

# Adsorption Behavior of Activated Carbon Composite Membrane

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## 1. Introduction

In recent years, activated carbon fibers (ACFs) have drawn great attention and widely used in various fields, such as water purification and removal of toxic gases, due to their superior adsorption properties [1-3]. The adsorption properties of ACFs contain micropores in a significant proportion, and these pores are depended on the manufacturing processes. It is well known that the activation process is essential to high performance ACFs, and the activation conditions are largely considered by two different courses; the physical (or thermal) and the chemical activation, depending on the nature of the raw materials used [4-7]. During activation, the enlargement of pore diameters and development of microporous structures are therefore introduced in the carbonized carbon, resulting in increasing the extreme large specific surface areas. Also, the chemically modified functional groups of the carbon surfaces can be created during activation process, which are related to the adsorption selectivity for polar organic and inorganic adsorbates [8].

The objective of this present work is to investigate the influence of activation temperature on the surface properties, such as specific surface area, porosity, pH and acid-base values of the activated carbon composite membrane prepared from mixtures of oxidative PAN-based carbon fabrics and phenolic resin binder.

## 2. Experimental

### 2.1. Sample preparation

The samples used for preparing activated carbon composite membrane (ACCM) were manufactured via molding process with oxidized PAN-based carbon fabrics (plain-type) and phenolic resin (resole type) at a ratio of 7:3 by weight. The greenbody was heated at 125 °C to cure the phenolic resin and they were carbonized in an inert environment at 1,000 °C for 2 hours. And then they were activated by CO<sub>2</sub>-gas at a heating rate of 5 °C.min<sup>-1</sup> for 2 hours, denoted as ACCM-700, ACCM-800, ACCM-900 and ACCM-1,000 to the activation temperatures followed as 700, 800, 900 and 1,000 °C.

### 2.2. Specific surface area and pore structure

Specific surface areas and pore structures of each ACCM were measured by nitrogen gas adsorption at 77K using ASAP 2400 instrument. Prior to analysis, the sample was outgassed at 298K for 6 hours to obtain a residual pressure of less than  $10^{-3}$  torr. The amount adsorbed was converted to liquid volume by assuming a molar volume of liquid nitrogen of  $35 \text{ cm}^3 \cdot \text{mol}^{-1}$ .

### 2.3. Surface properties

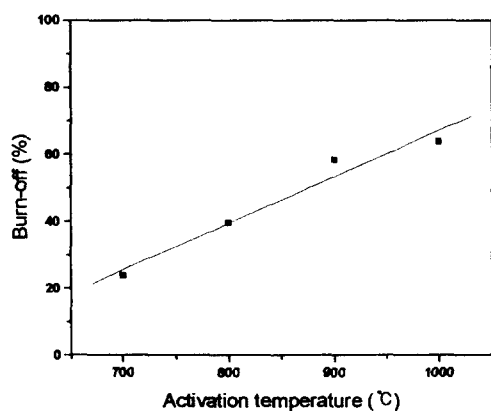
The surface pH of ACCM was measured according to the ASTM D3838. About 1.0 g of each sample was added to 20 ml distilled buffer solution (pH=7.0 at 25°C), and the mixture was shaken for 12 hours. Then the pH was measured after filtration with a nylon membrane filter (0.45  $\mu\text{m}$  in diameter).

The acid and base values on sample surfaces were determined by Boehm's method [9]. In case of acid value, about 1.0 g of sample was added to 100 ml of 0.1 N NaOH solution and shaking it for 24 hours. After filtration of the mixture with the nylon membrane filter, the mixture was then titrated with 0.1N HCl standard solution in the presence of 0.01 M phenolphthalein solution as indicator. Likewise, the base value was determined by converse titration.

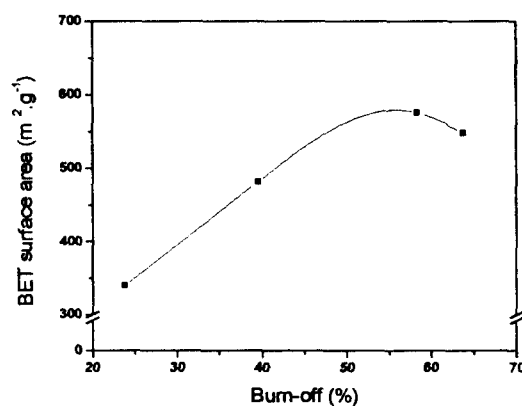
## 3. Results and Discussion

### 3.1. Surface and pore structures

Figure 1 shows the variation in burn-off of ACCM as a function of activation temperature. The weight loss is determined from a change in weight before and after activation. As expected, the burn-off increases in increasing the activation temperatures. This is explained by the fact that the weight loss of the resulting ACCM is largely influenced on the carbon

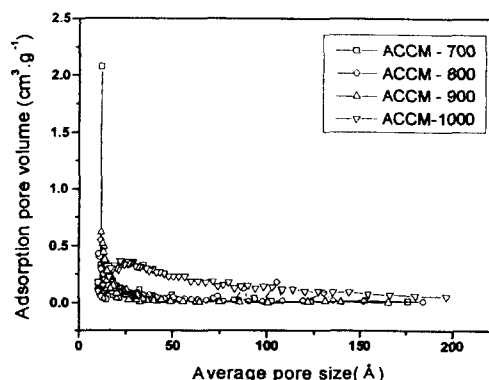


<Fig. 1> Burn-off vs. activation temperature for the ACCMs prepared by CO<sub>2</sub>-activation.



<Fig. 2> Specific surface area vs. burn-off of activated carbon composite membranes.

monoxide produced by reaction of the carbon constituent in the carbonized fiber. In Figure 2, the specific surface areas of ACCM are plotted as a function of burn-off value. As a result, ACCM carbonized at 1,000 °C is shown in the maximum specific surface areas being about 600 m<sup>2</sup>.g<sup>-1</sup>. But, a slightly marginal decrease in the specific surface area is shown. The developments of pore structure of ACCM studied with activation temperature are shown in Figure 3. In this work, the average pore diameters of the ACCM are decreased with increasing the activation temperature up to 90



<Fig. 3> Pore size distribution of ACCMs with activation temperatures.

0 °C. The observed variations of pore size distributions can be readily explained that the pore diameter substantially reduces during activation, indicating the fibers are radically consumed by a process of gasification on their external surfaces. In such a process, the pore structures are developed by gasification of the pore walls for the increases of the number of pores and the pore volume. With further burn-off, the fiber itself is consumed, thus destroying the pore structure.

### 3.2. Surface properties

The variation of surface properties for ACCM with different activation temperatures is shown in Table 1. The results of the Boehm's titration show that the surface natures of ACCM-700 and ACCM-800 are amphoteric with slight acidic properties, whereas ACCM-900 and ACCM-1,000 being to basic properties, respectively.

That is probably due to the existence of the chemically modified surface functional groups by the activation temperatures. As regard, when the activation temperature is lower than 80 °C, carbon-oxygen surface complexes are less stable and develop acid surface functional groups which are hydrophilic in character. On the other hand, the ACCM activated at higher than 900 °C develops basic surface functional groups due to surface carbon-oxygen

Table 1. Surface properties of ACCMs studied

Samples	pH	Acid value (mequiv.g <sup>-1</sup> )	Base value (mequiv.g <sup>-1</sup> )
ACCM-700	6.8	180	90
ACCM-800	6.9	135	70
ACCM-900	7.2	80	55
ACCM-1,000	7.4	65	50

resonance structure which is more stable and forms the electron pair donor structure. Also, these results indicate that the acid-base values are largely depended on pH of the ACCM studied.

#### 4. Conclusions

The evolutions of pore size, pore volume and specific surface area of ACCM can be observed in increasing the burn-off with activation temperature. Among them, the ACCM-900 produces a well-developed pore structure from the adsorption characteristics. And, the pore structures are developed by gasification of the pore walls of the resin, resulting in increasing the micropore volumes. Also, the activation temperature influences the surface nature of ACCM in the system studied.

#### 5. References

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