# Manufacturing of Activated Carbon Using Furfural Residue as Raw Material

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In a series of study, furfural residue that is a kind of waste byproduct was first utilized for manufacturing activated carbon, by self-activation. Pore characteristics and specific surface area of the resulting activated carbon was examined. It is noted that the self-activation with water content contributed mainly to the development of macropore volumes. This shows good trend for adsorption of sulfur dioxide in exhaust gas and other flue gas.

As a very important raw material to produce furan and its derivatives in chemical industries, furfural in China is commonly produced from corncobs, a kind of cheap agricultural byproduct, by acidic hydrolysis with sulfuric acid or hydrochloric acid. However, furfural residue was simultaneously generated during this production. It is mostly discarded except that its small portion is used for heating purpose.

In order to utilize this furfural residue, and eliminate environmental pollution as well, we have proposed a recycling system in which activated carbon was manufactured from the residue by self-activation method, sulfur dioxide in exhaust gas or other flue gas was adsorbed and concentrated by the activated carbon developed, sulfuric acid was then turned out from the concentrated sulfur dioxide, and used for the acid hydrolysis process. Thus the activated carbon used was also recovered for recycling use.

Here in this paper, the preparation of activated carbon from furfural residue by the self-activation method was tried, and pore volume as well as specific surface area of the activated carbon obtained was examined. At present, it seems that only one report on this study has been published by Wang et al[1]. in which phosphoric acid was used as an activator for the production of activated carbon.

A furfural plant in Liaoning Province of China supplied furfural residue used in this study. Proximate analysis showed that the residue had 73.5% of volatile content; 15.1% of fixed carbon; and 11.4% of ash content, respectively. This fixed carbon value is equal to that of coconut shell. However, the ash content is about 5 times that of coconut shell, so it was predicted that these inorganic substances would have some kind of effect on activated carbon to be prepared. Besides, pH of furfural residue was highly acidic or about 2.8.

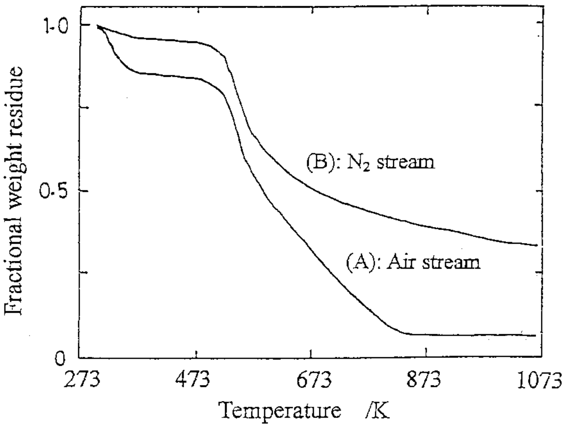

**Figure 1.** Thermal decomposition of furfural residue in $N_2$ and air streams at programmed temperature of 5 K/min.

Figure 1 shows the thermal decomposition curves of furfural residue in the air and under nitrogen flow, measured by TG8120 Thermal Balance (Rigaku Co.). The weight loss of furfural residue around 373 K in the air flow atmosphere is resulted from the evaporation of its water content, while that at a temperature range of 523–573 K is due to the evaporation of its resin content. From 573 K up to about 823 K the oxidative combustion of lignin, cellulose and others occurred. Nevertheless, even in the nitrogen flow atmosphere, a modest weight loss in the same temperature range was observed. It is attributed to the removal of water, the formation of carbon monoxide and carbon dioxide, and the escape of tar content due to the thermal decomposition of cellulose, lignin and others. Summing up the above, it is expected that if furfural residue is decomposed by heating at a temperature of above 873 K in the nitrogen flow atmosphere, high-quality carbonaceous material would be obtained.

For the carbonization of furfural residue, an external-heating rotary electric furnace was put into use as the case reported[2]. The carbonization of furfural residue was started at a temperature rising rate of 5 K/min and then a given temperature was kept for one hour. The temperature for carbonization was changed from 773 to 1273 K. Activated carbon was manufactured by the self-activation method in which the enrichment of water in furfural residue was used as the activation agent.

Pore volume and specific surface area of the activated carbon were determined by adsorption of nitrogen at 77 K. An automated adsorption apparatus (Omnisorp Model 100cx, Beckman Coulter Co.) was employed for these measurements. Then, by analyzing the adsorption isotherm data by BET method, specific surface area of the activated carbon was determined. The micropore volume of the activated carbon was calculated based on T-plot method[3] while its mesopore volume depended upon BJH method[4]. Hence, the macropore volume was obtained by subtracting the micropore and mesopore volumes from the total pore volume. The adsorption capacity of the activated carbon was evaluated by the Iodine Adsorption Amount Measuring Method, according to JISK-1474 Activated Carbon Testing Method.

Figure 2 reveals the relationship between the change in carbonization temperature and the specific surface area of obtained carbonized substance when the water content of furfural residue is 71.5 weight percent. It was found that specific surface area increases with the carbonization temperature to a maximum at

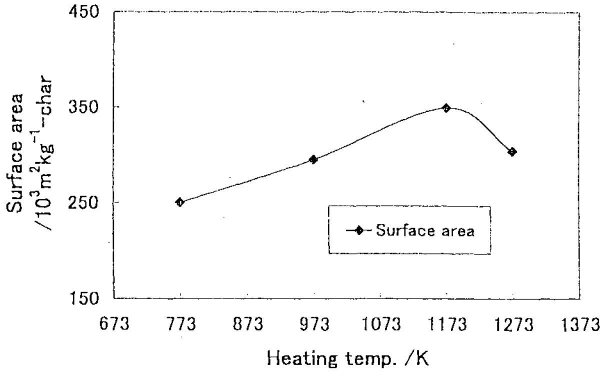

**Figure 2.** Relationship between surface area and heating temperature (heating rate of 5 K/min, heating time 1 h, water content of 71.5 wt%).

1173 K and then begins to decrease. The corresponding maximum of specific surface area was 350 × $10^3$ $m^2$/kg, which was almost equal to that of carbonaceous materials produced from wood pieces[5] or walnut shell[6].

Next, it was examined whether furfural residue would be activated by itself with its water content at a carbonization temperature of 1173 K. It was found that both the specific surface area and the amount of iodine adsorption increased with the water content. For instance, when the water content was 200 weight percent, the specific surface area reached the maximum value of 478 × $10^3$ $m^2$/kg, which was 1.2 times the specific surface area of 380 × $10^3$ $m^2$/kg when the water content was 20 weight percent. In addition, regarding the iodine adsorption amount, the similar tendency was observed. This is thought to have been due to the fact that the water content in furfural residue caused carbonization and the steam activation at the same time, namely, the self-activation took place.

Figure 3 indicates the relationship between the changes in the structure of micropore and other various pore volumes owing to this self-activation. It is noted that water content in furfural residue contributed markedly to the volume development of macropore. As shown in Figure 4, it was apparent from the shape of peak of pore distribution curve for the resulting activated carbon that when the water content was 200 weight percent, the pore distribution curve widened and the activated carbon had more macrospores. This agrees with the result in Figure 3. Additionally, pH value of the obtained carbon was 9.12–9.69 or alkaline regardless of the change in water content. The analysis by X-ray diffraction exhibited that the resulting activated carbon contained K, Ca, and Na.

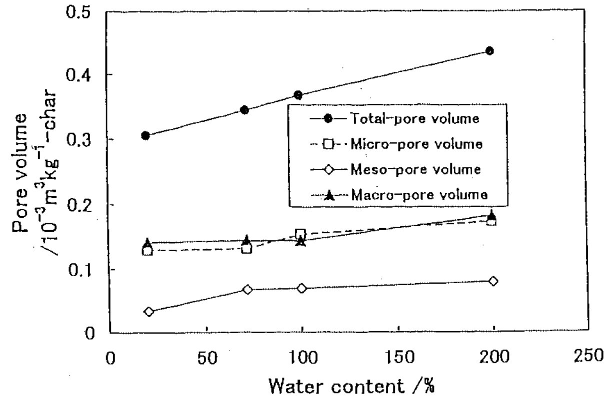

**Figure 3.** Relationship between pore volume and water content at 1173 K.

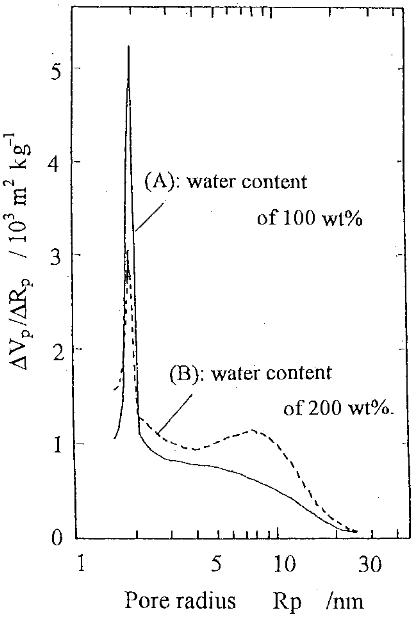

**Figure 4.** Pore-size distribution of the char prepared from furfural residue with various water content at 1173 K

From these results, it became apparent that furfural residue caused the self-activation with the water contained in it when its carbonization took place. At its carbonization temperature of 1173 K and when the water content in furfural residue was 200 weight percent, activated carbon whose yield for the raw residue, specific surface area, and iodine adsorption amount were 25.8%, 478 × $10^3$ $m^2$/kg, and 690 kg/kg, respectively, was obtained. Although furfural residue as raw material was highly acidic, activated carbon obtained was alkaline. About this matter, more study in detail needs to be carried out in future. So when furfural residue is converted to activated carbon by the self-activation method, some type of activated carbon catalyst containing alkali metal may be found.

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