

# Tubular carbon membrane for Hydrogen separation: Effect of Pyrolysis condition

Mohd Syafiq Sharip<sup>1</sup> and Norazlianie Sazali<sup>1,2,\*</sup>

\*Correspondence

azlianie@ump.edu.my

+This author contributes  
equally to this work

<sup>1</sup> Faculty of Mechanical &  
Automotive Engineering  
Technology, Universiti  
Malaysia Pahang, 26600,  
Pekan, Pahang, Malaysia

<sup>2</sup> Centre of Excellence for  
Advanced Research in Fluid  
Flow (CARIFF), Universiti  
Malaysia Pahang, Lebuhraya  
Tun Razak, 26300 Gambang,  
Kuantan, Pahang, Malaysia.

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## ABSTRACT

Hydrogen (H<sub>2</sub>)-based economy development is expected to create extensive need for efficient collecting strategies of fairly high purity H<sub>2</sub>. The aim of a H<sub>2</sub>-selective membrane is to manipulate H<sub>2</sub>'s high diffusivity characteristics as well as to restrict the outcome of lower solubility. Carbon membranes offer high potential in gas separation industry due to its highly permeable and selective. Therefore, this study aims to investigate the effect of pyrolysis temperature on the gas separation properties. Matrimid 5218 used as a precursor for carbon tubular membrane preparation to produce high quality of carbon membrane via pyrolysis process. The polymer solution was coated on the surface of tubular ceramic tubes by using dip-coating method. Dip-coating technique offer high potential in fabricating defect free carbon membrane. The polymer tubular membrane was then carbonized under argon atmosphere at 600, 700, and 800, and 900°C with heating rate of 2 °C/min. Matrimid 5218-based carbon tubular membranes were fabricated and characterized in terms of its structural morphology, chemical structure, thermal stability, and gas permeation properties by using scanning electron microscopy (SEM), Fourier transform infrared (FTIR), and pure gas permeation system, respectively. The highest H<sub>2</sub>/N<sub>2</sub> selectivity of 401.08±2.56 was obtained for carbon membrane carbonized at 800°C with heating rate of 2°C/min.

## 1.0 INTRODUCTION

Pyrolysis process is carried out after stabilization process. In pyrolysis process, the temperature of an appropriate carbon precursor is increased to the temperature of pyrolysis in a controlled atmosphere (inert or vacuum) at a particular rate of heating for an appropriate thermal soak time [1]. Pyrolysis parameters that give the most effects are final temperature of pyrolysis, rate of heating as well as atmosphere of pyrolysis. Matrimid is the most commonly utilized commercial in carbon membrane fabrication [2]. At the temperature of up to 800°C, Matrimid membrane pyrolysis will resulted in the production of carbon membrane with ultramicropores (<7 Å) as well as micropores of larger sizes. In general, carbon membrane of Matrimid-based exhibits low permeability with high selectivity in the processes of gas separation of H<sub>2</sub>/N<sub>2</sub>.

Various steps are needed in order to produce carbon membrane with high performance. Conversion of polymeric membranes to carbon membranes having interchanging porosity degrees as well as properties of structure and separation resulted in achieving the process of pyrolysis [3, 4]. Past researcher reported pore dimensions as well as its distribution in carbon membrane are provided by a modest treatment of thermochemical in order to acquire numerous separation aims and essentials. Moreover, Ismail, A. F. stated that Kapton and Matrimid are completely carbonized under 500-800°C temperature range in vacuum atmosphere [5]. Currently, aromatic polyimide for instance Matrimid is among the most thoroughly researched polymer precursor with successful usage in preparing carbon membranes. Utilization of aromatic polyimides as it is or mixed with other polymers such as polyethylene glycol (PEG), is also recognized as the finest materials in preparing the carbon membranes due to its great mechanical and separation capabilities [6]. Carbon membranes are the best membrane in constructing new technologies for membrane due to its molecular sieving abilities and stability. Reviews on the most distinguished benefits of carbon membranes compared to the polymeric membranes are recently available [7-9]. In the work of Miguel, T. et al., (2014), polymer solutions with  $\gamma$ -AlO(OH) as well as AgNO<sub>3</sub> were covered with  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> tubes before being carbonized under the temperature of 550°C in a single dipping-drying-pyrolysis step [10]. Hosseini, S.S. et al., (2014) reported the highest selectivity of gas pair for CO<sub>2</sub>/CH<sub>4</sub>, CO<sub>2</sub>/N<sub>2</sub> as well as O<sub>2</sub>/N<sub>2</sub> can be acquired via pyrolysis of PBI-Kapton carbonized under 800 °C at 10–7 Torr [11]. Therefore, it is crucial to emphasis on the attractive factors of carbon membranes that make it beneficial as a tool for separation.

The pyrolysis temperature has strong characteristic in decomposes the material. The high temperature effect on the decomposition of chemical bonding among the chemical compound that resulted on conversion of polymeric precursors into the carbon material [12]. Besides that, the effect of pyrolysis temperature can be observed when it reached the optimal temperature. In relation, the optimal temperature of pyrolysis temperature increase on the structural, separation properties, crystalline properties, compactness and the density of the membrane [13]. If the membranes were carbonized in inadequate temperature, the resultant membrane would contain sub domains in which the polymer/precursor structure can be identified. At minimal pyrolysis temperature, the resulted membrane possesses large pore and would not able to discriminate high molecule effectively [14]. In contrast, the permeability of gas will reduce as the membrane compaction structure decrease when the pyrolysis temperature is excessive [12].

The carbon membrane densifications are produced because of the shrinkage of the membrane as the effect of high temperature. This example is in concurrence with the way that the carbonized membrane had a microspore structure that is proficient to perceive the different kinetic diameter and the selectivity [15]. The agreement was supported by previous research in which resulted in the linear relationship between the permeability and the temperature. The research also provides the significant point in which the higher temperature effects the permeability of H<sub>2</sub> and the compactness structure of carbon membrane increase respectively due to the closed pore during pyrolysis process. Nonetheless, the same result of high permeability of H<sub>2</sub> cannot obtain by N<sub>2</sub>. The limitation occurs due to the slightly difference of kinetic diameter between both gases and their similarity in terms of molecular structure [16].

## 2.0 Materials

Chemicals such as polyimide Matrimid 5218 was obtained from Sigma Aldrich while N-methyl-2-pyrrolidone (NMP) was acquired from Merck (Germany). These chemicals were utilized as is with no additional purification step. An 8 cm long porous tubular ceramic support (Al<sub>2</sub>O<sub>3</sub>) was bought from Shanghai Gongtao Ceramics Co., Ltd. It has 0.2  $\mu$ m average pore size, inner and outer diameter of 10 mm and 13mm respectively as reported from previous study [10].

## 2.1 Preparation of carbon membrane

Preparation of polymer solution was done under continuous stirring at 80°C and it contained 15wt.% of Matrimid 5218 in NMP. In order to eliminate trapped bubbles in the solution, sonication was performed and the solution was let to rest for 12 hours. Plunged the tubular support into the polymer solution prepared earlier for 45 minutes and allowed it to go through aging at 80 °C for 24 hours. Immersed the membranes in methanol for 2 hours then rested them inside an oven for 24 hours long and temperature of 100 °C. Carbon membranes were produced from carbonized polymeric membranes. In order to go through carbonization process, the supported polymeric membranes were positioned at the centre of Carbolite

horizontal tubular furnace. Throughout the whole process of carbonization, 2°C/min heating rate was enforced as per reported by previous study [18]. Temperature of the processed membranes was then decreased to room temperature naturally upon the end of every heating cycle.

## 2.2 Preparation of carbon membrane

Carbon tubular membranes have been experimented in the gas permeation system as depicted in our preceding study [19]. The carbon tubular membrane was mounted inside a 14 cm long stainless-steel tubular frame. With the aim of avoiding leakages while housing the membrane inside the module, the membrane was equipped with rubber O-rings.

## 3.0 Results and Discussion

### 3.1 Effect of Pyrolysis Temperature

#### 3.1.1 SEM Results

The cross-section image carbon membrane and Matrimid-based polymeric are shown in Figure 3.1 using the SEM instrument. The SEM image show the various result of various pyrolysis temperature. From the Figure 3.1(a), the display by the instrument demonstrate such as finger structure of the polymeric precursor due to the heat factor. The membrane become reduce in structure and increase the density of the carbon membrane simultaneously when the heat was applied despite of any temperature [17]. In addition, the thickness of the membrane has been decreased as the higher temperature was applied linearly. The condition can be proved based on the thickness of CM-600 (Figure 3.1b), CM-700 (Figure 3.1c), CM-800 (Figure 3.1d), and CM-900 (Figure 3.1e) which are reduced from 83.8  $\mu\text{m}$  to 51.5  $\mu\text{m}$  as the temperature increased.

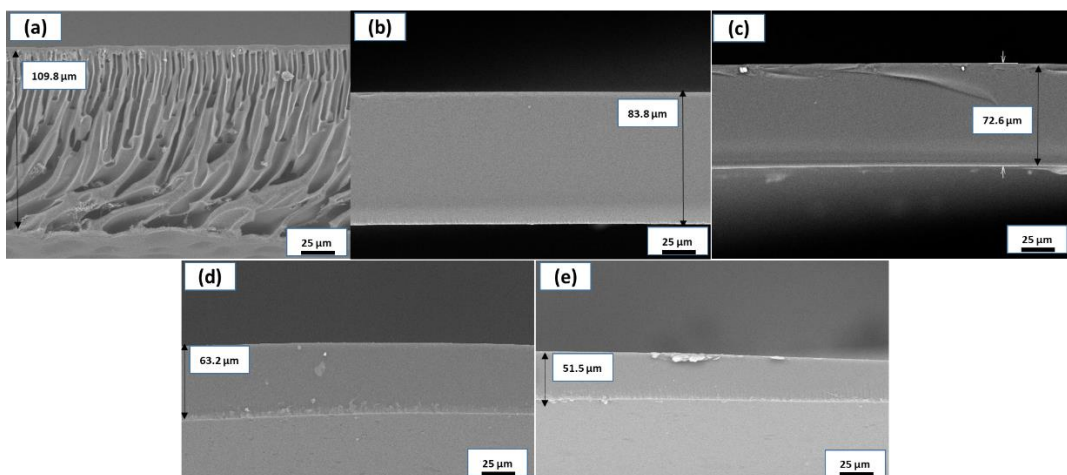


Figure 3.1: SEM images of the cross-sections for Matrimid/NMP-based (a) Polymeric membrane, (b) CM-600, (c) CM-700, (d) CM-800, and (e) CM-900

The heat element has significant role in order to decompose the bonding and structure of membrane and become denser and thinner respectively. The result obtained in this research also have the conjunction relation with previous study [18]. The previous research conducted have similar micrograph of carbon membrane and support the potentiality of this simple and relatively fast procedure, which offers new ways of designing and directly characterizing supported carbon membranes for the gas separation. The main different between carbon membrane from polyimide precursor and other polymer is the characteristic towards the gas separation performance. The polyimide precursor has high performance in gas separation because of their high selectivity characteristic. Based on all SEM results, the determination of the polymer precursor is a significant factor for carbon membrane and module preparation. In addition, the understanding of the relationship between the pore size and the permeability is crucial in order to

determine the transport mechanism of various carbon membrane under the different pyrolysis environment [19].

### 3.1.2 FTIR Results

Figure 3.2 shows the analysis of Matrimid 5218-based carbon membranes prepared by various pyrolysis temperature using FTIR instrument. The Figure 3.2 show the peaks being reduced linearly with the change of temperature compare to the polymer result.

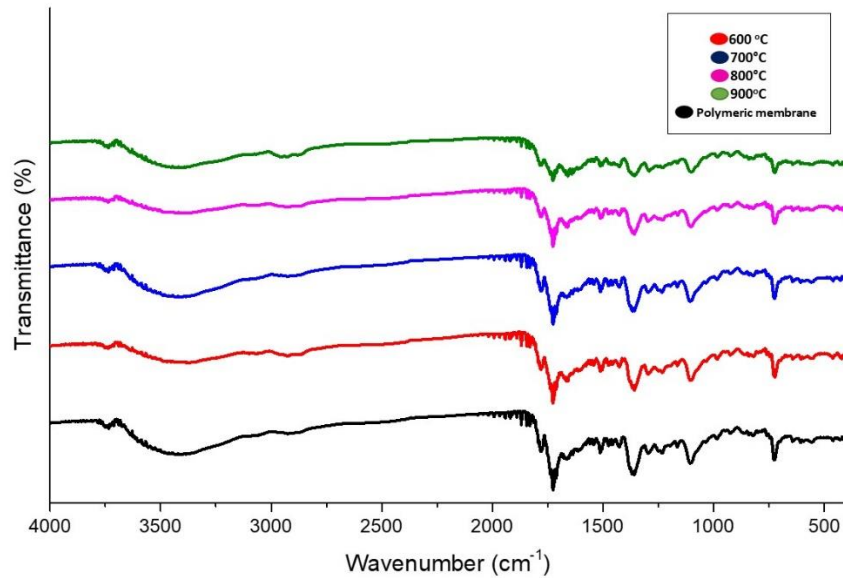


Figure 3.2: FTIR analysis of Matrimid-based carbon membranes prepared at different pyrolysis temperature.

The resulted prove that the temperature effect the bonding structure of the chemical compound. As the higher temperature use, the greater the decomposition of bonding take place in pyrolysis. The previous study also obtained the similar result that show insignificant peaks applied on several carbon membranes.

### 3.1.3 Gas Permeation properties

Table 1 display the carbon membrane permeability performance by using permeance test with room temperature and 8 bars parameters. In correlation, the permeance test show that the permeance increase as the temperature increase linearly. The permeance test resulted the change of membrane structure and the selectivity. According to previous research, basically the increase of temperature effect on contraction of gas permeance due to change of pores during the pyrolysis[4]. In contrast, the temperature ranges of 600°C- 800°C array the little increment of permeability for H<sub>2</sub> and N<sub>2</sub> gases because of the structural bonding for those gases become greater as the temperature increase and the compaction of selective layer occurred.

Table 1: Gas Permeation Properties of the Matrimid-based Carbon Tubular Membrane prepared at different pyrolysis temperature

Carbon membrane	Permeance (GPU)		Selectivity
	H <sub>2</sub>	N <sub>2</sub>	H <sub>2</sub> /N <sub>2</sub>
CM-600	986.22±2.37	3.21±2.11	307.23±2.19
CM-700	1045.87±3.74	3.25±2.89	321.81±1.86
CM-800	1247.37±1.52	3.11±3.59	401.08±2.56
CM-900	1128.57±2.78	3.29±3.28	343.03±2.67

Moreover, the comparison between small and larger molecule show that the small molecular have high tendency become more selectively as the temperature increase. The increases of permeance selectivity show that the inexistence of unselective crack during the high temperature of pyrolysis process [20]. The previous research shows the uses of Matrimid 5218 to study the pyrolysis temperature effect on the gas permeation performance of membranes. The resulted obtained provide a strong relationship between the pyrolysis temperature and the permeation performance. In addition, the study conducted also show that the pure gas H<sub>2</sub> permeance is consistently higher than that of N<sub>2</sub>. The pyrolysis temperature is defined as the highest temperature to which a precursor is heated during the pyrolysis process and was chosen in a range between the deconcentrating of the polymer and the graphitization temperature. Different temperature provides different effect on the permeation for the membrane as the Matrimid based membrane at 800°C produce high separation characteristic compared to the 600°C-700°C. This resulted in high pyrolysis temperature effect the size of cell membrane pores.

In relation, high temperature effect the carbon membrane become slightly small due to the actuality that carbonized layers have a micropore structure that able to perceive the distinctive kinetic diameter of gases and increases the selectivity. Comparable discoveries were likewise revealed by Sazali and co-workers [21]. For all gases, there will have the expansion in the unadulterated gas permeance with expanding pyrolysis temperature. This permeance increases was straightforwardly identified with the expansion in porosity with expanding pyrolysis temperature. Delightfully, despite the primary pyrolysis temperature, permeance will increase related to this pyrolysis temperature while the selectivity also increases. Consequently, further investigation on the impact of pyrolysis condition, the polymer film arranged from 15wt% of Matrimid and carbonized at 800°C was utilized.

### 3.2 Effect of Pyrolysis Environment

During the process, the surrounding is the priority to ensure and avoid any accident such as undesired burn off and chemical damage of the membrane precursor. Matrimid 5218 carbonized under vacuum was accounted for to create less penetrable yet progressively particular carbon layers contrasted with an inert gas pyrolysis system. In relation, the pyrolysis surrounding is significant to give perfect pyrolysis to Matrimid-based carbon layer manufacture [22]. Consequently, the test was done via carbonizing Matrimid-based polymeric layer under either N<sub>2</sub> or Ar air at 800°C.

#### 3.2.1 SEM Results

Figure 3.3 (a) shows the cross section for Matrimid 5218-based carbon membrane arranged under N<sub>2</sub> gas condition while Figure 3.3(b) demonstrates the cross section for Matrimid5218-based carbon membrane arranged under Ar gas condition by utilizing SEM. Based on the result, the dense carbon membrane can be achieving neglect any type of inert gases used and membranes from the both result showed a dense structure without any differences in the induced by pyrolysis environment. There is different thickness of membrane being prepared under the different type of inert environment such as the thickness of membrane for Ar environment and N<sub>2</sub> environment are 68.9 μm and 65.8 μm respectively.

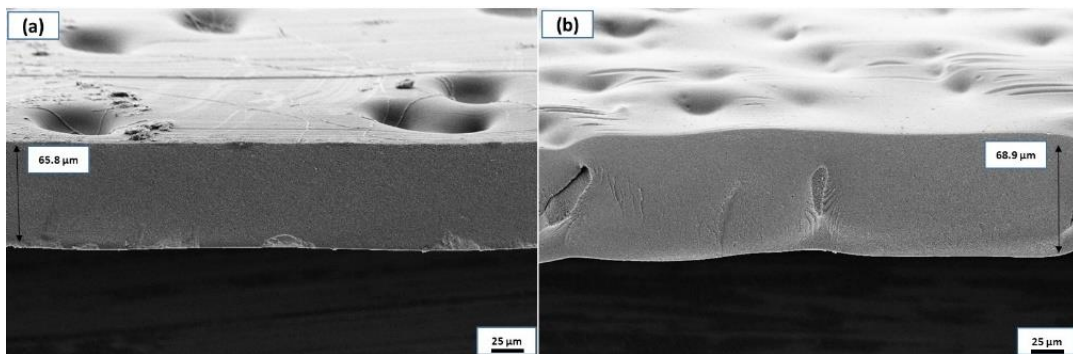


Figure 3.3: SEM images of the cross section of Matrimid-based carbon membranes prepared under (a) Nitrogen gas environment, (b) Argon gas environment.

In term of stability characteristic, Ar is more stable than N<sub>2</sub> due to the chemical configuration of Ar is octet which is the most stable inert gas than others. In contrast, the N<sub>2</sub> consist slightly unstable due to the

5 electrons on the outer shell that make  $N_2$  easily having reaction to gain stability with another compound. The pyrolysis utilizing Ar gas through ceramic materials produce better selectivity compared to other inert gas [23]. Be that as it may, their potential use is regularly constrained by the powerlessness to financially produce enormous or complex-formed parts showing solid performance. Plus, it is advantageous to manufacture parts in little sizes because of the impediment of the cylindrical layer module and heater [22]. Past examination expressed that in spite of the fact that these both dormant gas commonly produce palatable reproducible outcomes, every one of them requires high-temperature treatment, at times, high weight, in this manner lessening the practicality of the procedure particularly if the fabricate [24]. Past analyst likewise has been considered on the impact of pyrolysis condition and detailed that carbon layer arranged from polyimide Kapton® at 1000 °C in either argon or in a vacuum of 10<sup>-5</sup> Torr demonstrated moderately minor contrasts and thick in morphology structure [25].

### 3.2.2 FTIR Results

From the exploratory works, it tends to be demonstrated that the sub-atomic direction of the readied film is estimated by means of Frontier Transform Infrared spectroscopy (FTIR). Figure 3.4 demonstrates the decrease of peaks can be plainly observed at both pyrolysis environment and the antecedent polymeric membrane have changed to carbon membrane under both pyrolysis environments. No evident distinctive on utilitarian gathering of carbon film under various pyrolysis condition [26]. It shows, with high pyrolysis temperature however various pyrolysis environment, the changing patterns of the peaks somewhat were comparable.

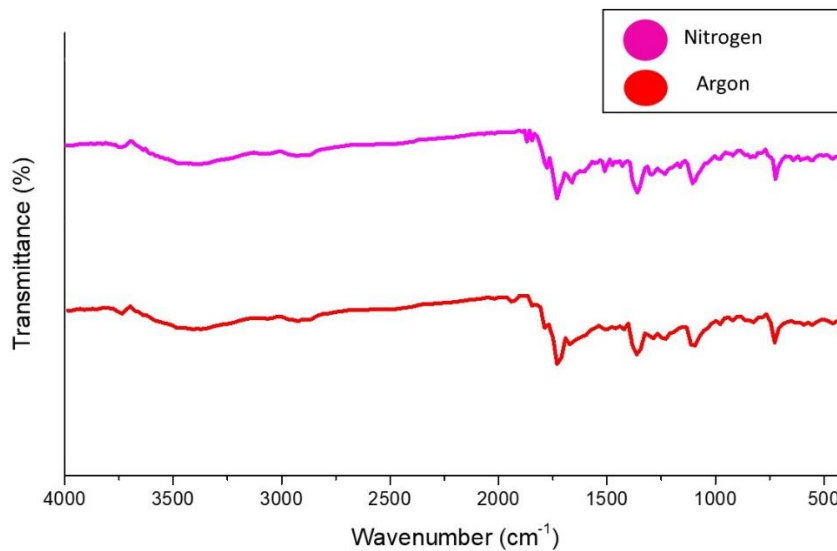


Figure 3.4: FTIR analysis of Matrimid-based carbon membranes prepared under Nitrogen and Argon gases.

### 3.2.3 Gas Permeation Properties

The performance of carbon membrane was effect by the pyrolysis environment respectively and prove by the results in Table 2. The Ar inert gas has higher permeability and selectivity with 1247.37±1.52 ( $H_2$ ), 3.11±3.59 ( $N_2$ ), and 401.08±2.56 ( $H_2/N_2$ ) for 800°C compared to the  $N_2$  gas. In addition, the Ar gas also originated higher permeable pores than  $N_2$ . At the 800°C, the permeability decrease contrast with the temperature at 600°C. It was observed since the dense layer of Ar carbon membrane are thinner compared to  $N_2$  carbon membrane, therefore it provides less transport resistance [27]. Surprisingly, the gas pair selectivity of Ar carbon membrane also higher than  $N_2$  carbon membrane. Carbon membrane produce under high thickness condition (Ar  $\rho$  = 1.784 g/l) will give preferable skin quality over carbon membrane produce under low thickness condition ( $N_2$   $\rho$  = 1.251 g/l).

Table 2: The performance of carbon membrane effected by the pyrolysis environment

Carbon membrane	Permeance (GPU)		Selectivity
	H <sub>2</sub>	N <sub>2</sub>	H <sub>2</sub> /N <sub>2</sub>
Argon	1247.37±1.52	3.11±3.59	401.08±2.56
Nitrogen	1229.85±2.88	3.16±2.54	389.19±1.86

Until this point in time, the layers that carbonized under idle environments produce a perfect gas permeance and selectivity aside from Helium gas. He did mention that Ar environment will show better selectivity compared with N<sub>2</sub> environment. In completion, the best pyrolysis environment for the readiness of the Matrimid 5218-based carbon membrane was under Ar gas stream [28].

#### 4.0 CONCLUSIONS

In completion, the carbon membrane was believed as the alternative for the separation process in near future. The use of carbon tubular membrane also shows that the characteristic and properties of carbon are the advantage for separation process compared to the other membrane material based on the research conducted using two gases such as H<sub>2</sub> and N<sub>2</sub>. The higher of pyrolysis temperatures effect on the increasing in permeability and selectivity. High selectivity was achieved at temperatures of 800°C. The results of gas permeance test indicate the linear relationship between the selectivity of the carbon tubular membrane with pyrolysis temperature. The highest H<sub>2</sub>/N<sub>2</sub> separation selectivity was achieved at 800°C of carbon membrane. Argon gas was the best condition during heat treatment process of pyrolysis environment because it can produce high selectivity of carbon membranes compared with nitrogen gas.

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