

Investigating the Performance of Membrane PEEK/PVA Modified with ZnO Nanoparticles in the Permeability and Selectivity of CO₂/N₂

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ABSTRACT

Membrane technology is applied in several industries due to the advantages of being environmentally friendly and energy efficient. An in-depth examination on the impact of membrane Polyetheretherketone/Polyvinylalcohol (PEEK/PVA) composite modified with nanoparticle zinc oxide (ZnO) on particle size, mechanical, and morphological properties. Scanning electron microscopy (SEM) was applied for studying the dispersion of ZnO particles in PEEK/PVA composite. The characterization of the obtained hybrids was done using Fourier transform infrared spectroscopy (FT-IR), and X-ray diffraction (XRD) with an increase in the solubility of the gas molecules within the polymer matrix, an increase in the CO₂ permeability is observed. The outcomes of the N₂ permeability confirmed that the permeability of this agent undergo reduction with increasing pressure. Separation process at high pressures was proven since the selectivity of CO₂/N₂ boosted from 93.8 at 1 bar pressure to 43.6 at 8 bar pressure, with increasing feed pressure. The careful and detailed examination of the permeability and selectivity of CO₂/N₂ for membrane PEEK/PVA/ZnO nanoparticle modified with PEEK/PVA composite was performed. An increase in the CO₂ permeability to the membrane PEEK/PVA from 19.6 to 87.7 upon modifying the surface area of the membrane PEEK/PVA/m-ZnO NPs (1.0 wt%, 2.0 wt% and 3.0 wt%), and increasing the percentage of this additive in the polymer matrix. Increase was due to the presence of cavities developed in the process of surface modification, and the increased of free volume and solubility of CO₂/N₂ inside the polymer membrane.

Keywords: Gas CO₂/N₂; Permeability; Membrane; Nanoparticles; Separation.

1. INTRODUCTION

Over the last few years, separation methods like absorption gained popularity in gas separation as successful methods. Chemical adsorption and cryogenic

distillation as well as novel technologies like membrane systems were practiced repeatedly [1-4]. Moreover, industries are seeking novel methods to tackle

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environmental pollution, tackle energy consumption and costs and therefore the use of membrane approach in industries like natural gas sweetening got the better of other methods [5]. Over several years, this technology, in comparison with other conventional gas separation processes, presented rapid growth [6]. The matter of gas separation in many professions and different applications is of significant importance [7,8]. Separation processes have drawn the attention of various industries due to their advantages requiring lower energy, having compact structure, having lower operating and maintenance costs, being user-friendly, most important of all, having the most negligible impact on the ecosystem [9,10]. In a study, the suitability of metal nanoparticles on the separation of N_2 and CO_2 pure gases for two nanocomposite membranes of polyesters was investigated. This fact that with increasing silica content, permeability improves was proven by net gas permeability. This phenomenon is explainable in this way that the separation of the molecular chain leads in the free volume of the polymer network [11]. In several studies the application of polysulfone (PSF) MMMs with mesoporous silica MCM-41 for gas separation has been reported. In addition, the improvement of gas permeability of PSF using incorporating mesoporous MCM-48. It was revealed that mesoporous materials because of excellent compatibility with the polymer matrix could improve the permeability of the polysulfone MMMs without decreasing selectivity. The combination of polyimide, and zeolite as a polymer membrane came under scrutiny by them. In that project, a mixed network membrane made up of polyimide, and 20wt% zeolite was prepared and analyzed to obtain data about the permeability of gases like O_2 , CO_2 , N_2 , and CH_4 . The outcomes showed an

increase in the permeability of CH_4 , and CO_2 and a decrease in the permeability of N_2 and O_2 gases. Changes in permeability are directly related to the changes in the permeability coefficient [12,13]. In another research, the impact of TiO_2 nanoparticles on 1, 2-polybutadiene (PB) was investigated. The permeability coefficients of CO_2 , CH_4 , N_2 , and H_2 gases in a membrane containing 27 vol.% TiO_2 nanoparticles was three times as big as the permeability coefficients of them in a membrane containing only the pure polymer. However, an increase in the solubility coefficient of the gases was observed when nanoparticles were added to the polymer, while a decrease in the permeability coefficients was detected by addition of the nanoparticles [14]. The impact of diverse variables by way of example adding zinc oxide (ZnO), aluminum oxide (Al_2O_3), and titanium dioxide (TiO_2) nanoparticles to PMP membrane on both structural properties of the membranes and function of the polymeric membranes has been evaluated utilizing statistical modeling as a method [15].

ZnONPs, amongst different nanomaterials, have grown in popularity due to their availability, photocatalytic activity, self-cleaning quality, ease of preparation, and affordability. At higher concentrations, ZnO results in the formation of agglomerates. Therefore, procuring a hybrid with fine NPs dispersion is a challenge [16-18]. The construction, of agglomerates is because of the high surface energy of ZnO, which provides a driving force for developing of particle bonding. The most appropriate method to attach a proper organic group on the surface atoms is silane coupling agents [19,20]. This surface modification not only supports the NPs, against accumulation but also helps their compatibility with the other phase [21]. Proper surface modification of NPs

results in the improvement of first dispersion and compatibility in polymer matrix and second chemical or physical interactions with the polymer matrix [21-23].

Here, a new macromolecular, based on the membrane PEEK/PVA/ZnO nanoparticle. Then, the functionalized ZnO nanoparticle (1.0 wt%, 2.0 wt%, and 3.0 wt%) into membrane PEEK/PVA /ZnO nanoparticle modified with PEEK/PVA composite. A detailed examination of the contact membrane PEEK/PVA/ZnO nanoparticle as an important parameter on the permeability and selectivity of CO₂/N₂ gas and the adsorption mechanism was carried out. It was revealed that the CO₂/N₂ selectivity and permeability of the mixed matrix membranes were remarkably enhanced by the membrane PEEK/PVA/ZnO nanoparticle. Sorption studies, FE-SEM, and gas permeation measurements were used to characterize the structure, and properties of the mixed matrix membranes based on membrane PEEK/PVA/ZnO nanoparticle.

2. EXPERIMENTAL

2.1. Reagents and materials

Merck Chemical Co and Aldrich Chemical Co provided us with all the materials, and solvent n-methyl pyrrolidone (n-MP) were provide and used nanosabz Co. (Tehran, Iran) provided us with the ZnO nanoparticles with the average particle size of 25–30 nm. To guarantee the complete drying, ZnO nanoparticles were dried at 500 °C for almost five h before utilization. the coupling agent; Poly ether ether keton (PEEK) from Chines Company, and membrane PEEK/PVA/ZnO nanoparticle modified with Poly vinyl alcohol (PVA) was purchased from merck Company.

2.2. Instrumentation

Scanning electron microscopy (FESEM:

MIRA III, TESCAN Company, of the Czech Republic) under an acceleration voltage of 26kV helped us in analyzing the morphology of samples. With the help of infrared spectroscopy (FTIR, Nicolet Co, USA), the functional groups on the surface of zinc oxide modified nanoparticles were determined. Instron (6025K) was utilized to do the tensile test of nanocomposites based on the ASTM-D3039 standards at room temperature on specimens 150 mm long, 25 mm wide and 3 mm thick, with a machine speed of 5 mm / min.

Also, the measurement of flexural strength was done according to ASTM-D790 standard using Instron (6025K) on specimens 90 mm long, 10 mm wide and 3 mm thick.

2.3. Synthesis of membrane PEEK/PVA/ZnO nanoparticle

A soluble casting method was employed to fabricate the separation membranes. Therefore, a few grams of membrane and a few ml of n-methyl pyrrolidone (n-MP) solvent were mixed and stirred for 3 hours through the instrumentality of a mechanical stirrer which ended in a uniform solution. The provided a homogeneously composite membrane with different quantities of m-ZnO NPs (1.0 wt%, 2.0 wt%, and 3.0 wt %). First, 0.1 g of polymer was dissolved in 2 mL n-methyl pyrrolidone (n-MP) solvent and second, under constant stirring for 12 h and at 25 °C, m-ZnO was separately dispersed in 2 mL n-methyl pyrrolidone (n-MP) solvent. Then, to obtain the desired weight percentages of m-ZnO from 1.0% to 2.0%, the solutions were mixed. The provided membrane PEEK/PVA/ZnO nanoparticle solutions were taken into glass Petri dishes to get rid of the solvent, and after than dried under vacuum at 60 °C for 12 h and 130 °C for 3h, membrane PEEK/PVA/ZnO nanoparticles, with Polyetheretherketon and Polyvinylalcohol (PEEK/PVA)

composite modified zinc oxide nanoparticle.

2.4. Measurement and calculation method

With the help of a constant pressure method (a soap bubble discharge gauge), the permeability of CO₂/N₂ in the membrane PEEK/PVA/ZnO nanoparticle was calculated. The trial entailed a gas seepage cell in which one side was exposed to the membrane and the other side to the gas feed. By making use of a regulator, and using, a barometer. The following formula to determine the gas permeability in the membrane [24,25]:

$$\frac{P_i}{l} = \frac{1}{\Delta P} \frac{dv_i}{dt} \quad (1)$$

P_i in the aforementioned formula stands for the permeability of gas i, the volume of gas discharged into soap bubble discharge or volume of Permeability gas into the membrane in cubic centimeters is shown by v_i, effective membrane surface area in square centimeter is shown by A, t refers to bubble motion the column in seconds, and 1P shows the pressure drop across the membrane in centimeters of mercury. In GPU, reports on the asymmetric membranes developed from the permeability division of the membrane thickness are available. Also, via using the following equation, the optimal selectivity of the two gases is calculable:

$$\alpha_{i/j} = \frac{\left(\frac{P_i}{l}\right)}{\left(\frac{P_j}{l}\right)} \quad (2)$$

Where α (i / j) is the optimal selectivity of gas i over j, P_i / l is the gas i permeability and P_j / l is the gas j permeability.

The excessive dependence of gas transfer properties of the organic/inorganic lattice membranes to the membrane structure is beyond all doubt, in contrast, the membrane structure is dependent on the surface area of contact between the particles and the polymer. Absorption

theory expresses the permeability of gas in glass polymers [26]. This approach, introduces the total concentration of permeates (C) in the glass polymer as a function of pressure (p). The following equation expresses the theory:

$$C = K_D p + C_H b p / (1 + b p) \quad (3)$$

In the abovementioned mathematical statement, K_D stands for the dissolution coefficient of artistic law, and C_H, shows the cavity saturation constant. Taking into considering the large porosity-to-surface ratio in the porosities. That a place for the interaction between gases, and the polymer network is required and the porosities can provide it, which provokes an increase in the permeability and solubility [26].

2.5. Investigation of permeability characteristics and selectivity of permeability measurement membranes

The pressure-volume-constant-variable method can be utilized to verify the permeability prosperities of carbon dioxide and nitrogen gases through polymer membranes. Taking into consideration the steady-state conditions, the polymer membranes in this method for a specific duration of time were pressurized and with the help of a digital flow meter, the volume flow rate through the polymer membrane was estimated. The effective surface area of all membranes was estimated to be 56/71 cm². The developed polymeric membrane in the membrane tubes, and with the help of a hollow circular rubber ring, was maintained. A gas feed inlet and two outlet portions were embedded in the membrane tube. From the two outlets, one is for the gas component leaked through the membrane and the other for the residual gas component. Fig. 1, represents the schematic of the devices applied to measure the leakage of carbon dioxide and nitrogen from the polymer membrane.

3. RESULTS AND DISCUSSION

3.1. Examination of ZnO Surface Modification Process

Fig. 2 represents the FTIR spectra of both unmodified and modified ZnO nanoparticles. The FTIR spectrum of membrane PEEK/PVA unmodified by ZnO nanoparticles and represents that of the membrane PEEK/PVA/ZnO nanoparticle coupling components. In close scrutiny, a sharp signal in the range of 1005-1345 cm^{-1} was detectable which could result from the vibration of the hydroxyl groups on the surface of the nanoparticles [27]. Considering the b

curve, it became apparent that the signals in the range 1300-1655 cm^{-1} was associated to the groups CH –CH and $(-\text{CH}_2)_n-\text{CH}_3$. Therefore, it can be assumed that the alphabetic groups of the components coupled onto the surface of the nanoparticles were Zn-O. The signal of the 2850 cm^{-1} region is coupled to the b-curve of the bond C=C at the component level. Consequently, the thriving coupling of coupling groups to the surface of zinc oxide nanoparticles was shown by the FTIR spectrum. In other terms, surface modification was successfully performed [27, 28].

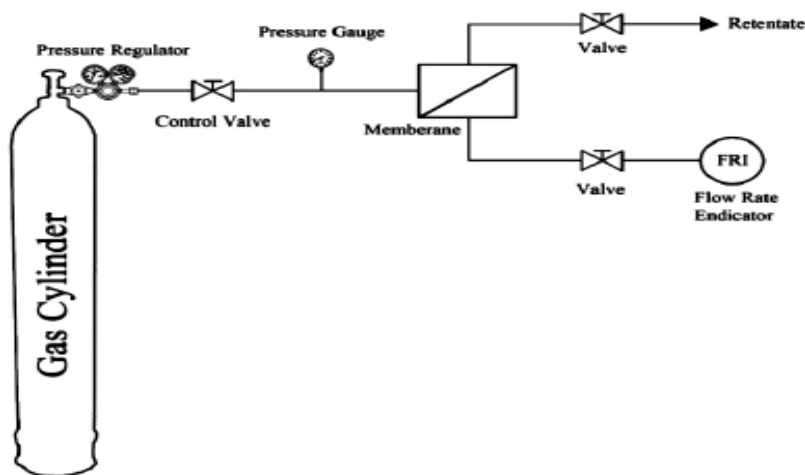


Fig. 1. Schematic permeability gas of the mixed matrix membranes.

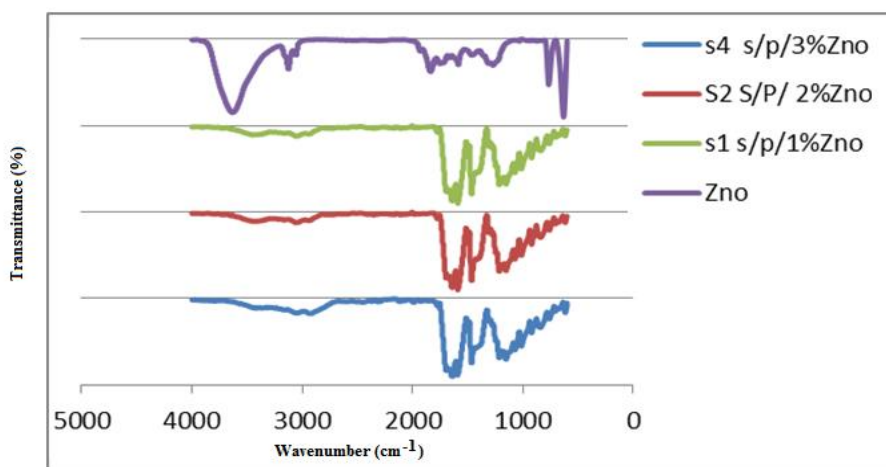


Fig. 2. FT-IR spectra of the aromatic membrane PEEK/PVA m-ZnO NPs (1.0 wt%, 2.0 wt% and 3.0 wt%).

3.2. X-ray diffraction

In Fig. 3, the XRD patterns of PEEK/PVA, m-ZnO and PEEK/PVA with (1.0 wt%, 2.0 wt% and 3.0 wt%) of m-ZnO NPs are exhibited. For pure PEEK/PVA (Fig. 3d), it is apparent that there is a lack of diffraction signal in the range of 2θ angle barring one crystalline signal suggesting the presence of a little proportion of crystalline phase. For m-ZnO (Fig. 3a), the apparent signals at 100, 002, 101, 102, 110, 103, 200, 112, 201, and 202 prove the presence of rutile phase which is a good evidence of the crystalline form of this compound [228]. The characteristic signals of m-ZnO and PEEK/PVA in the XRD patterns revealed that the preparation process and surface modification cannot change the morphology of NPs (Fig. a and b).

3.3. Morphology of prepared membranes

Filamentous fibers for pure membrane surface in SEM images. Also, in (Fig. 5), SEM images of membrane PEEK/PVA/ZnO nanoparticles display the result of this modification on the morph of the provided membrane.

Surface structure of the membrane PEEK/PVA/ZnO nanoparticles, metamorphosed into pseudo-flowering from fibrous. Additionally, in getting this image checked out, the transparent layers on the membrane surface confirm the hardening of the polymer-particle interface an indistinct separation when the modification was performed [28,29]. The nanoscale size and rounded shape of the m-ZnO nanoparticles have been shown in these images to be in the range of 40–60 nm.

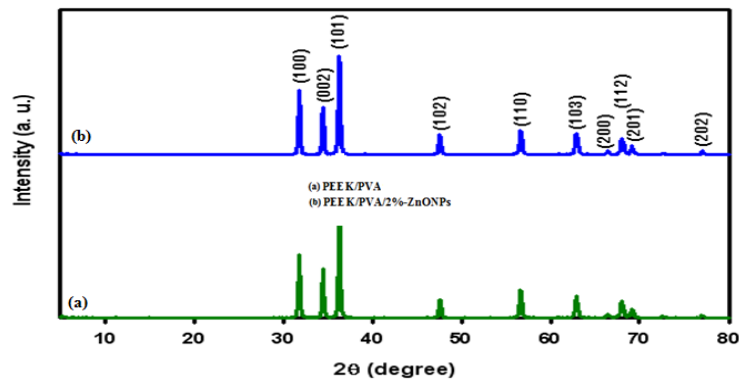


Fig. 3. XRD patterns of the surface ZnO nanoparticles (a), membrane PEEK/PVA/2.0% ZnO nanoparticles (b).

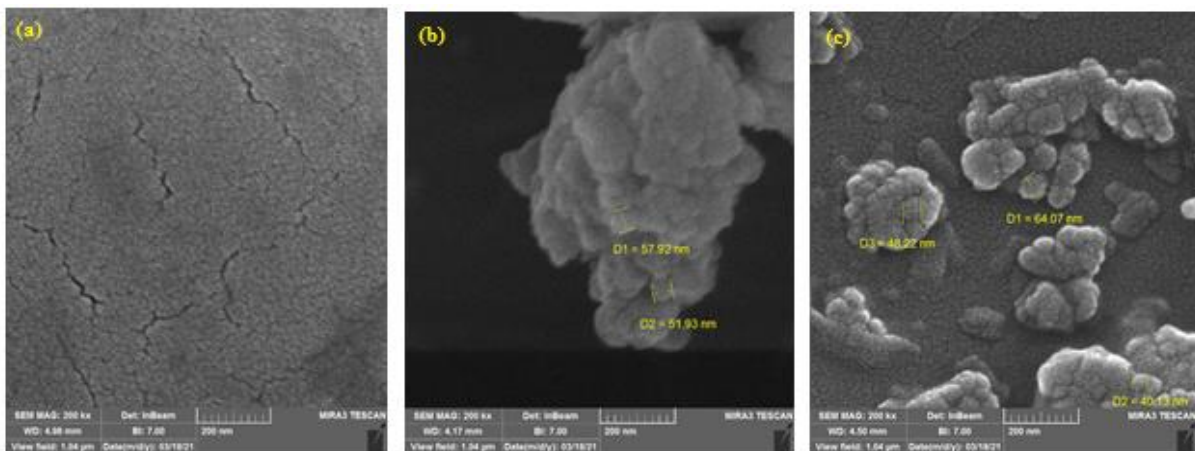


Fig. 4. SEM images of Surface (a) membrane PEEK/PVA (b) ZnO nanoparticles (c) membrane PEEK/PVA/ZnO nanoparticles.

3.4. Investigation of permeability properties and selectivity

The permeability and selectivity of carbon dioxide and nitrogen for membrane PEEK/PVA/ZnO nanoparticles. The ensuing diagram demonstrates the influence of these additives on the permeability of carbon dioxide and nitrogen. Surface modification with PEEK/PVA composite and expansion of the percentage of this additive in the polymer matrix can increase the carbon dioxide permeability to the membrane. The efficiency of this polymer membrane for separation at high pressures was proven since with increasing feed pressure, the permeability of CO₂/N₂ increased from 93.8 at 1 bar pressure to 43.6 at 8 bar pressure [30, 31]. In addition, the permeability and selectivity of CO₂/N₂ for membrane PEEK/PVA/m-ZnO nanoparticles was studied. The CO₂ permeability to the membrane PEEK/PVA increased from 19.6 to 87.7 upon modifying the surface area of the membrane PEEK/PVA/m-ZnO NPs (1.0 wt%, 2.0 wt% and 3.0 wt%), and increasing the percentage of this additive

in the polymer matrix. This increase is the result of the presence of cavities due to the increase of free volume and the increase of solubility of carbon dioxide inside the polymer membrane [31, 32]. However, the higher permeability of membranes to carbon dioxide rather than to nitrogen was evident. Thus, the recognition of the selectivity of CO₂/N₂ is in (Table 1). However, the perfect selectivity of the prepared membranes as well as the unique properties of the membranes based on the copolymer is confirmed by the results (Fig. 5 and Fig. 6) [32, 33].

3.5. Comparison of results for this work with other reported membranes

In order to illuminate the applicability and efficiency of membranes in this work, the results are compared with those of some of the recently reported methods for the reduction of variety of materials by membranes in (Table 2). Obviously, SPEEK/PVA/ZnO nanoparticles shows the high Permeability for the gas CO₂ and N₂ in comparison with literature other membranes.

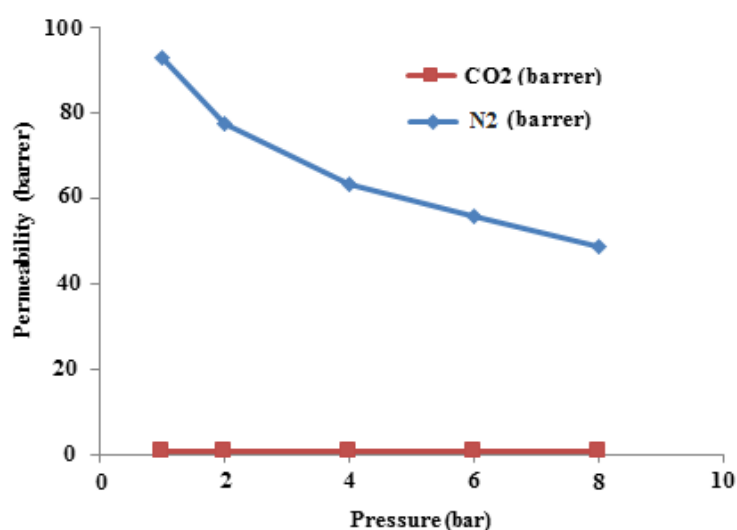


Fig. 5. The display of impact Permeability (barrer) CO₂/N₂ gas of the modified ZnO nanoparticles without Pressure percentage.

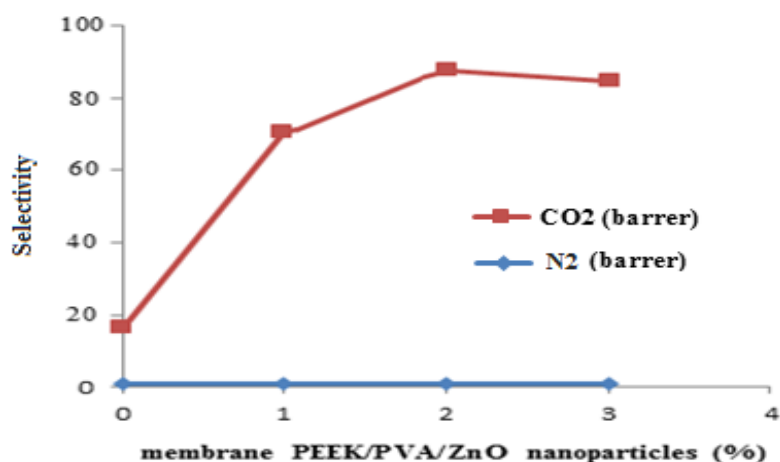


Fig. 6. The display of impact selectivity CO₂/N₂ gas of the modified ZnO nanoparticles at Pressure (1bar) percentage.

Table 1. Separation outcomes for membrane PEEK/PVA/m- ZnO nanoparticles

| Pressure (bar) | Permeability (barrer) | | Selectivity |
|----------------|-----------------------|----------------|-------------|
| | CO ₂ | N ₂ | |
| 1 | 93.8 | 1.51 | 19.6 |
| 2 | 77.8 | 1.32 | 33.15 |
| 4 | 62.4 | 1.06 | 69.2 |
| 6 | 59.4 | 0.94 | 77.8 |
| 8 | 43.6 | 0.73 | 87.7 |

Table 2. Comparison of results for this work with other reported membranes

| Samples | Membranes | Pressure (bar) | Permeability CO ₂ (%) | References |
|----------------------------------|--------------------|----------------|----------------------------------|---------------|
| CO ₂ /CH ₄ | Silicate-based MMM | 2 | 73.6 | [2] |
| CO ₂ | PEBA/MWCNT | 8 | 92.2 | [13] |
| CO ₂ /CH ₄ | PEEK/Zeolite 4A | 10 | 55.0 | [14] |
| CO ₂ /N ₂ | PVA/PEG 600 | 1.5 | 52.9 | [17] |
| CO ₂ | PEBA/PEG/CNT | 2 | 44.6 | [27] |
| CO ₂ | PVA/CNF | 8 | 39.0 | [34] |
| CO ₂ /N ₂ | PEEK/PVA/ZnO NPs | 1 | 93.8 | Present study |

4. CONCLUSIONS

The permeability of CO₂/N₂ gas for membrane PEEK/PVA/m-ZnO nanoparticles has been investigated at different pressures. The findings revealed that the direct relation of the permeability of CO₂ with pressure was relative to the elasticity of the polymer membrane. In other words, tensile strain could affect the permeability of polar CO₂ gas at high pressures. When high concentrations of CO₂ gas were absorbed onto the membrane

surface (sufficient concentration of CO₂ in the membrane), elongation was observed. Therefore, with an expansion in the solubility of the gas molecules within the polymer matrix, the CO₂ permeability showed an increase. Based on the obtained results, N₂ permeability and pressure were inversely related (an increase in pressure led to the reduction of N₂ permeability). The efficiency of this polymer membrane for separation at high pressures was proven since with increasing feed pressure, the

selectivity of CO₂/N₂ increased from 93.8 at 1 bar pressure to 43.6 at 8 bar pressure. In addition, the permeability and selectivity of CO₂/N₂ for membrane PEEK/PVA/m-ZnO nanoparticles was studied. The CO₂ permeability to the membrane PEEK/PVA increased from 19.6 to 87.7 upon modifying the surface area of the membrane PEEK/PVA/m-ZnO NPs (1.0 wt%, 2.0 wt% and 3.0 wt%), and increasing the percentage of this additive in the polymer matrix. This increase is the result of the presence of cavities due to the increase of free volume and the increase of solubility of carbon dioxide inside the polymer membrane. However, the membranes were high permeable to CO₂ but not permeable to N₂. To build up an empirical model, the neural network model was employed successfully. For permeability of CO₂/N₂ by membrane PEEK/PVA/m-ZnO nanoparticles, the lowest errors were observed. However, the CO₂/N₂ could not be determined. The results showed the perfect permeability of the prepared membranes as well as the unique properties of the membranes based on the membrane PEEK/PVA/m-ZnO nanoparticles.

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بررسی عملکرد غشاء PEEK/PVA اصلاح شده با نانوذرات اکسید روی در نفوذپذیری و گزینش

پذیری گازهای CO₂/N₂

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چکیده

فناوری غشاء و فرآیندهای غشایی به دلیل مزایای سازگار با محیط زیست و کارآمد بودن در مصرف انرژی در چندین صنعت کاربرد دارد. با این حال، در فرآیند جداسازی است. یک بررسی عمیق بر روی تاثیر کامپوزیت پلی اترکتون/پلی وینیل الکل غشایی (PEEK/PVA) اصلاح شده با نانوذرات اکسید روی (ZnO) بر اندازه ذرات، خواص مکانیکی و مورفولوژیکی ذرات انجام شد. میکروسکوپ الکترونی روبشی (SEM) برای مطالعه پراکندگی ذرات اکسید روی در کامپوزیت PEEK/PVA استفاده شد. خصوصیات هیبریدهای به دست آمده با استفاده از طیف سنجی فروسرخ تبدیل فوریه (FT-IR) و پراش پرتو ایکس (XRD) با افزایش حلالیت مولکولهای گاز در ماتریس پلیمری، افزایش نفوذپذیری گاز CO₂ انجام شد. نتایج نفوذپذیری گاز N₂ تأیید کرد که نفوذپذیری این عامل با افزایش فشار کاهش می یابد. مناسب بودن این غشای پلیمری در فرآیند جداسازی در فشارهای بالا از آنجایی که گزینش پذیری CO₂/N₂ از ۹۳/۸ در فشار ۱ بار به ۴۳/۶ در فشار ۸ بار افزایش یافت، با افزایش فشار خوراک، به اثبات رسید. بررسی دقیق و دقیق نفوذپذیری و گزینش پذیری CO₂/N₂ برای نانوذرات PEEK/PVA/ZnO غشایی اصلاح شده با کامپوزیت PEEK/PVA انجام شد. افزایش نفوذپذیری CO₂ به PEEK/PVA غشا از ۱۹/۶ به ۸۷/۷ با اصلاح سطح غشا PEEK/PVA/m-ZnO NPs (۰/۱ درصد، ۰/۲ درصد و ۰/۳ درصد) و افزایش درصد این افزودنی در ماتریس پلیمری. این افزایش به دلیل وجود حفره های ایجاد شده در فرآیند اصلاح سطح و افزایش حجم آزاد و حلالیت CO₂/N₂ در داخل غشای پلیمری بود.

کلید واژه ها: گاز دی اکسید کربن؛ گاز نیتروژن؛ نفوذپذیری، غشاء؛ نانوذرات؛ جداسازی

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