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Influence of Pretreatment and Activation Conditions in the Preparation of Activated Carbons from Anthracite

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Abstract–The influence of pretreatment and activation conditions on anthracite activation was investigated. Separation of low ash coals by using dense media was conducted to obtain appropriate raw materials for activation. Activated carbons were produced from crushed and granule coals by physical activation (steam or CO_2) and physical activation with chemical pretreatment in mild and strong conditions. Microporous activated carbons having a surface area of 900 m^2/g were produced by steam activation from granules with 60% burn-off for 3 hrs of activation. Chemical pretreatment at the strong condition increased the surface area by 30% as compared with non-treated activated carbons. Chemical pretreatment, in general, affected activation degree, so pore volume increased by 20% and burn-off increased remarkably at the identical activation conditions. CO_2 activation was proven to be an effective method for producing microporous activated carbons having an average pore diameter of 20 Å.

Key words: Anthracite, Anthracite Activation, Chemical Pretreatment, Alkali Pretreatment

INTRODUCTION

Anthracite, which is the highest rank coal among the four coal ranks from peat to anthracite, is further divided into three sub-ranks: semi-anthracite, anthracite, and meta-anthracite. Most of the Korean anthracite usually belongs to the anthracite and meta-anthracite rank, and has characteristics of low calorific value (<5,000 kcal/kg), low reactivity, high ash contents (>30%), and high ignition temperature (>550 °C) [Lee et al., 1994].

There are large amounts of anthracite deposits in many sites in the world, so anthracite accumulation is a worldwide problem at present because of its limited applicability as a fuel. Many researchers have worked on the combustion of blended coal with low rank coals such as bituminous coal and combustion in a fluidized bed [Choi et al., 1985; Jung and Park, 1988].

Recently, there has been increased interest in non-fuel uses of anthracites as summarized in Table 1, in which four main fields of anthracite uses are introduced. Graphite, carbon riser and carbon black may be possible uses of anthracites, but adsorbents such as activated carbons, as indicated in Table 1, are the most promising among the other possible uses for anthracite. Although the use of high-rank coals like anthracites as raw material to produce activated carbons is not very common because of their low reactivity, their fine pore structure and molecular sieve properties can be used for production of micro-porous activated carbons [Patel et al., 1972].

Many papers have been published for producing activated carbons by using anthracites as raw material and for developing molecular sieve properties. Gergova et al. [1993, 1995] have studied one step pyrolysis-activation of anthracites with steam to produce high surface area activated carbons with well developed porous structure. Preoxidation of anthracites with air or HNO_3 was introduced by Serrano-Talavera et al. [1997] who concluded that the more intense the preoxidation treatment is, the higher is the porosity of the resulting chars. Microporosity development by CO_2 activation was studied by Mittelmeijer-Hazeleger and Martine-Martinez [1992], and the mechanism of CO_2 adsorption in the micro-pores of activated anthracite was studied by Martine-Martinez et al. [1995]. To obtain activated carbons with good adsorptive properties by CO_2 activation of anthracites, very long activation time was required due to their low reactivity [Mittelmeijer-Hazeleger and Martine-Martinez, 1992].

Table 1. Non-fuel uses possible for anthracites

Raw materials for electrodes, anodes & specialty graphites
Carbon riser for steel making in electric furnaces and BOF furnaces
Filler material for black polymers
Development of advanced filters
Activated carbons
Molecular sieve carbons
Purification of water, organic chemicals, etc.
Pollution control

Chemical pretreatment with alkali hydroxides and chlorinated compounds was studied to increase the reactivity of anthracites [Daulan et al., 1998; Illán-Gomez et al., 1996; Walker and Almagro, 1995; Lyubchik et al., 1997]. The effect of perchloric acid pretreatment was introduced by Daulan et al. [1998] who concluded that the main effect of chemical pretreatment is to reduce the activation time. Illán-Gomez et al. [1996] studied the effects of alkali hydroxides on the activation of Spanish anthracite and found that the surface area increased to 1,700 m^2/g with alkali to coal ratio of

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2 at 700 °C. This process used molten alkali to activate and carbonize coals with evolution of large amounts of hydrogen. Chemical activation with molten alkali and changes in properties of raw materials have been introduced in the activation of coal and petroleum cokes [Otowa et al., 1993; Lee and Choi, 2000].

In this research work, Korean anthracite was activated. The effect of pretreatment conditions, such as air oxidation and alkali pretreatment and various activation conditions on the pore characteristics, were investigated.

EXPERIMENTAL

1. Raw Materials and Ash Control

Coal samples from Jangsung coal mine were used as raw material and their analyses are listed in Table 2. High ash contents (35%), low calorific value (about 5,000 kcal/kg), and low volatile matters (5%) are major characteristics of the coal samples. Carbon content and volatile matter in dry mineral matter free basis indicate that the coal samples are classified in an anthracite rank, on a relatively lower part of the rank among the Korean anthracite. Low sulfur and nitrogen contents in ultimate analysis are noticed.

Inorganic matters are known to have a negative effect on activation due to their inert property, although the minerals in coal itself do not have any effect on the pore development [Muñoz-Guillena, 1992]. Although major components of ash in coal samples are silica and alumina as shown in Table 2, iron and potassium contents are also comparatively large. To obtain suitable raw materials for activation, ash must be controlled as low as possible. Dense media separation by using $ZnCl_2$ as a medium was conducted. Specific gravity of solution was changed by varying $ZnCl_2$ concentration in the solution, and the float/sink fraction at each specific gravity was separated and analyzed.

Granule coals were made by mixing pulverized coal (under 200 mesh) and coal tar pitches at a coal to pitch ratio of 4. Resultant granule size is about $\phi 0.3\times 5$ mm. Granule coals were carbonized before activation so carbonized tar pitches should be coated on the pulverized coal surfaces to bridge the coal particles and to harden the granules, and the remaining tar pitches could be volatilized.

Table 2. Chemical analysis of Jangsung coal

Proximate (air dry, wt%)	
Moisture	2.5
Volatile matter	5.0
Fixed carbon	57.5
Ash	35.0
Ultimate (dry ash free, wt%)	
C	90.2
H	1.7
O	5.5
N	0.16
S	0.78
Ash analysis (wt%)	
SiO_2	52.1
Al_2O_3	31.5
TiO_2	1.46
P_2O_5	0.23
Fe_2O_3	4.41
CaO	1.0
MgO	0.89
Na_2O	0.17
K_2O	3.61
BaO	0.09
SrO	0.03
Calorific value (kcal/g)	4,800-5,200

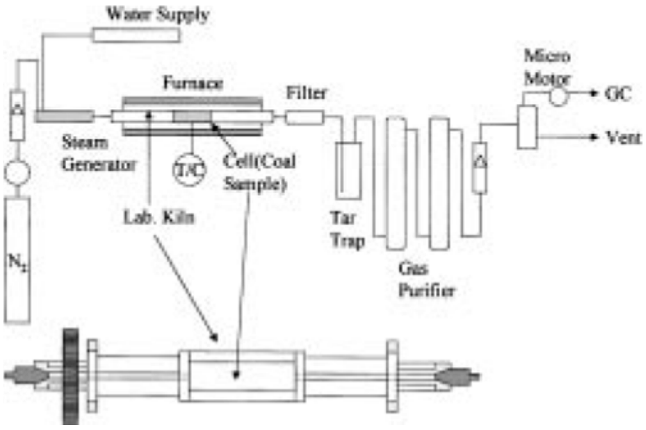

Fig. 1. Experimental apparatus with activation cell.

2. Pretreatment

For air oxidation, low ash coal samples were oxidized with air at 500 °C for 2 hrs in an air flow of 300 cc/min. In alkali pretreatment, coal samples were mixed with 2.5 M alkali solution to make a coal to alkali ratio of 1. After the mixtures had been maintained at 25 °C for 24 hrs stirring at 250 rpm during pretreatment process (denoted as mild condition) or treated in an autoclave at 250 °C and 6 MPa for 2 hrs (denoted as strong condition), the mixtures were filtered and washed with distilled water several times and dried in an oven for 24 hrs. The strong condition was very similar to the condition used in the BHC (Battel Hydrothermal Coal cleaning [Khoury, 1981]) process by which most of the coal ashes and 80% of organic sulfurs could be removed.

3. Activation

Activation was conducted with an activation cell (inner volume 75 cm^3, SUS 310S) specially designed to rotate during the experiment. The experimental apparatus with the activation cell is illustrated in Fig. 1. The activation cell containing coal samples (10-20 g) was placed in a tubular electrical furnace and heated by temperature controller to the activation temperature at a fixed heating rate of 50 °C/min in nitrogen flow of 50 cc/min. Then the activation gas (steam or CO_2) was introduced through an induction tube. The temperature of the induction tube was maintained at 250 °C by heating tape, and steam was produced in a horizontal glass column maintained at 250 °C by introducing water through a peristaltic pump at a rate of 0.15-1.0 cc/min. The flow rate of CO_2 was 250 cc/min in CO_2 activation.

Activation temperature was varied from 850 °C to 950 °C, and the activation time was from 0.5 to 10 hrs. After activation was completed, the products were cooled, weighed and stored in a desiccator. Surface area and pore characteristics were determined by N_2 adsorption at 77 °K (Micromeritics ASAP 2400 analyser). Morphological changes were observed by a Scanning Electron Micro-

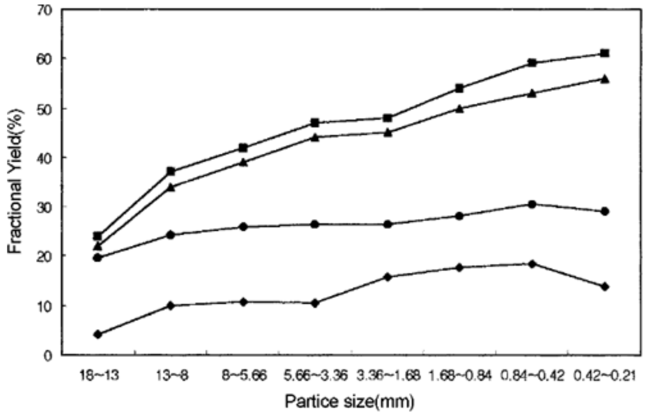

Fig. 2. Float fraction vs. particle size with variations of media specific gravity together with size distribution of raw coal (◆; raw coal, specific gravity ●; 1.8, ▲; 1.9, ■; 2.0).

scope (Philips XL-30).

RESULTS AND DISCUSSION

1. Ash Control

Results of dense media separation are illustrated in Figs. 2 and 3. Fig. 2 shows the relationship between the float fractional yields and particle size ranges at each specific gravity, and also the particle size distribution of raw coals. Fig. 3 shows ash contents of float fractions at each corresponding point in Fig. 2. It was found from Fig. 2 and Fig. 3 that the fractional yields and ash contents increased with specific gravity, and that the fractional yields increased with particle size decrease, while the ash contents had a minimum at the particle size range of 0.84-1.68 mm.

The fractions of 0.42-3.36 mm size ranges that have relatively higher fractional yields and lower ash contents were selected as raw material for the activation study. Float and sink fractional yields and ash contents with specific gravity at these size ranges are shown in Table 3. The cumulative float fraction was about 50% and its ash content was about 8% at the specific gravity of 1.85. At a specific gravity of 2.0, the differential ash content of float fraction was very high as 76%.

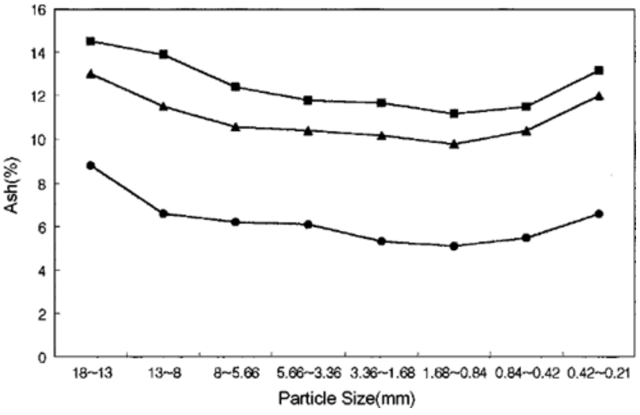

Fig. 3. Ash in float fraction vs. particle size with variations of media specific gravity (specific gravity ●; 1.8, ▲; 1.9, ■; 2.0).

Table 3. Results of heavy media separation with particle size ranges of 0.42-3.36 mm

Specific gravity	Float fraction (%)/Ash(%)		Sink fraction (%)
	Differential	Cumulative	
1.8	29.72/5.8	29.72/5.8	70.28
1.85	17.97/12.6	47.69/8.4	52.31
1.9	8.06/20.7	55.75/10.2	44.25
2.0	39.08/75.8	94.83/37.2	5.17

Table 4. Ultimate analysis of heavy media separated coal

Specific gravity	C	H	N	S	O
1.8	85.43	1.31	0.46	0.078	6.9
1.85	79.7	1.28	0.43	0.021	5.97
1.90	73.3	1.28	0.46	0.022	4.2
2.0	18.1	0.88	0.65	0.97	3.6

Ultimate analyses of differential float fraction at each specific gravity are presented in Table 4. Carbon content of float fraction at the specific gravity 1.85 was about 80%. Differential float fractions at the specific gravity 2.0 had sulfur content of 0.97% that was clearly discriminated from the other specific gravity.

2. Activation

Activation results are listed in Table 5. Series of activated anthracites were classified as four different groups as shown in the first column of Table 5. Activated carbons denoted as A-P were made from crushed coal by physical activation (steam or CO_2), and denoted as A-CP were made from crushed coals by physical activation with chemical pretreatment. AG-P and AG-CP were activated carbons from granule coals with same activation conditions with A-P and A-CP. Chemical pretreatment indicates the mild conditions if no special mention is given.

Table 5 shows activation results with various conditions such as activation temperature, activation time, activation medium and pretreatment conditions. It was found that the activated carbons from crushed coals (A-P, A-CP) had lower surface areas than those from the granule coals (AG-P, AG-CP) at the same activation conditions and burn-off. This difference might come from the differences of particle size. Granules were made from powdered coal sized under 200 mesh, so activation with granules had increased contact efficiency resulting in higher activation degree.

Air oxidation proved to be an effective method for increasing the surface area by 25% accompanied with an increase in burn-off as shown in the second row of Table 5. CO_2 activation with crushed coal showed little effect on the development of surface area within the experimental times of 5 hrs. However, it was so effective for granule coals that the surface area increased to 470 m^2/g for 5 hrs and further increased to 800 m^2/g for 10 hrs of activation.

The relationship between surface area and burn-off with different activation temperature is illustrated in Fig. 4. It was found that the surface area increased with burn-off due to enlargement of pores and that the surface area and burn-off increased with temperature, but marginal differences were observed above 900 °C. Surface area increased to about 900 m^2/g at the 60% burn-off, beyond which no further increase could be observed. Because of the relative increase in ash contents of activated carbons due to unexpected blow-off of

Table 5. Results of anthracite activation with various activation conditions

Series activated anthracite	Temp. (°C)	Activation time (hr)	Steam (cc/hr)	Burn-off (%)	Surface area (m^2/g)	Remarks
A-P	850	5	60	46.7	553.2	Air oxidation
		5	52	59.4	600.2	
	900	3	15.2	43	634.3	CO_2 activation
		3.5	40.6	-	719.7	
		5	-	12.6	168.4	
	950	3	33	45.3	706.9	
A-CP	850	5	51.9	42.5	650.7	NaOH
		5	39	47.0	688.1	KOH
	900	3	30	48.7	840.5	NaOH
						Strong Condition
	950	3	30	79.3	585.0	NaOH
AG-P	850	3	26	21.3	430.0	
		5	38	44.6	534.0	
		5	46	56.3	863.2	
	900	1.3	15	47	776.4	
		2	42	51	813.0	
		2.5	28	49	810.0	
		3	45.7	56	891.0	
		3	8.8	35.8	768.8	
	950	1	46	44	737.9	
		1.5	33.3	52.5	840.4	
		5	-	26	469.62	CO_2 activation
		10	-	51.4	799.27	CO_2 activation
AG-CP	850	3.5	62.8	45	813.2	NaOH
		4	52.5	50	800.7	KOH
		5	41	59	864	KOH
	900	2	51	54	709.4	NaOH
		2.75	67.3	92	-	NaOH
		3	36	73.4	812.6	KOH

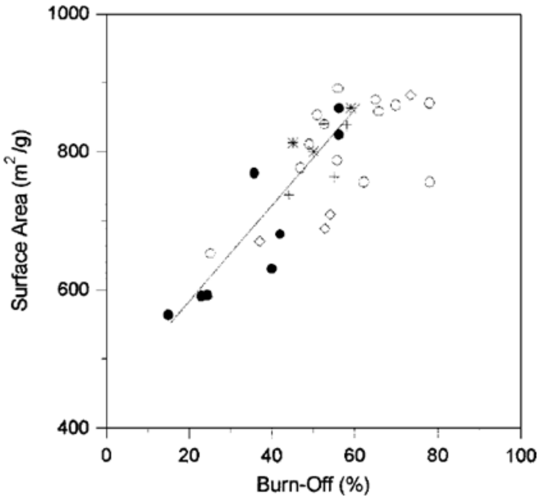

Fig. 4. Surface area vs. burn-off [●; 850, ※; 850 (chemical pretreatment), ○; 900, ◇; 900 (chemical pretreatment), +; 950].

carbons by excessive gasification at high burn-off, activation to the high burn-off above 60% could not produce high surface area activated carbons.

It was found from Table 5 that the chemical pretreatment with alkali hydroxides at the mild condition increased burn-off at high temperatures above 900 °C. This means that the activation time decreased with chemical pre-treatment at the identical activation condition as mentioned by Daulan et al. [1998]. Dualan et al. [1998] studied the chemical modification of anthracite by $HClO_4$ and concluded that the main effect of chemical pretreatment was to reduce the time of activation, and this reduction could be attributed to the creation of numerous channels in the anthracite macrotexture during chemical pretreatment.

KOH was proven to be more effective for coal activation than NaOH. Effectiveness of KOH was already introduced by Ehrburger et al. [1986] who showed that much more gas was evolved from coal pretreated with KOH than with NaOH during carbonization accompanied by more development of active site.

Chemical pretreatment at the strong condition had much stronger effect on the surface area development of crushed coals, so surface area increased about 30% as compared with physical activation. This is mainly due to the surface modification by alkali at the strong treatment condition as already mentioned by Senkan and Fuller [1979] and Lee and Shon [1997]. Senkan and Fuller [1979] studied NaOH effect on the surface properties of coal and showed that the water sorption and immersion heat into liquid water in-

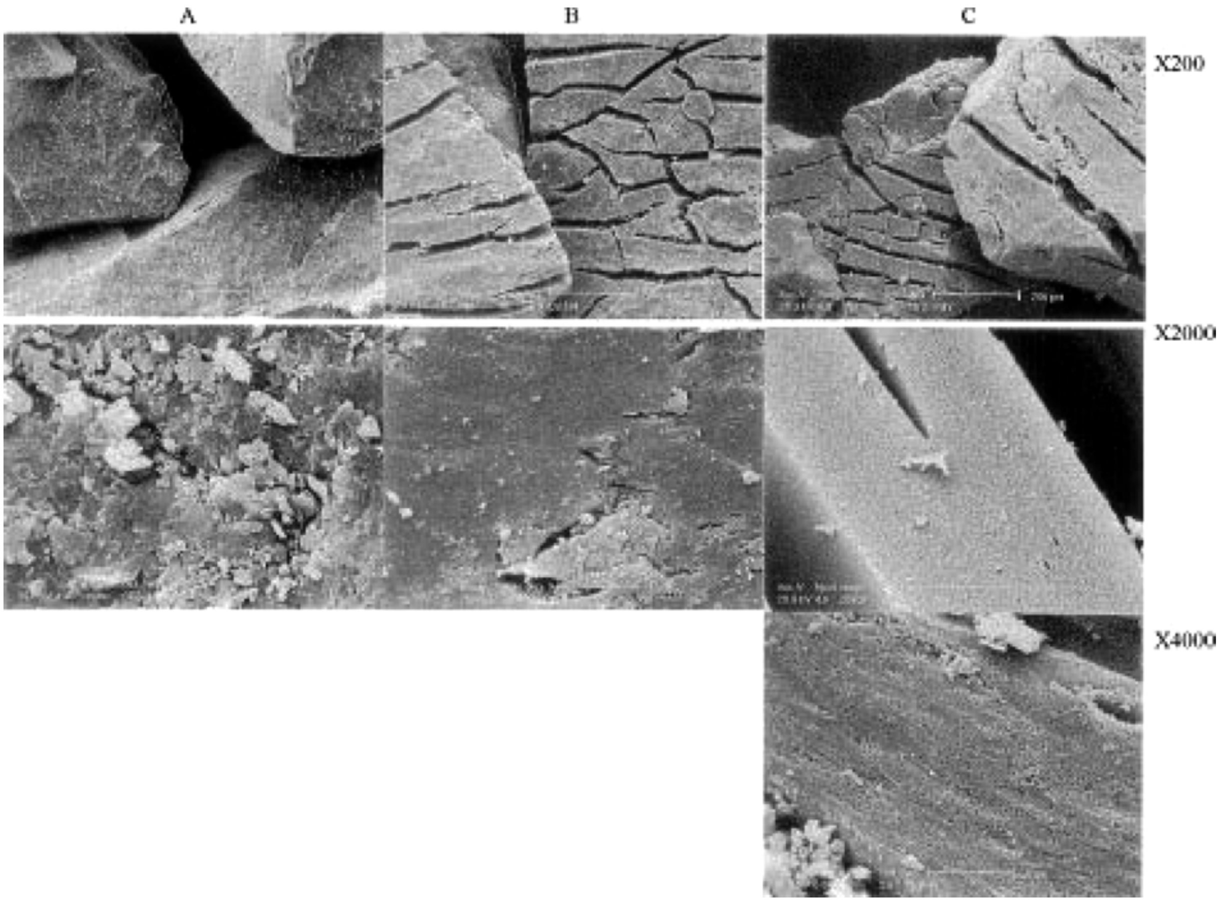

Fig. 5. SEM views of raw coal and activated anthracite [A column; raw coal, B column; physical activation, C column; physical activation with chemical pretreatment (strong condition)].

creased considerably after treatment with NaOH. Changes in the structure of coal were also introduced by Lee and Shon [1997] who worked with anthracite refining by alkali, and concluded that the physical changes such as the formation of defects were dominant factors for the increase of reactivity.

SEM views of the typical activated carbons are shown in Fig. 5. The surface of activated carbons by physical activation with steam shows little change compared with raw coals and is cleaner (×2,000) than that of raw coals due to the blow-off of reactive materials by gasification. Exfoliation of particle surface after activation is due to the thermal shock. Activated carbons with chemical pretreatment at the strong condition, however, show remarkable change as shown in Fig. 5. While no porous structure could be detected at the surface of activated carbons produced by physical activation, the surfaces of activated carbons by chemical pretreatment had many pits on the surface and had cleaner surfaces. The pits on the surface might come from the activation of initial defects formed by the alkali pretreatment and acted as an initial active site for activation.

3. Pore Characteristics

N_2 adsorption and desorption isotherms of five representative activated carbons are illustrated in Fig. 6. Five different types of activated carbons are the granule type activated carbons produced by steam activation (AG-P) and same ones with chemical pretreatment (AG-CP, mild conditions), granule type activated carbons by CO_2 activation (AG-P, CO_2), and crushed type activated carbons by steam activation (A-P) and same ones with chemical pretreatment (A-CP, strong conditions).

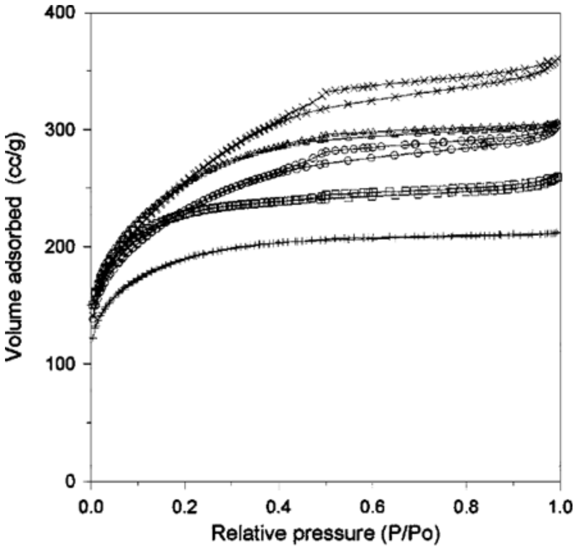

Fig. 6. N_2 adsorption-desorption isotherms (77 K) for five representative activated carbons (crushed, +; steam activation, ∧; chemical pretreatment, granule ○; steam activation, ×; chemical pretreatment, □; CO_2 activation).

Granule type activated carbons by steam activation and with chemical pretreatment (mild condition) have isotherms close to the BDT type II isotherms, and have hysteresis at a relative pressure of 0.5. Hysteresis is a typical characteristic of a wide range of porous adsorbents that usually have a broad distribution of pore sizes and shapes, and which introduces capillary condensation [Adamson, 1982]. Slopes to the relative pressure of 0.4 were comparatively high for these activated carbons, which means that the large portions of mesopores are involved.

Granule type activated carbons by CO_2 activation have different type of isotherm, closer to the type I isotherm, compared with activated carbons by steam, and its shape is rather close to that of the crushed activated carbons, that is, low volume increasing with relative pressure and small hysteresis. CO_2 activation of anthracite has been known to produce activated carbons with well developed microporosity, whereas meso- and macroporosity are poorly developed [Mittelmeijer-Hazeleger and Martine-Martinez, 1992].

Crushed type activated carbons by steam activation show a very sharp knee at low relative pressure and no marked increase further with increased pressure, which means that highly microporous characteristics are developed. These types of isotherms are referred to as type I, and zeolite-like adsorbents usually have these types of isotherms. Although it was found that the crushed type activated carbons by steam activation have a little lower adsorbed volume than the granule type activated carbons as shown in Fig. 6, their micropore characteristics may have selectivity for the special adsorption application. Chemical pretreatment of crushed coals at the strong condition increased the pore volume by 50% as can be seen in Fig. 6, and included larger portions of mesopore as inferred from the increased slope to the relative pressure 0.5. This means that the chemical pretreatment enlarges the pore.

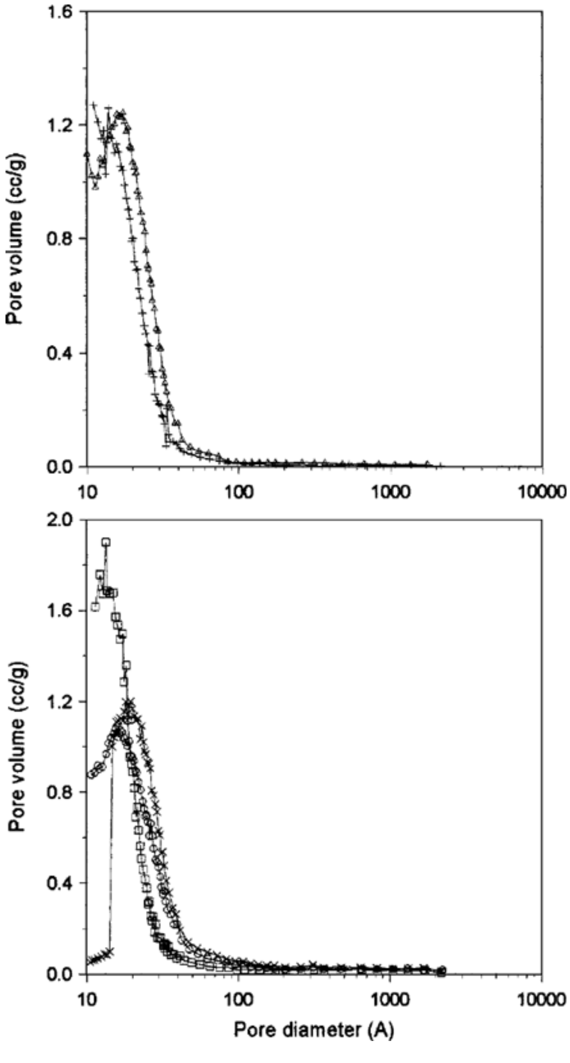

Fig. 7. Pore size distribution of activated carbons (upper part, crushed, +; steam activation, △; chemical pretreatment, lower part, granule ○; steam activation, ×; chemical pretreatment, □; CO_2 activation).

Table 6. Pore characteristics of activated carbons

AC Type	Surface area (m^2/g)	Average pore diameter (Å)
AG-P	768	24.88
AG-CP (mild)	800	27.94
AG-P (CO_2)	799	20.04
A-P	634	20.14
A-CP (strong)	840	22.56

Pore size distributions of the activated carbons are illustrated in Fig. 7, and the surface area and average pore diameter of these activated carbons are listed in Table 6. As implied in Fig. 6, granule type activated carbons by CO_2 activation show larger adsorbed volumes at the small pore diameter than the other granule type activated carbons as can be seen in the lower part of Fig. 7. Activated carbons by CO_2 activation had average pore diameter of about 20 Å; that is the smallest among the produced activated carbons. Activated carbons from granule coals have larger pore diameter than those from the crushed coals and the chemical pretreatment increases pore diameter for both of the coals (crushed and granules) as shown in Table 6.

CONCLUSIONS

We investigated the influence of pretreatment and activation conditions on the activation of Korean anthracite. Low ash coal samples were prepared by dense media separation of raw coals with $ZnCl_2$ as a dense medium. Float fraction of particle size ranges of 0.42-3.36 mm at the specific gravity 1.85 was selected as raw material. Activated carbons were produced from crushed and granule type coals by physical activation (steam or CO_2) and physical activation with chemical pretreatment with mild and strong conditions. It was found that the activated carbons from crushed coals had a little lower surface area than the activated carbons from granular coal at the same activation conditions and burn-off, but had well developed microporous characteristics. Air oxidation was proven to have the effect of increasing the surface area by 25%. Microporous activated carbons having a surface area of 900 m^2/g were produced by steam activation from granule type coals with 60% burn-off at 900 °C for 3 hrs of activation. Chemical pretreatment at the strong conditions increased surface area by 30% compared with non-treated activated carbons. Chemical pretreatment, in general, affected the activation degree so that the pore volume and burn-off increased at the identical activation conditions. CO_2 activation was proven to be an effective method for producing microporous activated carbons having a pore diameter of 20 Å.

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