

Effect of MEA on the performance of polysulfone/polyvinyl acetate blend membranes

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Received, January 9, 2018; Revised, August 2, 2018

Separation of CO₂ from natural gas within the field of membrane technology has been carried out for a long time. For efficient separation of CO₂/CH₄, high-performance gas separating membranes are desired. The blending technique enhances the chemical and thermal stability, also taking heed in the improvement of separation properties with the economic sustainability. This research studies the perm-selective performance of polymeric blend membranes, glassy polymer (polysulfone) and rubbery polymer (polyvinyl acetate) with monoethanolamine (MEA). The polymeric amine blend membranes with enhanced properties were synthesized with different blending ratios in dimethylacetamide (DMAc) solvent. In this study, various compositions of polyvinyl acetate (PVAc) ranging from 5 to 20 wt. % were blended with polysulfone (PSU) and MEA, and were used for the formation of flat sheet membranes *via* evaporation method. The gas permeation results showed that the CO₂ permeance increased with increasing feed pressure.

Keywords: Methane, Carbon dioxide separation, Polymeric blend, Enhanced polymeric blend membrane, Permeance, Selectivity.

INTRODUCTION

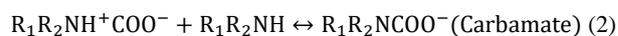
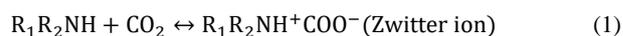
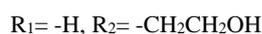
The separation of carbon dioxide from natural gas in the area of membrane technology has been a much-known practice. It provides multitudes of benefits in comparison with other techniques, which points to its feasibility for gas separation. The performance of gas separating membranes is quite important for effective separation of CO₂ from natural gas. Thus, in the development of the membrane, permeance and selectivity factors are essential that need to be addressed for efficient CO₂ separation [1, 2].

Numerous glassy polymeric materials were utilized for membrane fabrication in gas separation established over scientific or industrial research practices. Polysulfone (PSU) was selected due to its good mechanical, thermal and chemical stability with adequate gas performance [3, 4]. Combining these features with low price, polyvinyl acetate (PVAc) is a useful polymer displaying piezoelectric, ferroelectric and pyroelectric properties [5]. The typical benefits of polyvinyl acetate are formability, flexibility, and low density [6].

Monoethanolamine (MEA), which belongs to primary amines, is widely used for removing CO₂ from natural gas. It is a relatively strong base having high reactivity with CO₂, low cost of solvent and thermal stability. However, it was replaced by more efficient systems because of its corrosive properties, degradation of solvent issues, high heat of reaction with CO₂ and substantial energy consumption for

regeneration of the used solvent. The carbon dioxide loading capacity for primary and secondary amines is in the range from 0.5–1 mole of CO₂ per mole of amine, such as a fraction of the carbamate species is hydrolyzed to form hydrogen carbonates [7].

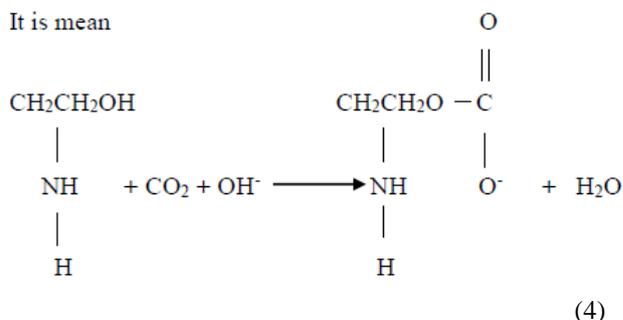
Numerous researchers have examined the behavior of CO₂-amine solutions because of their significant industrial application for removal of CO₂ from gas streams. The Dankwerts' zwitterions mechanism is widely accepted for amine reaction with CO₂ [8]. The reactions between CO₂ and primary amines, where R represents the functional group, for MEA are:



Overall reaction is:



It is mean



Monoethanolamine has the tendency to sanitize the acid gas from natural gas [9]. Hence, by blending

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a PSU/PVAc with monoethanolamine solutions, the separation capability is enhanced for the CO₂/CH₄ mixture. Diverse amines have unlike reaction rates with respect to various acid gases. Accumulatively, the separate amines differ in their equilibrium absorption characteristics for the various acid gases and hence display various sensitivities regarding solvent stability and corrosion factors.

On account of essential amines like MEA, the CO₂ is caught by a chemical absorption process in which the CO₂ reacts with the amine as a carbamate. The main objective of this study is to develop an enhanced polymeric membrane with the addition of

the amine. The different studies regarding blend membrane are summarized in Table 1 [5, 10-23].

METHODOLOGY

Materials and Membrane Synthesis

Polysulfone (PSU) Udel® P-1800, with a glass transition temperature (T_g) of 185°C, was attained from Solvay Advanced Polymers; L.L.C, U.S. in powdered form. Polyvinyl acetate (PVAc) was purchased from Sigma-Aldrich in the form of beads having a glass transition temperature (T_g) 30°C and average $M_w \sim 100,000$ by GPC.

Table 1. Different studies on polymeric blend membranes

Year	Polymers	Gas type	Remarks	Permeance	Selectivity
2006	PU based PAI-PEI (glassy-glassy) blend	CO ₂ /N ₂	CO ₂ /N ₂ selectivity of PU-based blend membranes is increased	P _{CO2} =20.06 Barrer	$\alpha \frac{CO_2}{N_2} = 22.56$
2006	PU-PDMS (rubbery-rubbery) cross-linked	CO ₂ /CH ₄	Highest permeability, diffusivity, and solubility values are found for the PDMS membranes	P _{CO2} =10 Barrer	$\alpha \frac{CO_2}{CH_4} = 0.41$
2008	PI-PES (glassy-glassy) blend MMM	O ₂ /N ₂	Ideal separation factors of O ₂ /N ₂ is increased	P _{O2} =4.2 Barrer	$\alpha \frac{O_2}{N_2} = 3.8$
2009	PVAm-PVA with porous PES support	CO ₂ /N ₂	Ultra-thin membrane with good permeability and selectivity	P _{CO2} =0.58 m ³ (STP)/(m ² h bar)	$\alpha \frac{CO_2}{N_2} = 74$
2010	PEG-PDMS	CO ₂ /CH ₄	Remarkably, the CO ₂ /H ₂ selectivity is enhanced	P _{CO2} =530 Barrer	$\alpha \frac{CO_2}{CH_4} = 10$
2010	PES-PI (glassy-glassy)	O ₂ /N ₂	Gas permeance of N ₂ increased with increase in feed pressure	P _{O2} =210.8 GPU	$\alpha \frac{O_2}{N_2} = 5.4$
2011	SPEEK-Matrimid	CO ₂ /CH ₄	Cross-linked for anti-plasticization	P _{CO2} =9.43 Barrer	$\alpha \frac{CO_2}{CH_4} = 27.98$
2011	PEI-PVP	CO ₂ /CH ₄ , CO ₂ /N ₂	Significant improvement in selectivity	P _{CO2} =1.66 GPU	$\alpha \frac{CO_2}{CH_4} = 55.33$
2012	PIM-1/Matrimid	CO ₂ /CH ₄ , CO ₂ /N ₂	Increased selectivity	P _{CO2} =50 Barrer	$\alpha \frac{CO_2}{CH_4} = 31$
2013	PSF-PVP	CO ₂ /CH ₄	Studied effect of solvents	P _{CO2} =275 GPU	$\alpha \frac{CO_2}{CH_4} = 5.75$
2013	PU-PVA	CO ₂ /CH ₄	Increased CO ₂ permeability	P _{CO2} =49.5 Barrer	$\alpha \frac{CO_2}{CH_4} = 10.1$
2013	PEG-PDMS	CO ₂ /N ₂	Significant improvement in selectivity	P _{CO2} =20.0 GPU	$\alpha \frac{CO_2}{N_2} = 18$
2014	CA-TiO ₂ blend	CO ₂ /CH ₄	Permeability of CO ₂ was found to be increased for CA-TiO ₂ blended	P _{CO2} =3.43×10 ³ Barrer	$\alpha \frac{CO_2}{CH_4} = 23.3$
2015	PES-PVAc	CO ₂ /CH ₄	CO ₂ permeance increase due to the addition of PVAc in PES	P _{CO2} =4.15 GPU	$\alpha \frac{CO_2}{CH_4} = 1.40$
2016	PSF/PI	CO ₂ /CH ₄	Improved selectivity	P _{CO2} =3.3 Barrer	$\alpha \left(\frac{CO_2}{CH_4} \right) = 13.06$

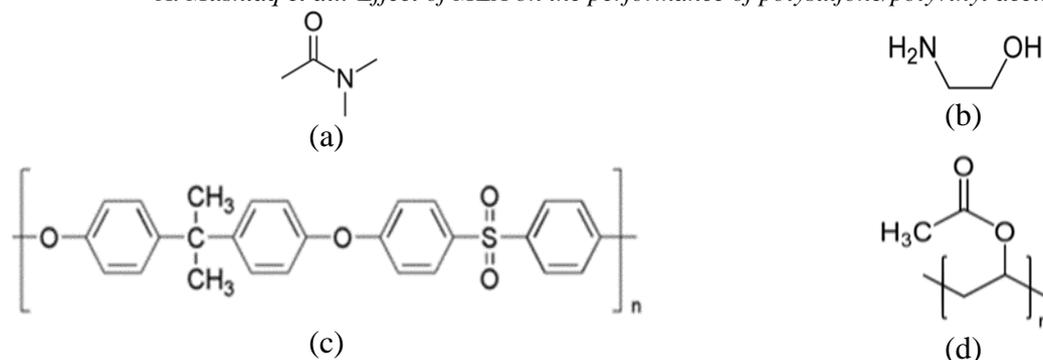


Fig. 1. Chemical structure of solvent, amine and polymers (a) DMAC (b) MEA (c) PSU (d) PVAc

Table 2. Composition of enhanced polymeric blend membranes

Membrane	Polymer, wt. %		Amine 10 wt. %
	PSU	PVAc	
1	100%	0%	-
2	0%	100%	
3	95%	5%	MEA
4	90%	10%	
5	85%	15%	
6	80%	20%	

Monoethanolamine (MEA) (boiling point 170.0°C) and dimethyl acetamide solvent (boiling point 160°C) with 99.99% purity were purchased from Merck. The chemical structures of the solvent, amine and polymers are shown in Figure 1. This study includes the blending of glassy (PSU) and rubbery (PVAc) polymers, and methyl diethanolamine was taken in DMAC solvent. Blending of 20% wt/wt was performed. The different compositions of enhanced polymeric blend membranes are presented in Table 2. Polysulfone was preheated for one night to eliminate moisture. Firstly, the polyvinyl acetate was allowed to dissolve completely in dimethylacetamide. Then PSU and MEA were added under continuous stirring for 24 h at room temperature to attain a homogeneous blend. To clear the solution from air bubbles, bath sonication in a Transonic Digital S, Elma® for one hour was performed. Polysulfone and amine were completely dissolved without any evidence of deposition, which qualified it as a miscible polymer blend. The blend was then cast on a glass plate *via* casting knife with an opening of 200 μm. These casted membranes were sited in a drying room at ambient temperature for five days to allow for the evaporation of the solvent. The membranes were then peeled from the glass plate to check the performance of CO₂ and CH₄ separation. Table 2

shows the composition of the polymer blended membranes. The equipment used for the gas permeation performance was a CO₂SMU unit, where CO₂/CH₄ flow rate (0.1cm³/sec) was measured by a flow controller meter. These trials were performed under ambient pressure (101 ± 2kPa) at room temperature (302 ± 2K). Figure 2 shows the methodology for fabrication of polymeric amine blend membrane.

The permeance of the CO₂ and CH₄ gases was calculated by the following equations [24]:

$$\frac{P_{CO_2}}{l} = \frac{J_{CO_2}}{\Delta P_{CO_2}} \quad (5)$$

$$\frac{P_{CH_4}}{l} = \frac{J_{CH_4}}{\Delta P_{CH_4}} \quad (6)$$

where J is the flux of CH₄ and CO₂ gases, ΔP is the differential partial pressure across the membrane for the gases and l denotes the thickness of the membrane. Thus, the gas selectivity (α_{CO_2/CH_4}) was calculated by taking the ratios of the CO₂ and CH₄ permeance [24]:

$$\alpha_{CO_2/CH_4} = \frac{P_{CO_2}/l}{P_{CH_4}/l} \quad (7)$$

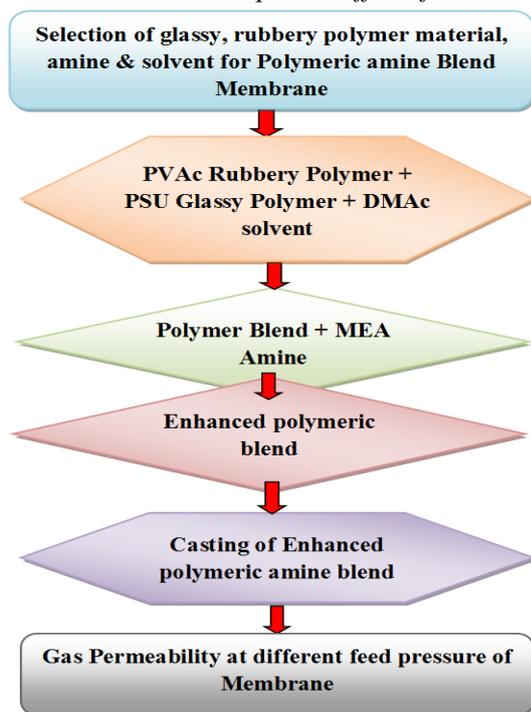


Fig. 2. Methodology for fabrication of polymeric amine blend membranes

RESULTS & DISCUSSION

Gas Permeance Evaluation

In this study, when the gases CO_2 and CH_4 will pass through the EPBM, the amine absorbs the maximum CO_2 . The CO_2 permeation rate will be higher as compared to CH_4 ; the membrane will not absorb the maximum amount of CH_4 so its permeation rate in the membrane will be less.

Figure 3 shows that base polysulfone membrane's permeance of CO_2 was reduced with increasing pressure due to the fact that the glassy polymer is a mixture of crystalline and amorphous phases. The crystallites act as effectual cross-links to decrease the area accessible for permeation, thus permeance of PSU decreases [25]. On the other hand, for the base PVAc membrane, the permeance of CO_2 (14.48 ± 0.01 to 40.24 ± 0.08 GPU) and CH_4 (3.34 ± 0.1 to 15.09 ± 0.08 GPU) increases with the

increase of pressure (2-10 bar) as shown in Figures 3 and 4. Due to PVAc membrane that exists above T_g there is a large intersegmental polymer chain motion [26, 27]. Figure 3 represents the increasing trend of CO_2 permeance when MEA amine is blended in PSU and PVAc. The CO_2 permeance increased in various compositions of PSU/PVAc with MEA amine membranes. The maximum CO_2 permeance for PSU80%/PVAc20%/MEA was 12.14 ± 0.02 GPU to 53.20 ± 0.29 GPU with an increase in the pressure from 2 to 10 bar as shown in Figure 3. The PVAc percentage increased in PSU with MEA 10 wt. %, the permeance of CO_2 also rose with pressure increase due to the fact that MEA amine has captured the CO_2 . The high CO_2 permeance of membranes with increased PVAc content in PSU was likely due to the blended membrane's rising affinity for CO_2 , especially since CO_2 has a non-polar linear structure and smaller kinetic diameter (3.3 \AA) [28-33]. In Figure 4 the CH_4 permeance slightly increased in various compositions of PSU/PVAc with MEA amine membranes, for PSU80%/PVAc20%/MEA from 1.33 ± 0.01 GPU to 1.78 ± 0.02 GPU with an increase in the pressure from 2 to 10 bar. As compared to CO_2 , the slow moving CH_4 molecule has a kinetic diameter of 3.8 \AA and a tetrahedral structure [28]. By addition of MEA amine in the PSU/PVAc blend membranes, the CO_2 permeance increased. Figure 5 shows that pure PSU selectivity CO_2/CH_4 is rising with pressure from 2 to 10 bar whereas selectivity (CO_2/CH_4) of pure PVAc membrane decreased with pressure increase. The enhanced polymeric membrane's selectivity also rose with increased feed pressure as shown in Figure 5. In the blend MEA amine molecules conjoint into the pores of the microporous materials. So for the blended membranes, the maximum selectivity of the PSU95%/PVAc5%/MEA is 9.43 ± 0.1 – 30.17 ± 0.7 from 2 to 10 bar. The membrane microporous structure incorporates MEA molecules. These amine molecules increase CO_2 adsorption due to interactions between CO_2 and $-\text{NH}_2$ groups [34, 35].

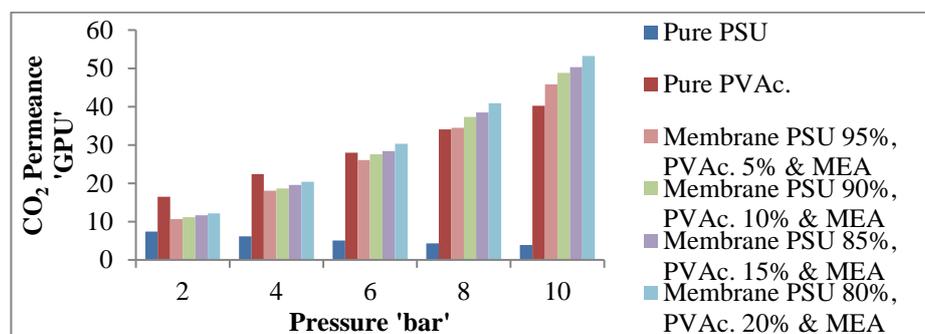


Fig. 3. Comparison of CO_2 permeance at various feed pressures for MEA amine polymeric blend membrane.

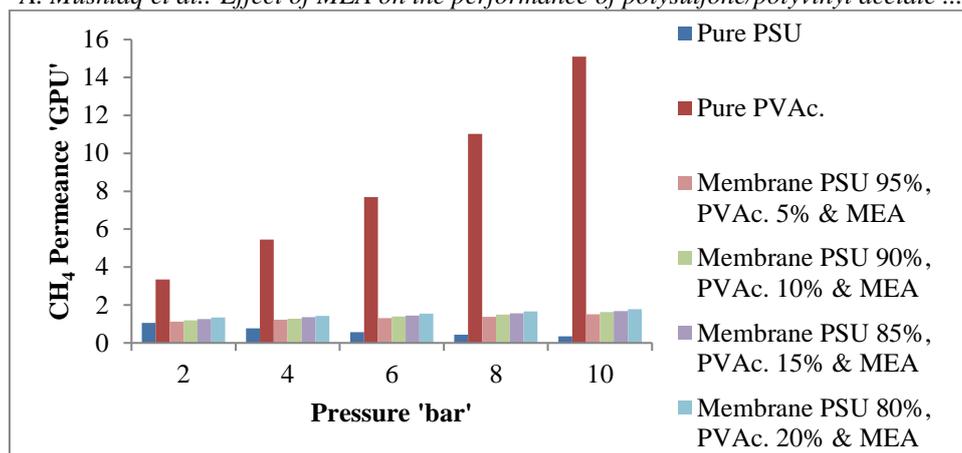


Fig. 4. Comparison of CH₄ permeance at various feed pressures for MEA amine polymeric blend membrane

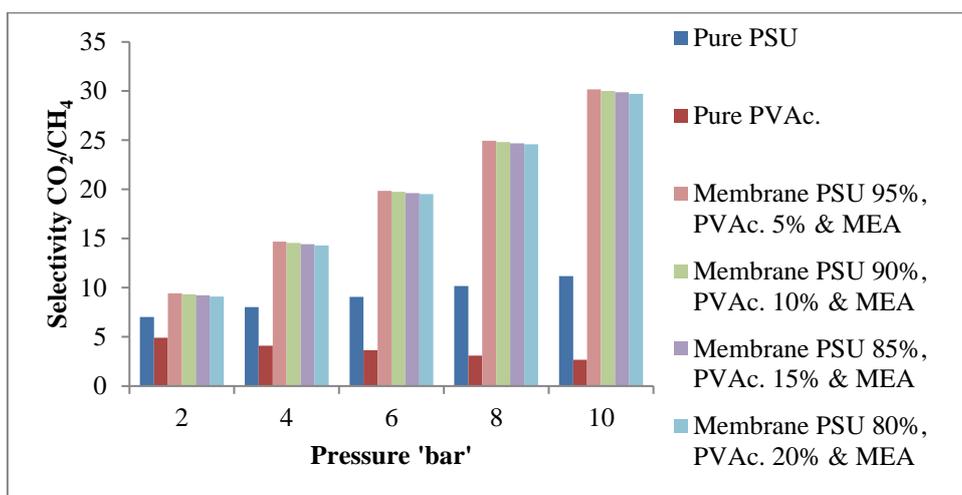


Fig. 5. Comparison of selectivity (CO₂/CH₄) at various feed pressures for MEA amine polymeric blend membrane

Hence, it was concluded that base polysulfone and polyvinyl acetate membrane and enhanced polymeric blend membranes were completely miscible and also characteristically increased CO₂ permeance and selectivity with rising feed pressure. The pressure difference across the membrane increased the volume of CO₂ gas across the membrane surface, increasing the formation rate of CO₂-amine complexes. Therefore, most amine molecules became involved in CO₂-amine interactions as the amine carrier concentration gently reached a state of saturation. Transporter saturation with a gradual reduction in gas flux under increased pressure describes the behavior of facilitated transport membranes. The observed results are in good agreement with former ones [29-33, 36].

Regression Graph between Pressure & Experimental Data for CO₂ Permeance, CH₄ Permeance & Selectivity CO₂/CH₄ of Different Polymeric Blend Membranes

Figures 6 and 7 show the correlation coefficients between the pressure and experimental values as given by a polynomial quadratic fit obtained with an R² value of 0.999. Hence, a proof is provided of the accuracy of the values obtained through the experimental setup based on the permeance performance of the different blend membranes.

Correlation Graph between Polynomial Predicted & Experimental Data for CO₂ and CH₄ Permeance & Selectivity CO₂/CH₄ of Polymeric Blend Membrane

Data analysis aims at understanding information gathered in an experiment and assessing the expected extent of one variable to rely on another which is autonomously known.

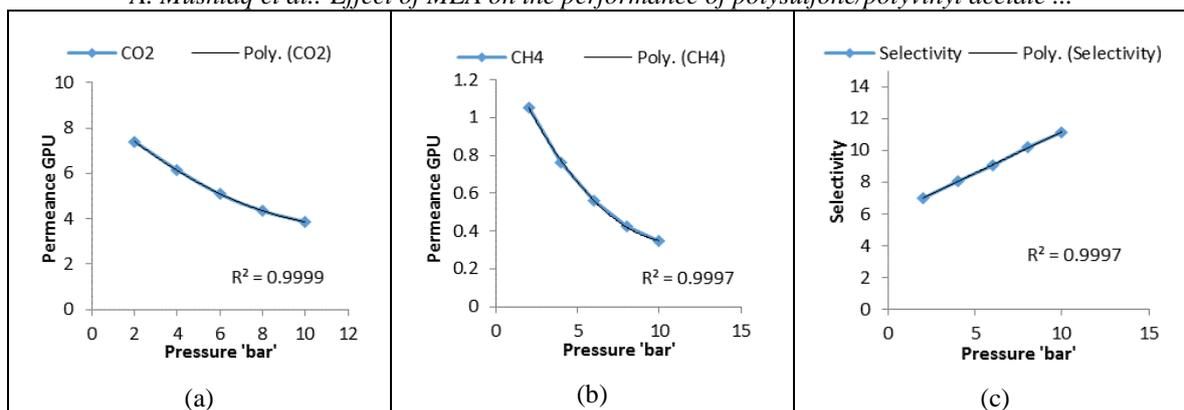


Fig. 6. Permeance (a) CO₂ (b) CH₄ in ‘GPU’ and (c) Selectivity CO₂/CH₄ against pressure ‘bar’ along with the correlation coefficient for base polysulfone membrane

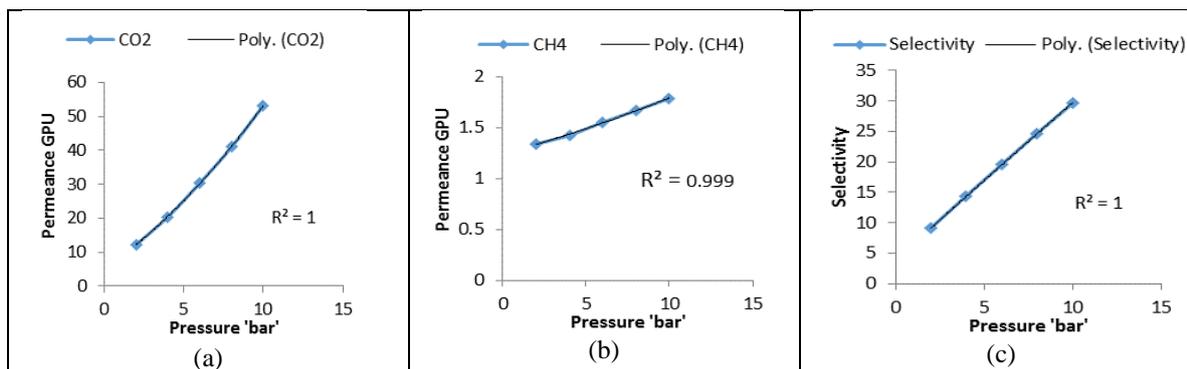


Fig. 7. Permeance (a) CO₂ (b) CH₄ in ‘GPU’ and (c) Selectivity CO₂/CH₄ against pressure ‘bar’ along with the correlation coefficient for PSU 80%/PVAc20%/MEA membrane.

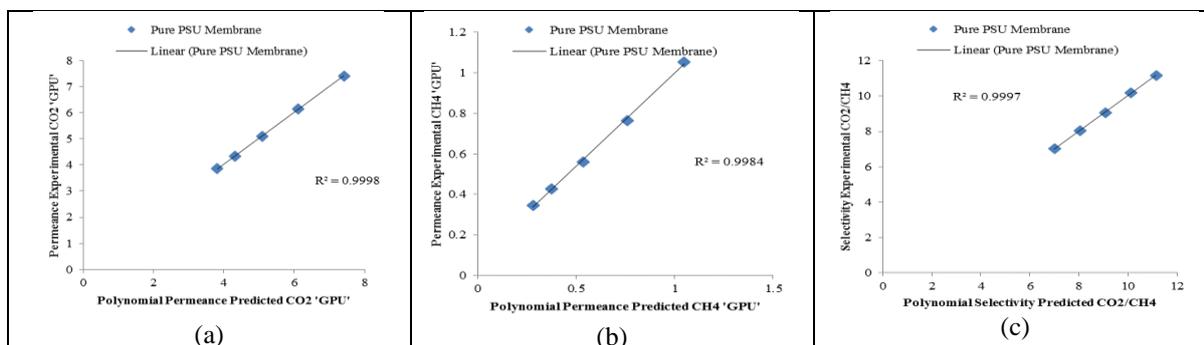


Fig. 8. Experimental permeance against polynomial predicted permeance and their selectivity along with the correlation coefficient for base polysulfone membrane

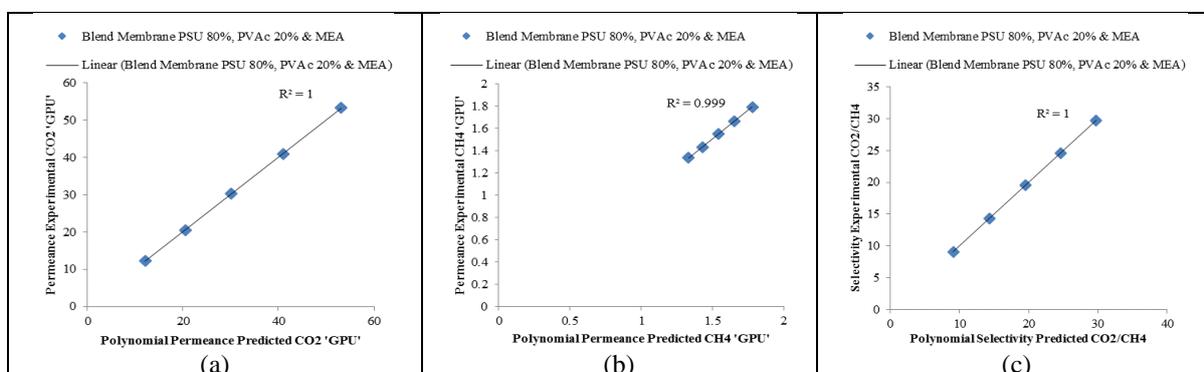


Fig. 9. Experimental permeance against polynomial predicted permeance and their selectivity along with the correlation coefficient for PSU 80%/PVAc20%/MEA membrane.

It will just manage the (regular) one-dimensional case in which the indigent variable is an element of one autonomous variable. The objective is to identify and evaluate the relationship between the two variables. The relationship between the polynomial-predicted, and experimental permeance values given in Figures 8 and 9 are in accordance with the linear fit obtained with an R^2 value of 0.999. Hence, a proof is provided of the accuracy of the values obtained through the experimental setup based on the permeance performance of the different blend membranes comprising pure PSU membrane and PSU 80%/PVAc 20%/MEA membrane. On the whole, a model fits the data if the difference between the experimental and the predicted values is small and unbiased.

CONCLUSION

An enhanced polymeric blend membrane was successfully developed. Moreover, the addition of MEA made an incredible improvement in the membrane's perm-selectivity. The soluble nature of CO_2 in MEA enhanced CO_2 solubility across the membrane. The enhanced polymeric amine blend membranes have a high proficiency of capturing CO_2 from natural gas. The relationship between the polynomial-predicted and experimental permeance values by a linear fit was obtained with an R^2 value of 0.999. For future work, propensity to add inorganic fillers such as zeolites or carbon molecular sieves in the blend would promote the enhanced polymeric blend membrane.

Acknowledgement: The authors would like to acknowledge the Universiti Teknologi PETRONAS for supporting this research work and the NED University of Engineering & Technology, Karachi, Pakistan for financial support to Asim Mushtaq studying at this University.

NOMENCLATURE

DMAc	Dimethyl acetamide
GPU	Gas Permeation Unit
MEA	Monoethanolamine
PSU	Polysulfone
PAI	Polyamide-imide
PDMS	Polydimethylsiloxane
PEA	Aromatic polyether amide
PEG	Polyethylene glycol
PEI	Polyether imide
PEO	Polyethylene oxide
PES	Polyethersulfone
PI	Polyimide
PIM-1	Polymer of intrinsic microporosity
PPO	Polypropylene oxide
PU	Polyurethane
PVA	Polyvinyl alcohol
PVAc	Polyvinyl acetate

PVAm	Polyvinyl amine
PVDF	Polyvinylidene fluoride
PVP	Polyvinyl propylene
SPEEK	Sulfonated aromatic poly(ether-ether-ketone)

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- A. Mushtaq et al.: *Effect of MEA on the performance of polysulfone/polyvinyl acetate ...*
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ВЛИЯНИЕ НА МОНОЕТАНОЛАМИН ВЪРХУ ЕФЕКТИВНОСТТА НА СМЕСЕНИ ПОЛИСУЛФОН/ПОЛИВИНИЛОВИ МЕМБРАНИ

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Постъпила на 9 януари, 2018; коригирана на 2 август, 2018

(Резюме)

Разделянето на CO₂ от природния газ с мембранна технология е използвано отдавна. За успешно разделяне на CO₂/CH₄ са необходими ефективни мембрани. Техниката на смесване повишава химичната и термичната стабилност, подобрява разделителната способност и икономическата устойчивост. В настоящата работа е изследвана ефективността на селективната проницаемост на смесените полимерни мембрани, стъкловидния полимер (полисулфон) и каучуковидния полимер (поливинилацетат) при добавяне на моноетаноламин (МЕА). Амин-съдържащи смесени полимерни мембрани с подобрени свойства са синтезирани при различни съотношения в разтворител диметилацетамид. Поливинилацетат с различна концентрация в интервала от 5 до 20 wt.% е смесен с полисулфон и МЕА и е използван за получаване на плоски мембранни филми по метода на изпаряване. Резултатите показват, че проницаемостта по отношение CO₂ нараства с увеличаване на захранващото налягане.