

## OXIDIZED COAL CHAR AS A CATALYST: I. CHARACTERIZATION

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### ABSTRACT

Oxidizing the surface of carbonaceous materials at 500°C to 700°C in air was shown in the 1960s (1-3) to produce an excellent dehydrohalogenation catalyst. One goal of the current research was to prepare this catalyst from an activated mild gasification coal char having good sorptive properties, test its activity as a catalyst for other elimination reactions, and characterize the catalyst and its precursors. The pore surface area (nitrogen adsorption/desorption, BET) of a mild gasification (MG) coal char was raised from <5 to 475-580 m<sup>2</sup>/g by steam gasification at 870°C and the catalyst properties were imparted by controlled oxidation with 10% O<sub>2</sub>/90% N<sub>2</sub> at 450°C. This material catalyzed not only dehydrochlorination but also dehydration and deamination reactions. Samples of the original coal and final oxidized activated char, as well as two intermediate samples (MG char and steam activated MG char) were fully characterized using Proximate and Ultimate Analysis, Inductively Coupled Plasma Analysis, X-Ray Diffraction, Nitrogen (BET) Surface Area, X-ray Photoelectron Spectroscopy, Diffuse Reflectance IR Spectroscopy, and Scanning Tunneling Microscopy.

### INTRODUCTION

Gasification of coal under mild conditions produces char, coal-derived liquids and some gases. Economic viability of this approach to coal utilization is unquestionably dependent on the marketability of the char. To have a reasonable impact on coal utilization via mild gasification, any market considered for the char must be large and should have a projected price greater than coal's fuel value. The activated carbon market has the required price structure, more than \$1000/ton for a high quality product, and a potentially large volume. Activated carbon is used not only for air and water purification but also for cleanup of nonaqueous solutions or suspensions. More than 300 chlorinated organic compounds have been identified in chlorinated potable waters, cooling waters, and sanitary effluents. Systems are needed which will trap and destroy unwanted organic compounds, both those occurring in industrial streams and those that are introduced into drinking water by chlorination.

Carbonaceous materials were converted in the 1960s to effective dehydrochlorination catalysts by heating material, like char, to 500°C to 700°C in air. Some of these catalysts produced linear olefins from chlorinated n-alkanes with little rearrangement of the carbon skeleton. Other dehydrochlorination catalysts studied later were also effective for producing olefins from alcohols and amines by elimination of water and ammonia, respectively (4-8). Oxidized activated char might have the capacity not only to adsorb waste molecules from a variety of streams, but also to catalyze chemical transformations of the unwanted adsorbed compounds into more manageable or even useful chemicals. This work was undertaken to better characterize and define the properties and methods for making the oxidized char catalyst.

### MATERIALS AND METHODS

**Char preparation and activation** - The mild gasification char was prepared in a pilot-scale, fixed-bed reactor at United Coal Co. (UCC), Bristol, Virginia, from a Herrin (Illinois No. 6) coal, IBC-103 in the Illinois Basin Coal Sample Program. This char was from a previous study on the burning behavior of partially devolatilized coal as a function of the amount of volatile matter (VM) in the char (9). Stepwise grinding and sieving of a composite of material having 11% and 15% VM produced 13.5 pounds of 10x30 mesh

particles, a size comparable to commercial granular activated carbon. This UCC char composite was subjected to additional pyrolysis in a 2.5-cm ID, batch, fluidized-bed reactor (FBR) (200-gram increments, N<sub>2</sub> gas, heated to 500°C at 20 C°/min) to drive off most of the VM. The pyrolyzed product is designated MG Char.

The FBR was used both for improving the porosity by steam gasification and for activating the material as a catalyst by oxidation with 10% O<sub>2</sub>/90% N<sub>2</sub>. In a typical steam gasification run, the MG char was heated in nitrogen to 870°C at a rate of 20 C°/min, and the fluidizing gas was switched to a 50:50 steam-nitrogen mixture. Fourteen steam-treatment runs, designated SA01 through SA14, were made. The SA01 and SA02 were used to determine the conditions for the final oxidation. Composites of SA03 through SA09 and SA10 through SA14 are designated SA3-9 and SA10-14, respectively. The catalyst surface was produced in the same reactor by heating batches in nitrogen to 450°C, and switching to 10% O<sub>2</sub>/90% N<sub>2</sub> for 15 minutes. The gas was switched back to N<sub>2</sub> while cooling to room temperature in runs SA01 through SA09 but the flow of 10% O<sub>2</sub>/90% N<sub>2</sub> was continued for SA10 through SA14. Composites from SA3-9 and SA10-14 are designated OSA3-9 and OSA10-14, respectively.

**Testing as a catalyst** - A 2.5 ml quantity of redistilled Wiley 3-chloro-3-methylpentane was heated 5 minutes at reflux temperature (120°C) in a 25 ml tube fitted with a micro reflux condenser before adding 0.25 g of the activated char to be tested. The temperature was maintained for another 10 minutes before the tube was removed from the heater. The liquid was separated from the char by filtration through a 10 ml gas-tight syringe connected to a filtration apparatus. The olefins in the filtrate were then analyzed using a Perkin-Elmer 8500 GC for a preliminary determination. A Hewlett Packard 5988A GC/MS (HP GC/MS) with a fused silica capillary column and a split (ratio 1:100) injection system was used for a molecular level identification. The preceding procedure was also used to dehydrate 2,3-dimethyl-2-pentanol.

Preliminary test data indicated that there was no catalytic activity exhibited by OSA3-9 for the deamination of t-butylamine or cyclooctylamine at a temperature lower than 220°C and a residence time of <10 minutes. Evidence that deamination requires a higher temperature than dehydrochlorination with other catalysts was found in the literature (5). Tests at higher temperatures were carried out in a Perkin-Elmer 8500 Gas Chromatograph equipped with a hot wire detector and liquid nitrogen trap. Helium was used as GC carrier gas. The GC injector glass liner was packed with 0.1 g of oxidized char and used as a catalytic column. The GC injector temperature was set at 295°C, the temperature found by Lycourghiotis to work. A 6' x 1/8" packed column was used as an analytical column for product separation. Deamination products eluted from the GC analytical column were collected at the exit of the detector at liquid nitrogen temperature. The expected olefins were identified on the HP GC/MS.

**Surface Areas** - Total surface area, micropore area and volume, and cumulative pore volume were determined from nitrogen adsorption data obtained at 77 K using a Micromeritics (ASAP 2400) apparatus. Adsorption isotherms were analyzed using the BET equation to obtain total surface area. Micropore area and volume were obtained from t-plots using the Halsey equation (10). Adsorption/desorption isotherms were analyzed using the BJH method (11) to calculate cumulative pore volumes.

## RESULTS AND DISCUSSION

**Catalyst activity.** The oxidized char (OSA3-9 and OSA10-14) catalyzed the production of the expected olefins (identified by GC/MS) from 3-methyl-3-chloropentane at its boiling point, 2,3-dimethyl-2-pentanol at about the same temperature (120°C), and cyclooctylamine at 295°C. The methods used showed some differences in catalytic activity among char samples made by methods not described here, but better analytical procedures will be required to quantify the differences in catalytic activity.

**Proximate and Ultimate analyses** - Proximate analyses (Perkin Elmer TGA 7 thermogravimetric analyzer), and Ultimate analyses (Leco CHN-600 and Leco SC-132) appear in Table 1. There are significant differences among the samples. While the percent VM decreased from about 51% to 2.5% from coal to OSA char on a dry-ash-free basis (not shown in the table), the ash and fixed carbon

contents increased. The carbon content increased from a low of 80%, on a dry-ash-free basis, for the original coal to a high of 95% for the OSA char sample and, at the same time, the hydrogen decreased tenfold. The atomic H/C ratio was decreased from 0.87 to 0.28 by mild gasification and from 0.28 to the minimum of 0.07 when the MG char was steam treated (SA3-9 or SA10-14).

As expected, as the coal is pyrolyzed and gasified, it loses oxygen functional groups. Therefore, when IBC-103 was pyrolyzed to 500°C, it lost over 40% of its oxygen (from 9.27 to 5.24%, direct analysis) and then when this char (MG char) was steam gasified at 870°C, it lost another 60% to 75% of its oxygen content (down to 1.94% and 1.23% for SA3-9 and SA10-14). When this steam treated char was oxidized in air, to make it a catalyst, its oxygen content increased significantly (up to 3.32% and 3.33%, respectively). The cooling in the presence of 10% O<sub>2</sub>/90% N<sub>2</sub> for OSA10-14 did not appear to increase the oxygen over that of OSA3-9.

**Inductively Coupled Plasma (ICP) analysis** - ICP spectroscopy determines the inorganic elements present in the bulk of a sample. Only the elements commonly present in the coal were determined. The data are shown in Table 2. As expected, the concentration of all metallic elements was increased as the original coal (IBC-103) was pyrolyzed to produce the MG char and even more as the char was steam treated to obtain SA3-9 or SA10-14.

**X-Ray Diffraction (XRD)** - All samples were found to be mostly amorphous and showed a graphite-like structure. Quartz (SiO<sub>2</sub>) was the major crystalline phase in all samples. XRD data, like the data from ICP analysis, indicate that significant amounts of oxides of Si, Fe, Al and Ti are present in the oxidized chars (OSA3-9 and OSA10-14). In addition to phase and crystallinity information, XRD analyses provided some important correlations between the small-angle X-ray scattering (SAXS) intensity and microstructure of the samples. The SAXS intensity for the SA char was approximately 480 cps, and 550 cps for the OSA char samples OSA3-9 and OSA10-14, respectively. The conclusion one can reach solely on the basis of these two data sets is that the microporous structure in the OSA char is more fully developed than that for SA char. This is in contrast to the data on micropore volume, cumulative pore volume and average pore size radius data obtained from N<sub>2</sub> gas adsorption/desorption (BET) surface areas which indicated no significant differences between SA chars and the OSA chars.

**Nitrogen (BET) surface area** - Char activation has two purposes. One is to develop adsorption capacity, as estimated by pore surface area, and the other is to give the char the type of oxidized surface that catalyzes elimination reactions. Thermogravimetric analysis (TGA) was used for preliminary assessment of two methods for increasing surface area: 1) oxidation with 10% O<sub>2</sub>/90% N<sub>2</sub> at 450°C and 2) steam gasification at 870°C. Because steam gasification generated higher nitrogen BET surface areas, steam was used. Adsorption/desorption data with N<sub>2</sub> at 77°K and the Brunauer, Emmett, and Teller (BET) equation were used to calculate pore surface areas and the micropore surface areas (Table 3). Due to the small surface area, the micropore area, micropore volume, cumulative pore volume and average pore radius were not measured for the original coal (IBC-103) and MG Char.

Steam activation raised the N<sub>2</sub> surface area from <5 m<sup>2</sup>/g in the mild gasification char (MG char) to 460 m<sup>2</sup>/g at 41% weight loss and to a high of 625 m<sup>2</sup>/g at 54% weight loss as measured at the ISGS. The average weight losses and pore surface areas generated were 51% and 542 m<sup>2</sup>/g for the 7 batches mixed to produce SA3-9 and 54% and 575 m<sup>2</sup>/g for the composite of 5 batches designated SA10-14. Amoco's values for these composites, 473 m<sup>2</sup>/g and 585 m<sup>2</sup>/g, respectively, confirmed order but showed a greater difference. The oxidized steam-activated (OSA) char having catalyst properties was made by oxidation at 450°C with 10% O<sub>2</sub>/90% N<sub>2</sub> for 15 minutes. The pore surface areas of the catalyst composites, OSA3-9 and OSA10-14, were about equal, 557 m<sup>2</sup>/g and 552 m<sup>2</sup>/g, respectively, despite the difference in the pore surface areas and micropore surface areas of their precursors (473 m<sup>2</sup>/g and 187 m<sup>2</sup>/g for SA3-9 and 584 m<sup>2</sup>/g and 230 m<sup>2</sup>/g for SA10-14). The micropore surface area of the catalyst which during preparation was cooled in 10% O<sub>2</sub>/90% N<sub>2</sub> to leave more oxygen on the surface (OSA10-14) had a lower micropore surface area than the one (OSA3-9) cooled in nitrogen (207 m<sup>2</sup>/g and 242, respectively).

Clearly the steam treatment and air oxidation steps opened up some very fine micropore structures in the MG char. This fine microstructure could account for the greatly increased surface area of the steam treated and final activated chars.

**X-ray Photoelectron Spectroscopy (XPS)** - Elemental surface composition (relative weight percent) and a summary of their binding energies (BE), as determined by XPS, are shown in Tables 4 and 5, respectively. Table 4 shows the concentration of several elements, including oxygen, on the surface of the samples which is typical for these types of materials. Binding energy data, Table 5, indicate the presence of hydrocarbon species such as C-C and C-H ( $284.6 \pm 0.2$  eV), oxidized carbon species such as C-O and C=O ( $286.2 \pm 0.2$  eV), and carbonate/carboxylic species O-C=O ( $289 \pm 0.6$  eV). The binding energy of 163.6 eV ( $\pm 0.6$  eV) is typical for elemental sulfur and/or organic sulfur species such as mercaptans (R-SH), disulfides (R-SS-R) and thiophenes, and the binding energy of 168.0 eV ( $\pm 0.3$  eV) is typical for species such as sulfonates (R-SO<sub>3</sub>), alkylsulfonates (RO-SO-OR) and dialkylsulfates (RO-SO<sub>2</sub>-OR). The nitrogen binding energies suggest the presence of amines (RNH<sub>2</sub>) including alkylamines, aromatic (phenyl) amines and pyridine derivatives at 398.6 eV ( $\pm 0.6$  eV), pyrolic nitrogen species at 400.4 eV ( $\pm 0.3$  eV), and ammonium derivatives (RNH<sub>3</sub><sup>+</sup>) at 402.0 eV ( $\pm 0.4$  eV) with one unidentifiable species at about 404.0 eV. However, numerous nitrogen-containing organic compounds (i.e. nitrobenzene and dinitrobenzene) fall into the binding energy range of 404-405 eV ( $\pm 5$  eV). The presence of binding energies at approximately 711.5 eV ( $\pm 0.5$  eV) for Fe, 103.0 eV ( $\pm 0.1$  eV) for Si, and 75.0 eV ( $\pm 0.6$  eV) for Al is most likely due to the respective oxides.

Based on the data in Table 5, the chemical species concentrated on the surfaces of the catalytic variety of char differ from those not oxidized. For example, while the concentration of hydrocarbon species such as C-C and C-H ( $284.6 \pm 0.2$  eV) on the surfaces of steam activated chars (SA3-9 and SA10-14) were 75% and 65%, respectively, the concentration of the same species on the surfaces of the air oxidized chars (OSA3-9 and OSA10-14) were 66% and 60%. On the other hand, the concentration of oxidized carbon species such as C-O and C=O ( $286.2 \pm 0.2$  eV) on the surfaces of steam treated chars were 16% and 23%, respectively, and on the surfaces of air oxidized chars were 23% and 30%. The concentration of carbonate/carboxylic species O-C=O ( $289 \pm 0.6$  eV) remained constant at about 10% for both steam treated and air oxidized chars.

The surface binding energies, Table 5, obtained from the XPS studies also suggest that the surface concentration of oxidized carbon species (C-O and C=O) is lower for OSA3-9 (23%) than for OSA10-14 char (30%), the sample that was cooled in 10% O<sub>2</sub>/90% N<sub>2</sub> atmosphere rather than N<sub>2</sub>. While four types of nitrogen species were identified on the surface of OSA3-9, only two were identified on the surface of OSA10-14, and this too may be the result of the cooling regime.

**Diffuse Reflectance IR Spectroscopy** - Spectra were obtained on a Mattson Cygnus 100 FTIR using a Harrick Diffuse Reflectance accessory. All spectra were acquired by co-adding 1024 scans at a resolution of 8 cm<sup>-1</sup>. Powdered KBr was used as a background. Bands elucidated by second derivative treatment are broadly assigned as: 1867 cm<sup>-1</sup> and 1809 cm<sup>-1</sup>, C=O, anhydride, probably cyclic and unconjugated; 1740 cm<sup>-1</sup>, C=O, ester; 1707 cm<sup>-1</sup>, C=O, possibly ketone, aldehyde, or COOH; 1655 cm<sup>-1</sup>, C=O, highly conjugated, Ar-(C=O)-Ar; and 1613 cm<sup>-1</sup>, highly conjugated, hydrogen bonded C=O. Mild gasification of the IBC-103 resulted in a higher ratio of aromatic C-H functional groups to aliphatic C-H functional groups which is consistent with a graphitization process. The pyrolysis process resulted in modest band shifts, which may be due to differences in electronegativity or level of conjugation in the substituents bonded to the various carbonyl groups. The spectrum obtained from the air oxidized char (OSA3-9) had features that were very faint and broad. A band centered near 1590 cm<sup>-1</sup> suggests the same aromatic character along with carboxylic acid salts. The former assignment is consistent with the broad feature centered near 3150 cm<sup>-1</sup> encompassing the aromatic C-H stretch region. A band near 1590 cm<sup>-1</sup> along with a very broad feature centered near 1170 cm<sup>-1</sup> may also be indicative of coordinated inorganic carbonate, which is consistent with the XPS results.

**Scanning Tunneling Microscopy (STM)** - STM and the newer technique for imaging and resolving surface detail at atomic and near atomic level, atomic force microscopy (AFM), are becoming increasingly

important in studies of molecules deposited on the surfaces of catalysts. The original coal sample (IBC-103) could not be imaged by STM, most likely because of the low intrinsic conductivity of the material. Chars had sufficient conductivity. Images were obtained with 1-2 nA tunneling current and 750 mV-1750 mV positive bias on the tip and sample at ground which is tunneling from occupied  $\pi$  states of the carbon to the probe tip. Images from the oxidized char (OSA3-9) suggest aggregated particles as large as several thousands of Angstroms which clearly are aggregates of still smaller particles. Particles were usually made of an aggregated state of several 200-400 Å particles. Increasing magnification indicated a further heterogeneity with smaller aggregates of size in the order of tens of Angstroms. At even higher magnification (less than 10 nm) an apparent lattice imaging became evident. The partially ordered region seen in this image was in the order 50x70 Å with a very poor registry. A well-ordered domain of size 20x40 Å was also seen at this magnification.

### CONCLUSIONS

The oxidized, steam-activated (OSA) char catalyzed dehydration of an aliphatic tertiary alcohol and deamination of a primary aliphatic amine, in addition to dehydrochlorination of an alkyl halide. PS data indicated a significant amount of oxygen was in the surface of the OSA char. The ICP and XRD data indicated the presence of significant amounts of Si, Fe, Al and Ti oxides. In addition to phase and crystallinity information, XRD data provided an important correlation between the small angle X-ray scattering (SAXS) intensity and the microstructure of the samples. The strong presence of SAXS at low angles in OSA char is evidence that OSA char is a microporous material, and a comparison of intensity measurements suggests that the microporous structure becomes more fully developed during the oxidation of steam-activated (SA) char. XPS data suggest the chemical species concentrated on the surfaces of the chars are indeed different and these differences are expected to be related to variations in catalyst reactivity among samples. The surface binding energies from XPS work, and IR data supported the speculation that the cooling under  $N_2$  atmosphere after the oxidation step causes the removal of some of the CO and C-O complexes.

Both IR and  $^{13}C$  NMR data confirm that the ratio of aromatic C-H groups to aliphatic C-H groups increased significantly when volatile matter was removed during mild gasification. An oxidation index is defined as the ratio of the integrated area of the carbonyl region (1635-2000  $cm^{-1}$ ) to the integrated area of the region associated with the aliphatic and aromatic C-H stretches (2675-3135  $cm^{-1}$ ). The calculated oxidation index was about 0.48 and 1.09 for the coal and MG char, respectively. This index may prove useful in characterizing mild gasification chars and correlating final pore development with thermal histories.

In general, STM imaging of the samples revealed a highly aggregated hierarchical structure. A very fine sheet-like microstructure could be seen in steam-activated and oxidized steam-activated chars. STM data clearly support the BET data that the steam-activation opened up a very fine micropore structure on the originally small, several hundred Angstrom, features of MG char. Certainly, this fine microstructure could account for the greatly increased active surface area of the steam-activated char. The OSA chars have been shown to have good sorption properties and results will be reported in a subsequent publication.

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## REFERENCES

1. Kruse, C.W.; Ray, G.C., U.S. Patent 3,240,834, March 15, 1966.
2. Mahan, J.E., Reusser, R.E.; Kruse, C.W., U.S. Patent 3,352,935, Nov. 14, 1967.
3. Kruse, C.W., U.S. Patent 3,437,695, April 8, 1969.
4. Lycourghiotis, A. *React. Kinet. Catal. Lett.* **1976**, *5*(4), 453-7.
5. Lycourghiotis, A.; Vattis, D.; Katsonos, N.A.; *Z. Phys. Chem. (Wiesbaden)*, **1981**, *126*(2), 259-67.
6. Suarez, A.R.; Mazzieri, M.R. *J. Org. Chem.* **1987**, *52*(6), 1145-7.
7. Mochida, I.; Watanabe, H.; Uchino, A.; Fujitsu, H.; Takeshita, K.; Furuno, M.; Sakura, T.; Nakajima, H. *J. Mol. Catal.* **1981**, *12*(3), 359-64.
8. Misono, M. *J. Catal.* **1973**, *30*(2), 226-34.
9. Rostam-Abadi, M.; DeBarr, J.A.; Chen, W.T. *Prepr. Pap.-Am. Chem. Soc., Div. Fuel Chem.* **1989**, *35*(4), 1264-71.
10. Halsey, G.D. *J. Chem. Phys.* **1948**, *16*, 931.
11. Barrett, E.P.; Joyner, L.G.; Halenda, P.P. *J. Am. Chem. Soc.* **1951**, *75*, 373-380.

Table 1. Proximate and Ultimate Analyses

	IBC-103 Coal	MG Char	SA3-9 Char	OSA3-9 Char	SA10-14 Char	OSA10-14 Char
<u>Proximate Analysis (wt%, as-received)</u>						
Moisture	3.35	1.01	2.28	0.93	2.01	1.62
Ash	5.92	10.42	19.81	21.81	21.43	24.58
V. Matter	49.2	16.02	12.36	2.44	11.38	2.72
Fixed C	41.53	72.55	65.55	74.82	63.18	71.08
<u>Proximate Analysis (wt%, dry)</u>						
Ash	6.12	10.53	20.27	22.02	21.87	24.98
V. Matter	50.91	16.18	12.65	2.46	11.61	2.76
Fixed C	42.97	73.29	67.08	75.52	66.52	72.26
<u>Ultimate Analysis (wt%, as-received)</u>						
%C	71.82	80.06	69.61	72.14	69.82	70.48
%H	5.63	1.98	0.65	0.53	0.67	0.58
%N	1.69	2.22	1.58	1.47	1.43	1.37
%S	2.2	1.84	1.23	1.21	1.19	1.24
%O <sup>a</sup>	11.39	5.54	3.54	3.39	2.73	3.9
<u>Ultimate Analysis (wt%, dry)</u>						
%C	79.16	90.39	89.35	93.37	91.2	95.5
%H	5.79	2.11	0.51	0.55	0.58	0.54
%N	1.86	2.51	2.03	1.9	1.87	1.86
%S	2.42	2.08	1.58	1.57	1.55	1.68
%O <sup>b</sup>	10.77	2.91	6.53	2.61	4.8	0.42
%O <sup>a</sup>	9.27	5.24	1.94	3.32	1.23	3.33
H/C	0.87	0.28	0.07	0.07	0.07	0.07

<sup>a</sup>Oxygen values were obtained by direct analysis.

<sup>b</sup>Oxygen values were obtained by difference (100 - (%C + %H + %N + %S) = %O).

Table 2. Metal Composition (Wt.%) Data from ICP Analyses

Sample I.D.	IBC-103 Coal	MG Char	SA3-9 Char	OSA3-9 Char	SA10-14 Char	OSA10-14 Char
Fe	1.04	1.59	4.1	2.85	3.14	3.27
Si	0.67	1.31	2.13	4.30	4.80	4.3
Al	0.74	1.38	2.72	2.45	2.95	2.92
Na	0.30	0.04	0.11	0.10	0.08	0.08
K	0.14	0.25	0.5	0.46	0.46	0.45
Ca	0.07	0.14	0.26	0.27	0.27	0.21
Mg	0.04	0.06	0.13	0.13	0.16	0.14
Ti	0.04	0.06	0.12	0.16	0.16	0.16

Table 3. Nitrogen Surface Area Data (Adsorption/Desorption)

	IBC-103 Coal	MG Char	SA3-9 Char	OSA3-9 Char	SA10-14 Char	OSA10-14 Char
N <sub>2</sub> - BET Surface Area (m <sup>2</sup> /g)	1.3	1.1	473	557	584	552
Micropore Area (m <sup>2</sup> /g)	N.D. <sup>a</sup>	N.D. <sup>a</sup>	187	242	230	207
Micropore Vol (cc/g)	N.D.	N.D.	0.08	0.11	0.11	0.10
Cumulative Pore Vol (cc/g)	Ad.	N.D.	N.D.	0.17	0.19	0.22
	De.	N.D.	N.D.	0.18	0.20	0.23
Average Pore Radius (Å)	Ad.	N.D.	N.D.	20	20	20
	De.	N.D.	N.D.	18	18	19

<sup>a</sup> Not Determined. Surface areas are too small to measure pore volumes by this technique.

Table 4. Surface Composition (Wt.%, Relative) Data from XPS Analyses

	IBC-103 Coal	MG Char	SA3-9 Char	OSA3-9 Char	SA10-14 Char	OSA10-14 Char
C	71.3	74.1	78.7	78.8	80.	80.
O	17.6	15.7	12.9	13.3	10.4	10.2
N	1.6	1.6	0.9	0.9	0.5	0.8
S	1.4	1.2	0.7	0.7	3.7	3.9
Al	2.8	2.4	2.1	2.3	1.6	1.6
Si	4.6	3.6	3.4	3.0	2.6	2.3
Fe	0.4	0.8	0.9	0.4	0.8	0.8
Mg	0.2	0.2	0.2	0.2	Tr.	Tr.
Ca	---	0.3	0.3	0.3	0.3	0.3

Table 5. Binding Energies (eV) Data from XPS Analyses

Sample Identification	C 1s	%	O 1s	Si 2p	Al 2p	S 2p	N 1s	%	Fe 2p	Ca 2p
IBC-103 (coal)	284.5	84	533.0	103.5	75.0	163.8	398.5	38	712.0	-----
	286.2	14				168.9	400.2	57		
	289.2	2					402.1	5		
MG Char (United Coal Company)	284.5	81	533.2	103.6	75.7	163.7	398.6	28	712.1	349.0
	286.4	16					400.4	62		
	288.8	3					402.4	10		
SA3-9	284.6	75	533.2	104.0	75.7	163.8	398.6	19	711.7	347.7
	286.3	16					400.7	68		
	288.7	9					402.3	13		
OSA3-9	284.6	66	533.2	104.1	75.7	163.9	398.4	13	711.2	347.7
	286.1	23					400.7	56		
	289.7	10					402.3	23		
SA10-14	284.6	65	533.2	103.6	75.4	163.9	398.6	22	711.6	348.2
	286.3	23					401.2	78		
	289.7	12								
OSA10-14	284.6	60	533.2	103.9	75.7	163.9	398.7	22	711.9	347.9
	286.0	30					402.3	78		
	290.0	10								