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Production and Screening of Carbon Products Precursors From Coal

**Quarterly Report
January 1 - March 31, 1998**

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QUARTERLY TECHNICAL PROGRESS REPORT

NUMBER 13

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1. Processing and Characterization of HTI Bottoms by NMP Extraction

It was reported last quarter that attempts to reduce the ash content of the Hydrocarbon Technology, Inc. (HTI) bottoms material following N-methyl pyrrolidone (NMP) extraction and centrifugation produced a coal-derived pitch with an ash content of 0.7 wt%. Additional processing by pressure filtration through a 0.2 μm Teflon filter produced a coal-derived pitch with an ash content of 0.5 wt%. The latter result indicates that the ash-forming constituents are sub-micron in dimensions.

Because the ash exhibited the color of an iron oxide, the assumption was made that the ash-forming matter is primarily iron based. Thus, a portion of the inorganics may be soluble in a dilute solution of HCl acid. To test this assumption, 200 g of the coal liquid were redissolved in 1.5 L of NMP. To this was added a solution comprised of 10 mL of concentrated HCl in 100 mL of deionized water. The mixture was stirred overnight at 60°C. Afterward, the mixture was cooled to room temperature and 2 L of deionized water added to precipitate the coal liquid. The supernatant liquid was decanted and the precipitate washed an additional two times with deionized water. Finally, the precipitated coal material was vacuum dried overnight at 170°C.

Table 1 compares some data of the properties of the acid-washed coal liquid with those from centrifugation alone. It is evident that the acid treatment was effective in reducing the quantity of ash significantly. In addition, because some of the lighter coal components were discarded with the supernatant liquid, the coke yield, real density, and softening point temperature all increased.

Table 1. Properties of acid-washed coal liquid and centrifuged coal liquid.

	Acid Washed	Centrifugation
Softening Point, °C	127.1	101.8
Density, g/cm ³	1.203	1.187
Coke Yield, wt%	43.9	37.3
Ash Content, wt%	0.11	0.70

Figure 1 is a photomicrograph of the coke structure developed by the acid-washed coal liquid. Note the degree of anisotropy does not seem to be adversely affected by the acid treatment.

Figure 1. Optical texture developed by the acid-washed HTI coal liquid

The acid treatment was repeated in batches several times until about 4 kg of cleaned coal product was produced. About 1 kg of the coal-derived pitch was sent to Koppers for examination by their battery of tests. A portion of the pitch was carbonized into green coke and also sent to Koppers for petrographic analysis, the results to be reported when they become available. Table 2 shows the results of the tests of the HCl-treated coal pitch.

Table 2. Properties of acid-treated HTI coal-derived pitch as determined by Koppers.

Softening Point, °C	130.5
Quinoline Insolubles, wt%	0.3
Toluene Insolubles, wt%	21.4
Conradson Coking Value, wt%	40.6
Ash Content, wt%	0.15
Volatility, wt%	6.04
Specific Gravity	1.189
C/H Atomic Ratio	1.10

The values determined by Koppers for the softening point temperature, coke residue, ash content, and specific gravity agree well with those determined by West Virginia University.

The viscosity of the pitch at different temperatures was also determined by Koppers, with the results presented in Table 3.

Table 3. Viscosity of acid-treated HTI coal-derived pitch at selected temperatures.

Temperature, °C	Viscosity, centipoise
160	14,400
180	3,385
200	805

About 0.4 kg of the acid-treated material was converted into green coke and samples prepared for examination by Alcoa. Results will be reported when they become available.