PRODUCTION AND SCREENING OF CARBON PRODUCTS PRECURSORS FROM COAL

Quarterly Technical Report

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Executive Summary

This quarterly report covers activities during the period from October 1, 1996 through December 31, 1996. The technical work has focused on enhancing equipment and instrumentation in the WVU Carbon Products Laboratory. Development work on coal-based precursors for carbon foams, pitches, cokes, and fibers continues. The effects of carbon powders and chopped fibers as additives to the foam precursor are being evaluated. Extensive coordination and technology transfer activities have been undertaken and are described in Section 5 of this report.

1. Project Planning and Administration

The purpose of Task 1 is to prepare and submit to the DOE, a Project Management plan for the WVU portion of the Carbon Products Consortium (CPC) workplan. This Management Plan was submitted to PETC on July 2, 1995. It has been accepted by the COTR and by the contracts management staff. A work plan for year 2 of the project has been developed and is attachment no. 1 to this report.

2. Consortium Administration and Reporting

The purpose of Task 2 is to establish a Participants Agreement (PA) and a Proprietary Information Agreement (PIA) for members of the CPC, to facilitate communications between CPC participants and the COTR, and to help secure, maintain and manage CPC funds obtained under this contract.

The PA and the PIA were finalized on September 1, 1995. It was necessary to revise the PA to define the category of Affiliated Participant for an organization which does not sign the PA or the PIA, but is involved with the work of the CPC. A copy of the revised PA was included with Monthly Status Report No. 8, September 1, 1995 - September 30, 1995.

All monthly status and quarterly technical reports have been submitted as required by the contract. In addition to the required reports, regular communications with the COTR have been maintained.

Extensive efforts have been made to maintain funding through Fossil Energy, as well as, to broaden the base of funding for the work of the CPC. Programs are under development with the Office of Heavy Vehicle Technologies, the Office of Industrial Technologies, as well as, with the Navy Surface Warfare Center.

3. Coal Extraction

Under Task 3.0 and subtasks, WVU will provide samples of coal extracts and hydrogenated coal pitches to the CPC participants and affiliates. Also, samples of coal-derived green and calcined cokes will be developed and provided as needed. A summary of the technical accomplishments and other progress follows:

Upgrading of the Instron capillary rheometer continues. The instrument is being modified to perform a variety of tasks in addition to the measurement of the viscosity of coal-derived pitches as a function of temperature. Special sample-compression-test holders were designed and fabricated in-house and have undergone preliminary evaluation. The new fixtures will give WVU the capability to conduct compression tests on carbon foam samples, which are currently being developed. A computerized data acquisition system is also being mated to the rheometer to collect and analyze strength-crushing profiles. Moreover, the instrument has the capability for spinning coal-based isotropic and anisotropic carbon fibers.

A large reactor system for producing carbon foams is now fully functional. Process parameters have been changed in order to produce a variety of foams with controlled densities, cell structures, and porosity. Several specimens were prepared and sent to C. Baker at FMI for impregnation and graphitization. WVU is also conducting experiments to determine the effects of additives, such as carbon powders and fibers, on the mechanical properties of the foams. Moreover, WVU is now undertaking its own procedures for impregnating foams with pitches and other monomeric additives. These efforts on coal-based foam technology are being developed concurrently with the CPC program.

A subcontract to WVU on AMOCO's NSWC project continues. Several coal-based pitches have been developed at WVU and have been tested for mesophase development. These materials may find application in high-thermal conductivity carbon fibers.

A 20-L extractor unit is currently being used to extract unhydrogenated coal with NMP. The goal is to produce a sizable quantity of coal extract so that Koppers and UCAR will have sufficient material for their testing.

Michael A. Nowak of DOE/FETC contacted Richard A. Winschel of CONSOL, Inc. for a sample of coal liquid for evaluation as a feedstock material for use as either a binder pitch or anode coke. CONSOL subsequently sent several grams of a solvent-refined coal (SRC) product to WVU which is identified as "Kerr-McGee Light Phase". The material was produced using the Kerr-McGee critical solvent de-asher (CSD, also known as the ROSE process). The CSD unit produced three streams; an ash concentrate, heavy SRC, and light SRC products. The sample sent to WVU is claimed to be the light-stream SRC.

The light-stream SRC material has undergone preliminary testing. The available data are presented in the table below. Also included are data for a commercial coal-tar pitch produced by Koppers Industries.

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 Microscopy of Green Coke	Course-Flow Anisotropy	----

+oxygen by difference

At this time, further testing will be required to establish whether or not the light CSD material would be suitable for use as a binder or a precursor for anode coke. A perusal of the data indicates that the high softening point, low aromaticity, and low coke yield would suggest otherwise.

If specimens could be made available, it may be worthwhile to test the heavy as well as the ash-laden fractions of the CSD process for applicability as carbon products precursors. These materials would be expected to have a greater carbon content, contain

more aromatic hydrogen, and produce a higher coke yield. Ash reduction can be accomplished by WVU's solvent extraction procedure in case the mineral matter content is unacceptably high.

In addition, several isotropic pitches have been produced at WVU. The pitches were made from coal under different conditions of hydrogenation severity, solvent extraction and partitioning, heat treatment, or blending. The coal-derived pitches are currently being evaluated for physical properties, mesophase formation, and coke structure.

4. Technical/Economic Evaluation of WVU Extraction Process

WVU provided all requested information to the MITRE Corporation for their economic analysis of the coal extraction process. A draft of the MITRE report was received in mid July, 1995 and a revised version was received in September, 1995.

The MITRE report suggests several process changes whose implementation on a larger scale could substantially reduce the cost of the coal extraction process. MITRE finds that coal extract based calcined coke for anodes could be produced for approximately \$177 per ton. A February 1 article in the Financial Times reported that world aluminum production could be restricted by shortages of petroleum coke. The article said that coke prices have doubled the past year to between \$240 and \$250 a ton. This is because it is more profitable for refineries to make liquids than coke. MITRE also recommends research on the production of isotropic carbon fibers from coal extracts of unhydrogenated coal. Such fibers are in the \$8 to \$10 per pound range and the market for low cost fibers is expected to double in the future.

5. Technology Transfer

A confidentiality agreement between a large chemical company, Koppers and WVU is being negotiated. The agreement will facilitate discussions on enhancing the economics of the coal extraction process and scaling up production of certain types of coal-based extracts for further evaluation.

A Carbon Products Coordination Meeting was held in Senator Byrd's Office in Washington, DC on October 18, 1996. The purpose of the meeting was to provide a common information base and to develop a cooperative approach to funding the coal-based carbon products program. See attached agenda and attendee list.

Carl Irwin was invited to the 1996 Fall Meeting of SACMA (Supplies of Advanced Composite Materials Association) to give a talk on the results of the Chicago Workshop on carbon products and heavy vehicles.

Carl Irwin represented the CPC at the Aluminum Industry Outreach Conference, sponsored by the Aluminum Association.

On November 1, Carl Irwin met with Howard Feibus in Germantown to discuss possible synergy of the program on coal-based carbon products with DOE coal refinery concepts. This was a follow-up meeting to the carbon products coordination meeting held October 18 in Senator Byrd's office.

The Carbon Products Consortium (CPC) had an exhibit at the November 9 tribute to Senator Byrd which was held on the WVU campus at the National Research Center for Coal and Energy. The work on coal-derived carbon products was referred to in a talk given by WVU President David Hardesty.

The CPC 1996 business meeting was held at WVU on November 19. See attached agenda. A substantial part of the meeting was devoted to discussing a workplan for year two of the PETC sponsored project. The objective for year two is to focus on development of the most likely commercially successful coal-based carbon products. Notes from a CPC conference on this topic were attached to the November Monthly Status Report. The consensus of the CPC industrial participants is that the first commercial successes could well be binder pitches and anode cokes for the aluminum industry, and pitches and isotropic cokes for specialty molded graphites. The Year 2 Workplan and Budget for this program is attached.

Following the CPC business meeting, a Carbon Industry Vision Workshop, sponsored by the U.S. DOE Office of Industrial Technologies (OIT), was held at WVU on Tuesday evening November 19 and November 20. An agenda and list of companies and agencies represented at the vision workshop is attached. The Governor-elect of West Virginia, Cecil H. Underwood, made his first post-election talk at the vision workshop, expressing strong support for the carbon program and for industry-government-academic partnerships.

Carl Irwin was invited to discuss the OIT vision workshop and the CPC at the November 22 meeting of the National Laboratory Coordinating Council.

6. Attachments

1. Workplan and Budget for year two
2. Agenda and Attendee List for Carbon Products Coordination Meeting on October 18, 1996
3. Agenda for CPC 1996 Business Meeting on November 19
4. Agenda and Attendee List for the Carbon Industry Vision Workshop