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

CARBON PRODUCTS CONSORTIUM

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Quarterly Report**

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Michael A. Nowak of DOE/FETC contacted Richard A. Winschel of CONSOL, Inc., for samples of coal-derived liquids for evaluation as feedstock materials for anode coke and binder pitch. Two samples have undergone preliminary evaluation by the Chemical Engineering Department at West Virginia University. The results of the tests are presented herein.

The first sample is identified as “Kerr-McGee Light Phase”. The material was produced using the Kerr-McGee critical solvent deasher (CSD, also known as the ROSE process). The CSD unit produced three streams; an ash concentrate, heavy solvent refined coal (SRC), and light SRC products. The sample examined by WVU is claimed to be the light-stream SRC.

A summary of the available data is presented in Table 1. Also included are data for a commercial coal-tar pitch produced by Koppers Industries.

Table 1. Characterization of light-stream SRC coal liquid.

Supplier	CONSOL	Koppers
Date Produced	Circa 1979, Wilsonville, AL	----
Material	SRC, Illinois Basin, Bituminous Coal	30 Hard, Coal-Tar Pitch
<u>Elemental Composition</u>		
C (wt%)	85.71	93.83
H (wt%)	6.73	3.92
N (wt%)	1.80	0.92
S (wt%)	0.68	0.59
O ⁺ (wt%)	5.08	0.74
C/H Atomic Ratio	1.06	1.99
¹ H _{aromatic} NMR (%)	38.5	86.0
Density (g/cm ³)	1.197	1.340
Softening Point (°C)	133.5	110.3
Coke Yield (wt%)	41.1	58.0
Toluene Insoluble (wt%)	23.5	28.3

Ash (wt%)	0.17	0.21
Optical Texture of Green Coke	See Figure 2	----

+oxygen by difference

To demonstrate the extreme differences in optical texture observed in green cokes, refer to Figure 1. The top photomicrograph is an example of fine-grain anisotropy, indicative of cokes which are highly isotropic in properties. In this instance, the isochromatic units are small. Such a coke would be totally unacceptable for anode manufacture because, among other things, its coefficient of thermal expansion would be too high. The bottom photomicrograph shows the development of large isochromatic regions for a coke with highly directional properties. The molecular constituents have arranged themselves into regions of long-range order. This particular coke would be expected to exceed the requirements for anode application, approaching those for needle coke.

The light-stream SRC was converted to green coke by heating to 600 °C and a specimen prepared for examination by polarized-light microscopy. Figure 2 is a photomicrograph of the optical texture of the green coke. It can be seen that the light-stream SRC produces a green coke that is anisotropic, but the domain size of the isochromatic units is judged to be of an insufficient size for anode manufacture. Moreover, the low aromaticity, low coke yield, high softening point, and high oxygen content of the light-stream SRC would suggest that it is also not suitable as a binder pitch.

Figure 1. Optical texture of representative green cokes; Left isotropic, Right anisotropic.

Figure 2. Optical texture of light-phase SRC green coke.

The second sample supplied by CONSOL is representative of a two-stage direct coal liquefaction process stream. The specimen is described as a composite of individual samples taken over extended time periods of what is called Run 259 made in 1990 at the Wilsonville integrated two-stage coal liquefaction pilot plant. This particular run was made in a continuous unit which had catalysts in both stages of the reactor. As explained by CONSOL, samples were removed regularly at a point called the “ interstage ” which was located between the first and second reactors of the two-stage liquefaction unit. After each sampling, the coal liquid was distilled to an atmospheric equivalent boiling point of 850 °F. Finally, the 850 °F⁺ fractions were combined to make a composite sample. It is this material , designated by CONSOL as R1235, which was tested by WVU.

The ash content of R1235 reported by CONSOL (about 9 wt%) was confirmed by our laboratory. Presumably the contribution to the high-ash level is from that inherent in the starting coal and the addition of liquefaction catalysts.

To produce a clean, low-ash material, Sample R1235 was deashed using WVU’s NMP-solvent extraction procedure for comparison with the characteristics of the as-received composite coal liquid. Simple filtration of the NMP-R1235 mixture revealed that the organic portion of the coal liquid was nearly 100 wt% soluble in NMP, with only the insoluble inorganic solids remaining on the filter paper. A summary of the available analyses is presented in Table 2.

Table 2. Characteristics of coal liquid R1235 before and after extraction with NMP.

Material	Coal Liquid R1235	Coal Liquid R1235 After NMP Extraction
Product Precursor	Pittsburgh Seam Bituminous Coal	Pittsburgh Seam Bituminous Coal
Date Produced	1990, Wilsonville, AL	1997, WVU
Elemental Composition (wt%)	MAF ¹	
C	90.12	----
H	6.19	----
N	1.15	----
S	1.50	----
O ²	1.04	----
C/H Atomic Ratio	1.21	----
¹ H _{aromatic} NMR (%)	63.4 ³	----
Density (g/cm ³)	1.303	1.215
Softening Point (°C)	152.6	151.8
Toluene Insoluble (wt%)	32.9	26.2
Ash Content (wt%)	8.74	0.10
Coke Yield (wt%)	62.3	54.6
Optical Texture of Green Coke	See Figure 3	See Figure 3

¹MAF = moisture and ash free, ²oxygen by difference, ³proton distribution of tetrahydrofuran solubles.

The as-received R1235 and its NMP-extracted product were converted to green coke and specimens prepared for optical microscopy. Photomicrographs are presented in Figure 3.

Cursory examination of the data in Table 2 suggests that R1235 is a more suitable material for anode coke and binder pitch because its carbon content, aromaticity, and coke yield are higher than the values for the light-stream SRC. However, the ash content of R1235 is nearly 9 wt%. This quantity of ash forming inorganics would preclude the use of R1235 in most carbon product precursors. Indeed, the ash forming materials are not only undesirable contaminants but also inhibit the development of anisotropy, as can be seen by the isotropic coke structure in Figure 3.

On the other hand, removal of the mineral matter by NMP extraction produces a pitch-like substance more amenable for coke making than either the light-stream SRC or the as-received R1235. The development of molecular alignment in the NMP-extracted coal liquid after carbonization is considerable, producing optical structures seen similarly in conventional anode cokes. Moreover, the ash content, coke yield, toluene solubility, and anisotropic development of the NMP-extracted material are judged to be more in line with commercial pitch precursors.

Although the preliminary results look promising thus far, further testing of the NMP-extracted R1235 on a larger scale will be required to establish whether or not the coal liquid will be a suitable precursor for both anode coke and binder pitch.

Figure 3. Optical texture of coal liquid R1235; top as received R1235, bottom NMP-extracted R1235.

Vivek Pradhan of Hydrocarbon Technologies, Inc. (HTI) has prepared a larger quantity of coal liquid produced under relatively mild hydrogenation conditions. No processing conditions, coal and catalyst type, or any other information is available at this time. However, the material is identified as 0-6 bottoms material collected during run 227-102 Periods 17, 18, 19, and 20 (sic).

The coal-derived material is a thick liquid at room temperature. WVU conducted preliminary characterization of the product. The results are presented in the table below.

**Initial Characterization of HTI
Coal Liquid**

Softening Point, °C	Room Temperature
Density, g/cm ³	ND
Coke Yield, wt%	47.0
THF Insolubles, wt%	17.2
Toluene Insolubles, wt%	20.5
Ash Content, wt%	12.9

Work is currently underway to modify an autoclave in order to perform vacuum distillation of HTI's coal-liquid product to remove liquefaction vehicle and to increase softening point.