

## Effect of heating rates on the microstructure and gas permeation properties of carbon membranes

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### Article history

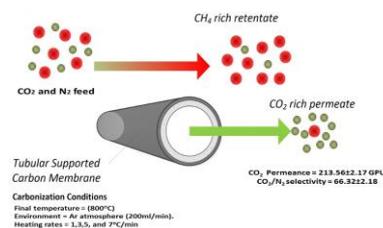
Submitted 14 February 2018

Revised 9 April 2018

Accepted 28 May 2018

Published Online 3 September 2018

### Graphical abstract



### Abstract

High performance tubular carbon membrane (TCM's) for CO<sub>2</sub> separation were prepared by controlling the carbonization heating rates in range of 1-7 °C/min carbonized at 800 °C under Argon environment. A single permeation apparatus was used to determine the gas permeation properties of the membrane at room temperature. Fine turning of the carbonization condition was necessary to obtain the desired permeation properties. The preparation of PI/NCC-based TCM at low heating rate caused the gas permeance for the examined gas N<sub>2</sub> and CO<sub>2</sub> decreased whereas the selectivity of CO<sub>2</sub>/N<sub>2</sub> increased. It was also identified that the gas permeation properties of the resultant TCM and its structure was highly affected by the heating rate. The best carbonization heating rate was found at 3°C/min for the fabrication of TCM derived via polymer blending of PI/NCC for CO<sub>2</sub>/N<sub>2</sub> separation.

**Keywords:** Gas permeation, heating rates, polyimide, nanocrystalline cellulose, carbon membrane

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## INTRODUCTION

Membrane for gas separation processes has gained significant interests due to its lower energy requirements, ease of operation, low operating and capital cost, continuous process and unnecessary regeneration process like those in the absorption and adsorption process (Adewole et al., 2013, Robeson, 2016). For the past 30 years, membrane technology has been prominently used as a method to obtain various types of gases with high purity. In order to attain membranes that exhibit high selectivity, high permeability, high chemical and thermal steadiness, membranes are developed using polymeric materials (Sridhar et al., 2007). Separations by utilizing membranes are getting to be progressively important in the process industries. Due to the advantages of traditional methods, membranes for gas separation are grown rapidly. There are several traditional methods that have been used by previous researchers for instance amine absorption, pressure swing adsorption and cryogenic distillation. However, there are some drawback of all these methods such as expensive, energy intensive, and has negative impact to the environment (Wang et al., 2003). Semipermeable wall or known as membrane can control the rate of movement of various molecules for separation process such as between two liquid phases, two gas phases, or a liquid and gas phase (Mat and Lipscomb, 2017).

Membrane separation-based carbon membrane is become important drastically in several processes especially in gas separation process. Nowadays, many efforts have been done to develop new membrane materials with excellent selectivity and permeability. Carbon membrane is resulted from carbonization of polymeric

precursors. The presence of benzene ring and other functional groups in the polymeric precursor will result in amorphous materials after undergoing carbonization process. These resultant carbon membranes consist of disordered sp<sup>2</sup> hybridized condensed hexagonal sheets serves an idealized pore structure, with pores formed due to packing imperfections (Bhuwania et al., 2014). The idealized pore structure serves as channels for gas permeation while providing good selective features through molecular sieving. Carbon membrane has superior advantages over polymeric membranes i.e thermally and chemically resistance, and thus results in no contamination, physical aging and plasticization as polymeric membrane (Adewole et al., 2013). In addition, the high pore volume of carbon membrane has provided higher selectivity and permeability in the separation of gas that has similar gas molecules size such as CO<sub>2</sub>/CH<sub>4</sub>, O<sub>2</sub>/N<sub>2</sub>, and CO<sub>2</sub>/N<sub>2</sub> (Korsh and Soffer, 1986, Jones and Koros, 1994, Tanihara et al., 1999, Hunt et al., 2010).

Moreover, carbon membrane has been recognised as a very favourable candidate for gas separations, due to their attractive characteristics particularly in terms of their separation abilities with excellent permeability and selectivity (Ismail et al., 2018, Sazali et al., 2018). In addition to that, the cooling step for the carbon membrane can be eliminated and therefore will reduce the processing cost and does not increase the energy (Hamm et al., 2017). Ultra-micro porosity can be obtained by the orientation dislocation of aromatic micro domains in like glasslike matrix in which gives rise to free volume. The pore mouth dimensions are similar to the diameter of gas molecular kinetic diameter and the microspores are generally considered to be nearly slit-shaped (Swaidan et al., 2014). Generally, the carbonization of aromatic

polymers and its derivatives are used to prepare the carbon membrane to obtain excellent separation performance for gas pair such as  $O_2/N_2$  and  $CO_2/CH_4$  (Yoshimune and Haraya, 2013). Carbonization is the hearth process in fabricating carbon membrane due to its determine the pore characteristic of a carbon membrane. Three main factors need to be considered to enhance the separation performance of carbon membrane which are carbonization conditions, precursor selection, as well as membrane preparation (Briceño et al., 2013). The polymer precursor used ought to various standards for instance high aromatic carbon content, high glass transition temperature,  $T_g$ , chemically stable and give predominant separation properties (Saufi & Ismail, 2004). Glass transition temperature is used to specify the capacity of polymers to cope the high temperature applies throughout carbonization process. Suitable precursor materials for carbon membrane preparation won't result in any pore openings or defects to show up after the carbonization stage (Ismail & Li, 2008).

Carbon membrane that prepared with the addition of nanocrystalline cellulose (NCC) had promoted the enhancement of the diffusion pathway for the gas species in the domain of the thermally liable polymer. The advancement in technology and separation requirements as well as its applications lead to further challenges, therefore new materials to fabricate membrane are required to develop and obtained a membrane which can be optimized for the separation purpose and has a good productivity and efficiency. Thus, the application on blending of NCC with polymeric precursor solution can tailor significant effort on many areas of research.

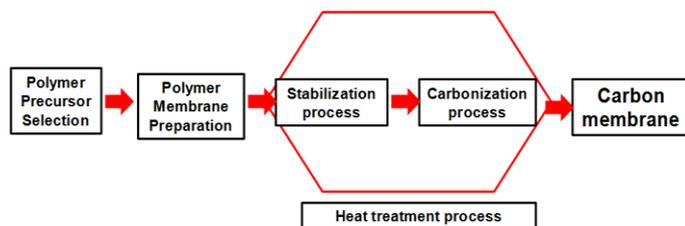


Figure 1: Schematic diagram of the carbon membrane preparation.

Heating rate has substantial effect on the gas permeation properties and the structure of the resultant carbon membrane (Centeno et al., 2004) (Salleh and Ismail, 2012). According to literature, generally the carbonization heating rate is conducted at range of 1-13  $^{\circ}C/min$  (Salleh and Ismail, 2012). However this rate is highly dependent on the type of polymeric precursor membrane used. According to Centeno et al. (2004) conducted a research on different carbonization heating rate of phenolic resin to produce the carbon membrane with maximum carbonization temperature was 700  $^{\circ}C$ . The result showed that at 10  $^{\circ}C/min$  heating rate, the pore size of the membrane shifted to smaller pores which was very useful for the enhancement of molecular sieve of the resultant carbon membrane characteristic and was examined for the permanent gas pairs separation ( $O_2/N_2=5$ ,  $CO_2/N_2=27$ , and  $CO_2/CH_4=23$ ) as well as olefin/paraffin ( $C_2H_4/C_2H_6=3$  and  $C_3H_6/C_3H_8=6$ ). At heating rate of 1-5  $^{\circ}C/min$  was the most effective rate for the polymer-based carbon membrane and for PEI/PVP-based CHFMs prepared at low heating rate, the selectivity of  $CO_2/CH_4$  and  $CO_2/N_2$  was increased and the gas permeance ( $N_2$ ,  $CH_4$ ,  $CO_2$ ) was decreased

The focus of current work was to investigate the preparation and characterization of tubular carbon membrane (TCM) derived from P84 co-polyimide (PI) blended with NCC, specifically on heating rate. The presence of NCC within the membrane is expected to alters the morphology and subsequently influence the membrane performance.

## EXPERIMENTAL

### Materials

P84 co-polyimide from Sigma Aldrich was employed as the main precursor and the solvent used was N-methyl-2-pyrrolidone (NMP) which obtained from Merck (Germany). All chemicals were used directly without further purification. The in-house synthesized

nanocrystalline cellulose (NCC) as explained in previous study (Sazali et al., 2017) was used as the filler for the carbon membrane. The porous tubular ceramic support ( $TiO_2$ ) with dimension of 8 cm x 3 cm with average pore size of 0.2 $\mu m$  (porosity of 40-50%) was purchased from Shanghai Gongtao Ceramic Co., Ltd.

### Carbon membrane preparation

A homogenous solution was prepared by mixing 15 % of P-84 (relative to total weight) and NMP under constant stirring condition of 80  $^{\circ}C$  and 7 wt% of NCC was added gradually into the mixture. After that, the polymer solution was placed in the sonication bath for few hours to remove the bubble in the solution that formed during the mixing process. Then, the tubular support was dip-coated where this coating technique has been known as a simple method to produce a very thin top layer. The tubular membranes were produced by immersing the ceramic tubes into the dope solution for 45 minutes and then were immersed in methanol for 2 hours. The resultant membranes were then placed in the oven for 24 hours at 100  $^{\circ}C$  to remove the solvent. Carbon membrane was obtained by the carbonization process of the supported polymeric membrane. Therefore, the polymeric carbon membrane was heated in carbolite horizontal tubular furnace at 800  $^{\circ}C$  under Argon gas flow (200ml/min) at different heating rate of 1, 3, 5, and 7  $^{\circ}C/min$ . The experimental procedures were employed based on the previous studies (Sazali et al., 2017) and the characterization for the flat sheet carbon membrane (without substrate) was following the similar procedures. The heating cycle was controlled by Eurotherm 2500  $^{\circ}C$  temperature control systems as shown in Figure 2.

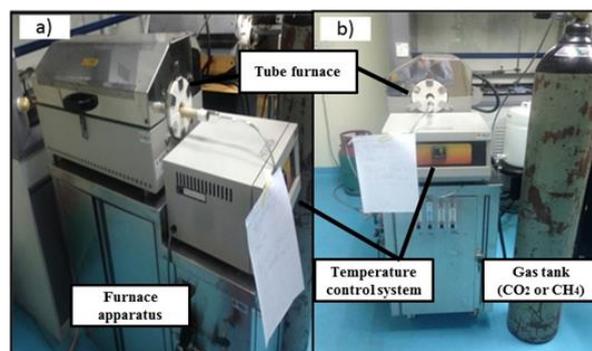


Figure 2 (a): Furnace operation, (b) Complete furnace operation.

### Membrane characterization

Scanning electron microscopy (SEM) was used to observe the membrane structure and morphology. Prior observation, the samples were coated with gold by employing a scanning electron microscope (TM3000, Hitachi) with a potential of 10 kV.

### Pure gas permeation measurements

Gas permeation system as explained in our previous study was employed to test the carbon tubular membranes (Sazali et al., 2018, Sazali et al., 2017) as in Figure 3.



Figure 3: Tubular membrane module

A tubular stainless-steel module of 14 cm in length was used and the carbon tubular membrane was put inside the module. An O-rings was fitted to the membrane to avoid leakage on the module. Trans-membrane pressure of 8 bars was applied and pure nitrogen (N<sub>2</sub>) gas (0.364 nm), carbon dioxide (CO<sub>2</sub>) (0.330 nm) were fed separately into the module. Equation to calculate the permeance, P/l (GPU) and selectivity,  $\alpha$  of the membranes were calculated using the following equations:

Permeance, P:

$$(P/l)_i = \frac{Q_i}{\Delta p \cdot A} = \frac{Q}{\pi D l \Delta P} \quad (1)$$

Selectivity,  $\alpha$ :

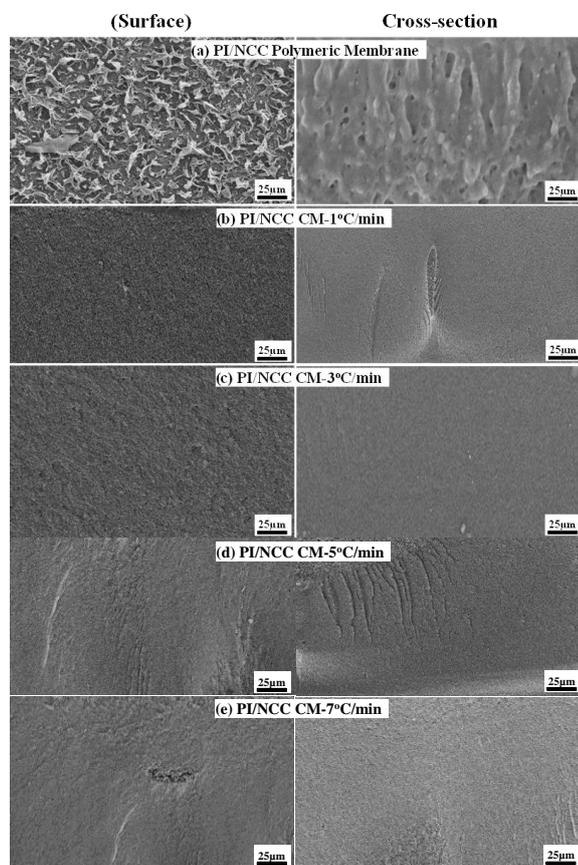
$$\alpha_{A/B} = \frac{P_A}{P_B} = \frac{(P/l)_A}{(P/l)_B} \quad (2)$$

where P/l is the permeance of the membrane, Q<sub>i</sub> is the volumetric flow rate of gas i at standard temperature and pressure (cm<sup>3</sup> (STP/s)), p is the pressure difference between the feed side and the permeation side of the membrane (cmHg), A is the membrane surface area (cm<sup>2</sup>), D is the outer diameter of the membrane (cm) and l is the effective length of the membrane (cm). The permeate volumetric flow rate was measured by the soap bubble flow meter reading of 1.0ml. This permeation test was repeated with two other different membrane samples. The gas permeation test was conducted at ambient temperatures. Similar gas permeation was also used in previous studies (Sazali et al., 2017)

## RESULTS AND DISCUSSION

### Scanning electron microscopy analysis

The heating rate used in this experiment was in the range of 1 to 7°C/min. The micrograph and outer surface images of PI/NCC-based carbon membranes were shown in Figure 4.



**Figure 4:** Surface and cross-section microphotographs of the (a) PI/NCC polymeric membrane, PI/NCC CM prepared at different carbonization heating rate (b) 1°C/min, (c) 3°C/min, (d) 5°C/min, and (e) 7°C/min.

The image showed that the PI/NCC polymeric membrane owned a few microporous structure and blocked pores on the outer surface. A defect free surface of PI/NCC carbon membranes were obtained after the heating temperature was increased up to 800 °C where the micro pore size became smaller and invisible. This is because the heat treatment does not influence the physical structure arrangement of the membrane. In addition to that, it is also postulated that lengthen the heat treatment will ominously increase the thickness of the sin layer and reduce the diameter of the precursor membrane.

### Gas permeation measurements

In literature, the rate of evolution of the volatile compounds can be determined by carbonization heating rate as it is believed could affect the microstructure of the carbon membranes (Salleh and Ismail, 2012). The variation in carbonization heating rates have showed different gas separation results on the PI/NCC carbon membranes as represented in Table 1. The data obtained showed an average value from at least three different PI/NCC carbon membranes with small error analysis of  $\pm 10\%$  for both selectivity and permeance value.

**Table 1:** Gas separation results for PI/NCC carbon membranes carbonized at different heating rate.

Sample	Carbon membrane		
	Permeance (GPU)		Selectivity
	N <sub>2</sub>	CO <sub>2</sub>	CO <sub>2</sub> /N <sub>2</sub>
PI/NCC 1°C/min	3.08±3.65	174.12±1.99	56.53±3.87
PI/NCC 3°C/min	3.22±3.21	213.56±2.17	66.32±2.18
PI/NCC 5°C/min	3.18±4.16	196.44±2.77	61.77±1.54
PI/NCC 7°C/min	3.11±3.98	181.65±1.36	58.40±3.21

As tabulated in Table 1, it can be seen that the gas permeance of N<sub>2</sub> and CO<sub>2</sub> decreasing when the heating rate increasing from 1 to 3°C/min but start to decreased from 5°C/min to 7°C/min due to the decreasing in pore size distribution and concurrently, due to the carbon structure densification. Previously, numerous studies have reported that increases in carbonization heating rate can affect the pore size distribution by producing pores with smaller size and this is believed to cause another limitation in the degree of gases rotation freedom (Wang et al., 2014, Yong et al., 2016). According to Centeno et al. (2004), they found that random smaller pore size distribution polymer-based carbon membranes can be affected by the higher carbonization heating rate (Centeno et al., 2004). This is might due to the loss of most of the volatile compounds during the increases in the heating rate and subsequently, the partial carbon vapor deposition occurred in the pores that were formed previously. Moreover, it is believed any further increment to the heating rate might cause pinholes creation and microscopic crack on the membrane surface. During the heat treatment, the membrane are expected to deform.

As mentioned earlier the most effective heating rate for PI/NCC carbon membrane was 3 °C/min and this was presumed because of its favourable and excellent characteristics in term of gas permeation performance. The selectivity of CO<sub>2</sub>/N<sub>2</sub> for the carbon membrane was 66.32 ± 2.18 and this result is comparable with the other polymeric membrane with the selectivity ranging from 600 to 800°C (Sazali et al., 2017). In spite of all the good thing about carbon membrane, there is one major concern which is extra careful is needed when handling the carbon membrane. It was found that, at heating rate of 1 and 3 °C/min, the PI/NCC carbon membrane are easily mounted as compared to 5 and 7 °C/min heating rate. This is probably due to the deformation of cross-section or irregularities happened in the post-heat treatment. These membranes are able to be used in module at laboratory scale for gas permeation testing, even though it has low mechanical properties. Salleh and Ismail (2012) stated that defects and cracks on the carbon membranes surface were designated as membrane fragility (Salleh and Ismail, 2012). The authors also stated that there is less information about mechanical properties of the carbon membranes were discussed in the previous study. However, it is important to mention that there are other factors that might affect the separation performance such as

variation of precursor types, synthesis procedure, and configuration of membrane. Hence, the study on carbon membrane optimization for specific gas system might be not incompatible for another system.

The implement of low heating rate to the carbon membrane have resulting reduction in the values of gas permeance. In comparison to the other tested membranes, these membranes possessed lower separation value, however the value obtained is acceptable as its value is still comparable with previous hydrogen separation studies (Favvas et al., 2015). Furthermore, the highest CO<sub>2</sub>/N<sub>2</sub> separation was obtained by carbon membrane carbonized at heating rate of 3°C/min as compared to 5 and 9°C/min. This was caused by long contact of membrane with the inert gas that consequently, narrow down the pore size distribution. The increment of carbonization heating rate from 5 to 7°C/min will decreasing the selectivity of the CO<sub>2</sub>/N<sub>2</sub>. Sazali and co-workers (2017) have also stated that the pore size distribution was also attributed by selection of types of the polymer precursors such as the chemical composition of the polymer precursor (Sazali et al., 2017).

## CONCLUSION

Carbonization parameters such as rate of heating have affected the performance of the PI/NCC carbon membrane. In this study, the properties and performance of tubular carbon membranes (TCMs) that were fabricated by blending of PI/NCC has been studied. It was found that carbonization conditions can affect the morphologies of the membrane and also their separation capabilities. The results suggest that the membrane selectivity can be improved by applying optimum heating rate of 3°C/min during carbonization process. In CO<sub>2</sub>/N<sub>2</sub> separation, it was found that carbon membrane prepared at 3 °C/min exhibited the selectivity of 66.32±2.18. From this study, it can be concluded that PI/NCC carbon membrane have potential characteristics in various gas separation applications including H<sub>2</sub> recovery, O<sub>2</sub> enrichment and also CO<sub>2</sub> capture.

## ACKNOWLEDGEMENT

The authors gratefully acknowledge the financial support from Ministry of Higher Education and Universiti Teknologi Malaysia (UTM) under Higher Institution Centre of Excellence Scheme (Project Number: R. J090301.7846.4J188) and Research University Grant Scheme (Project Number: Q. J130000.2546.18H97). The authors would also like to acknowledge technical and management support from Research Management Centre (RMC), Universiti Teknologi Malaysia. The authors gratefully acknowledge the financial support from the Ministry of Higher Education and Universiti Malaysia Pahang under Fundamental Research Grant Scheme (Project Number: Rdu170125).

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