Separation of n-hexane/acetone mixtures by pervaporation using high density polyethylene/ethylene propylene diene terpolymer rubber blend membranes

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ABSTRACT

Polymer membranes were prepared by blending high density polyethylene (HDPE) with ethylene propylene diene terpolymer rubber (EPDM). These blend membranes were evaluated for the selective separation of n-hexane from acetone. The flux and selectivity of the membranes were determined both as a function of the blend composition and feed mixture composition. Results showed that polymer blending method could be very useful to develop new membranes with improved selectivity. Pervaporation properties could be optimized by adjusting the blend composition. The effects of blend ratio, feed composition, and penetrant size on the pervaporation process were analyzed. The permeation properties have been explained on the basis of interaction between the membrane and solvents and blend morphology. Flux increases with increasing alkane content in the feed composition.

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1. Introduction

Separation of organic–organic mixtures using membrane separation technique is being investigated extensively owing to its great importance in chemical and petrochemical industries. Pervaporation (PV) is an energy-efficient membrane separation process, which has gained acceptance by chemical industries over the years because of its favourable economics, easy maintenance, and simplicity of the process [1–7]. The pervaporation process can be defined as a selective evaporation of a liquid mixture through a dense polymeric membrane. This method is applicable for the separation of azeotropic mixtures which are difficult to be separated by distillation, extraction of aromatic compounds from dilute solutions, separation of close boiling liquids, recovery of dissolved substances and the separation of organic–organic mixtures. High permeability, good selectivity, and stability are the important factors in choosing a suitable pervaporation membrane [8].

Pervaporation is defined as the selective evaporation of a component from liquid mixture through a membrane. In pervaporation, the liquid mixture to be separated is in direct contact with a membrane on one side and the permeated product is removed as vapor from the other side by applying a low pressure. Unlike other membrane separation processes, pervaporation involves a phase change of permeating species from the liquid to the vapor state. The mass transfer in a PV membrane is based on a solution-diffusion mechanism. The solution-diffusion mass transport involves three steps: (1) sorption of permeant from the feed liquid to the membrane; (2) diffusion of the permeant in the membrane; and (3) desorption of the permeant to the vapor phase on the downstream side of the membrane. The sorption and diffusion are considered as the rate-determining step of the mass transfer.

In recent years, there has been increased interest in the use of the pervaporation membrane separation process for the separation of organic liquid mixtures. Many researchers reported on the separation of binary liquid mixtures by this technique. Sinha et al. [2] reported the use of chemically modified polyvinyl alcohol membranes for the separation of methanol from its mixtures with toluene over the concentration range of 0.5–20 wt % methanol. Mixed matrix membranes based on chitosan and silicalites were used to separate toluene from its mixtures with methanol [9]. Both Polydimethyl siloxane(PDMS) as well as Polyoctylmethyl siloxane (POMS) membranes were applied for the pervaporation of industrial wastewater containing toluene [11]. The pervaporation dehydration of water–ethanol mixtures was investigated using the mixed matrix membranes prepared from natural rubber and crosslinked poly (vinyl alcohol) semi-IPN embedded with the zeolite [10]. A comprehensive review of polymeric membranes

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for pervaporation was published by Shao and Huang [12]. They investigated the potential pervaporation had for separating liquid mixtures in the areas of alcohol and solvent dehydration, organic(s) removal from water and organic/organic separations.

Generally, homopolymer membranes could not meet the demand of pervaporation. Polymer blend membranes are promising materials that can overcome the major difficulties associated with homopolymer system in the pervaporation process. Polymeric blend membrane is of great interest because it is the most versatile way of achieving materials with new desirable properties and also the least expensive [13]. Polymer blends are considered as physical mixtures of two or more polymers. In polymer blends, the properties are controlled by the microstructure of the blends. Polymer blend membranes are extensively used in the pervaporation membrane field for obtaining high-performance [14–19]. High density polyethylene (HDPE)/ethylene propylene diene terpolymer rubber (EPDM) blend membranes possess very good properties. To the best of our knowledge, the use of HDPE/EPDM blend membranes for the pervaporation process has not yet been examined.

The main objective of this work is to develop a new polymer membrane based on the HDPE/EPDM blend for the selective separation of aliphatic hydrocarbons from alkane–acetone mixtures by pervaporation. The alkane/acetone mixture has been chosen to perform a basic study on the pervaporation process using the membrane. The separation efficiency has been evaluated as a function of blend ratio and feed composition.

2. Experimental

2.1. Materials

High density polyethylene (HDPE-Relene, M60 200) of density $932 \, kg \, m^{-3}$ and melt flow index $20 \, g/10 \, min$ (at $230 \, °C/2.16 \, kg$) was obtained from Reliance Industries Ltd. Hazira Gujarat, India. EPDM with an E/P ratio of 62/38 and a diene content of 3.92% supplied by Herdillia Unimers, New Mumbai was used. The solvents n-pentane, n-hexane, n-heptane and acetone (Merck India, Ltd., Mumbai, India) were distilled twice before use. All other ingredients were of laboratory reagent grade, supplied by Bayer India, Ltd., Mumbai, India.

2.2. Preparation of membranes

The blends were prepared in a Brabender plasticorder by melt mixing of the components at 160 °C and a rotor speed of 60 rpm. HDPE was melted for 2 min and then EPDM was added. The mixing was continued for 5 min. Dynamically vulcanized blends were also prepared by using three different vulcanizing systems such as sulphur, peroxide (DCP) and a mixture of sulphur and peroxide (mixed). In the case of dynamically crosslinked blends, after blending HDPE with EPDM, the curing agents were added and mixing is continued for 3 min. Membranes were prepared by compression molding the melt mixed blends in a hydraulic press at 170 °C (at $200 \, kg/cm^2$ pressure). The thin membranes thus obtained were used for pervaporation experiments. The average thickness of the membrane was 0.2 mm.

The binary blends with varying compositions are noted as H_{100}, H_{70}, H_{50}, H_{30} and H_0 where the subscripts denote the weight % of HDPE in the blend. The formulation of the mixes used is given in Table 1.

2.3. Phase morphology studies

Scanning electron microscope (JEOL JSM 35C) was used to study the phase morphology of blends. The compression-molded samples were cryogenically fractured under liquid nitrogen and the

Table 1
Formulation of mixes in phr.[a]

Ingredients	HDPE/EPDM 50/50 blend (crosslinking systems)		
	Sulphur	Mixed	Peroxide
EPDM	50	50	50
HDPE	50	50	50
Stearic acid	2	2	–
Zinc oxide	5	5	–
MBTS[b]	0.05	0.05	–
TMTD[c]	0.1	0.1	–
Sulphur	0.2	0.1	–
DCP[d]	–	0.5	1

[a] Parts per hundred rubber.
[b] Dibenzothiazole disulphide.
[c] Tetramethyl thiuram disulphide.
[d] Dicumyl peroxide.

EPDM phase was preferentially extracted from the samples using cyclohexane at room temperature for 5 days. Fracture surface was sputter coated with gold in a sputter coating machine (Balzers SCD 050) for 150 s.

2.4. Swelling studies

The swelling behavior of the membranes was assessed by immersing them in mixtures of n-hexane and acetone of different compositions at 28 °C for 72 h. After reaching equilibrium, the membranes were taken out from the mixtures, their surfaces wiped with a filter paper, and then they were weighed immediately in an electronic balance. The swelling ratio (S) was determined as

$$S = \frac{W_s - W_d}{W_d} \tag{1}$$

where W_d and W_s are the weight of dry and swollen membranes, respectively.

2.5. Pervaporation experiments

The pervaporation experiments were performed using the apparatus shown in Fig. 1. The permeation cell was assembled from two half-cells of column couplers made of glass and fastened with bolted clamps. The capacity of each half cell was around 100 mL, and the effective surface area of the membrane was $19.4 \, cm^2$. The membrane was supported on a finely porous stainless steel plate with holes drilled in it. Vacuum at the downstream side was measured with a vacuum gauge. The membrane was kept in the
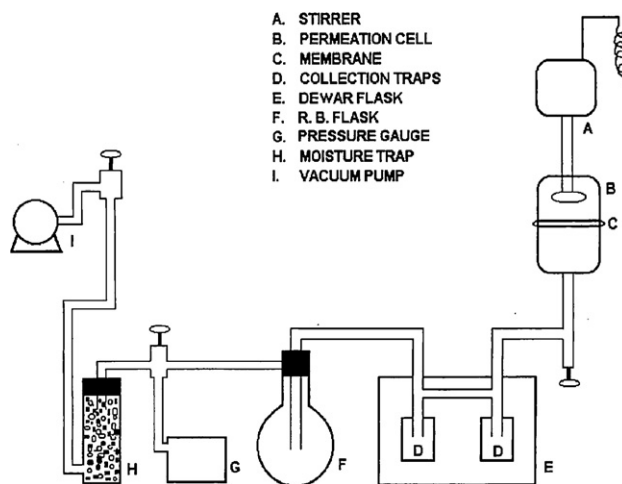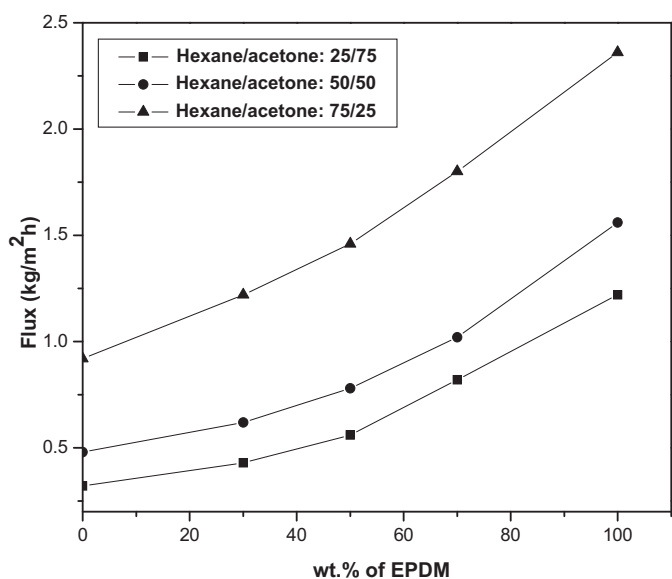

A. STIRRER
B. PERMEATION CELL
C. MEMBRANE
D. COLLECTION TRAPS
E. DEWAR FLASK
F. R. B. FLASK
G. PRESSURE GAUGE
H. MOISTURE TRAP
I. VACUUM PUMP

Fig. 1. Pervaporation apparatus.

Fig. 2. Effect of the blend ratio on the pervaporation flux at three different hexane–acetone compositions.

Fig. 3. Effect of blend composition on the pervaporation selectivity at 50/50 hexane–acetone compositions.

Pervaporation cell for about 2 h in each run to reach equilibrium conditions before collecting the permeate. The permeated vapors were completely condensed in a trap placed in a Dewar flask cooled to −77 °C using dry ice–acetone mixture. The permeate and feed compositions were analyzed by a gas liquid chromatography. Pervaporation experiments were carried out in triplicate at 28 °C using freshly prepared feed solution each time to check the reproducibility of measurements.

The performance of a membrane in pervaporation is characterized by permeation rate or flux (J) and selectivity (α_{AB}). Flux was determined by measuring the weight of liquid collected in the cold traps during a certain time at steady-state condition. The pervaporation selectivity α_{AB} is defined as

$$\alpha_{AB} = \frac{Y_A/Y_B}{X_A/X_B} \tag{2}$$

where Y_A and Y_B represent the weight fraction of n-hexane and acetone in the permeate and X_A and X_B those of the n-hexane and acetone in the feed, respectively.

The Pervaporation separation index (PSI) has been used to evaluate the overall performance of the membranes. It can be determined from the relation [20,21]

$$PSI = J \ (\alpha_{AB} - 1) \tag{3}$$

3. Results and discussion

3.1. Effect of blend composition

The separation of hexane–acetone mixtures through HDPE/EPDM blend membranes was evaluated to investigate the polymer blend concept in developing membrane materials for pervaporation. Effects of blend ratio on the pervaporation flux at three different hexane–acetone compositions are shown in Fig. 2. From the figure, it can be observed that the transport properties of the HDPE/EPDM blends depend strongly on the HDPE/EPDM ratio in the blend. Pervaporation flux is lowest for the 100/0 HDPE/EPDM membranes and it slowly increases with an increase in the wt% of EPDM in the blend up to 50% followed by a sharp increase. This figure shows that by controlling the EPDM content in the blend a variety of fluxes can be obtained for any feed composition. In the case of a feed mixture of hexane–acetone (50/50), for example,
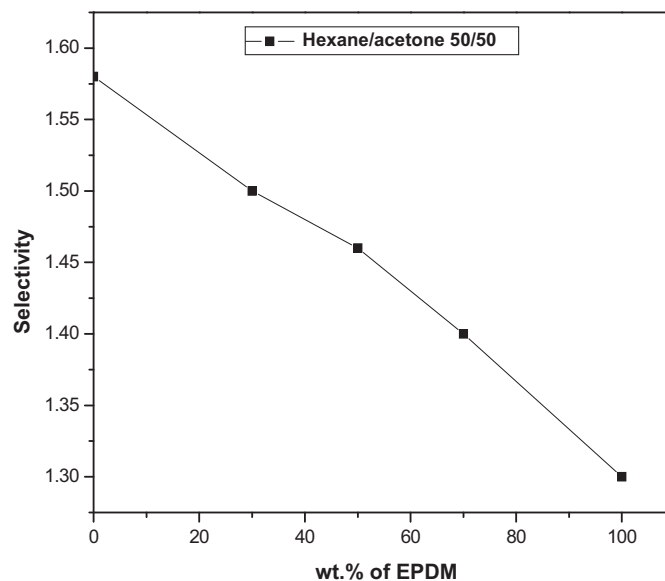
the flux increases from 0.62 to 1.02 kg/m² h as the EPDM content increases from 30 to 70 wt%.

The effect of the blend ratio on the pervaporation selectivity at 50/50 hexane–acetone composition is shown in Fig. 3. The increasing EPDM content in the membrane has a retarding effect on the selectivity. All membranes are selective to alkane, and the selectivity decreases with an increasing EPDM content in the blend. Upon increasing the concentration of EPDM from 50 to 70 wt % there is a sudden change in the value of selectivity. This Pervaporation behavior can be explained based on the extent of interaction between the membrane and the solvents and the morphology of the blend. Based on vapor pressure data, one would expect that the acetone is to pass first the membrane and evaporated. However, the driving force in the pervaporation process is the solubility parameter, which overshadows the effect of vapor pressure. The solubility parameter values of EPDM, HDPE, n-hexane and acetone are 16, 16.96, 14.9 and 20.3 (J/m³)^{1/2}, respectively. Due to the closer solubility parameter values, there is a strong interaction between EPDM and n-hexane. Therefore, with increasing EPDM content in the blend, the interaction between the blend and n-hexane increases. Interaction effectively increases the frequency and amplitude of rubber chain motions, thereby allowing the permeate molecules to pass through the membrane easily. As a result, the permeation rate increases and the selectivity decrease. Similar results were reported earlier [22].

A slow increase in the flux is observed initially even though the interaction between the membrane and alkane increases with increasing EPDM content in the blend. This can be correlated to the phase morphology. The scanning electron micrographs of H_{70}, H_{50} and H_{30} blends are given Fig. 4(a–c). It is seen that in H_{70} and H_{50}, the EPDM phase gets dispersed as spherical domains in the continuous HDPE matrix. The two phase morphology retards the passage of the solvent mixture. Hence, the flux is less and selectivity is higher. But, in H_{30} both exhibit a co-continuous morphology (Fig. 4(c)). Due to the co-continuous morphology, passages of the penetrant become easier and hence flux increases and permselectivity decreases. A similar behavior has been observed in the case of heptane/acetone and pentane/acetone, as shown in Fig. 5. From Fig. 5, we can see that the flux increases with the size of the hydrocarbon molecule. The Pervaporation separation index (PSI) was calculated using Eq. (3) and is plotted as a function of the blend
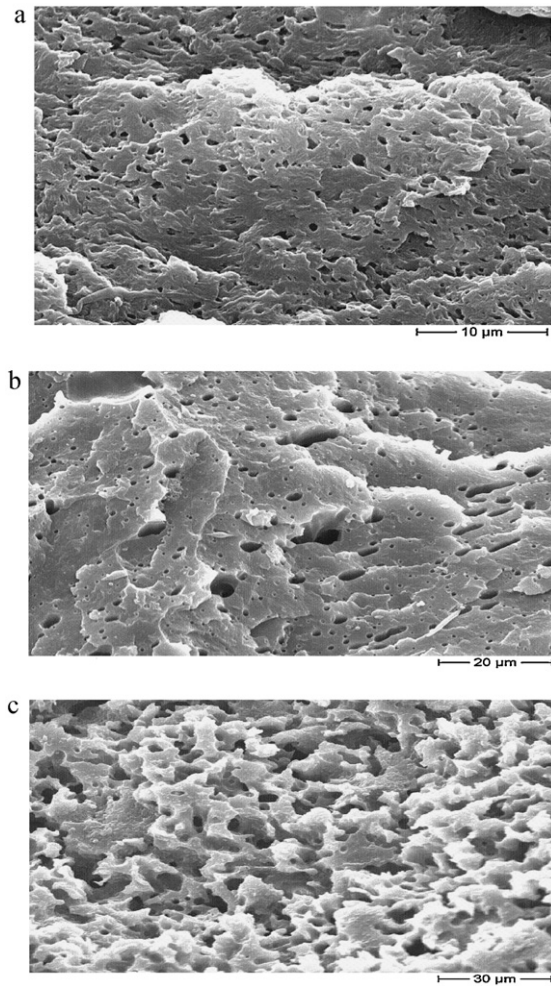
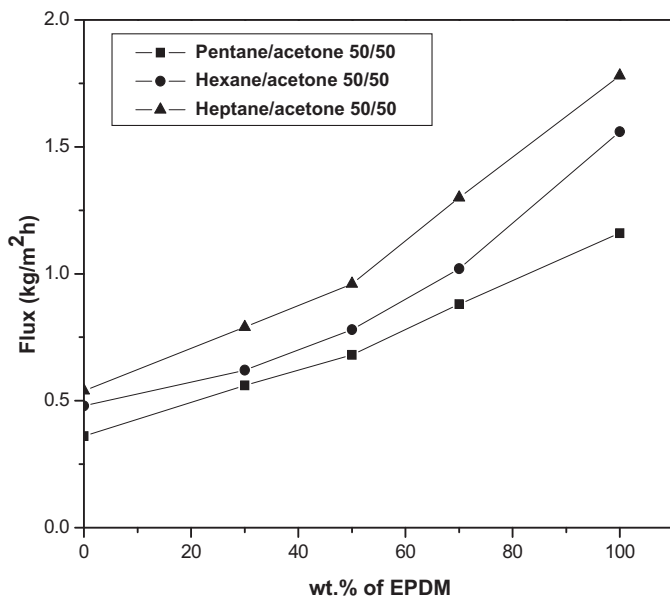**Fig. 4.** (a–c) SEM micrographs of H_{70}, H_{50}, and H_{30} blends.

Fig. 5. Variation of flux with blend ratio for two different alkane/acetone mixtures.
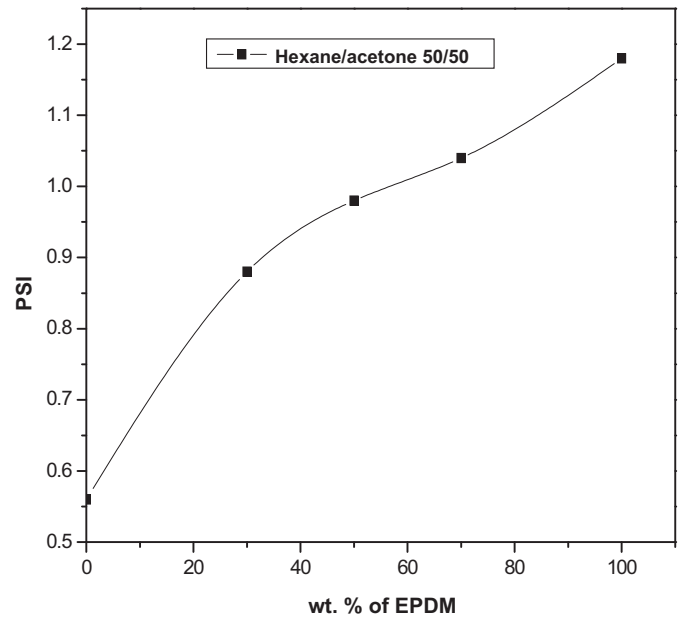

Fig. 6. Effect of blend ratio on the pervaporation performance of the membranes.

composition in Fig. 6 for hexane/acetone 50:50 composition. From the figure, we can see that the overall performance of the blend membrane increases with the increase of EPDM content in the blend. The positive deviation shown by the blend membranes suggest that they can be used as potential membranes for separating liquid mixtures.

3.2. Effect of dynamic vulcanization

Figs. 7 and 8 show the effect of crosslinking on the permeation rate (flux) and selectivity of the hexane in the hexane/acetone mixture respectively. It can be seen from the figure that the HDPE/EPDM blend vulcanized by the sulphur system exhibits the highest permeation rate and then mixed system followed by DCP. But, a reverse trend was observed for the selectivity, as shown in Fig. 8. The peroxide vulcanized HDPE/EPDM blend membrane exhibits the highest
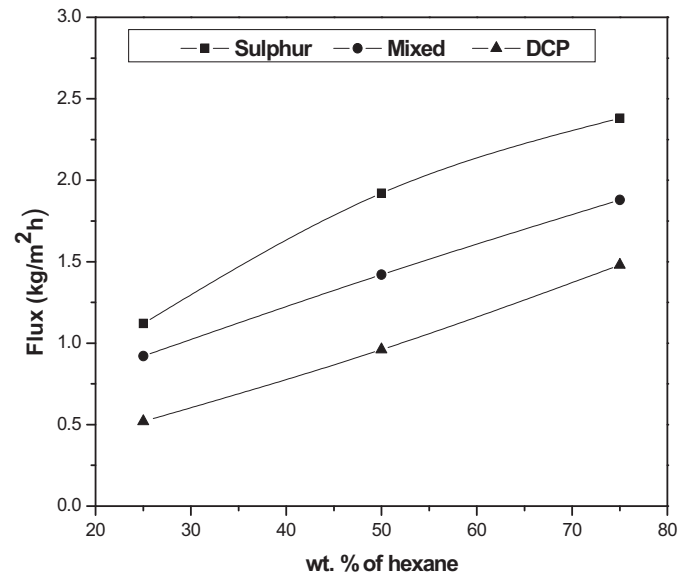

Fig. 7. Effect of feed composition on the permeation rate of hexane in hexane/acetone mixture.
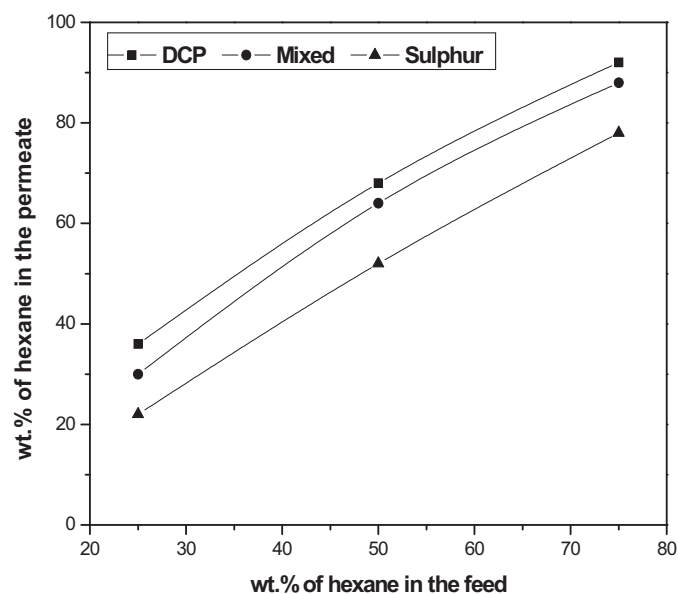
Fig. 8. Effect of feed composition on the selectivity of hexane in hexane/acetone mixture.

selectivity and the sulphur vulcanized blend membrane the lowest. Mixed system occupies intermediate position. In order to closely investigate this observed behavior, the degree of crosslinking was determined from stress–strain measurements. Mooney-Rivlin [23] equation was used for calculating the amount of crosslinks by stress–strain measurements.

$$\nu = \frac{F}{2A_0 \rho RT(\alpha - \alpha^{-2})} \tag{4}$$

where ν is the physically effective crosslink density; F is the force at extension ratio α; A_0 is the area of cross-section; R is the universal gas constant; and T is the absolute temperature. Calculated values of the crosslink densities are given in Table 2. Membranes vulcanized by the sulphur system have the lowest degree of crosslinking and that by the peroxide (DCP) system the highest. As the degree of crosslinking decreases, the flexibility of the rubber chains increases and thereby the free volume available in the membrane increases. Thus, the permeation rate decreases from sulphur to peroxide. The high selectivity exhibited by the peroxide membrane is probably due to the high diffusivity of the hexane molecules. The swelling behavior of the blend membranes shown in Fig. 9 is also in agreement with this observation. The swelling ratio is highest for HDPE/EPDM blends vulcanized with sulphur system and lowest for the peroxide system. That is, the swelling ratio is highest for systems with lowest degree of crosslinking and lowest for systems with highest degree of crosslinking.

3.3. Effect of feed composition

The total flux J can be divided into the component flux of alkane, J_{alkane} and that of acetone, $J_{acetone}$ using the permeate composition data [22].

$$J_{alkane} = J \times Y_{alkane} \tag{5}$$

Table 2
Values of crosslink density (ν).

Vulcanization system	Crosslink density (ν) $\times 10^4$ (g mol/cm^3)
Sulphur	3.18
Mixed	5.74
Peroxide	6.98

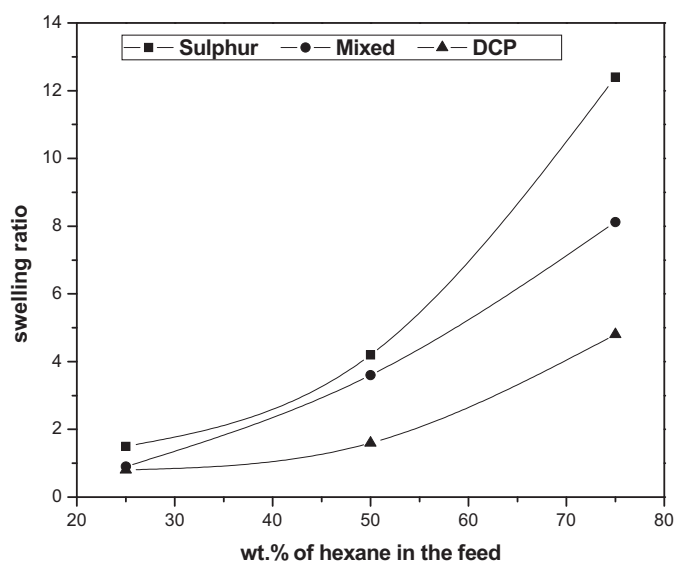**Fig. 9.** Effect of feed composition on the swelling ratio of membranes.

$$J_{acetone} = J \times Y_{acetone} \tag{6}$$

where Y_{alkane} and $Y_{acetone}$ are the mass fractions of the alkane and acetone in the permeate respectively. For example, the component fluxes of hexane/acetone (50:50) mixture calculated from these equations are shown in Fig. 10 as a function of feed composition for the DCP membrane. From the figure, we can see that with the increase of feed composition, the hexane flux increases and acetone flux decreases. The increase in selectivity with increase of feed composition is due to the increase of hexane flux compared to acetone flux. Similar behavior is observed in the case of sulphur and mixed systems too. Therefore the high selectivity of peroxide membrane is also attributed to the higher percent of hexane flux compared to the other systems. The high hexane flux and very low percent of acetone flux contribute significantly towards the high selectivity of DCP membranes.

According to the solution-diffusion model previously described, the permselective properties of pervaporation membranes are
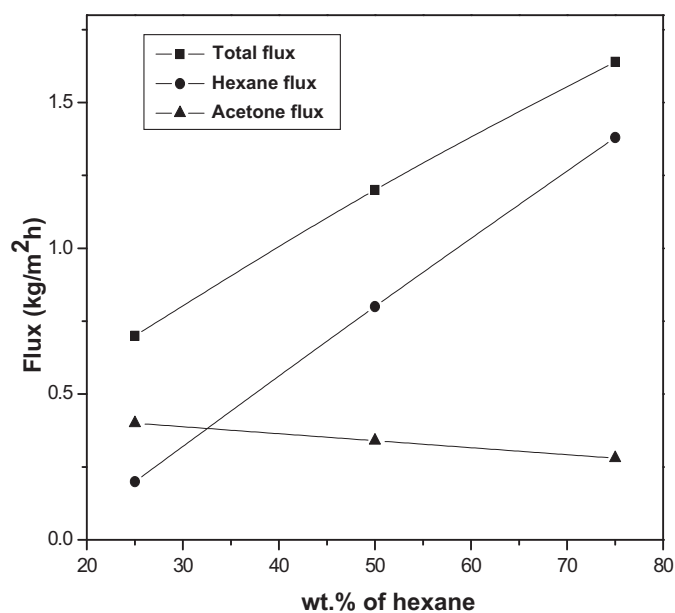

Fig. 10. Effect of feed composition on total and component fluxes of hexane/acetone mixture.
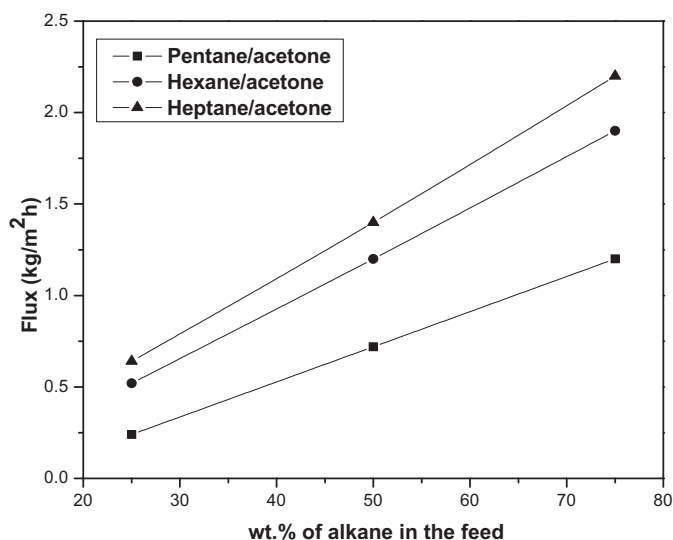
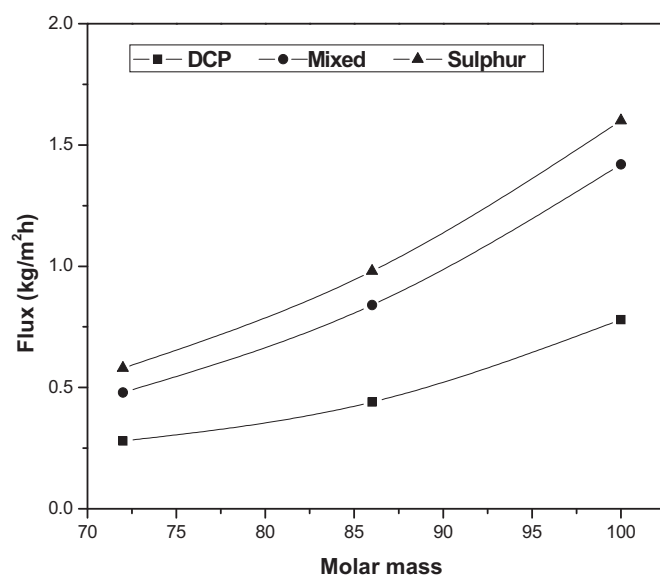Fig. 11. Effect of feed composition on the pervaporation flux.

Fig. 12. Effect of molecular size of alkane in the alkane/acetone feed mixture on pervaporation flux.

Fig. 13. Diffusion profile of the 50/50 HDPE/EPDM blend membrane in n-alkanes.

determined by an increase of the solubility and diffusivity of the permeating components in the membrane. As both sorption and diffusion phenomena are dependent on the composition of the liquid mixture, permeation characteristics of membranes are usually strongly influenced by the feed composition. The effect of feed composition on the pervaporation fluxes is shown in Fig. 11. From the figure, it can be seen that low fluxes are obtained when the alkane content in the feed composition is low. The fluxes increase with an increase of the alkane concentration in the feed. Among the three curves shown, the flux is highest for the heptane/acetone mixture and lowest for the pentane/acetone mixture. This behavior can be explained on the basis of a general pervaporation equation as well as the interaction between the membrane and penetrants.

When a penetrant i diffuses through a membrane, the flux J_i is the product of the concentration C_i and the linear velocity v_i of the penetrant inside the membrane. The velocity is the product of mobility B_i and driving force. In the case of pervaporation, the driving force is a gradient in the chemical potential across the membrane (i.e., $-d\mu_i/dx$). Therefore, the following relation expresses the flux in a pervaporation process [24].

$$J_i = v_i C_i = -C_i B_i \frac{d\mu_i}{dx} \qquad (7)$$

According to this relation, the flux should decrease as the activity (i.e., the chemical potential of a mixture component in the feed) decreases because the activity in the permeate side is kept constant by continuous evacuation. Thus, as the concentration of alkane in the feed increases, the flux also increases. It is also significant to note that as the alkane concentration in the alkane/acetone mixture increases, the interaction between the alkane and the membrane also increases. This interaction increases the flexibility of the rubber chains, which results in increased permeation rate.

3.4. Effect of penetrant size

In order to study the effect of molecular size of the penetrants on the Pervaporation properties, pentane/acetone, hexane/acetone and heptane/acetone mixtures were examined through HDPE/EPDM blend membranes in pervaporation. Fig. 12 shows the effect of the molecular size of the alkanes on the permeation rate (flux) of alkane/acetone mixture through HDPE/EPDM blend membranes. The factors which influence the pervaporation process are molecular size, interaction between the polymer and solvent etc. Normally pervaporation rate decreases with increasing
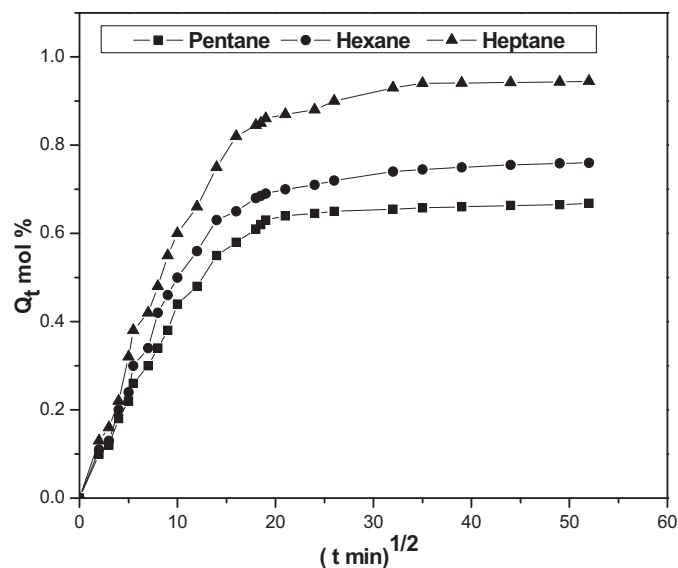
molecular size. Contrary to our expectation, the permeation rate is increasing with increasing molecular size (Table 3). This might be due to the different degree of interaction between the polymer and the solvent. The HDPE/EPDM blend membranes seem to have higher affinity for heptane than pentane and hexane, because

Table 3
Values of flux for various penetrants.

Vulcanizing system	Penetrant	Flux (kg/m² h)
Sulphur	Pentane	0.58
	Hexane	0.98
	Heptane	1.6
Mixed	Pentane	0.48
	Hexane	0.84
	Heptane	1.42
Peroxide	Pentane	0.28
	Hexane	0.44
	Heptane	0.78

the solubility parameter difference between the blend and solvents is lowest for heptane ($\Delta\delta = 1.38$) than for other alkanes ($\Delta\delta$ pentane = 2.18, $\Delta\delta$ hexane = 1.58). This stronger interaction results in higher sorption, and thereby a higher penetration of the heptane mixtures than the other solvents. Lloyd and Meluch [25] used the ratio Δ_{AM}/Δ_{BM} (ratio of solubility difference between the permeant and membrane) as a measure of preferential sorption for the permeants A and B in the membrane M and as an index for membrane material selection. According to them, component B permeates more compared to A if Δ_{AM}/Δ_{BM} is maximum. Here A represents acetone and H, alkane. The Δ_{AM}/Δ_{BM} values for heptane, hexane and pentane blend systems are 2.77, 2.42 and 1.75, respectively. From these values it is clear that the permeation rate increases with increase in molecular size. The diffusion profile shown in Fig. 13 is also in agreement with this observation.

4. Conclusion

HDPE/EPDM blend membranes were prepared and their pervaporation performance investigated in the alkane–acetone mixture. The flux increased with the increase in EPDM content in the blend. However, the selectivity decreased. The interaction between the membrane and the solvents increases with increase in EPDM content in the blend due to the closer solubility parameter values. This behavior is associated with the interaction between the membrane and the penetrants, besides the morphology of the blend system used. In H_{70} and H_{50}, the EPDM phase gets dispersed as spherical domains in the continuous HDPE matrix. The two phase morphology retards the passage of the solvent mixture. Hence, the flux is less and selectivity is higher. But, in H_{30} both exhibit a co-continuous morphology. Due to the co-continuous morphology, passage of the penetrant becomes easier and hence flux increases and selectivity decreases.

The separation efficiency for different vulcanizing systems varies in the order DCP > Mixed > Sulphur. The differences in the pervaporation performance of the membranes were explained in terms of the extent of crosslinking. This behavior is also attributed to the variation in component fluxes. The Pervaporation separation index values indicate that the best overall pervaporation performance is exhibited by DCP membrane.

It was also observed that the membrane performance was strongly influenced by the feed mixture composition. For all the alkane–acetone mixtures the flux increased with increasing alkane concentration in the feed. As the alkane concentration in the feed increases, the interaction between the membrane and penetrant increases and thus permeation rate.

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