

Preparation of the activated carbon for the canister from cokes

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Introduction

Activated carbons are the microporous carbonaceous adsorbents which are prepared from carbon-containing source materials such as wood, coal, lignite, petroleum and sometimes synthetic high polymers.[1-2] Activated carbons shows an ability to adsorb hydrocarbons of the gas phase. Activated carbons are used in the purification of many kinds of gas phases like hexane, benzene, toluene, gasoline, phenol etc.[3] In this study, cokes from bituminous coal were activated for the purpose of preparing the activated carbons by steam activation. The effect of the activation temperature, time, steam concentration and flow rate on the *n*-butane adsorption, burn off, surface area and average pore size of the activated carbons, were investigated. The adsorption characteristics of the activated carbons for gasoline are indirectly estimated by *n*-butane adsorption.

Experiment

The cokes obtained from bituminous coal were used in this study. The result of the elemental analysis is summarized in Table 1. Cokes were activated in a tube furnace. Steams with concentration of 25~75%, flow rate of 1~3 l/min, was supplied to the tube furnace through gas mixing apparatus which was preheated at 450°C. Activation was performed for 10~250 minutes at the temperature between 800~1000°C. Adsorption of *n*-butane on activated carbon was evaluated by the weight change after adsorption and desorption at 25°C. The flow rate of *n*-butane was 105ml/min in adsorption, and that of air was 230ml/min in desorption. Adsorption and desorption were kept for 20 minutes respectively. Effective adsorption(W_E) was determined by the following

$$W_E = (M_1 - M_2) / V \times 100$$

where M_1 is the weight of the sample after adsorption, M_2 is the weight of the sample after desorption, V is volume of the samples.

Table 1 Element Analysis of the Cokes

Ash	C	H	N	S
2.8%	86.25%	1.89%	2.80%	0.19%

Results and discussion

Activation processes of cokes are carried out by using oxidizing gases, such as steam at above 800°C or carbon dioxide at higher temperatures. Most of commercially available activated carbons are prepared by steam activation. Oxidation of carbons by steams is an endothermic reaction. The oxidation, therefore, is affected by activation temperature and time. The steam concentration in carrier gas and the gas flow rate through the reactor is important, too.

Fig. 1 shows burn offs of the cokes which were activated at various temperature and time. At the beginning of activation, the rapid increment of burn off was thought to be due to the volatile matters which were still remained in the cokes after carbonization. It can be seen that burn offs of the cokes were considerably influenced by the activation temperature.

The adsorption isotherms of nitrogen on activated carbon obtained at various temperatures and times were presented in Fig. 2. The shape of adsorption isotherms corresponded to the characteristic type I shape. Although there were no great difference in specific volumes with relative pressure, it could be seen that adsorption was mainly performed by micropores. It was observed that the increase in activation time gave high specific volumes, which was thought to be due to increase in micropores.

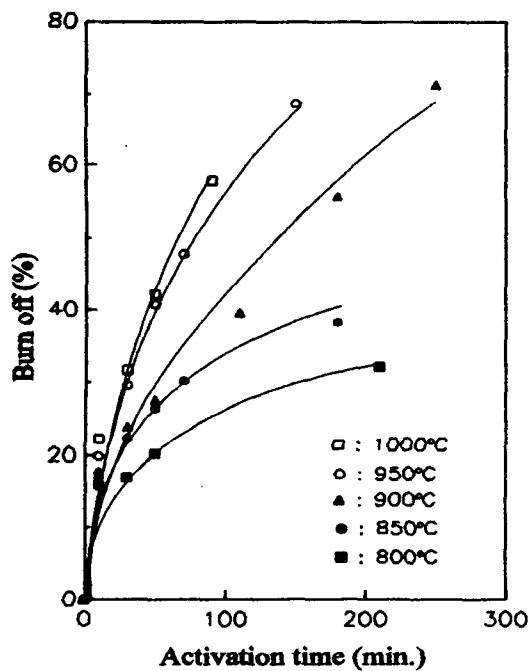


Fig.1 Burn offs of activated carbons obtained from cokes

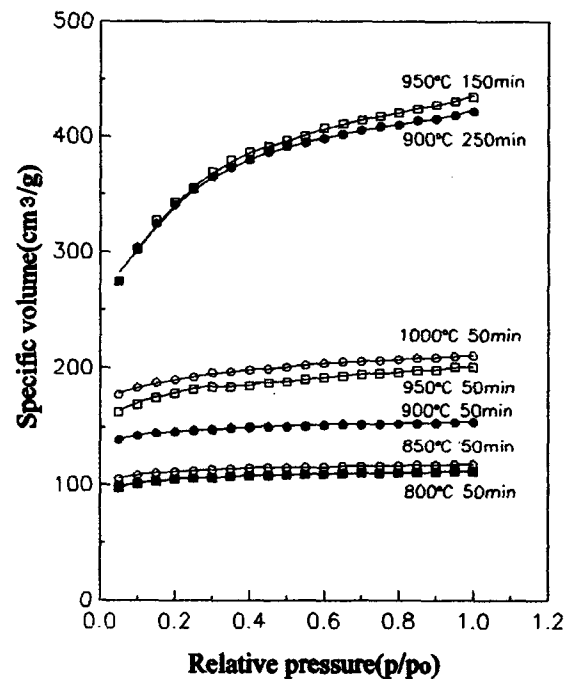
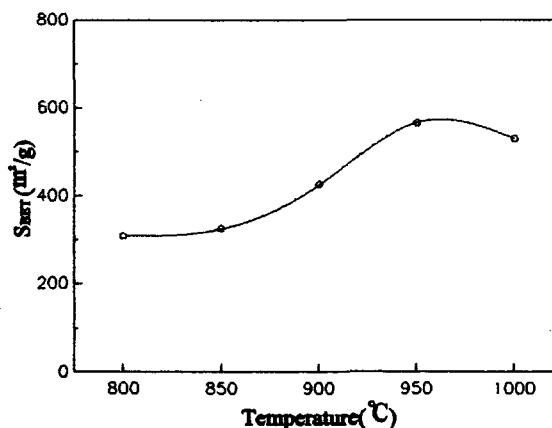


Fig.2 Adsorption isotherms of N₂ at 77K on the activated carbons obtained from cokes

Table 2 Adsorption characteristics for the activated carbons

Activation Temp. (50min)	BET Surface area(m ² /g) by nitrogen	Total Pore Volume (cc/g)	Average Pore Size(Å)
800°C	309	0.16	10.2
850°C	326	0.17	10.3
900°C	426	0.22	10.4
950°C	566	0.28	10.0
1000°C	530	0.26	9.8
900°C (250min.)	1,097	0.47	8.5
950°C (150min.)	1,116	0.47	8.4

**Fig. 3 Specific surface area of activated carbons with activation temperature**

The total volumes of micropores, BET surface area of the carbons obtained by the activation of 30 minutes, are shown in Table 2. It was known that micropores and mesopores were well developed in the activated carbon obtained at 600~900°C and micropores were homogeneously developed in the carbons obtained at above 1000°C.[4] Table 2 and Fig. 2 shows that average pore size below 900°C increase with elevated activation temperatures and decrease above 950°C, which means that micropores were developed at higher temperature. However, Fig. 3 and Table 2 show that specific surface area and total pore volumes decreased at above 950°C. These were thought that exhaustion of carbons by oxydation was very serious at higher temperature above 950°C. In table 2, it can be seen that adsorption properties are strongly depended on activation time as well as activation temperature. Higher specific surface area and total pore volume were obtained by prolonged activation time at 900°C and 950°C. And average pore size progressively decreased, which was expected to give improved adsorption ability of activated carbons on *n*-butane.

In Fig. 4, *n*-Butane adsorption of activated carbons is plotted versus activation temperature and time. It was observed that the amount of *n*-butane adsorbed increased considerably with activation time and reached maximum value at 950°C and decreased in the carbons obtained at 1000°C, as expected in table 2. Fig. 5 and 6 show the amounts of *n*-butane adsorbed with steam concentrations and steam flow rates during activation of 950°C 90 minutes. It can be seen that the steam concentration and steam flow rate greatly affected on the *n*-butane adsorption properties, and that almost constant above 2l/min. of steam flow rate, 50% of steam concentration.

Although there was a large difference in the amounts of *n*-butane adsorbed with activation

temperature and time, there was not a sharp difference in average pore sizes of the activated carbons, as shown in Table 2. This indicates that the amount of *n*-butane adsorbed is directly influenced by surface area and total pore volume rather than by average pore size. The surface area of the activated carbons which is related to the burn offs, needed to be increased in order to increase the amount of *n*-butane adsorbed.

The relationship of the amount of *n*-butane adsorbed and burn off for all the samples prepared in this study, is illustrated in Fig. 7. It is interesting that without regard to activation temperature and time, the amount of *n*-butane adsorbed is directly proportional to the burn off.

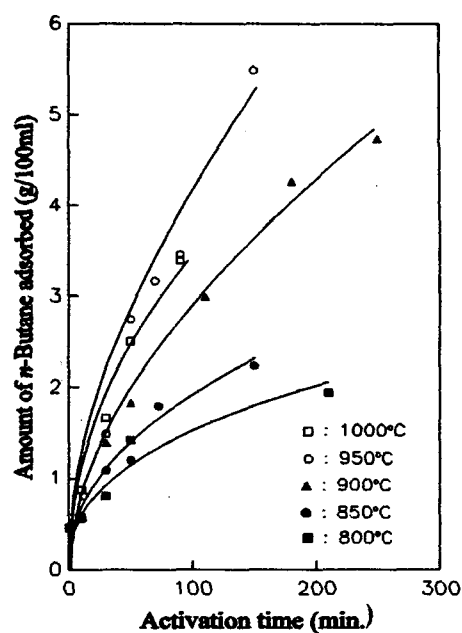


Fig. 4 *n*-Butane adsorption of the activated carbons obtained from cokes

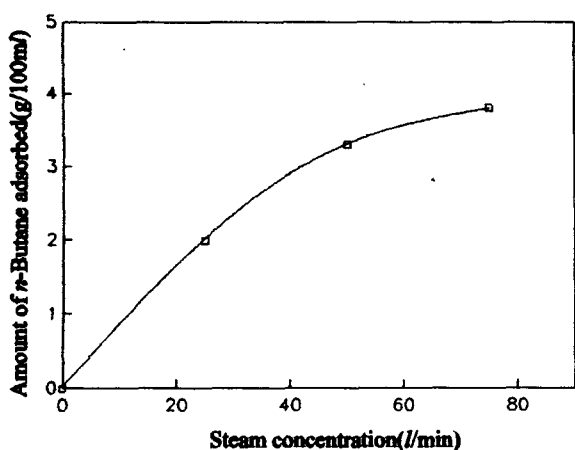


Fig. 5 *n*-Butane adsorption of the activated carbons with steam concentration

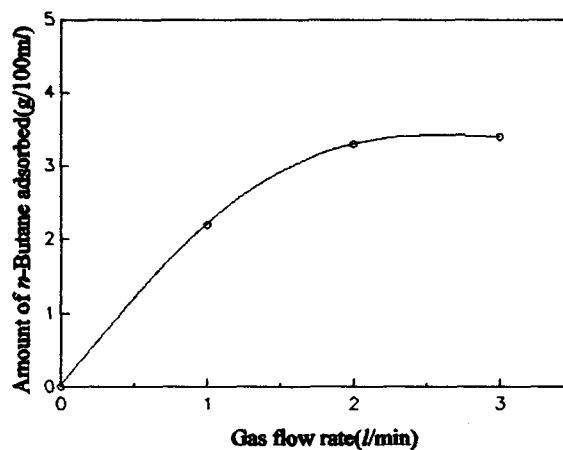


Fig. 6 *n*-Butane adsorption of the activated carbons with gas flow rate

Conclusion

n-Butane adsorption and surface area of the activated carbons obtained by steam activation from cokes, gradually increased up to 950°C, but decreased above 950°C. The amount of *n*-butane adsorbed on the activated carbons was directly related to the burn off.

Reference

1. Motoyuki Suzuki, Adsorption engineering, p8, published by KODANSHA Tokyo, 1990
2. Robert M. Clark, Benjamin W. Lykins, Jr., Granular Activated Carbon, p1-p31, published by Lewis Publishers, 1989
3. Kirk-Othmer, Encyclopedia of Chemical Technology, Vol. 3, p561, published by Wiley Interscience, 1978
4. Ermolenko, I., Morozova, A. A., Fridman, L. I., Savel'ev, G. G., Stas, N. F. and Gorina, T. S., Vesti Akd. Navuk, BSSR, Ser. Khim. Navuk, 5, 20(1975)

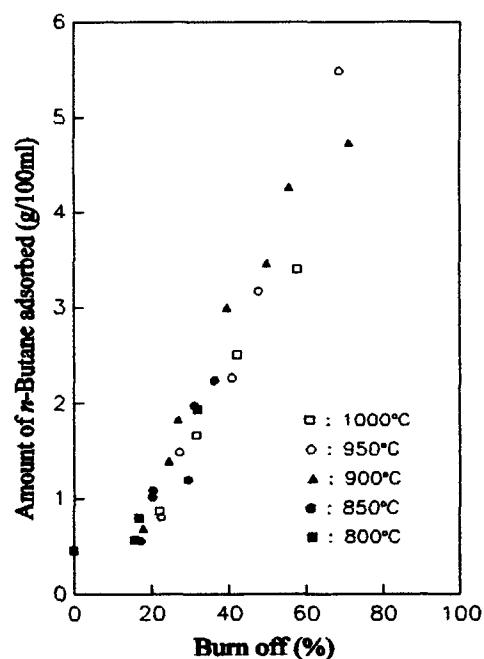


Fig. 7 The relationship of burn off and *n*-Butane adsorption for the activated carbon from cokes