DEVELOPMENT OF CONTINUOUS SOLVENT EXTRACTION PROCESSES FOR COAL DERIVED CARBON PRODUCTS
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ABSTRACT

The purpose of this DOE-funded effort is to develop continuous processes for solvent extraction of coal for the production of carbon products. These carbon products include materials used in metals smelting, especially in the aluminum and steel industries, as well as porous carbon structural material referred to as "carbon foam" and carbon fibers. There are a number of parameters which are important for the production of acceptable cokes, including purity, structure, density, electrical resistivity, thermal conductivity etc. From the standpoint of a manufacturer of graphite electrodes such as GrafTech, one of the most important parameters is coefficient of thermal expansion (CTE). Because GrafTech material is usually fully graphitized (i.e., heat treated at 3100 ^{o}C), very high purity is automatically achieved. The degree of graphitization controls properties such as CTE, electrical resistivity, thermal conductivity, and density. Thus it is usually possible to correlate these properties using a single parameter. CTE has proven to be a useful index for the quality of coke. Pure graphite actually has a slightly negative coefficient of thermal expansion, whereas more disordered carbon has a positive coefficient.

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1.0 EXECUTIVE SUMMARY

The purpose of this DOE-funded effort is to develop continuous processes for solvent extraction of coal for the production of carbon products. These carbon products include materials used in metals smelting, especially in the aluminum and steel industries, as well as porous carbon structural material referred to as "carbon foam" and carbon fibers.

During this reporting period, efforts have focused on the development of continuous processes for hydrogenation as well as continous production of carbon foam.

2.0 EXPERIMENTAL

2.1 UV-Vis Spectrophotometry

An experimental procedure for the reactions of coal and their analysis using UV-Vis spectrophotometry is described. This research requires coal samples of various sizes to be reacted at various temperatures and times. The quantitative specifications for the different coal sizes are listed in

Table 1. Specifications for Different Coal Sizes.

Coal Classification	Tyler Mesh Size	Microns	Inches
Small	Sub 150	Less than 106	Less than 0.00417
Medium	60 by 150	250 to 106	0.00984 to 0.00417
Large	45 by 60	355 to 250	0.01397 to 0.00984

Seven temperatures and twelve times were selected, resulting in a total of 3 x 7 x 12 = 252 samples.

After these reactions were run, an additional 36 samples were run, at a temperature of 185°C. This was done because most of the dissolution observed occurred somewhere between 170°C and 200°C. An additional 33 reactions were run under agitation to observe the possible effect on dissolution rates. However, there was no observable difference between agitated and non-agitated coal dissolution.

Table 2. Naming Conventions for Test Tube Samples

Size			Time (minutes)		
s1	Small		t1	5	
s2	Medium		t2	15	
s3	Large		t3	30	
			t4	60	
			t5	90	
Temperature (°C)			t6	120	Temperatures 140°C or Less
m1	50		t7	150	
m2	80		t8	180	

m3	100		t9	210
m4	120		t10	240
m5	140		t11	270
m6	170		t1	2
m7	200		t2	4
m8	140		t3	6
m9	140		t4	8
m10	185		t5	10
			t6	12-14
			t7	14-16
			t8	16-18
			t9	18-20
			t10	20-22
			t11	22-24
			t12	60

Temperatures Above 140°C

A set of 10ml graduated test tubes specifically matched to each other were obtained for use in the research. The samples were run in batches of twelve, which is the number of time levels. For instance, all of the small coal size are run at a temperature of 140°C, and are removed at the appropriate time until the batch of twelve samples are complete.

Each test tube is weighed empty to one milligram accuracy. The test tube is then filled with 1ml of coal of the appropriate size, and is then weighed again to the nearest milligram. The graduated test tubes were then filled with 6 ml of coal, and were again weighed to the nearest milligram. The above procedure is completed when all twelve samples of a particular experimental batch are filled. The test tubes are placed in a custom test tube rack that was designed specifically to fit into the sand bath available in the Engineering Research Laboratory. The rack was lowered into the sand bath, which was pre-heated to the necessary temperature.

After a specific test tube's time was reached, it was removed from the test tub rack in the sand bath and allowed to air cool. The test tubes were removed at the specified times one at a time until no more test tubes were left remaining in a specific experimental batch. After all test tubes cooled, the test tubes were centrifuged. The swelling ratio was measured by noting which graduation mark the coal had swelled to in the 10ml test tube relative to the original 1 ml mark. For example, if the coal had expanded to the 2.5 ml mark, that would indicate a swell of 150%.

One milliliter of the coal-NMP solution was withdrawn after centrifugation. The one milliliter of solution was placed in a 100 ml Ehrlenmeyer Flask. NMP was added to the flask until the 100 milliliter mark was reached. This dilution of the coal-NMP solution was necessary to obtain usable UV-Vis spectrophotometer data, which will be described in more detail in the following section.

Next, the coal-NMP solution had to be analyzed to determine how much coal was present in solution. A UV-Vis spectrophotometer is used.

A solution exposed to UV-VIS spectroscopy must be absorbent enough to absorb some light, but not so absorbent that too little light is transmitted. Without the proper absorbance, useful data will not be obtained. The quantity we are interested in, the absorbance, is presented as Equation 4.2.2.

$$A = \log \frac{P_0}{P}$$

Where P_0 and P is the power of a beam of monochromatic radiation before and after passing through the solution respectively.

Another important parameter for UV-Vis spectrophotometry is the path length the radiation must pass through. The path length is referred to as b. These quantities are illustrated graphically below.
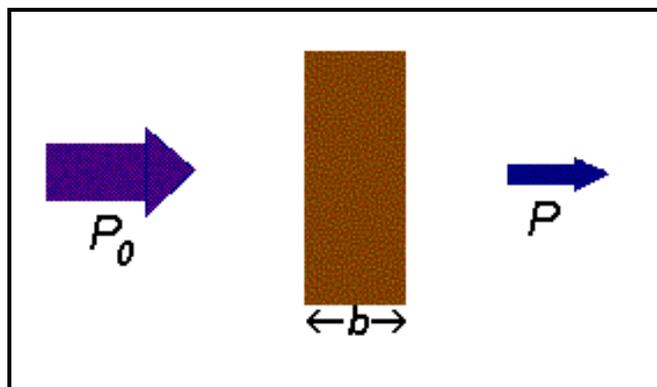

Figure 1. Conceptual drawing of radiation passing through a solution with path length b.

There are two parameters that can be adjusted to tailor the absorptivity of a solution. The first is the path length of the cell in which the solution resides during testing. If cell A is ten times the width of cell B, the solution in cell A will appear to have one tenth of the transmittance ($T = P / P_0$) of the solution in cell B. This is why Beer's law requires a path length parameter. The second parameter we can adjust is the dilution of the solution. A relatively dilute solution will absorb less (have a higher transmittance) than a relatively concentrated solution.

Adjustment of both of these parameters was necessary to obtain solutions with the proper absorbance. A cell width (path length) of 0.1 mm was required, which is small compared to most cells. Dilution of the coal-NMP solution was also necessary. Some coal-NMP samples were diluted by a factor of 50, while most were diluted by a factor of 100. The dilution factor was chosen based on how relatively dark the coal-NMP solution appeared. Light solutions were diluted 50:1, while average and dark solutions were dilute 100:1. These dilution factors placed absorbance readings in an acceptable range. Absorbance is then given by Beer's Law,

$$A = \varepsilon bc \quad,$$

where ε is the molar absorptivity, L mol^{-1} cm^{-1}, b is the path length in cm, and c is the concentration in mol/L.

Beer's Law is not a truly linear relationship at higher concentrations, only at lower concentrations. Therefore, samples were chosen such that the absorbance of the samples could be no higher than about 3.

The path length is known, and the spectrophotometer records the absorbance. Thus the molar absorptivity must be calculated in order to can obtain the concentration of coal in the coal-NMP solution. The molar absorptivity, a measure of the amount of light absorbed per unit concentration, is calculated as follows:

Soxhlet extraction was performed on measured quantities of all sample coals for for 24 hours at reflux conditions (202°C for NMP). The product was filtered and vacuum dried at ambient temperature. The product and residue weights were added and mass closure was achieved. The coal was then dissolved in a known amount of NMP. The extract dissolved fully in the NMP, and was then diluted 100:1. The absorbance was measured and plotted as a function of the concentration. A linear regression was performed and the slope is the product of the molar absorptivity and the path length. To obtain the molar absorptivity the slope was simply divided by the path length. The results indicate that coal size had no noticeable effect on molar absorptivity.

2.2 Thermo Gravimetric Analysis (TGA) and Elemental Analysis (EA)

TGA analysis was used to determine the amount of moisture, volatiles, and ash for several products. Elemental analysis is used to determine the amounts of hydrogen, carbon, and sulfur in the products. Graduate students Laura Eddy and James Rhoades have produced samples from biological waste as well as coal, to be evaluated as a potential fuel in addition to carbon feedstock. Technician Jennie Wheeler has carried out TGA and EA measurements in support of both efforts.

Table 3. Elemental analysis start up table.

Sample #	Command	Solid	Slurry	Liquid
1	bypass	vanadium pent oxide	empty capsule	empty capsule
2	bypass	sample + vanadium pent oxide	sample	chemosorb + sample
3	bypass	vanadium pent oxide	empty capsule	empty capsule
4	bypass	vanadium pent oxide	empty capsule	empty capsule
5	standard	bbot + vanadium pent oxide	bbot	bbot
6	unknown	bbot + vanadium pent oxide	bbot	bbot

2.3 Kinetics Measurements

Three solvents, Carbon Black Base (CBB), Tar Oil, and a 1:1 blend of the two, were tested to determine how well they can donate H_2 to Kingwood coal. Tube Bombs

were used to perform the reactions. Each tube bomb contained 8 grams of solvent to 3 grams of Kingwood coal and 6 metal balls. Each tube bomb was purged with N_2. The reactions were carried out at 400°C, for a duration of 1 hour, in a heated fluidized bed.

Below are the measured weights (in grams) for CBB.

Table 4. Mass Bbalance for CBB Extraction of Kingwood Coal.

	Carbon Black Base (run 1)	Carbon Black Base (run 2)
Mass of empty Tube Bomb	804.21	874.59
Mass of solvent	8.0948	8.0130
Mass of Kingwood Coal	3.0115	3.0316
Mass of balls	1.5665	1.5669
Mass of full Tube Bomb	816.84	887.19
Reaction	829.74	899.99
Mass of Tube Bomb Pre-vent	816.63	886.84
Mass of Tube Bomb Post-vent	816.58	886.76

Table 5. Heated Fluidized Bed (Sand Bath) Conditions for CBB Extraction

Time	Temp (°C)	Gauge Pressure (Run 1, Run 2)
2:50	410	0,0
3:05	400	0,400
3:20	400	0,400
3:35	400	0,400
3:50	401	0,400

The next procedure was to separate the insoluble portion from the soluble portion using Tetrahydrofuran (THF). The soluble and insoluble products, were separated in soxhlets overnight. The results are seen below for CBB.

Table 6. THF Extraction Results for CBB Oil.

	Run 1	**Run 2**
Flask wt.	115.5142	96.2477
Boiling Chip wt.	0.0996	0.0765
Thimble wt. empty	1.7501	1.7874
Thimble wt. Full	3.5972	3.6679
THF Insoluble	1.8471	1.8805
% Conversion (Coal wt.- Insoluble)/(Coal wt.)	**38.67**	**37.97**

After the solubles and insolubles were separated, a rotary evaporator was used to recover the THF. Next, a vacuum was pulled on the rotary evaporator to help extract additional THF. And finally the solubles were placed in a vacuum oven to separate the remaining THF.

The percent coke in the soluble product was measured twice to ensure reliability.

Table 7. Coking Tests for CBB Extract.

	Run 1	Run 2
Crucible wt. empty	8.0790	9.1863
Pitch wt.	0.6487	0.6227
Crucible wt. after reaction.	8.1743	9.2772
% Coke	**15.30**	**14.01**

The experiments were repeated using Tar Oil in place of Carbon Black Base Oil, and the results are recorded in Tables 8-11.

Table 8. Mass Balance for Tar Oil Extraction of Kingwood Coal.

	Tar Oil (run 1)	Tar Oil (run 2)
Mass of empty Tube Bomb	861.11	877.59
Mass of solvent	8.0241	8.0430
Mass of Kingwood Coal	3.0210	3.0058
Mass of balls	1.5671	1.5704
Mass of full Tube Bomb	873.70	890.13
Reaction	886.51	903.21
Mass of Tube Bomb Pre-vent	873.71	890.29
Mass of Tube Bomb Post-vent	873.59	890.11

Table 9. Heated Fluidized Bed (Sand Bath) Conditions for CBB Extraction

Time	Temp (°C)	Gauge Pressure (Run 1, Run 2)
10:40	407	0,0
10:55	400	300,200
11:10	400	400,300
11:25	400	400,300
11:40	400	400,300

Table 10. THF Extraction Results for Tar Oil.

	Run 1	Run 2

Flask wt.	114.3596	119.135
Boiling Chip wt.	0.0944	0.0823
Thimble wt. empty	1.8939	1.6153
Thimble wt. Full	5.4591	5.1
THF Insoluble	1.9981	1.9143
% Conversion (Coal wt.-Insoluble)/ (Coal wt.)	**33.86**	**36.31**

Table 11. Coke Tests for Tar Oil Extract.

	Run 1	**Run 2**
Crucible wt. empty	9.2138	8.6944
Pitch wt.	0.6640	0.5963
Crucible wt. after reaction.	9.4092	8.8852
% Coke	**29.43**	**31.99**

The results for a 1:1 ratio of Tar Oil to CBB are shown below in Tables 12-15.

Table 12. Mass Balance for Solvent Blend Extraction of Kingwood Coal.

	Tar Oil : CBB (run 1)	**Tar Oil : CBB** (run 2)
Mass of empty Tube Bomb	861.21	877.34
Mass of solvent	4.0615 : 3.9791	4.0003 : 4.0284
Mass of Kingwood Coal	3.0024	3.0109
Mass of balls	1.5656	1.5679
Mass of full Tube Bomb	873.63	889.96
Reaction	886.75	903.07
Mass of Tube Bomb Pre-vent	873.87	890.21
Mass of Tube Bomb Post-vent	873.76	890.14

Table 13. Heated Fluidized Bed (Sand Bath) Conditions for Solvent Blend Extraction

Time	Temp (°C)	Gauge Pressure (Run 1, Run 2)
2:34	405	0,0
2:49	401	200,200
3:04	402	200,200
3:19	402	200,200
3:32	400	200,200

Table 14. THF Extraction Results for Solvent Blend.

	Run 1	Run 2
Flask wt.	99.2151	113.6584
Boiling Chip wt.	0.1014	0.0948
Thimble wt. empty	1.6173	1.8481
Thimble wt. Full	5.5759	5.8095
THF Insoluble	2.393	2.3935
% Conversion (Coal wt.-Insoluble)/ (Coal wt.)	**20.29**	**20.51**

Table 15. Coke Test for Solvent Blend Extract.

	Run 1	Run 2
Crucible wt. empty	11.9673	8.6751
Pitch wt.	0.5847	0.6402
Crucible wt. after reaction.	12.0455	8.7675
% Coke	**13.29**	**14.43**

2.4 Kinetics Calculations

The Donor-Solvent Process Simulation needs certain data for a successful simulation.

a. Stirred Tank Reactor.

Reaction:

Dehydrogenated Creosote Oil + $H_{2(g)}$ $\longrightarrow$ Hydrogenated Creosote Oil
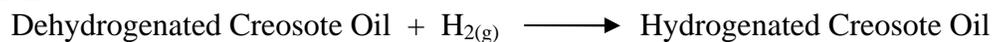
Data Needed:
1. X vs t over many T
2. Heat of Reaction
3. Components of Creosote Oil used in process.
4. C_p and MW data for Creosote Oil
5. Reactor Geometry
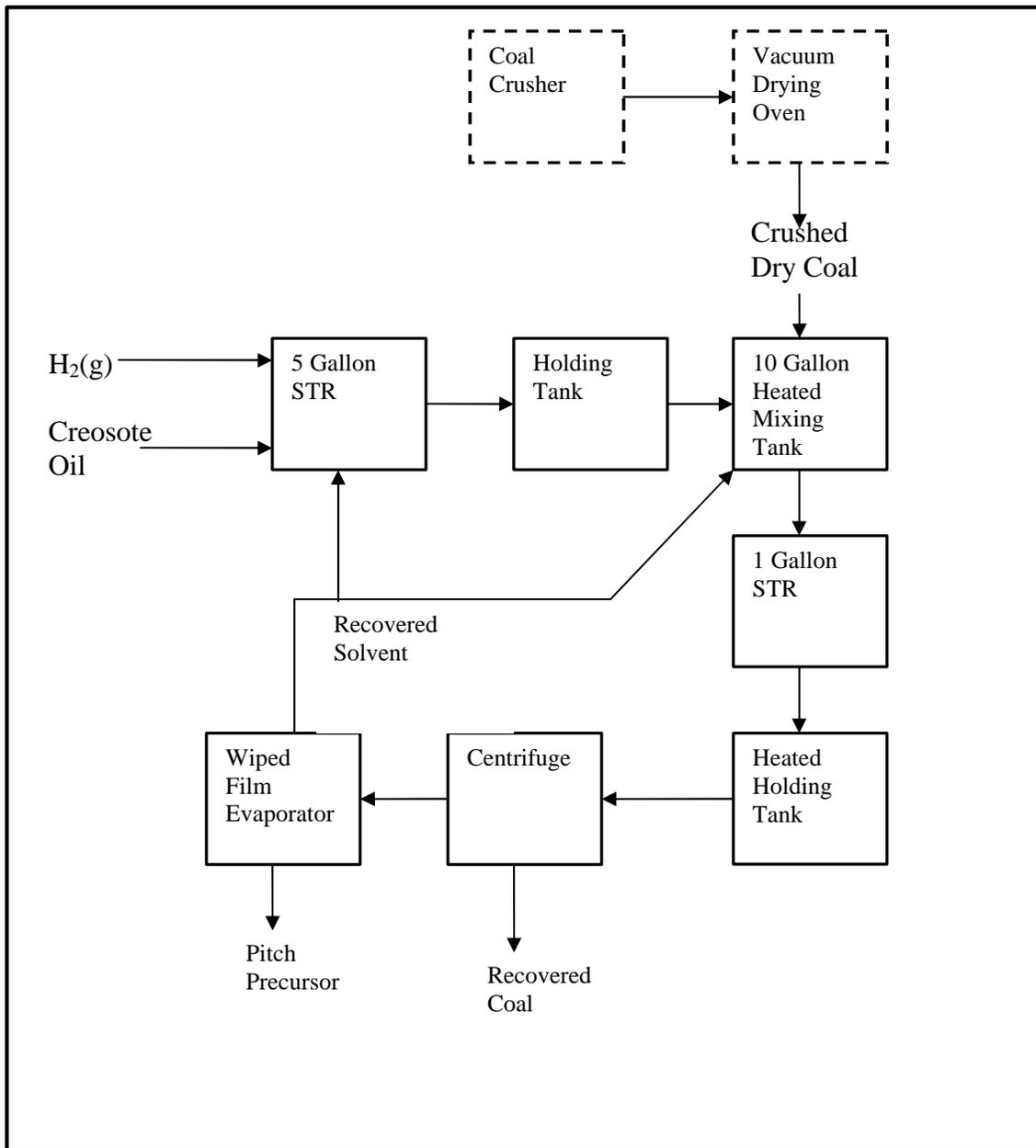6. C_{DC0} and C_{H0} or initial mass and density data

Figure 2. Process Diagram.

b. One Gallon Stirred Tank Reactor.   The reactor digests coal into a binder pitch precursor.

Coal + Solvent $\longrightarrow$ Digested Pitch Precursor + Unconverted Coal

Data Needed:
1. *X* vs *t* over many *T*
2. Heat of Reaction
3. Components of Coal used in process.
4. C_p and MW data for Coal, Solvent and Products
5. Reactor Geometry
6. C_{C0} and C_{S0} or initial mass and density data

c. Centrifuge. The centrifuge separates the unconverted coal from the product.

Data Needed:
1. Density of Coal
2. Average Particle Size
3. Viscosity Data for Liquid

d. Wiped Film Evaporator
   -BP@ 1 atm is 315-355 °C.

2.5 Pilot Plant Instrumentation

The data acquisition computer has been readied for networking. In order to do this, a wireless networking system was purchased. The wireless networking system consists of a wireless 802.11g router (with 4 wired ethernet ports) along with a wireless 802.11g PCI type network adapter.

Sharing TCP/IP protocols (internet access) proved to be easy, however, one of the computers in the office has to be networked through the wireless router and the computer attached to the router is no longer able to see the local network.

Currently, a work-around is being researched, but chances are that it will not be possible to use the network print sharing. The consequences of this are minor, though, when compared to the necessity of having the data acquisition computer networked to the High Bay office.

2.6 Ultrasonic Hydrogenation

Ultrasound is considered as a means for enhancing the dissolution rate of coal in solvent, as well as a means for enhancing the rate of hydrogenation of solvents and transfer of hydrogen to coal. However, recently the Hielscher Sonotrode unit has

undergone some minor changes to address operational problems. These problems included:

1. The frequent removal of the sonotrode for reagent loading was damaging the rubber o-ring.
2. There existed no good way of removing a sample for analysis. So, analysis of products for a series of reactions was almost impossible.
3. Injection of hydrogen gas was not well controlled.

These problems were solved by installing a series of valves and tubing which is diagramed in Figure 3 below.
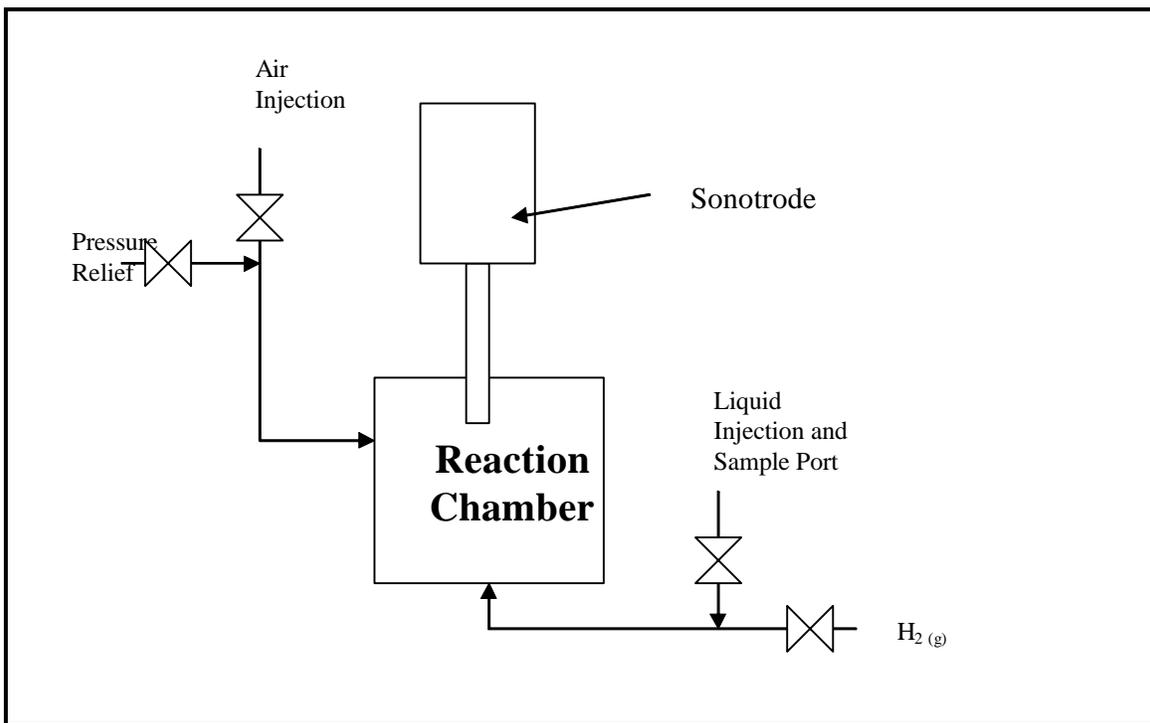

Figure 3. Ultrasound Modifications.

3.0 RESULTS AND DISCUSSION

3.1 Production Definition

Information from GrafTech and Koppers Industries was used to compile the pitch and coke specifications in the tables below.

Table 16. Binder Pitch Specification

Property	Value

Viscosity @ 160 ^{o}C	20 poise max
Mettler Softening Point	110- 115 ^{o}C
Ash	0.5% max
MCC	55% min
Tg	40 ^{o}C min
QI	4-16%
Size of QI	25 microns max
Mesophase	0%
S content	1% max
Penetration Temperature	165 ^{o}C max
Flash Point (Cleveland Open Cup)	200 ^{o}C min

Table 17. Additional GrafTech Binder Pitch Characteristics

Property	Comment
Storage Stability @ 160 ^{o}C	Long term storage is required at 160 ^{o}C, chemical change would be a concern. Pitch is often transported in a molten state.
Additive Sensitivity	Compatibility required with other materials to be combined with as-received binder pitch.
Bake Structure	Must result in a carbon structure acceptable for electrodes (in steelmaking case) or anodes (in aluminum manufacturing case).
In Stock Coking Value > 65%	Self-explanatory.
Graphite Properties	Properties of graphite product must remain within spec.
HSEP (PAH characterization)	HSEP requirements are stringent for any new material, and carcinogenic chemicals or other health-adverse chemicals are usually avoided.

In addition to binder pitch, it was suggested that solvent extraction processes might be interesting for impregnation pitches, which are similar to binder pitches in many respects.   However, the impregnation pitch must exhibit low viscosity at higher temperatures than binder pitch. In this case, the lack of quinoline insolubles from solvent extracted material would be a distinct advantage.

Table 18. Impregnation Pitch Specifications

Property	Comment
Mettler Softening Point	90-120 ^{o}C
MCC	52% min
Viscosity @ 225 ^{o}C	50 centipoise max
Flash Point (Cleveland Open Cup)	270 ^{o}C min
Solids	1% max

Table 19. Additional GrafTech Impregnation Pitch Characteristics

Property	Comment
Storage Stability @ 160 ^{o}C	Long term storage is required at 160 ^{o}C, chemical change would be a concern. Pitch is often transported in a molten state.
Additive Sensitivity	Compatibility required with other materials to be combined with as-received binder pitch.
Bake Structure	Must result in a carbon structure acceptable for electrodes (in steelmaking case) or anodes (in aluminum manufacturing case).
In Stock Coking Value > 65%	Self-explanatory.
Graphite Properties	Properties of graphite product must remain within spec.
HSEP (PAH characterization)	HSEP requirements are stringent for any new material, and carcinogenic chemicals or other health-adverse chemicals are usually avoided.

There are a number of parameters which are important for the production of acceptable cokes, including purity, structure, density, electrical resistivity, thermal conductivity etc. From the standpoint of a manufacturer of graphite electrodes such as GrafTech, one of the most important parameters is coefficient of thermal expansion (CTE). Because GrafTech material is usually fully graphitized (i.e., heat treated at 3100 ^{o}C), very high purity is automatically achieved. The degree of graphitization controls properties such as CTE, electrical resistivity, thermal conductivity, and density. Thus it is usually possible to correlate these properties using a single parameter. CTE has proven to be a useful index for the quality of coke. Pure graphite actually has a slightly negative coefficient of thermal expansion, whereas more disordered carbon has a positive coefficient.

Table 20 illustrates how CTE may be used to segregate different qualities of coke. The highest quality of coke, referred to as Super Premium grade, might carry a sales price per ton of 3 – 4 times higher than conventional anode coke.

Table 20. Coke Specifications

Designation	ppm/^{o}C @ 30 - 100 ^{o}C
Super Premium Needle Coke	0.05 - 0.15
Normal Premium Needle Coke	0.15 - 0.25
Intermediate Premium Needle Coke	0.25 – 0.40
Base Premium Needle Coke	0.40 - 0.65
Anode Grade Coke	0.65 - 1.00

3.2 Qualification of a Synthetic Binder Pitch Using Solvent Extraction

An electrical resistivity cell is used as an inexpensive, simple method to characterize the electrical resistivity of carbon and graphite, from which the index of graphitization may be correlated. A piston type lid was machined so that the cell can be loaded with free weights. The resistivity of granular as well as two samples of solid graphite was measured. In addition, various SECO samples were analyzed, as well as agricultural waste samples. The agricultural waste samples are of interest as possible solvents and have been also suggested as candidate additives to diesel fuel. Representative thermogravimetric analyzer data illustrates the behavior of the raw waste as well as blends with diesel fuel.

Each graph consists of two runs for the same sample. The curve is interpreted as the moisture percent (up to 100°C), volatiles percent (100°C to 950°C), and ash percent (575°C to 750°C).
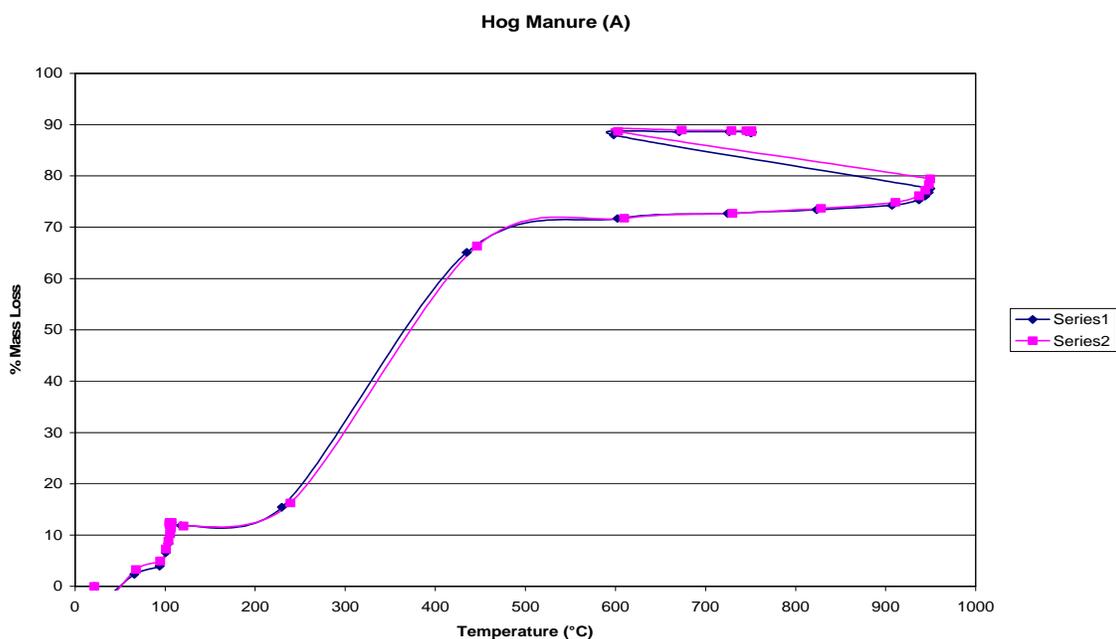

Figure 4. TGA plot of mass loss versus temperature for agricultural waste (hog manure).
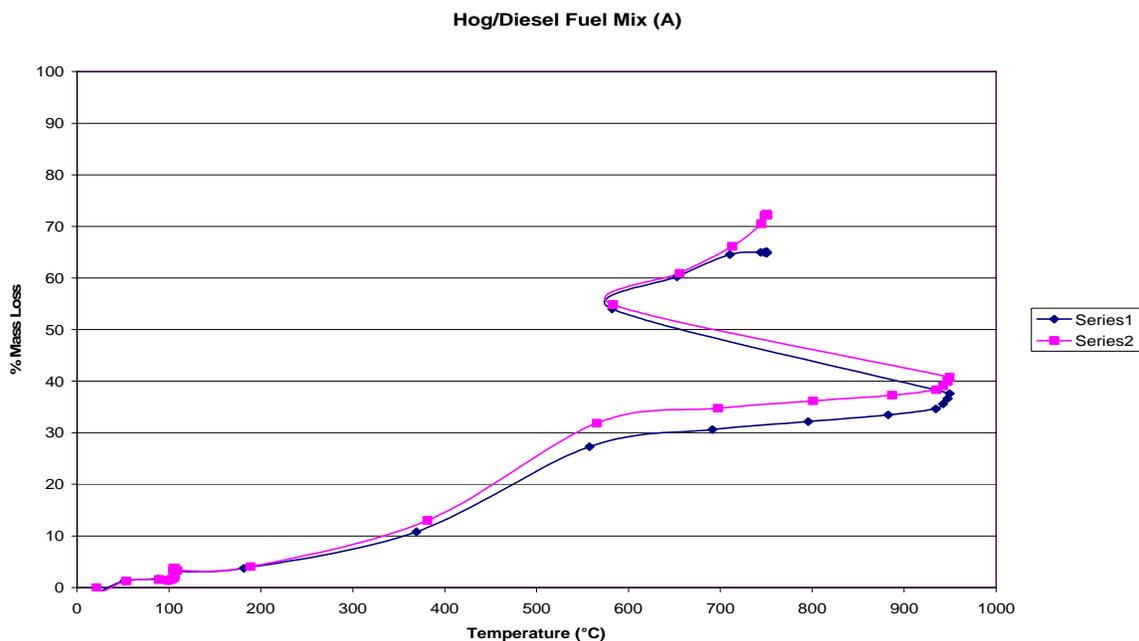
Hog/Diesel Fuel Mix (A)

Figure 5. Hog manure and diesel fuel mix.

3.3 Foam and Fiber Synthesis

Foam Table Manufacture, Troubleshooting, and Operation

A pilot scale foaming/coking furnace has been constructed that will enable the Carbon Products Group to make foam samples 1.0 m by 1.0 m by 2 cm and to make coke in batches of approximately 200 lbs in a run. The furnace is reasonably sealed with minor leaks around the gasket material at elevated temperatures. A ventilation system is being designed to suck up the material that leaks and prevent it from entering the air within the high bay.

Small scale tests have been done to determine the proper protocols to use when using the new foam table. Very slow foaming at 0.1°C/min results in a phase separation within the foam sample. The denser mesophase portion is believed to settle to the bottom of the matrix, resulting in a dense foam with very small pores, while the less dense material forms relatively large pores. It appears that devolatilization at approximately 475°C, followed by a period of oxidation will produce reasonably good foam at atmospheric conditions.

Fluidity and expansion (dilation) data were taken for several types of coal for use in continuous carbon foam production tests.
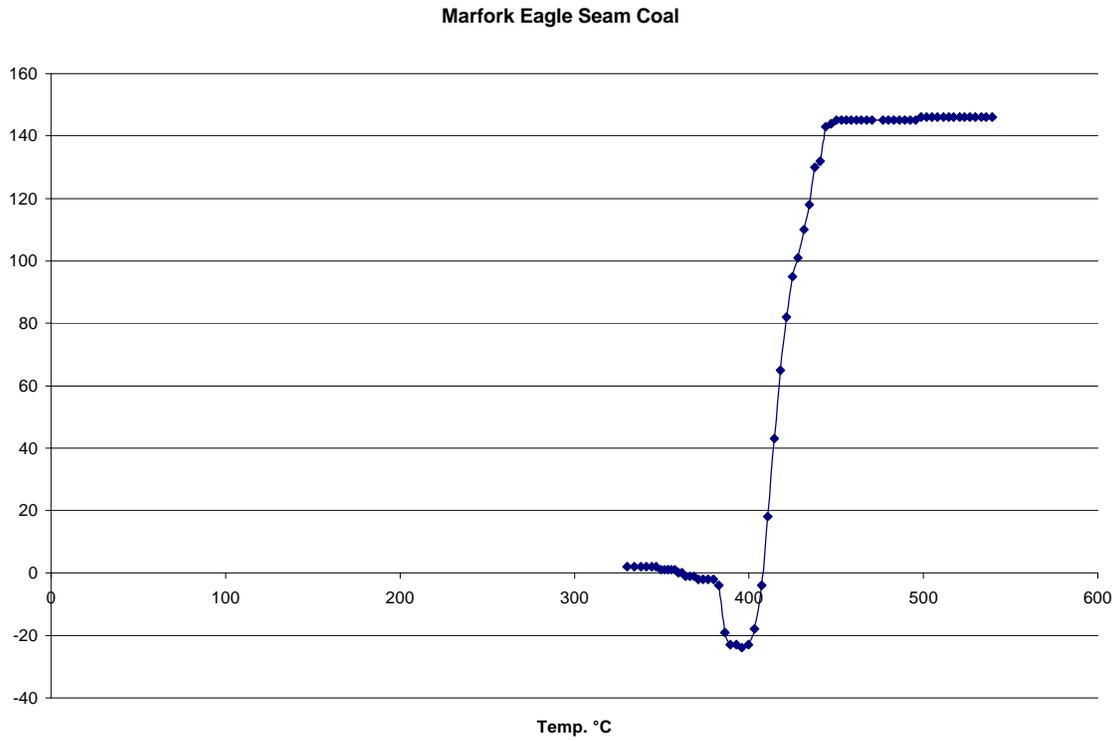
Marfork Eagle Seam Coal

Figure 6. Dilation (percent elongation) versus temperature for Marfolk coal.
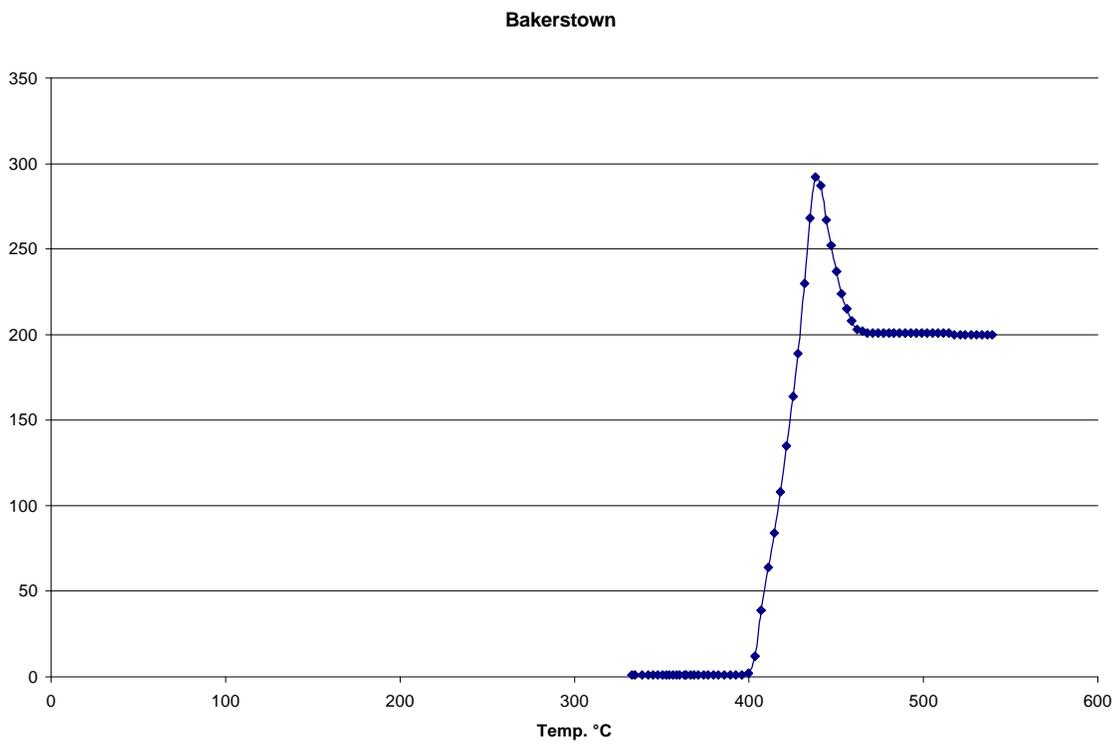
Bakerstown

Figure 7. Dilation (percent elongation) versus temperature for Bakerstown coal.

20

Box furnace for coal-derived carbon fiber processing.

The high temperature box furnace is used for the processing of coal-derived carbon fibers during the carbonization step. The carbonization step in processing the stabilized carbon fibers requires an atmosphere of nitrogen in the sample heating chamber. To date, fibers have not been successfully calcined in this oven due to excessive leakage of oxygen into the sample chamber. The following steps have been taken to remedy this problem:

- Installation of a higher capacity rotometer for increased nitrogen flow.
- Sealing of the furnace chamber top exhaust port.
- Sealing around the furnace chamber nitrogen entrance port.
- Adjustment of the furnace door.

The furnace is still allowing excess oxygen into the sample area. The next step is to construct a gas restricted internal sample processing box that will fit inside the heating chamber.

3.4 Characterization of carbon and graphite via X-Ray Diffraction

NIST standards (LaB_6 and Si) for the Panalytical XRD were analyzed to benchmark its capabilities. The results are shown in Table 21 and 22.

Table 21. X-Ray Diffraction Data using LaB_6 Samples

LaB6 Standard			LaB6 A	LaB6 B	LaB6 C	Averages	LaB6 A	LaB6 B	LaB6 C
Pos. [°2Th.]		Peak No.	Pos. [°2Th.]	Pos. [°2Th.]	Pos. [°2Th.]	Pos. [°2Th.]	%Error	%Error	%Error
21.3578		1	21.3584	21.3578	21.3577	21.3580	0.00281%	0.00000%	-0.00047%
30.3847		2	30.3820	30.3849	30.3850	30.3840	-0.00889%	0.00066%	0.00099%
37.4417		3	37.4444	37.4419	37.4413	37.4425	0.00721%	0.00053%	-0.00107%
43.5064		4	43.5067	43.5066	43.5065	43.5066	0.00069%	0.00046%	0.00023%
48.9573		5	48.9564	48.9577	48.9569	48.9570	-0.00184%	0.00082%	-0.00082%
53.9886		6	53.9874	53.9888	53.9882	53.9881	-0.00222%	0.00037%	-0.00074%
63.2182		7	63.2189	63.2194	63.2182	63.2188	0.00111%	0.00190%	0.00000%
67.5474		8	67.5467	67.5480	67.5466	67.5471	-0.00104%	0.00089%	-0.00118%

Table 22. X-Ray Diffraction Data using Silicon Samples

Si Standard			Silicon 1	Silicon 2	Silicon 3	Averages	Silicon 1	Silicon 2	Silicon 3
Pos. [°2Th.]		Peak No.	Pos. [°2Th.]	Pos. [°2Th.]	Pos. [°2Th.]	Pos. [°2Th.]	%Error	%Error	%Error
28.4409		1	28.4408	28.4409	28.4409	28.4409	-0.0004%	0.0000%	0.0000%
47.3003		2	47.301	47.3002	47.3003	47.3005	0.0015%	-0.0002%	0.0000%
56.1193		3	56.1185	56.1194	56.1193	56.1191	-0.0014%	0.0002%	0.0000%
69.1261		4	69.1263	69.1261	69.1261	69.1262	0.0003%	0.0000%	0.0000%

GrafTech graphite samples are being analyzed in work toward developing an ASTM method for characterizing the crystallite dimensions of graphite. These round robin of tests involve GrafTech, Penn State University, Oakridge National Laboratories, and Alfred University.

Mineral samples containing $NaCO_3$ and CaO from NETL (Morgantown) are being analyzed. Peak position, crystallite sizes and quantitative analysis will be conducted on these samples. Quantities of lab grade NaCO3 and CaO have been acquired to perform the quantitative analysis of these samples.

Physical testing was performed for the following:

Graphite embedded thermoplastic coupons were tested for strain versus electrical resistivity.

Thermoplastics embedded with clay and Kevlar fibers were tested by extension and three point bending.

Plastics embedded with nanofibers were tested via the three point bending test.

4.0 CONCLUSION

Discussions with Alcoa lead to the possibility of enhancing the capabilities of this project. Alcoa has supported WVU efforts in solvent extraction for many years, and has been responsible in no small way for the success of the pilot operation. Among other things, Alcoa has donated a calcining furnace, ceramic lined continuously stirred tank reactor, centrifuge and other equipment to the program. Accordingly, it is recommended that Alcoa be included in the present program as an Industrial Advisor.

5.0 REFERENCES

None