

# A Review of Fabrication Methods of Carbon Membranes and Applications Related to Their Hydrophobic and Electrically Conductive Properties

Yuan-Yao LI\*, Akiyoshi SAKODA\* and Motoyuki SUZUKI\*

## 1. Introduction

In the past few decades, membrane technologies have emerged as an important means of separation in a variety of industries. This is because, in comparison with other separation processes, membrane separation offers the advantages of, at least, high selectivity, low energy consumption, moderate cost to performance ratio, and compact and modular design<sup>1)</sup>. Membranes are traditionally divided into organic and inorganic types. The former type contains carbon-hydrogen bonds such as polymeric and biological membranes. All other membranes—ceramic, metal, carbon, and glass—are inorganic. Inorganic membranes have been used commercially for years in food, water/wastewater, biotechnology, pharmaceutical, and petrochemical processing because of their characteristics of thermal stability, chemical compatibility, pH resistance, and good mechanical strength<sup>2)</sup>. Many studies of inorganic membranes including membrane synthesis, characterization, applications, and theoretical modeling<sup>2-5)</sup> have been conducted to achieve a better understanding of membrane materials and processes. This, in turn, should lead to the creation of novel membranes and new membrane processes for enhancing membrane permeability and permselectivity. Depending on pore size, membranes are categorized as microfilters (pore size  $> 0.1\mu\text{m}$ ), ultrafilters ( $1\mu\text{m} >$  pore size  $> 0.1\text{nm}$ ), and nanofilters (pore size  $< 0.1\text{nm}$ ). Microfilters and ultrafilters are generally used in separation processes of the liquid phase, while nanofilters are used in gas separation.

This paper is a review of the fabrication methods and applications of carbon membranes. Its primary focus is on issues related to our recently developed membrane called the “carbon coated ceramic membrane,”<sup>6)</sup> shown in Figure 1. This membrane consists of a ceramic substrate and a layer of deposited carbon thin film covering the surface of the substrate. By reviewing and comparing



Fig. 1 Carbon coated ceramic membranes by chemical vapor deposition.

the characteristics of a variety of carbon membranes, we expect to discover new applications for carbon coated ceramic membranes.

## 2. Fabrication methods

### 2.1 Microfiltration and Ultrafiltration

Carbon membranes can be made by (1) *in situ* pyrolysis of polymers, (2) chemical vapor deposition of hydrocarbons on a porous substrate, and (3) activation of carbon fibers. Table 1 is a list of the literature published during the past decade. *In situ* formation of a carbon membrane is achieved by the deposition of a polymeric film such as phenolic resin<sup>7)</sup> on the top of a support followed by thermal treatment to pyrolyze the polymer. Depending on the temperature, the pore size of the membrane can be controlled, and the membrane can become slightly hydrophobic (low temperature) or more hydrophobic (high temperature). In addition, the carbon film is firmly bound to the support so that it can be used in micro/ultrafiltration applications. Carbon membranes can also be formed by *in situ* methods without any support. Schindler *et al.*<sup>8)</sup> used acrylonitrile polymer and hydrazine solution as the starting

\* Institute of Industrial Science, University of Tokyo

materials to form tubular or flat membranes. It was suggested that the carbon membrane could be used wherever electrical conductivity of membranes is required. Figure 2 depicts the formation of carbon membranes by the *in situ* method and the CVD method. The *in situ* method involves building a second layer on the substrate while the CVD method generates a carbon film coating on a porous substrate, which is usually made of interconnected granular particles. Precursors for CVD are generally hydrocarbons in vapor form such as ethylbenzene, propylene<sup>9</sup>, methane,<sup>6</sup> etc. Carbon coated membrane is formed by the decomposition of hydrocarbon under high temperature followed by the diffusion and deposition of carbon species on the surface of the substrate to form a carbon thin film. The pore size of the membranes decreases as the thickness of the carbon film increases. In addition, with a carbon film on the membrane, the properties of the membrane such as hydrophobicity, thermal conductivity, and electrical conductivity can be modified due to the characteristics of carbon. The other method of fabricating carbon membrane is activation of natural carbon materials<sup>10</sup> or hollow carbon fibers by a burn-off method. Carbon dioxide or steam is commonly used to react with the nitrogen, hydrogen, and oxygen atoms in the material causing the

material to release gases and create interconnected pores. The porosity and pore size depend on the ratio of the components in the material and the percentage of burn-off.

## 2.2 Nanofiltration

In nanofabrication, carbon membrane is sometimes referred to as carbon molecular sieve membrane (CMSM) because the membrane pores are very small. The membrane acts as a sieve enabling the passage of certain micromolecules. Study of the fabrication of carbon nanofilters for gas separation has increased rapidly in the past few years. The methods are listed in Table 2. Apart from the *in situ* formation method (dipping&coating)<sup>10-19</sup> and the CVD formation method<sup>20-22</sup>, new technologies have been developed including spray coating<sup>23</sup>, ultrasonic deposition<sup>24</sup>, and plasma deposition<sup>20</sup>. Polyfurfuryl alcohol (PFA) in acetone was used as a precursor in making carbon membrane by a spray coating process. The precursor was sprayed onto a porous stainless steel support using an airbrush followed by pyrolysis at 600°C. The resulting membrane was found good for air separation. Recently, a high selectivity nanoporous carbon membrane was made by ultrasonic deposition of polyfurfuryl alcohol on porous stainless steel tubes followed by pyrolysis at 450°C. The ultrasonic deposition technique enables formation of a carbon thin membrane on the support without cracking. As compared to that for nitrogen, it was suggested that the ideal separation factors for oxygen, helium, and hydrogen are 30:1, 178:1, and 331:1, respectively. Plasma deposition of thermosetting polymers on a porous matrix membrane, which could be of a varied configuration including sheet form, tubular form, and asymmetrical, is another method of fabricating nanoporous carbon membranes. Pyrolysis at a temperature ranging from 500 to 800°C was suggested as a means of creating a narrow range of pore sizes (0.25 to 0.5nm) for gas separation. Gaseous mixtures can also be separated by a modified

Table 1 Fabrication methods of carbon membranes for microfiltration and ultrafiltration.

No.	Technique	Carbon precursor	Support	Applications	Ref.
1	<i>In situ</i> pyrolysis/carbonization	Phenolic resin	Porous carbon tube (OD, 8mm, ID, 6mm)	Liquid phase separation	7
2	<i>In situ</i> pyrolysis/carbonization	Acrylonitrile polymer (to form tubular or flat membranes) + hydrazine solution	None	Liquid phase separation	8
3	Chemical vapor deposition	Ethylbenzene, propylene	Ceramic membrane	Liquid phase separation	9
4	Activation	None	Hollow carbon fibers, natural carbon material	Liquid phase separation	10

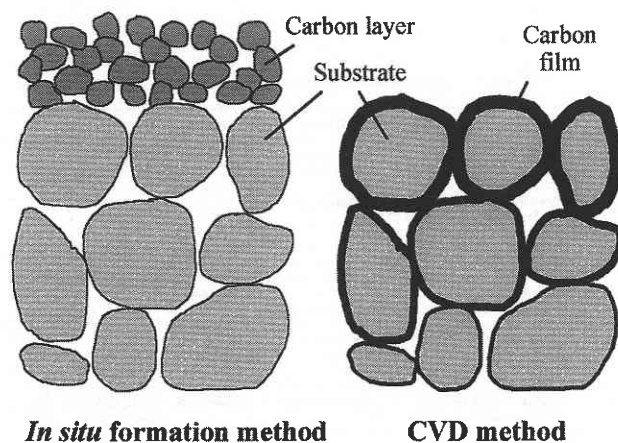


Fig. 2 Formation of carbon membrane by *in situ* and carbon coated membrane by CVD method.

Table 2 Fabrication methods of carbon membranes for nanofiltration.

No.	Technique	Carbon precursor	Support	Applications	Ref.
1	Dipping & coating <i>in situ</i> pyrolysis	Polymeric precursors	Porous membrane, activated carbon fiber	CO <sub>2</sub> /CH <sub>4</sub> , O <sub>2</sub> /N <sub>2</sub> , CO <sub>2</sub> /N <sub>2</sub> , H <sub>2</sub> /CH <sub>4</sub> , H <sub>2</sub> /CO <sub>2</sub> , He/N <sub>2</sub>	11-19
2	CVD	Cellulose, a cellulose derivative, a thermosetting polymer	Hollow carbon fibers	Gaseous mixtures	20
3	CVD	CCl <sub>3</sub> CH <sub>3</sub> and (CH <sub>3</sub> ) <sub>2</sub> C or any carbon-containing gas	Porous hollow fibers	H <sub>2</sub> /N <sub>2</sub> , gas separation	21
4	CVD	Propylene	Alumina support + polyimide	N <sub>2</sub> /CO <sub>2</sub> , N <sub>2</sub> /O <sub>2</sub>	22
5	Spray coating	Poly(furfuryl) alcohol (PFA) in acetone	Porous S.S. disk	O <sub>2</sub> /N <sub>2</sub>	23
6	Ultrasonic deposition	Poly(furfuryl) alcohol (PFA)	Porous S.S. tube	He/N <sub>2</sub> , He/N <sub>2</sub> , O <sub>2</sub> /N <sub>2</sub>	24
7	Plasma deposition	Cellulose, a cellulose derivative, a thermosetting polymer	Hollow carbon fibers	Gaseous mixtures	20

ceramic membrane formed by the CVD technique. These include the palladium<sup>25,26</sup>, SiC<sup>27</sup>, silica<sup>28</sup>, and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub><sup>29</sup> membranes listed in Table 3.

### 2.3 Comparison of carbon membranes

*In situ* pyrolysis of polymers, chemical vapor deposition of hydrocarbons, and activation of carbon-contained materials are the three principal methods of forming carbon membranes. In comparing these three methods in terms of the control of pore size, the CVD method provides the most accurate control. This is because carbon atoms are deposited on the membrane, and the pore size decreases by increasing the thickness of the pyrocarbon film. The *in situ* method is capable of controlling pore size by the rate and temperature of the pyrolysis. Table 4 compares the physical properties of carbon coated membranes formed by the CVD method, carbon membranes, ceramic membranes, and polymer membranes. As is shown in the table, inorganic membranes generally offer the advantages of thermal stability, chemical compatibility, and good mechanical strength. However, the porosity of the membrane, which affects permeability is relatively poor, especially in the case of the carbon coated membrane. This is because the carbon film coated on the membrane decreases the

Table 3 Modified ceramic membranes fabricated by the CVD technique.

No.	Precursor	Support	Membrane	Application	Ref.
1	Palladium acetylacetonate and palladium chloride (together with hydrogen)	Gamma -Al <sub>2</sub> O <sub>3</sub> supports	Pd membrane	Gas separation	25-26
2	Trisopropylsilane	Alpha -alumina support tube	SiC membrane	H <sub>2</sub> /H <sub>2</sub> O (steam)	27
3	Tetraethoxysilane	Porous glass tubes	Silica membrane	CH <sub>3</sub> OH vapor/He	28
4	Trimethylaluminum	$\alpha$ -Al <sub>2</sub> O <sub>3</sub> supports	$\gamma$ -Al <sub>2</sub> O <sub>3</sub> membrane	O <sub>2</sub> /H <sub>2</sub> O	29

Table 4 Comparison of types of carbon membranes.

	CVD carbon coated membrane	Other carbon membranes*	Ceramic membrane	Polymer membrane
Thermal stability	Good	Good	Good	Poor
Chemical stability	Good	Good/ Satisfactory	Satisfactory	Poor
Mechanical strength	Good	Good/ Satisfactory	Good	Poor
Hydrophobicity	Good	Good/ Satisfactory	Poor	Poor
Thermal conductivity	Good	Satisfactory	Poor	Poor
Electrical conductivity	Good	Satisfactory	Poor	Poor
Porosity	Poor	Satisfactory/Poor	Poor	Good

\* Other carbon membranes could be hollow carbon fiber membranes, carbon membranes, or carbon coated ceramic membranes made by the *in situ* carbonization technique

void fraction of the membrane<sup>6</sup>). A thicker carbon film leads to poorer membrane porosity. Compared to the other types, the carbon coated membrane possesses the advantageous properties of good hydrophobicity, electrical conductivity, and thermal conductivity because of the graphite-like structure of the non-porous carbon film. Therefore, utilization of these unique properties is strongly recommended to assist CVD carbon coated membranes for better separation. The review of the literature on membrane applications is focused on hydrophobic membranes and electrically conductive membranes because they are relevant to potential applications of CVD carbon coated membranes.

## 3. Applications

### 3.1 Applications of hydrophobic membranes

Hydrophobic membranes are generally used for the separation of biphasic mixtures or organics from aqueous mixtures. Table 5 lists hydrophobic membranes and their applications in separation processes as well as the separation techniques used. Kujawski<sup>30</sup> used polymer membranes or zeolite membranes to separate water/butyl acetate and water/methanol mixtures by the pervaporation technique. It was found that a high separation factor of over 900 could be achieved for a water-butyl acetate mixture. Pervaporation was also employed for the separation of ethyl esters-water mixtures with a poly (ether block amide) (PEBA) copolymer membrane reported by Djebbar *et al.*<sup>31</sup>. The results suggest that it is advantageous to use the PEBA membranes of the highest polyester content for extraction of volatile organic compounds (VOC). Isono *et al.*<sup>32</sup> utilized PTFE membranes and polyethylene membranes to separate seven different alcohol-water mixtures with a conventional filtration method. Nomura *et al.*<sup>33</sup> used carbon membranes fabricated by *in situ* pyrolysis to separate activated sludge. The results of the sludge experiments demonstrated that the carbon membrane produced better detach-

Table 5 Applications of hydrophobic membranes.

No.	Hydrophobic membrane	Applications	Technique	Conditions	Ref.
1	Polydimethylsiloxane membrane (PERVAP-1060) PDMS ZSM-5 zeolite filled membrane (PERVAP-1070) Poly membrane (ether-block-amide) (PEBAX-4033)	Methyl/ethyl/butyl acetate-water Methanol-water	Pervaporation	0-30 wt% organics	30
2	Poly(ether block amide) copolymer membranes	Ethyl esters/water, VOC	Pervaporation	-	31
3	PTFE, polyethylene membrane	Alcohol-water mixtures.	Conventional	40°C, pressurized by N <sub>2</sub> gas	32
4	Poly(tetrafluoroethylene) (PTFE) on ceramic support	Activated sludge	Conventional	Sludge: 6500 glucose mg/l	33
5	Silica membranes/dip coating	Gas separation	Conventional	Pressure drop: 0.5-3 bar Temperature: 100°C - 300°C	34

bility of the cake layer formed on the membrane, which could result from the hydrophobic nature of the carbon layer. A study of gas separation by a hydrophobic silica membrane was also conducted by de Vos<sup>34</sup>. The membrane shows high degrees of permeability for H<sub>2</sub>, N<sub>2</sub>, O<sub>2</sub>, CO<sub>2</sub>, and CH<sub>4</sub>, and permselectivities of 20–50 for these gases referring to SF<sub>6</sub> and larger alkanes.

### 3.2 Applications of electrically conductive membranes

For the purpose of preventing cake generation and membrane fouling, membrane filtration with an external DC electric field was studied. Huotari *et al.*<sup>35</sup> employed hollow carbon fibers as a filter as well as a cathode for separation of oily wastewater. The flux increased about 4.5 times with an electric field applied under the limited conditions of the study. However, the study suggested that using the membrane as a cathode instead of applying an electric field across the membrane caused foaming on the membrane, which is a disadvantage of flux enhancement. Several studies of electrically controlled separation of biomaterials with polymeric membranes were also reported. Kokufuta *et al.*<sup>36</sup> used poly (vinyl alcohol) and poly (acrylic acid) composite membranes with an electric field to separate maleic acid and fumaric acid, and Yamauchi *et al.*<sup>37</sup> used the same method to control protein permeation. All results showed a very positive effect on separation filtration in the presence of an electric field (2–6V) and different pH values (1–8). Bowen and Sabuni<sup>38</sup> proposed a pulsed electric field to remove deposited titanium dioxide from stainless steel microfilters. This technique offered the advantages of cleaning the membrane without interrupting the filtration process and of eliminating the need for high cross-flow velocities. A study on minimizing the accumulation of particles and colloids on the membrane surface with a cross-flow electrofiltration (CF/EF) technique was conducted by Visvanathan and Aim<sup>39</sup>. They found that CF/EF offers a better promise in reducing membrane fouling by charged colloids and particles than do other methods such as high cross-flow velocity and periodic membrane backwashing. In addition to the electrically conductive membrane

mentioned above, detailed reviews of electrofiltration (also called “electrically enhanced membrane filtration”) that cover its synthesis and applications can be found in the literature<sup>40–43</sup>.

## 4. Conclusions

The methods of fabricating carbon membrane and the applications associated with the hydrophobic and electrically conductive properties of the membrane have been reviewed. In terms of microfiltration and ultrafiltration, which are primarily applied to separation processes in the liquid phase, in situ pyrolysis, chemical vapor deposition, and fiber activation are the three major methods of fabricating carbon membranes. These membranes can be used in wastewater treatment, bioseparation, and food processes with less fouling and less formation of a cake layer. In comparing the membranes reviewed with our recently developed membrane—a carbon coated ceramic membrane exhibiting superior hydrophobicity and electrical conductivity—it was suggested that our membrane has a greater potential for separation applications that require these properties. A study of potential applications of carbon coated ceramic membrane will be conducted in the future.

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Table 6 Applications of electrically conductive membranes.

No	Membrane	Separation of mixture	Note	Ref.
1	Carbon fibers	Oily wastewater	Membrane as a cathode	35
2	Poly(vinyl alcohol)/poly(acrylic acid) membrane	Maleic acid and fumaric acid	Gel membrane	36
3	Poly (vinyl alcohol)/poly (acrylic acid) composite membrane	Protein permeation	Gel membrane	37
4	Stainless steel microfilters.	Filtration of titanium oxide	<i>In situ</i> electrochemical membrane cleaning (IEMC)	38
5	0.2 μm microfilters (cross-flow)	Wide range of particles and colloids	Anti-fouling	39

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