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Production and Screening of Carbon Products Precursors From Coal

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For
U.S. Department of Energy
Office of Fossil Energy
Federal Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

By
West Virginia University Research Corporation
on behalf of West Virginia University
617 N. Spruce Street
Morgantown, West Virginia 26506

MASTER *JWT*

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1. Processing and Characterization of HTI Bottoms by NMP Extraction

For this quarter, West Virginia University has completed the extraction of the Hydrocarbon Technology, Inc. (HTI) O-6 bottoms coal product. Recall that the material was vacuum distilled earlier at the Koppers Technical Center in Harnarville, PA to remove liquefaction solvent and other low-boiling substances. There are now approximately 25 kg (about 55 pounds) of extracted coal product available for further testing and evaluation.

The steps involved in the removal of ash and other solids were carried out by placing about 800 g of the distilled O-6 bottoms in a 10-L flask along with 6 L of N-methyl pyrrolidone (NMP). The solution was stirred for 1 hour at elevated temperature while at atmospheric pressure. The mixture was cooled and transferred to a centrifuge. Solids were removed under a force of 2000 G and the supernatant liquid then decanted into a large rotary evaporator to strip off NMP for recycle. The deashed pitch was finally vacuum dried at 170 °C overnight under a slight purge of nitrogen before weighing and storage in a cold room. This batch procedure was repeated several times until all of the distilled O-6 bottoms was processed. Table 1 gives nominal values for some of the properties of the processed coal liquid.

Table 1. Nominal properties of HTI O-6 bottoms after solvent extraction

Softening Point, °C	101.8
Density, g/cm ³	1.187
Tetrahydrofuran Insolubles, wt%	1.1
Toluene Insolubles, wt%	12.8
Coke Yield, wt%	37.3
Ash Content, wt%	0.7

2. Laboratory-Scale Conversion of Processed HTI Bottoms into Green Coke

A portion of the extracted HTI coal liquid was converted into green coke in a laboratory-scale atmospheric still coker. Five hundred grams of coal liquid were placed in the coker reactor and the reactor inserted in a programmable furnace. Carbonization

conditions were 3 °C/min to 400 °C and held at this temperature for 5 hours; 1 °C/min to 430 °C/min and held 5 hours; finally, 1 °C/min to 550 °C/min and held 3 hours. The coker reactor contents were allowed to cool to room temperature before removal of the green coke for weighing. The coke yield under these carbonization conditions was 47 wt%. Figure 1 is a photomicrograph of a polished specimen of the green coke, showing the development of anisotropy.

Figure 1. Photomicrograph of laboratory-scale green coke from extracted HTI O-6 bottoms

3. Ash Reduction of HTI Bottoms

As can be seen in Table 1, the ash content of 0.7 wt% may be too high for the extracted coal liquid to be used directly in carbon products. To reduce the ash-forming constituents further, a series of filtrations was performed using a Millipore pressure filter apparatus. About 500 g of the coal material were redissolved in 4 L of NMP and the mixture transferred to the pressure filter unit. The mixture was conveyed through the filter by the application of 20 to 30 psig of nitrogen gas, the filtrate collected, and the NMP removed by rotary evaporation. The filtered product was then vacuum dried at 170 °C overnight while under a slight purge of nitrogen. After cooling, samples were removed

and their ash content determined as proscribed by the method ASTM D2415.

Table 2 lists the results of the ash determinations for the coal material after filtration through filters of different particle retention capability.

Table 2. Ash content of filtered HTI O-6 bottoms coal product

Particle Retention of Filter, μm	Ash Content, wt%
Centrifugation alone	0.7
>20	0.6
5-10	0.6
2-5	0.6
1-3	0.6
0.2	0.5

As the data in the table indicate, the ash forming substances must be submicron in diameter. Even after filtration through a filter designed to retain particles of $0.2 \mu\text{m}$, the ash content is still 0.5 wt%. Efforts are now being pursued to remove the ash forming material by selective extraction.