, and (c) GZSA membranes.

3.1.2. SEM Analysis

Figure 3 shows the SEM surface images of (a) ZSM-5 nanosized zeolite particles; (b) SA; (c) ZSA; and (d) GZSA composite membranes. Notice that ZSM-5 nanosized zeolite particles are distributed uniformly at the molecular level in the SA matrix (Figure 3b and 3c). In addition to this, there is no change in particle symmetry/uniform arrangement after crosslinking the ZSA membrane with glutaraldehyde (Figure 3d). During PV experiments, most of the water molecules get adsorbed through ZSM-5 particles that are embedded in the SA matrix.

3.2. Sorption Studies

The effect of the equilibrium sorption percentage of ZSA and GZSA membranes are shown in Tables 1 and 2, respectively in different feed compositions of 1,4-dioxane/water mixtures. From these experimental data, it is evidenced that the sorption percentage increases steadily from 11.89 to 85.68 for the ZSA membrane and from 5.76 to 63.61 for the GZSA membrane with increasing water concentration from 4.31 to 35.11 wt.%. At 100 wt.% water composition, the sorption is very high for both the membranes i.e., 246.45 for the ZSA membrane and 124.66 for the GZSA membrane, respectively. This shows the hydrophilic nature of the membrane, which has an affinity for water and is capable of being selective towards the same during separation. Comparatively, the sorption percentage is less for the GZSA membrane than the ZSA membrane due to the compact nature of the membrane after cross-linking.

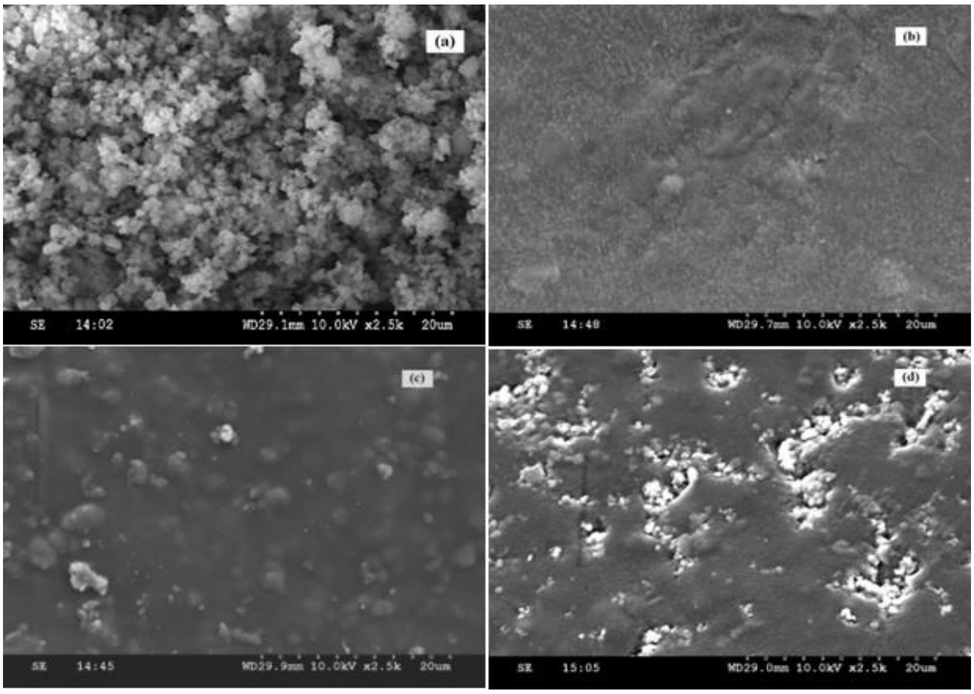


Figure 3: SEM micrographs of (a) ZSM-5 zeolite particles, (b) SA, (c) ZSA, and (d) GZSA membranes.

Table 1: Effect of feed concentration on DS, % of sorption, normalized flux, selectivity, and PSI values for ZSA membrane.

Water in feed (wt %)	Degree of swelling	% of sorption	Normalized flux (j)	Selectivity (α)	PSI (j.α)
0.00	1.0527	5.27	-	-	-
4.31	1.1189	11.89	0.7694	430.78	331.44
10.95	1.2478	24.78	0.8944	253.77	226.97
18.10	1.4845	48.45	1.0903	178.83	194.98
26.02	1.6472	64.72	1.1801	52.62	62.09
35.11	1.8568	85.68	1.2765	29.09	37.13
100.00	3.4645	246.45	-	-	-

Table 2: Effect of feed concentration on DS, % of sorption, normalized flux, selectivity, and PSI values for GZSA membrane.

Water in feed (wt %)	Degree of swelling	% of sorption	Normalized flux (j)	Selectivity (α)	PSI (j.α)
0.00	1.0132	1.32	-	-	-
4.31	1.0576	5.76	0.1648	931.56	153.52
10.95	1.1174	11.74	0.1954	584.09	114.14
18.10	1.2390	23.90	0.2229	219.00	48.83
26.02	1.4885	48.85	0.2566	87.92	22.56
35.11	1.6361	63.61	0.2775	45.64	12.67
100.00	2.2466	124.66	-	-	-

3.3. Ion Exchange Capacity

IEC plays an important role in the values of PV parameters such as flux and selectivity. IEC studies of the GZSA membrane give the residual hydroxyl groups present in the membrane after cross-linking. The IEC values of ZSA and glutaraldehyde crosslinked ZSA is 1.52 mequiv./g and 0.65 mequiv./g, respectively. The IEC results show that more than 57% of the hydroxyl groups are crosslinked with glutaraldehyde and there are still some hydroxyl groups left for sorption and diffusion of water molecules through hydrogen bonding.

3.4. Pervaporation Results

3.4.1. Effect of Feed Composition

PV experiments were performed over a wide range of concentrations of 1,4-dioxane/water mixtures to study the separation behavior of ZSA and GZSA membranes. Experimental results on the effect of feed composition on flux and selectivity are given in Table 1 for the ZSA membrane and Table 2 for the GZSA membrane. From the data, it is noticed that normalized flux increases with the increasing water component of feed concentration in the mixture. These investigations are carried out over a wide range of feed compositions at a temperature of 30°C, pressure of 0.5 mmHg, and a membrane thickness of 60 μm, respectively.

The water normalized flux increases from 0.769 to 1.277 $kg.m^{-2}.h^{-1}.10$ μm for ZSA membrane and 0.165 to 0.278 $kg.m^{-2}.h^{-1}.10$ μm for GZSA membrane for varying water feed concentration from 4 to 35 wt.% water. Mass transport through the hydrophilic membrane occurs by solution diffusion mechanism. At higher feed water concentrations, the membrane swells appreciably because of the presence of more water molecules that are available for sorption and diffusion. On swelling, the polymer chains become more flexible, and hence the transport through the membrane becomes easier for both the feed components resulting in high flux.

Selectivity, on the other hand, decreases from 431 to 29 for ZSA and 932 to 46 for GZSA membranes as the feed water composition increases from 4 to 35 wt.%. The high selectivity values, 431 and 932 for ZSA and GZSA membranes, respectively due to the presence of zeolite particles in both membranes. For GZSA membranes, a normalized flux of 0.223 $kg.m^{-2}.h^{-1}.10$ μm is lower and a selectivity of 219 is higher than that of the uncrosslinked blend (normalized flux; 1.090 $kg.m^{-2}.h^{-1}.10$ μm and selectivity; 179) at the azeotropic composition of 82 wt.% 1,4-dioxane. This is due to the reduced degree of swelling occurring on crosslinking the membrane with glutaraldehyde. On crosslinking, the free volume of the polymer reduces due to the proximity of the chains concerning one other thus enabling preferential permeation of the water molecules thereby increasing the selectivity and decreasing the flux.

PSI values are calculated using Equation (6) and presented in Tables 1 and 2. As it is reflected in the Tables the PSI values of the membranes increase with the increase in 1,4-dioxane concentration in the feed composition. This observation indicates that the performance of both ZSA and GZSA membranes is good at high solvent concentrations.

3.4.2. Effect of Membrane Thickness

The effect of varying membrane thickness on separation performance was studied at constant azeotropic feed composition (82 wt.% 1,4-dioxane) and permeates pressure (0.5 mmHg) by synthesizing membranes of different thicknesses. With an increase in the membrane thickness, a gradual reduction in the flux from 0.182 to 0.140 $kg.m^{-2}.h^{-1}$ for ZSA (Figure 4) and 0.037 to 0.032 $kg.m^{-2}.h^{-1}$ for GZSA (Figure 5) membranes were observed. 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. With an increase in membrane thickness from 60 to 150 μm the selectivity increases from 179 to 324 for the ZSA membrane and 219 to 406 for the GZSA membrane, respectively. In the pervaporation process, the upstream layer of the membrane is swollen and plasticized due to the 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.

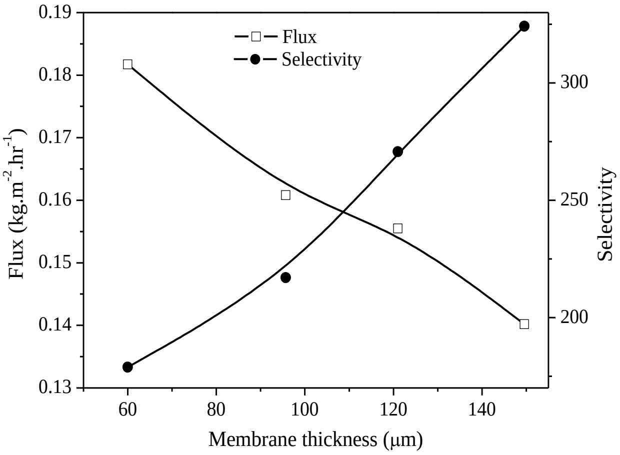


Figure 4: Effect of thickness of ZSA membranes on flux and selectivity.

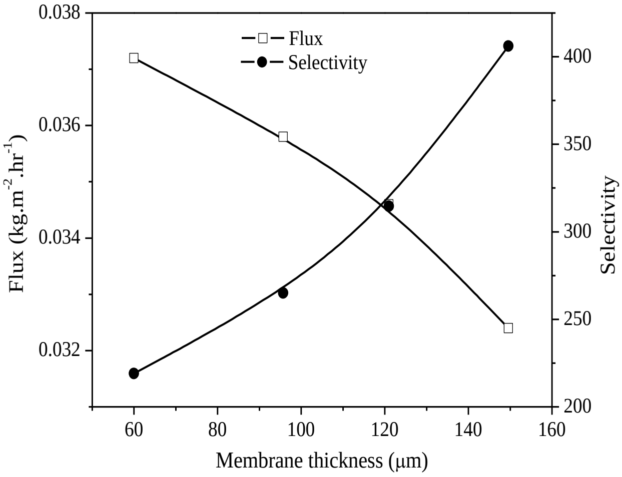


Figure 5: Effect of thickness of GZSA membranes on flux and selectivity.

4. CONCLUSIONS

In this study, ZSM-5 nanosized zeolite-incorporated sodium alginate membranes were prepared and further crosslinked with glutaraldehyde. The two, uncrosslinked (ZSA) and crosslinked (GZSA) filled membranes were used for PV-based dehydration of 1,4-dioxane/water mixture. With increasing feed water compositions, the membranes performance exhibited a reduction in selectivity and an improvement in flux due to increased swelling. As expected, with increasing membrane thickness, selectivity improved, but flux decreased. Though both ZSA and GZSA membranes have a good ability for processing the aqueous azeotrope of 82 wt.% 1,4-dioxane, the crosslinked membranes show better PV performance than that of uncrosslinked membranes. Based on the results, both membranes could be used with PV as an alternative to the traditional distillation process at aqueous azeotrope composition of 1,4-dioxane.

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