**QUARTERLY TECHNICAL PROGRESS REPORT
AND KEY PERSONNEL STAFFING REPORT
NUMBER 11**

**PRODUCTION AND SCREENING OF CARBON PRODUCTS
PRECURSORS FROM COAL**

CARBON PRODUCTS CONSORTIUM

CONTRACT NO. DE-AC22-95PC94063

REPORTING PERIOD:

July 1, 1997 to September 30, 1997

Submitted to:

**AAD Document Control Center
U.S. Department of Energy
Pittsburgh Energy Technology Center
PO Box 10940, MS 921-143
Pittsburgh, PA 15236-0940**

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**October, 1997
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1. Executive Summary

The U.S. DOE Office of Fossil Energy is interested in the coproduction of coal liquids and carbon precursors. The DOE project manager has arranged for Hydrocarbon Technologies, Inc. (HTI) to provide the WVU researcher team with samples of stage-one, mildly hydrogenated coal liquids. Distillation and extraction process are being developed which produce a potential carbon anode precursor from the HTI material. The results obtained thus far seem to indicate that a coal-derived, pitch-like material can be developed from the HTI liquefaction product after distillation to remove liquefaction solvent and NMP extraction to eliminate mineral matter. The physical properties of the liquid are not unreasonable when compared to other types of available pitches. The molecular arrangement and the optical structures present in the green coke of the de-ashed coal liquid may be suitable for the production of anodes for aluminum manufacture, but more testing needs to be conducted to confirm this.

2. Processing of HTI 0-6 Bottoms

As reported previously, initial characterization of the Hydrocarbon Technology, Inc. (HTI) coal liquid (designated 0-6 Bottoms, Run 227-102) was promising for its use in making calcine coke for anode production after removal of the distillate fraction and deashing by NMP extraction. However, testing of a larger quantity of material will be required to confirm the suitability of the HTI liquid for coal-derived carbon products.

To accomplish this goal, Koppers has graciously allowed WVU to use its pilot-scale vacuum distillation unit and personnel located at the technical center in Harmarville, PA. Approximately 32 kg total of vacuum distilled coal liquid have been prepared in two batches at the Harmarville facility.

2.1 Distillation

About one-half of the HTI coal liquid was distilled in the first batch with no unusual difficulty or problem. The pot area of the distillation unit was gradually brought to 350 °C while under vacuum and samples withdrawn at regular time intervals for the measurement of softening point temperature. After a softening point temperature of about 125 °C was reached, power to the heaters on the distillation unit was removed. The contents remaining in the distillation unit were allowed to cool slightly before transfer of the pitch to a receiving container.

About 30 wt% of distillate was removed from the initial amount of HTI coal liquid charged to the distillation unit. This quantity of distillate is somewhat lower than that produced earlier in the WVU laboratory, possibly because of the size and scale of the Koppers distillation unit. Table 1 below gives the results of mass considerations for batch number 1.

Table 1. Material balance for pilot-scale distillation of HTI O-6 bottoms, batch number 1

Amount of HTI Coal Liquid Charged, g	23,035
Total Distillate, g	6,228
Total Pitch, g	16,222
Softening Point of Pitch, °C	124.9
Yield of Pitch, wt%	70

The second batch was obtained under similar conditions by raising the distillation pot temperature gradually to 350 °C while the unit was under vacuum. Samples of pitch were again withdrawn from the pot area at regular intervals for the determination of softening point temperature. When the softening point temperature of the pitch had reached about 130 °C, distillation was stopped and the pitch allowed to cool somewhat before transferal to a metal container. Mass balance data following the distillation of the second batch are provided in Table 2.

Table 2. Material balance for pilot-scale distillation of HTI O-6 bottoms, batch number 2

Amount of HTI Coal Liquid Charged, g	23,131
Total Distillate, g	6,472
Total Pitch, g	16,353
Softening Point of Pitch, °C	130.0
Yield of Pitch, wt%	70.7

The distilled coal liquid was subsequently shipped to the chemical engineering laboratory at West Virginia University for additional processing. WVU continues to make good progress in deashing and purifying the distilled coal liquid. Between 3 to 4 kg per week are being treated by solvent extraction with N-methyl pyrrolidone (NMP). At the time of this reporting period, nearly 15 kg of extracted and deashed pitch are available for

evaluation or coking. We anticipate completion of the NMP-extraction process sometime in October.

2.2 Solvent Extraction

The steps involved in the removal of ash and other solids are carried out by placing about 800 g of the distilled O-6 bottoms in a 10-L flask along with 6 L of NMP. The solution is stirred for 1 hour at elevated temperature while at atmospheric pressure. The mixture is cooled and transferred to a centrifuge. Solids are 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 is finally vacuum dried at 170 °C overnight under a slight purge of nitrogen before weighing and storage in a cold room. This batch procedure will be repeated several times until all of the distilled O-6 bottoms is processed.

2.3 Other HTI Coal Products

HTI has also provided several samples of pressure filter liquids (PFL) and pressure filter solids (PFS) for examination by WVU. Table 3 identifies what the samples are as labeled on their containers.

Table 3. Designation of HTI coal-derived products

Material Type	Designation
Pressure Filter Liquid	227-100-10,11,14
Pressure Filter Liquid	227-102-13A
Pressure Filter Solid	227-102-14A
Pressure Filter Solid	227-102-14B
Pressure Filter Solid	227-102-15B

Samples PFL 227-100-10,11,14 and PFS 227-102-14A have undergone preliminary testing. The results for the former are presented in Table 4 and a photomicrograph of the green coke shown in Figure 1.

Table 4. Characteristics of as-received PFL

Designation	227-100-10,11,14
Softening Point, °C	Room Temperature
Coke Yield, wt%	24.9
Ash Content, wt%	1.4

This particular PFL material flows readily at room temperature. Currently, it is not known whether the low softening point temperature is inherent to the sample or because of the inclusion of liquefaction solvent. Efforts are being made to contact HTI for additional information on the processing conditions used and will be reported on later.

The coke yield is rather low, being only about 25 wt%. The low yield is consistent with the low softening point temperature, indicating the light nature of the PFL. The ash content appears to be high for a filtered product. As is evident in Figure 1, the ash content seems to interfere with the development of anisotropy. It is suggested that the low coke yield, high ash content, and restricted molecular alignment would preclude this PFL product as a candidate for anode coke or binder pitch.

Figure 1. Photomicrograph of green coke from PFL

The PFS sample is the filter cake formed after passage of liquefied coal through HTI's filtration unit. Some properties of the as-received PFS are presented in Table 5 and a photomicrograph of its green coke shown in Figure 2.

Table 5. Characteristics of as-received PFS material

Identification	PFS-227-102-14A
Quinoline Insolubles, wt%	62
Coke Yield, wt%	71.4
Ash Content, wt%	48.6

Because the PFS is essentially a residue, its ash content and quinoline insolubility are very high. Moreover, examination of the green coke structure reveals that the PFS develops a coke that is highly isotropic.

Figure 2. Photomicrograph of PFS-227-102-14A green coke

A sample of the PFS was dissolved in NMP and the solids removed by centrifugation. The NMP-soluble portion was isolated and tested for physical properties, Table 6. A portion of the NMP-solubles was also converted into green coke and a photomicrograph presented in Figure 3.

Table 6. Characteristics of NMP-soluble portion of PFS-227-102-14A

NMP Solubles, wt%	37
Softening Point, °C	82.1
Density, g/cm ³	1.195
Coke Yield, wt%	37.5
Ash Content, wt%	0.7

Figure 3. Photomicrograph of NMP-soluble portion of PFS-227-102-14A green coke

It is evident from the data in Table 6 and the optical texture in Figure 3 that NMP extraction of the PFS produced a low-ash-pitch-like material. The coke yield and softening point temperature are rather low for use as binder pitch, but these values could possibly be improved with additional processing. The development of anisotropy in the green coke is also extensive, suggesting that the NMP extract from the PFS could possibly after further testing find use in the manufacturing of carbon products.

HTI has also sent about 40 kg of pressure filter solids for testing, as requested by WVU. Preparations are underway to process a sizable quantity of the PFS for examination as a possible precursor for carbon products.