The Development of Carbon Membrane for Gas Separation: A Review

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Abstract: The applications of carbon membranes are becoming more important than ever due to their resilient mechanical strength, stability, and separation performance. The production of carbon membranes involves a suitable precursor that is being subjective to a pyrolysis process under controlled operating conditions that will result a porous structure to be utilized in many different separation applications. Thus, by understanding the preparation aspects of this fabrication process, the product can be manipulated, to optimize the best preparation procedure for obtaining the desired properties of a given type of membrane. This paper reviews the preparation aspects of carbon membranes, that can be manipulated to enhance the overall separation performance.

Keywords: Carbon, Membranes, Gas separation, Molecular sieve.

INTRODUCTION

The continues mandate for high efficient processes to limit the emissions of gases that contribute to the greenhouse has triggered the global enthusiasm to embrace new green technologies with a long-term potential solution [1-3]. Within the context of this worldwide environmental concerns, membrane technology has attracted a considerable attention as it require limited amounts of chemicals compared to other standard unit operations [4-8].

Among the many types of porous inorganic membranes, carbon membranes have many distinct advantages and have been looked into and developed noticeably in the recent years, mostly for gas separation applications. [9-12]. For many applications, carbon membranes have a high potential for separation due to their micropore structure and amorphous properties. Moreover, the use of carbon membranes in many applications has been demonstrated to be very effective for cost and energy reduction instead of the conventional ones [5, 14-16].

Currently, polymer-based membranes are being the most popular type used in the membrane industries. However, their poor chemical and temperature stabilities limit their applications [17-19]. Therefore, many efforts have been made to explore other materials that exhibit molecular sieving properties, such as silica, zeolites and carbon. Carbon molecular sieve membranes (CMSM) acquire many attractive characteristic properties due to their selective planar molecule shape and hydrophobicity. The CMSM is produced by converting a given precursor by pyrolysis process [20-23].

Koresh and Soffer were the first to report a successful preparation of CMSM using organic cellulosic and phenolic resins as precursors [24-26]. They have reported and studied the temperature effect on the formation of the produced CMSM pore structure, followed by developing CMSM to improve gas separation performances. Nowadays, membranes performance is being investigated and developed to overcome the flux/selectivity trade-off and limitation.

The production of CMSM can be classified into different precursor essential treating parameters, illustrated in Figure **1**, 1) Selection 2) Pre-treatment, 3) Pyrolysis, and 4) Post-treatment. As a result, CMSM properties (including pore size) and performance are determined by manipulation of these parameters.

CLASSIFICATION

Different geometries and classifications have been reported in the literature to improve the permeability and selectivity of gas mixtures [28-33]. Carbon membranes can be produced in similar configurations as the polymeric membranes, such as hollow fiber, flat sheet, tubular, etc. Therefore, An unsupported carbon membrane can be distinguished from a supported carbon membrane. As for the configurations of supported structure, flat membranes and tubular membranes are the main categories. Conversely, unsupported structure can occur in three flat, hollow fiber, and capillary form. Supported membranes tend to have more surface areas resulting better separation capability [31-33]. Also, the supported membranes have better structural integrity compared to the non-supported membranes [33, 34]. The carbon membrane

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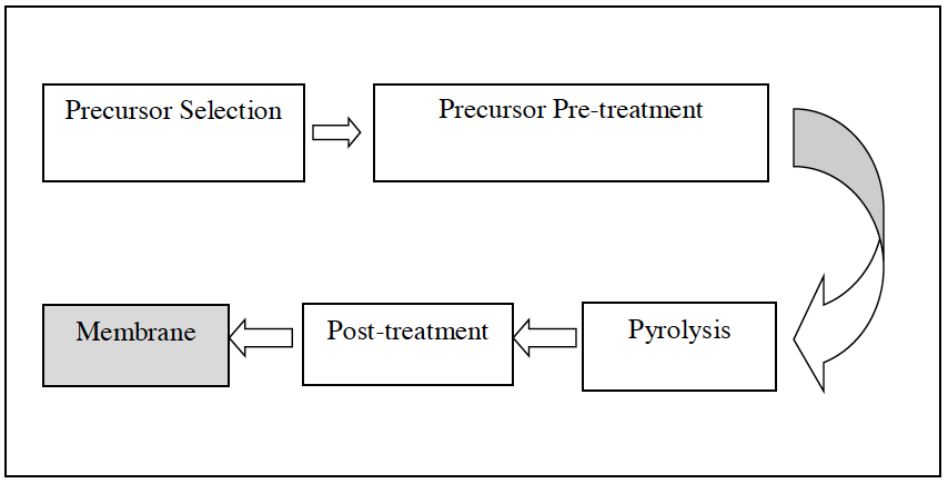

Figure 1: Fabrication steps of carbon membrane.

classification is summarized in Figure **2**. Membranes are categorized structurally as either isotropic (symmetric) or anisotropic (asymmetric). A symmetric membrane can be a porous membrane or a nonporous membrane (with a dense structure).

As a result of having a uniform structure and identical properties, the properties of symmetric membranes are identical over the entire cross section [35-38]. Conversely, asymmetric membranes contain an very thin surface layer based on a porous substructure with a higher thickness. The main purpose of using porous support is to provide the mechanical support without affecting the separation/permeation rates. Where the separation properties and permeation rates of the anisotropic membrane are determined exclusively by the surface layer. The higher fluxes resulted from using Asymmetric membranes are so great that almost all commercial processes use such type of membranes. Asymmetric membranes usually have a thin layer between 0.1 and 30μm thick, while supporting layers are usually above 200 micrometers (μm) in thickness [35-42]. The development of asymmetric membrane structure was one of the major innovations made in the separation technology during the last 30 years [41, 43]. In general, carbon membranes exists in asymmetric geometry (flat or tubular).

SELECTION OF PRECURSOR

A number of studies have concluded that the choice of the precursor is very significant as it can affect the final composition's structural properties [44-47]. In general, carbon materials are produced by carbonize a given precursor using an inert atmosphere or vacuum during the pyrolysis process. The most popular precursor currently used for manufacturing carbon membranes is polyimide [48, 49]. Although polyimide offers a promising selective carbon structure, however its cost is too high and its commercial availability is sometimes very limited. Therefore, in order to reduce the overall cost and production time during carbon

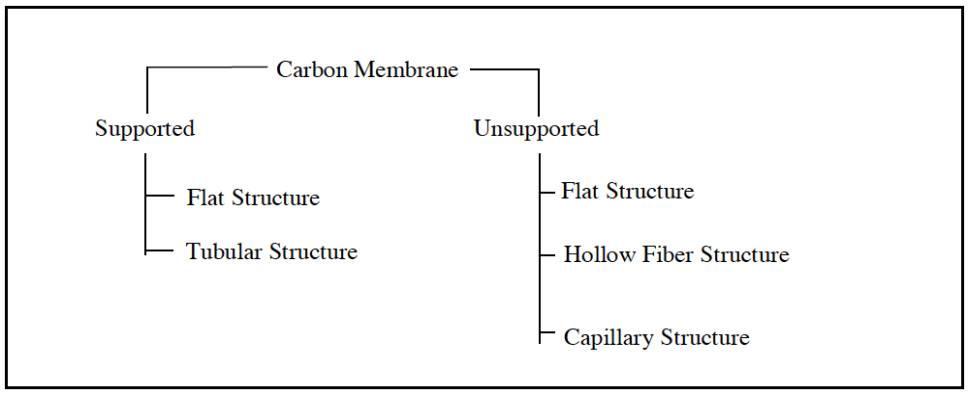

Figure 2: Carbon membrane classifications and structures.

membrane fabrication, alternative polymers were looked into and investigated as listed in Table **1.**

Table 1: List of Different Polyimides Precursors Prepared at Different Operating Conditions

Precursor	Configuration	Temperature Range (°C)	Exposure Duration	Ref.
Acrylonitrile	Hollow fiber	800-1600	5-60 min; N_2	50
Acrylonitrile	Hollow fiber	600-1200	10 min; N_2	51
Polyacrylonitrile	Hollow fiber	250	160 min; N_2	52
Polyacrylonitrile	Hollow fiber	260	50 min; N_2	53
Polyacrylonitrile	Hollow fiber	900	30 min; N_2	54
Cellulose	Hollow fiber	400-900	1-10 min; Ar	55
Cellulose	Hollow fiber	500-800	12 h; Ar	56
Cellulose	Supported film	120-400	30 min; Ar	57
Coal tar pitch	Plate	600-900	1h; N2	58
Condensed polynuclear aromatic	Supported film	400-1000	N2	59
Kapton and matrimid	Supported film	450-700	1h; Vacuum	60
Kapton and matrimid	Film	500-800	2h; Vacuum	61
Kapton polyimide	Film	1000 oC	2h; Vacuum	62
Kapton polyimide	Film	600-1000	2h; Vacuum or Ar	62
Phenol formaldehyde	Flat	800-950	120-180 min; N_2	63
Phenol formaldehyde	Tubluar	800	N2	64
Phenol formaldehyde	Support film	900	60 min; Ar	64
Phenolic resin	Support film	700-850	3h; N2 or CO2	65
Phenolic resin	Supported film	900	1h; N2	66
Phenolic resin	Supported film	500-1000	Vacuum	67
Phenolic resin	Supported film	700	Vacuum	68
PVDC-AC	Supported film	600	3h; N_2	69
PVDC-AC	Supported film	1000	3h; N_2	69
PVDC-AC	Supported film	1000	3h; N_2	69
PVDC-AC	Supported film	600	3h; N_2	69
PVDC-VC	Supported film	500-1000	Vacuum	69
PEI	Supported film	800	1h; Vacuum	70
PEI	Supported film	350	30 min; Ar	70
PFA	Supported film	500-800	2h; He or N_2	71
PFA	Supported film	600	1-2 h; He	72
PFA	Supported film	300	2h; N_2	73
PFA	Supported film	150-600	2h; He or N_2	73
PFA	Supported film	450	120 min; He	73
PFA	Supported film	150-600	120 min; He	74
PFA	Supported film	200-600	2 h; He	74
PFA	Supported film	600	120 min; He	74
PFA	Supported film	450	1 h; He	75
PFA	Supported film	450-600	1 h; He	76
PFA	Supported film	500-700	30 min ; N2	76
Polyimide	Supported film	550-700	1h; vacuum	77
Polyimide	Supported film	800	N_2	77
Polyimide	Supported film	600-900	Ar	78
Polyimide	Hollow fiber	500-550	2h; He	78
Polyimide	Hollow fiber	800	2h; vacuum	79
Polyimide	Hollow fiber	600-900	1h; N_2	80

Polyfurfuryl alcohol (PFA) precursor has been extensively used widely for nanoporous carbon membrane preparation [71, 72]. PFA polymerization process is very flexible as it can be achived under a wider range of temperate. Moreover, this precursor can also be used for simulation studies because it has a simple mechanism of structure formation. In the carbon membrane industry, PFA is used as a support film since it exists at room temperature as a liquid. Chemical stability and pore size distribution are desirable properties of PFA.

Using the porous surface of a stainless steel disk for support, Shiflett and Foley demonstrated the use of PFA in nanoporous applications. They demonstrated a successful procedures with separation factors of 600, 45, 17 and 14 for H_2/CH_4, CO_2/CH_4, N_2O/N_2 and H_2/CO_2, respectively [81].

Wang and his coworkers developed a composite carbonaceous polyfurfuryl alcohol (CPFA) on polysulfone support in 2010. This was achieved by carbonizing PFA partially. The application of this product was subjected for water desalination applications, achieving ratios of water permeability of 1.54, 2.01 and 0.17 $L \cdot \mu m \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$. Their study has concluded that due to the hydrophilic structure of the membrane, the flux is considerably higher than that of PFA membranes. [82]. Polyacrylonitrile (PAN), with the chemical formula $(C_3H_3N)_n$, is a well-known polymer type that has been used widely in the separation industry. Among its many uses, PAN is a complex of monomer used in reverse osmosis, textile manufacturing and high-quality carbon fiber production; 93% of the world's carbon fiber comes from this polymer [83]. Among its many advantages are PAN has a high melting point, high thermal stability, and a wide variety of mechanical properties. These properties have attracted considerable attention alongside other advantages, including a high melting point, thermal stability, and significant yields. The production of carbon membrane using Polyimides are usually done by condensing dianhydrides with diamines. Recently, this polyimide as a precursor has been used in glassy carbon production as it has performed exceptionally well in its mechanical stability and separation performance. One of the most mutual byproducts of this polymer is Kapton polyimide. Kapton has been subjected widely in the production of CMSM films. This is achieved by carbonizing Kapton precursor at 800 ^oC. Another polymer type *i.e*., Polyetherimide (PEI), has a great chemical properties. PEI is considered as an shapeless polyimide used in membrane preparation industry mostly for gas separation processes. Phenolic polymer, is another well-known polymer consists of mixture of chemical compounds (phenol and aldehyde). Phenolic polymers has a wide range of applications including the manufacture of carbon membranes at high yield with molecular sieve properties at low cost. Cellulose ($C_6H_{10}O_5$) polymer is also considered as a low-cost precursor and can be found in most types of plants [84].

PRE-TREATMENT

As the polymeric structure tends to shrink during the pyrolysis conditions, pre-treatment process is a vital step to avoid this as it has a significant impact in maintaining the precursor stability during pyrolysis [85]. Also, the pre-treatment process of a given precursors plays an essential role in the transportation of properties and could deliver a production of CMSM with the desired performance. Pretreatment of precursors will result in altering the cooperative segmental mobility of the polymer [86], which will significantly affect the structural organization of the structure during pyrolysis. In other words, the aim of the pre-treatment is to stabilize the repeat units' structure of the precursors, to obtain the molecular structure of the carbon chains during the pyrolysis process. Also, a given pre-treatment procedure could enhances the uniformity of pore formation during the pyrolysis process. Current pre-treatment methods include chemical (oxidation and chemical treatment) and physical methods (stretching). In general, the chemical methods can be summarised in exposing the polymeric precursor to a number of chemical reagents to obtain the desired final structure to achieve desired separation properties, while the physical pre-treatment is achieved by applying a stretching mechanism [87]. Oxidation (thermostabilization) is considered to be the most popular and commonly method used pretreating the polymeric precursors before involving it in the pyrolysis process [88-90]. The oxidation treatment has a excessive impact in stabilizing the structure of the precursors to withstand the high temperatures of pyrolysis and rise the final yield of the precursor's carbon by preventing excessive volatilization.

In 1997, Kusuki *et al* have tested the performance of a number of membranes that were not pre-treated prior to the pyrolysis process. The study has concluded the poor performance of these membranes due to not pre-treating them [91], emphasizing the great importance of this process. Other studies have reported this process using different precursors as listed in the Table **2**.

Table 2: Pre-Treatment (Oxidation) at Different Operating Conditions and Precursors

Precursor	Configuration	Temperature Range (^{o}C)	Exposure Duration	Ref.
Acrylonitrile	Hollow fiber	200-300	3 h	92
Acrylonitrile	Hollow fiber	180-350	1-20 min	92
Polyacrylonitrile	Hollow fiber	250	30 min; O_2	93
Polyacrylonitrile	Hollow fiber	260	30 min; N_2	94
Polyacrylonitrile	Hollow fiber	270	30 min; air	95
Phenol resin	Supported film	200-300	2 h; air	96
Phenol resin	Supported film	150	2 h; air	97
Phenol resin	Supported film	300	1h; air	97
PVDC-VC	Supported film	150	6h; air	98
Polyfurfuryl alcohol	Supported film	90	3h; air	99
Polyimide	Hollow fiber	400	30 min; air	100
Polyimide	Hollow fiber	400-450	1h; air	100
Polyimide	Hollow fiber	400-500	1-100 h; air	101
Polyimide	Hollow fiber	400	1h; air	102
Polyimide	Supported film	500	0-1h; air	103

Another aspect that should be considered in pretreating the membrane precursor with certain chemicals enhances the uniformity and distribution of the channeling structure during the pyrolysis step. The most common chemicals used to fulfill this task are hydrazine, dimethylformamide (DMF), hydrochloric acid and ammonium chloride [104, 105]. In the chemical pre-treatment process, the given membrane is fully immersed in a chemical solution then washed and dried before undergoing the separation application. Pretreatment of a membrane with certain chemicals can provide enhanced uniformity of the pore system formed during pyrolysis. Another pretreatment technique that is being used is the stretching pretreatment. This is a physical pre-treatment technique that has been adapted from spinning treatment and applied to many different hollow fiber precursors. The stretching method expose the surface defects and make them reachable so that they can be treated to become rigid fibers. Also, this method reorient the molecular structure to produce more balanced fiber arrangement. A previous study reported that the pretreatment by heating and stretching was conducted on PAN precursor fibers in a steam bath media, to convert the flexible linear chain molecule to a stiff ladder like structure by intramolecular cyclization process [106].

PYROLYSIS

Pyrolysis can be defined as a heating process, at a certain heating rates, that is being conducted at an elevated temperature in a vacuum/ inert atmosphere. This process aims to produce a carbon sturcture with molecular sieve properties [107]. In this process, most of the heteroatoms are removed, including; nitrogen, iodine, chlorine. This process aims to result a cross-linked carbon structure to avoid the formation of tapered pores the are produced by the graphite-like crystals.

A deep comprehension of the operational conditions *i.e*., pyrolysis temperature, gas flow, heating rates, and thermal soaking period, will make the desired structure of a given carbon membrane easy to achieve. Therefore, conducting the pyrolysis process in a proper conditions, will result a membrane with appropriate pore size. This is obviously of great importance to establish a high performance in terms of permeability and selectivity [108].

Generally, carbon membranes contain a pore system that is non-homogeneous as the size and shape of the pore system depend on the precursor type and the operating conditions of the pyrolysis process. Figure **3** illustrates a typical carbon pore structure,

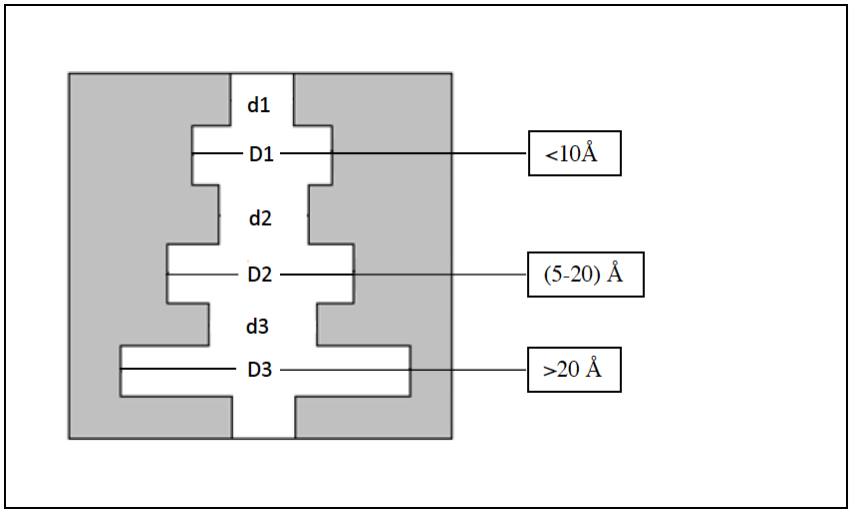

Figure 3: Illustration of a pore structure of carbon materials.

presenting the ultramicropores structure with a diameter less than 10Å (D1). This channel perform the sieving properties for a given molecule. On the other hand, micropores structure with diameter of 5-20 Å (D2) permit the molecules to be diffused through this channel. It can be concluded that the carbon membrane has the ability to deliver a significantly higher flux of permeated molecules than the sieving property solely [109].

In a controlled atmosphere, the pyrolysis process is usually performed at 500-1000 ^{o}C depending on the type of precursor to prevents any unwanted burnoff that might ruin it. As a result, heteroatoms are removed in order to form a cross-linked porous carbon structure. [110]. There has been extensive research revealing that temperature affects the production of carbon structure, including its separation performance selectivity and permeability, and that an increase in pyrolysis temperature leads to more crystallinity and smaller layer spacing [109-120]. By controlling the heating rate sequence, pyrolysis produces products with different volatilities and results in a final weight loss [130, 131]. By determining the evolution rate of volatile components, the heating rate contributes to the formation of the final shape. According to the findings, lower heating rates increase the carbon crystallinity and facilitate the formation of small pores. On the other hand, higher the heating rate, the more likely it will produce a structure with low separation selectivity due to pinholes, cracks, and deformations [121-132]. Another aspect in the pyrolysis process that affects the final properties is the thermal dwell period. This is a key player in the rearrangement of the microstructures, which leads to different distribution of pores in the final formation of the membrane. The period of thermal dwell depends on two main aspects *i.e*., pyrolysis temperature and precursor type.

In general, with longer thermal soak periods, a membrane's separation selectivity generally increases [107, 132]. The gas flow plays an important role in the pyrolysis process. Gas flow is introduced into the pyrolysis process in order to prevent any undesired burn-off, which can negatively impact membrane formation [134].

In general, a vacuum or inert atmosphere can be used to perform the pyrolysis process. In case of the use vacuum, a less permeable structure is formed that has a better separation selectivity than an inert atmosphere [135]. Meanwhile, inert gas flow can affects the overall permeability performance without affecting the membrane selectivity. It is established that the gas flow is proportional with the permeability of the carbon membrane [135, 136]. Different precursors with using different inert gas with dwell period are listed in Table **3**.

POST-TREATMENT

Oxidation process has also been utilized a post treatment process in the preparation of carbon membrane. Post-oxidation, is a popular post-treatment utilized in many reported studies to regulate the pore structure of the prepared carbon membrane. In which, the average pore size increases when the membrane is exposed to an oxidizing atmosphere afterward the pyrolysis process. The atmosphere involved in this

Table 3: List of Different Types of Precursors Under Pyrolysis Process Conditions

Precursor Type	Heat Rate	Temperature (°K)	Inert Gas	Dwell Period	Ref.
PFA	10 C/min	773-1072	He/N_2	2 hr	73
PFA	5 C/min	423-871	He/N_3	1-2 hr	75
PFA	6 C/min	723	He	2 hr	121
PAN	1 C/min	1223	N_2	3 hr	122
PAN	9 C/ min	523 -1073	N_2	3 hr	123
Polyimide	5 C/min	773-1173	N_2	N.A.	124
Polyimide	6 C/min	973-1073	Ar	N.A.	125
PEI	0.5 C/min	1073	Vacuum	1 hr	126
Phenolic resin	25C/min	1173	N_2	1 hr	127
Phenolic resin	50 C/min	1073	N_2	N.A.	127
Cellulose	0.5 C/min	393-673	Ar	N.A.	128
Cellulose	1-10 C/ min	673-1173	Ar	N.A.	129

process can be pure oxygen or mixed with other gases, or air [88, 89]. In recent years, the post-oxidation effect on the permeation performances of the carbon membrane under different oxidation conditions as listed in Table **4**.

The growing research on carbon membrane technology, especially in gas separation, indicates that carbon membranes is an alternative potential candidate to fulfill the industrial-related separation process due to their stability and molecular sieving capabilities. The results of carbon membrane performance that have been reported by different investigators in different gas separation applications as presented in Table **5**.

CONCLUSIONS AND FUTURE CONSIDERATION

Carbon membranes with its useful characteristics and advantages will compete with polymeric membranes and other porous inorganic membranes. However, further intensive research work should be carried out to develop the carbon membranes to meet the industrial standards and to commercialize carbon membranes in the international market. The production process of carbon membranes should be developed as it currently involves a relatively costly procedure. Also, the cost of a carbon membrane per unit of membrane area is reported to three times the typical polymeric membrane [18]. Therefore, carbon membranes must achieve a superior performance in order to compensate for their higher cost. Optimizing the fabrication parameters during the pyrolysis process is arguably the best way to achieve this goal. These parameters will tailor the pore properties of the resulted carbon membrane, which eventually determine the overall separation mechanism. In addition, the effect of pretreatments and posttreatments during the membrane fabrication process should also be

Table 4: Examples of Post-Oxidation Treatments of Selected Carbon Membranes

Precursor	Configuration	Temperature Range (°C)	Exposure Duration	Ref.
Cellulose	Hollow fiber	200-300	3 h; N_2	137
Phenol formaldehyde	Supported film	180-350	1-20 min; air	138
Phenol formaldehyde	Supported film	800	30 min-6h; air	138
Phenolic resin	Supported film	100-475	30 min; air	139
Phenolic resin	Supported film	300-400	30 min; air	139
Phenolic resin	Supported film	75-350	30 min; air	139
Phenolic resin	Supported film	800	3 h; O2	139
Polyimide	Supported film	300	3 h; O2	140
Polyimide	Hollow fiber	250-455	0.2–50 h; air	140

Table 5: Examples of Post-Oxidation Treatments of Selected Carbon Membranes

Precursor	O_2/N_2	CO_2/CH_4	CO_2/N_2	H_2/CH_4	H_2/N_2	He/N_2	Ref.
Sucrose		1.64	1.41	31.34			164,165
PAN							166
PEI		12.5					167
PEI	3.9		17.5				168
PEI	4.6		13.7				169
PEI	7.4						170
PEI / PVP	4.6						169
PEI/MWCNTs	24.2						169
PFA							171
PFR	10.65						172
PFR	1.8-2.8						173
Phenolic resin	6.8						174
Matrimid (M1)	0.72	0.86	0.7			26.5	175
Polyimide (AP)	12.3						176
Matrimid	8.8	78	27				177
Kapton	19.69	138.53	60.87				178
LARC-TPI Polyimide	3.0-3.3	>104					179
Polypyrrolone		180	16				180
PPES	5.5		16.3		22.6		181
PPESK	23.8-27.5		150.4 -213.8				182
PVDC-PVC	14						183
PPO		33	14			80	184
PPO	11.4	127					184
TMSPPO	10	102					185
PPO		138					185
PFR/PEG	9.7	20.2		693.6	406.9		186
PFR/CMSM	12.8	17.86		504.93	471.27		187
PPESK/PVP	6.5		25.7		38		188
PPESK/zeolite	6.9		102.5		26.6		189
PI/MWCNTs	3.3		4.1				190
PBI/Matrimid	6.67	56.41	28.06		466.1		191

considered. These steps provide a great opportunity for optimize the separation properties of a given carbon membrane. The data obtained from the experimental work should be utilized and simulated using computer software to provide an ideal pyrolysis condition that can be subjected in to a pilot scale.

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