DEVELOPMENT OF CONTINUOUS SOLVENT EXTRACTION PROCESSES FOR COAL DERIVED CARBON PRODUCTS

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DISCLAIMER:

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. A process has been developed which results in high quality binder pitch suitable for use in graphite electrodes or carbon anodes. A detailed description of the protocol is given by Clendenin.[1] Briefly, aromatic heavy oils are hydro-treated under mild conditions in order to increase their ability to dissolve coal. An example of an aromatic heavy oil is Koppers Carbon Black Base (CBB) oil. CBB oil has been found to be an effective solvent and acceptably low cost (i.e., significantly below the market price for binder pitch, or about $280 per ton at the time of this writing). It is also possible to use solvents derived from hydrotreated coal and avoid reliance on coke oven recovery products completely if so desired.

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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, specifications were developed for the major types of pitches to be developed in this effort, including binder pitch, impregnation pitch, needle coke and anode coke.

2.0 TECHNICAL

2.1 Development of a Continuous Process

A process has been developed which results in high quality binder pitch suitable for use in graphite electrodes or carbon anodes. A detailed description of the protocol is given by Clendenin.[2] Briefly, aromatic heavy oils are hydro-treated under mild conditions in order to increase their ability to dissolve coal. An example of an aromatic heavy oil is Koppers Carbon Black Base (CBB) oil. CBB oil has been found to be an effective solvent and acceptably low cost (i.e., significantly below the market price for binder pitch, or about $280 per ton at the time of this writing). It is also possible to use solvents derived from hydrotreated coal and avoid reliance on coke oven recovery products completely if so desired.

Typical hydrogenation conditions are 375 ^{o}C temperature with 500 - 750 psig cold starting pressure. Pressure at peak temperature is controlled by the vapor pressure of the aromatic heavy oil, and is generally less than 1000 psi for oils used in WVU reactors to date. The process requires approximately one hour, plus cool-down time. The amount of hydrogenation is nominally 0.3% by weight, considered to be a quite mild condition compared to other direct coal liquefaction processes (most of which has been directed at producing synthetic crude oil. Syncrude requires much higher temperature and pressure).

The second step is to combine the hydrogenated solvent with coal, which is digested at nominal conditions of 425 ^{o}C and 0-1200 psi. Referring to this process as a "digestion reaction" is an oversimplification. In reality, the process likely transfers some of the hydrogen to the coal, resulting in a complex combination of chemical reactions as well as dissolution. Solids must then be removed from the combined solvent and coal solution, either by centrifugation or filtration. Solvent recovery and thermal processing can be accomplished via a combination of distillation or air blowing.

To summarize the perceived benefits of the protocol compared to previously piloted processes,

i. The cost of the solvent is about $150 per ton, whereas previously studied solvents were about ten times more expensive.

ii. Coal is extracted at about 90% by weight compared to about 60% for other coals tested prior to ~2003.

iii. The level of hydrogenation required is about 0.3% compared to earlier estimates of 1.0%.

iv. Only about 85% of the solvent needs to be recovered, versus 99% for earlier processes. Chemically, a fraction of the solvent can be left in the final product, and actually enhances the material properties.

v. If desired, it is possible to accomplish this process with zero makeup solvent, because the final product can be re-hydrogenated and used as a solvent.

vi. Based on the above, the material costs can be under $100 per ton versus the commodity selling price of $280 per ton for binder pitch, indicating that this can be a potentially viable process if processing costs are reasonable.

2.1.1 Hydrotreating

Although at the beginning of this effort there had been sentiment to seek means to avoid hydrogenation by using high performance solvents at lower temperature, the experimental data strongly supports the use of hydrogenation as the best method to increase the solubility of coal. Based on the above rationale, the lower-temperature blending concept has been largely abandoned in favor of the hydrogenation-based protocol. Essentially the trade-off is achieving greatly reduced material feedstock costs while accepting higher temperature and higher pressure processing conditions.

For the Pilot Plant to adequately serve its function; i.e., to demonstrate these laboratory-scale processes using representative technologies that industry would use, then some modifications are needed in the current system. Specifically, the CSTR is limited to 200 ^{o}C, with pressure in the CSTR limited to about 350 psi, which may be too low to carry out the coal digestion reaction as it has been demonstrated on the laboratory scale.

Although a significant consideration is to utilize as many existing unit process components as possible in order to minimize the amount of equipment money required, it nevertheless is worthwhile to consider how the pilot plant might be configured in the absence of constraints. Accordingly, an idealized process diagram is shown in Figure 1, with a block diagram of pilot line equipment in Figure 2. The main product for this project is binder pitch. However, it is recognized that other potential product streams exist as well. Specifically, solid materials separated from the hydrogenated coal solution can be coked, also potentially producing a tar as a byproduct. Alternatively, this material could be gasified to produce a syngas, which in turn might be used for liquid fuels or other indirect liquefaction products. In addition, during the distillation step, the lower boiling point liquids could be recycled as solvent, or recovered as a separate product. For this project, we assume that the low boiling point liquids would be recycled and re-used as coal solvents, while recognizing the possibility that other applications may also exist. In particular, laboratory scale experiments seek to determine whether some of the distillation products may be useful additives for producing gasoline or diesel additives.

There are practical constraints which force compromises in the idealized process. Most importantly, from the standpoint of safety, it was decided early on that the hydrogenation and digestion process would be carried out separately from the Pilot Plant. Originally it had been intended to accomplish solvent hydrogenation in the Hydrotreater Facility. Unfortunately, the extent to which the entire facility would have to meet code requirements for handling gaseous hydrogen around high temperature apparatus made that option unattractive. For that reason, the hydrogenation and liquefaction steps are currently performed separately in batch processes. Demonstrating the coal liquefaction process in a continuous production mode becomes the logical next step for the Pilot Plant. Direct feeding of coal would have been a useful step had the blending concept prevailed. However, because our setup is limited to batch production of hydrogenated solvents, coal will be fed in slurry form from a holding tank.

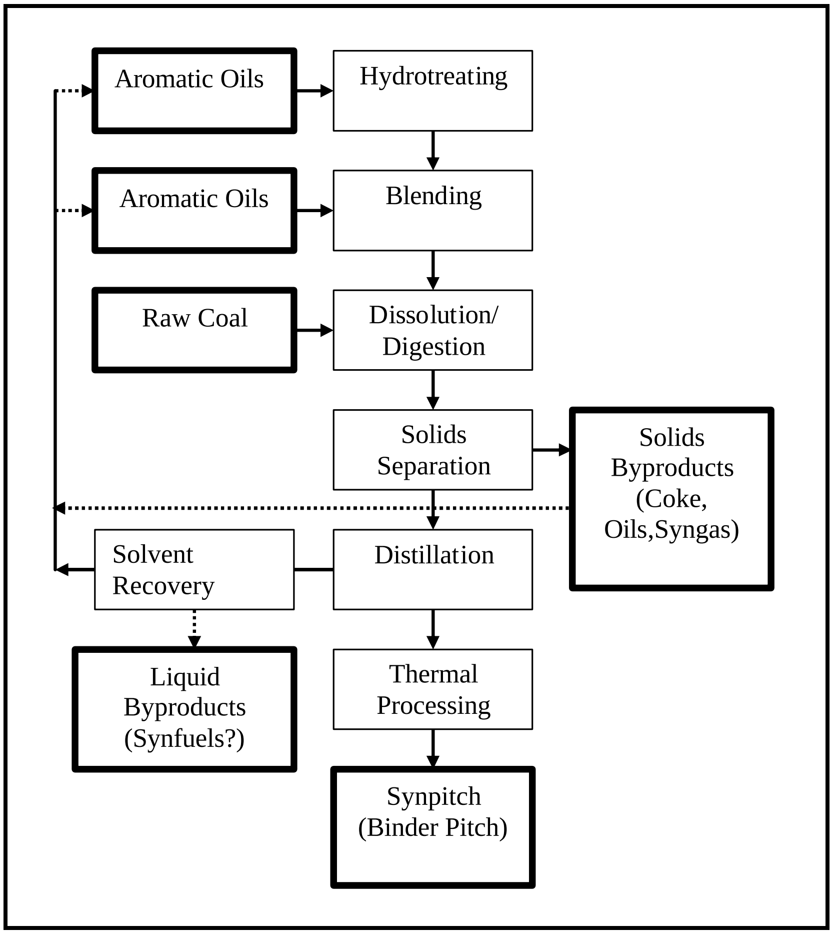

Figure 1. Process Diagram for Producing Synthetic Binder Pitch.

Referring to the equipment block diagram in Figure 2, most of the major pieces of equipment already exist. The main difference is that coal and solvent will be fed in slurry form to a hydrogenation unit. The slurry pump must be capable of producing high pressure while also handling a large amount of particulates (for example, typical coal/solvent ratios are 1:2).

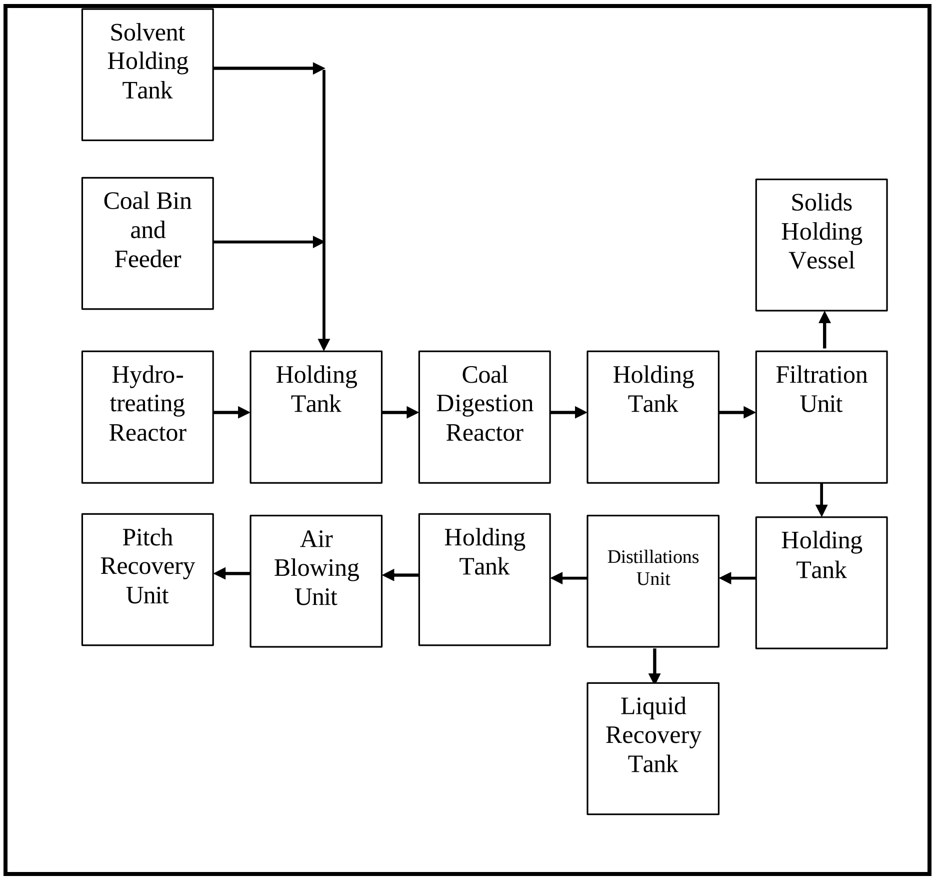

Figure 2. Major equipment block diagram.

A key item is the addition of a high temperature, high pressure digestor. The nominal requirement is to produce 55 gallons of pitch product per eight hours of facility operation. There are a few options being studied for this purpose.

a. One option is to convert an existing autoclave reactor, capable of up to 3300 psi at 375 ^oC. This would be sufficient to carry out hydrogen digestion, although extraction efficiencies will be somewhat lower compared to laboratory trials, most of which have been carried out at 400 ^oC – 425 ^oC. The autoclave reactor would need a magnetic stirrer as well as an additional feedthrough to accept the feed from a slurry pump, if operated continuously.

b. A second option under consideration is some variant of a pipe reactor design, based upon 1” nominal pipe reactor design using Stainless Steel. Compression fittings (e.g., Swagelok or other manufacturer) are suitable for up to 3000 psi, well in excess of the requirements for this task. Because the temperatures exceed the limitations of oil heating, electric band heaters will be used. The slurry will flow through approximately 25 separate pipes, approximately ten feet in length. At the present time, a decision has not been made whether to manifold the pipes together or to unite them in a single flow path. In the former case, the potential for clogging is reduced because the flow path is linear and no u-bends are present. However the flow velocity is only 10 feet per hour, resulting in concern about the uniformity of flow in all the pipes. Conversely in the latter case, the flow velocity would be 250 feet per hour, eliminating concerns about flow velocity but elevating the concern that solid residue could clog the pipes. Both cases result in the need for about 21 kilowatts of electric power, based upon 50% thermal efficiency, which is likely conservative. Analysis will determine whether internal convection and conduction is sufficient to assure a reasonably isothermal condition within the secondary containment. Similarly, the entrainment of solid particles within the liquid will be studied to determine whether a critical velocity exists such that all particulates can be suspended, and thus avoiding clogs. Still, these results will have to be interpreted in the context of actual plant operation, which would involved planned and unplanned shutdowns. Hence scenarios involving settling of particulates must be considered. The power and flow requirements may cause us to consider a smaller scale design, at least until such time as we can convince ourselves that the solid phase inventory can be successfully managed. In any case, local approval may be needed if changes are required in our Chemical Hygiene Plan (CHP).

2.1.2 Kinetics Measurements

Kinetics measurements are essential in order to determine the material throughput requirements and processing equipment capacities.

The simulation project has been initiated and preliminary data is being collected. An overall process basic Block Flow Diagram was constructed based on the process illustrated in Figure 2. Efforts were focused on obtaining kinetic data for a ChemCad® simulation.

Process Description:

a. 5 Gallon Stirred Tank Reactor. This reactor partially hydrogenates creosote oil using Hydrogen gas. The tank is filled with four gallons of Creosote Oil and then the head space is purged with H_2 (g) for about 5 minutes. The reactor is then pressurized to 500-950 psi and is heated to 350-375 °C by an electric heating sleeve for one hour. At reaction temperature the pressure is around 1200 psi. The reactor employs a Ni-Mo catalyst on an Al_2O_3 substrate which is suspended in the reactor by a basket. The reactor effluent, which is partially hydrogenated creosote, hereinafter “solvent”, is drained into a holding tank.

b. 10 Gallon Heated Mixing Vessel. This vessel mixes solvent and crushed, dried coal in a 2:1 ratio by weight with a total volume of nine gallons. Kingwood Coal, which is 8.92 wt.% ash, will be used in the process. The composition of dry, ash-free Kingwood Coal is detailed in Table 1.

Table 1. Composition of Dry, Ash-Free Kingwood Coal.

Element	Weight %
Carbon	77.44
Hydrogen	4.95
Sulfur	1.58
Nitrogen	1.18
Oxygen	14.85

The crushing and drying will be performed by the supplier for the pilot plant operation. However, a scaled-up operation may include these steps. The mixing vessel is heated to 350 °C by an electric sleeve. The slurry is then pumped to into the one gallon STR. A N_2 (g) head pressure assists the pump.

c. One Gallon Stirred Tank Reactor. The reactor digests coal into a binder pitch precursor. The overall reaction is shown in Equation 1.

$$\text{Coal + Solvent} \xrightarrow{\qquad} \text{Digested Pitch Precursor + Unconverted Coal} \qquad \textit{Eq. (1)}$$

The reaction is time is 1 hour with a temperature of 425 °C and pressure of 1200 psi. The conversion is expected to be 90%. The reactor effluent feeds is pumped into a heated holding tank.

d. *Centrifuge*. The centrifuge separates the unconverted coal from the product. More details to come.

e. Wiped Film Evaporator. The evaporator separates the solvent and the binder pitch precursor. The recovered solvent will be recycled into the 10 gallon mixing vessel or the 5 gallon STR. The set up of the recycle loop is yet to be determined. This will depend on the percent hydrogenation of the solvent after the reaction. The solvent recovery is expected to be 90%. The boiling point of the solvent at 1 atm is 315-355 °C.

2.1.3 Air Blowing

Work has been undertaken to produce air-blown pitch on a pilot scale. The original design is shown in Figure 3.

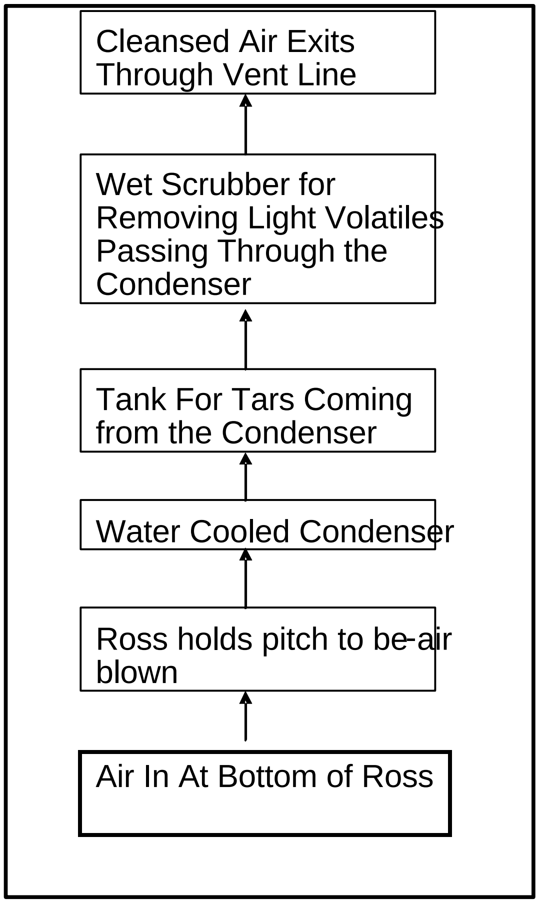

Figure 3. Original Air-Blowing Setup

With this design, air-blowing proceeded relatively slowly. Several weeks were required to increase the coke yield to 17%. Part of the slow progress was the result of the vent lines clogging with condensed tar. The clogging problem was exacerbated by limited pressure rating of the Ross Mixer. At approximately 3 psi, the Ross would begin to vent into the high bay. Another limiting factor in the rate of air-blowing was the size of the bubbles coming into the Ross. Relatively large bubbles entered the Ross because of its setup and there was no practical way to disperse the bubbles to increase the surface area to speed up the oxidation reaction.

Because of the difficulties experienced with the original design, a modified air-blowing setup was developed as shown in Figure 4. The main difference is a dedicated air-blowing reactor. The air-blowing reactor is a 4 inch tube filled with heated pitch that is circulated through the reactor from bottom to top. At the bottom of the reactor is an inlet port for air. The inlet port is covered with a screen to create a multitude of tiny bubbles, thus providing a large amount of surface area for the oxidation reaction.

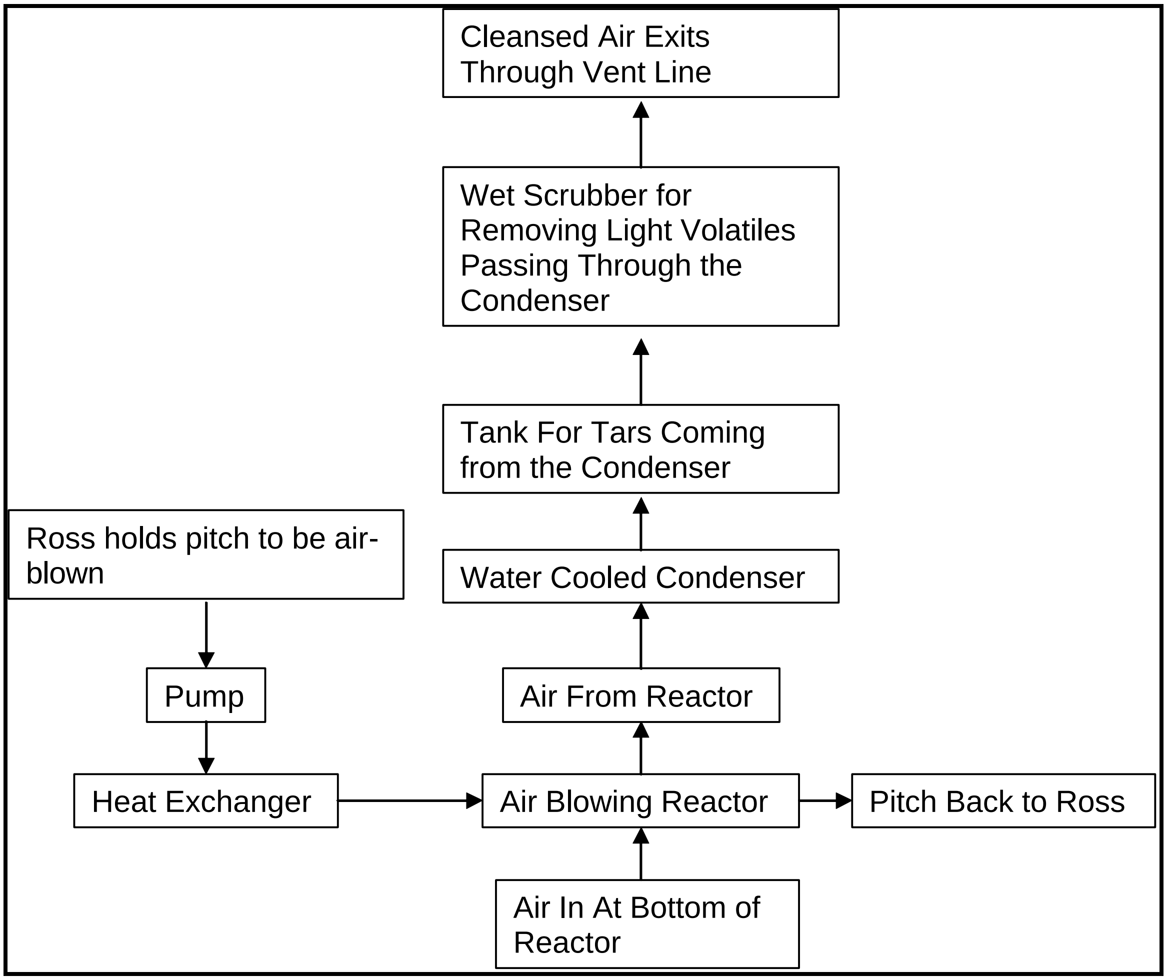

Figure 4. Modified Air-Blowing Process

With this setup the rate of air blowing increased. While the process was working properly, the softening point was increased to 39°C in approximately 16 hours of air-blowing. Unfortunately, the line carrying pitch back to the Ross clogged and the pitch was pumped into the tank for the tars coming from the condenser. At the point that the Ross was almost empty, the softening point of the remaining pitch was 120°C. Another problem was also encountered. With the softening point of the pitch, the pump no longer would turn, even with heat tape around the pump head. Thus, manufacturing high softening point pitch (or even relatively moderate softening point pitch) is not possible with this pump. For this reason, the possibility of purchasing a heated head pump is being investigated. As part of establishing the specifications for the heated head pump, the maximum target softening point should be established. A modified air-blowing setup has also been designed that should allow for rapid air blowing without associated plugging problems.

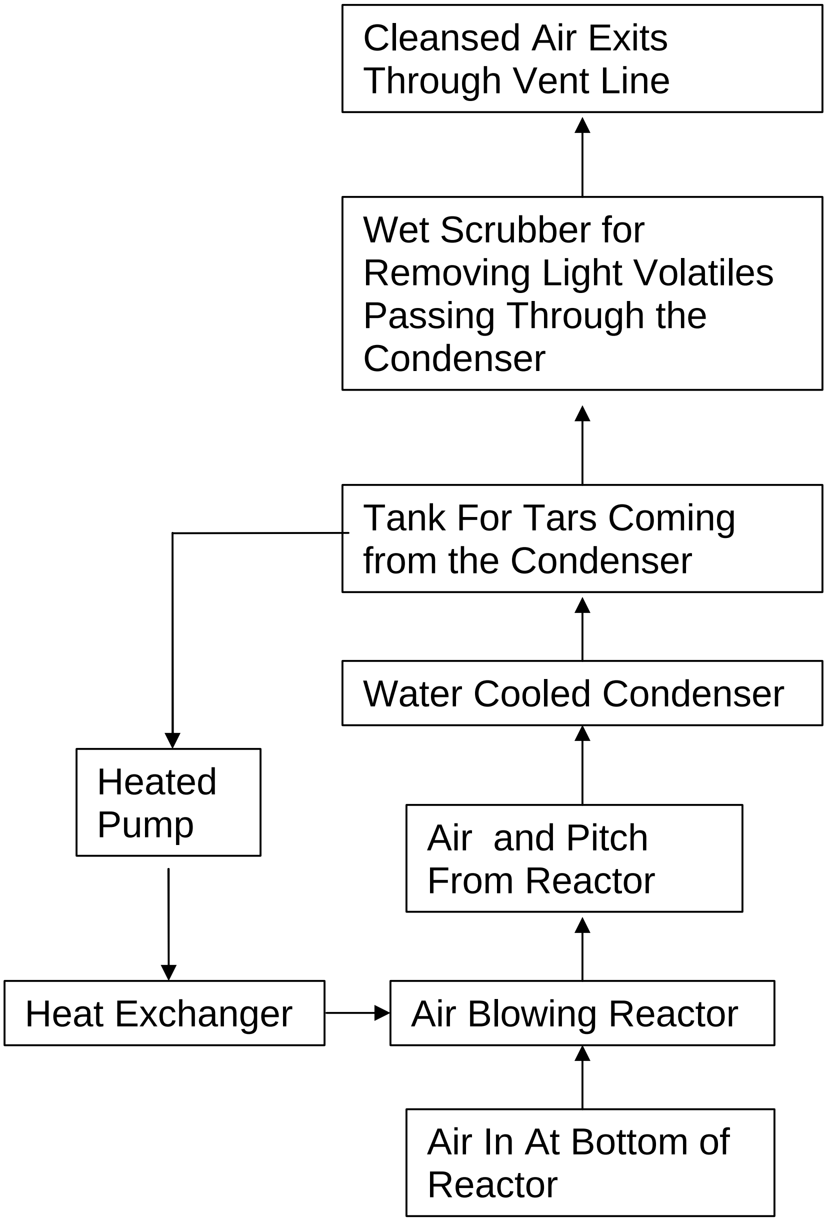

Figure 5. Anticipated Air Blowing Setup

2.1.4 Pilot Plant Instrumentation

A National Instruments PCI 6033E multifunction data acquisition card was ordered. The card is currently being set-up and tested. The next step will be to set up a program to monitor general inputs to the card. After that, the card will be connected to live thermocouples installed throughout the Pilot Plant.

2.2 Carbon Product Manufacture

2.2.1 Production Definition

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

Table 2. Binder Pitch Specification

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

Table 3. Additional GrafTech Binder Pitch Characteristics

Property	**Comment**
Storage Stability @ 160 °C	Long term storage is required at 160 °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 4. 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 5. 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 6 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 6. 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

2.2.2 Qualification of a Synthetic Binder Pitch Using Solvent Extraction

No data this reporting period.

2.2.3 Foam and Fiber Synthesis

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 currently being fitted with the necessary heaters and being connected to the electrical supply. Work is also ongoing in an effort to seal the furnace so that volatiles do not escape and instead pass through the scrubbing system to remove them before the inert gas/volatile mixture is released into the atmosphere.

3.0 RESULTS AND DISCUSSION

3.1 Carbon Foam for Armor Applications

Preliminary tests were carried out on carbon foam to determine whether it could be useful as part of personnel armor (“bulletproof vest) strategy. These tests represented our first attempt at proof of concept for this application. Literature review indicates that there are a number of personnel armor products which are available to the military and public servants. In many cases, such devices are also available on the open market.

The term “bulletproof vest” is often used by the popular media to describe personnel armor, but this is a misnomer. In fact, there is no such thing as a completely "bulletproof" material. Any vest material can be destroyed by a combination of sufficiently powerful rounds and repeated shots. Moreover, even if the bullet is stopped by the vest, a substantial amount of the bullet's momentum and kinetic energy are transferred to the target. Table 7 summarizes the National Institute of Justice Ratings for personnel armor. In order to pass the test, the armor should not only stop the bullet, but also limit the blunt trauma to the target. Blunt trauma is measured by the indentation suffered by a soft clay backstop to the vest – a maximum of 1.7" *(44 mm)* is allowed.

Samples were prepared by attaching Kevlar sheets to carbon foam samples with different geometries as listed in Table 8. Face sheets were attached to the front and back of the samples.

Table 9 summarizes the types of weapons which were used.

Table 7. National Institute of Justice Standard Rating for Personnel Armor.[3]

Protection Level	Caliber	Projectile Description	Weight (Grains)	Velocity (Ft/sec)
I	.22 Long Rifle	Lead	40	1080
I	.380 ACP	Full Metal Jacket	95	1055
II-A	9 mm	Full Metal Jacket	124	1120
II-A	.40 S&W	Full Metal Jacket	180	1055
II	9 mm	Full Metal Jacket	124	1205
II	.357 Magnum	Jacketed Soft Point	158	1430
III-A	9 mm	Full Metal Jacket	124	1430
III-A	.44 Magnum	Jacketed Soft Point	240	1430
III	7.62 mm NATO	Full Metal Jacket	138	2780
IV	.30-06	Armor Piercing	166	2780

Table 8. Carbon Foam Sample Matrix.

Designator	Short Name	Foam layers - ***F***	Foam thick-ness (inches)	Kevlar layers - ***K***	Steel layers - ***S***	Poly-urethane Vol % - ***P***	Poly coating -***C***	PL - ***PL***	No
F2-0.50-K2-S0-P0-C1-PL0-070204	Sandwich 2	2	0.50	2,2,2	0	0	1	0	1
F2-0.50-K4-S0-P0-C1-PL0-070204	sandwich 4	2	0.50	4,4,4	0	0	1	0	
F1-0.25-K4-S0-P100-C1-PL0-070204	impregnated ¼ inch (100%)	1	0.25	4,4	0	100	1	0	
F1-0.50-K4-S0-P30-C1-PL0-070204	impregnated ½ inch (30%)	1	0.50	4,4	0	30	1	0	
F1-1.00-K4-S0-P30-C1-PL0-070204	Impregnated 1 inch (30%)	1	1.00	4,4	0	30	1	0	
F1-1.00-K6-S0-P30-C1-PL0-070204	Impregnated 1" 30%	1	1.00	6,6	0	30	1	0	1
F1-1.00-K6-S0-P30-C1-PL0-070204	Impregnated 1" 30%	1	1.00	6,6	0	30	1	0	2
F1-0.25-K2-S0-P0-C1-PL0-070204	Poly 2, ¼ inch	1	0.25	2,2	0	0	1	0	
F1-0.50-K2-S0-P0-C1-PL0-070204	Poly 2, ½ inch	1	0.50	2,2	0	0	1	0	
F1-1.00-K2-S0-P0-C1-PL0-070204	Poly 2, 1 inch	1	1.00	2,2	0	0	1	0	
F1-0.25-K4-S0-P0-C1-PL0-070204	Poly 4, ¼ inch	1	0.25	4,4	0	0	1	0	
F1-0.50-K4-S0-P0-C1-PL0-070204	Poly 4, ½ inch	1	0.50	4,4	0	0	1	0	
F1-1.00-K4-S0-P0-C1-PL0-070204	Poly 4, 1 inch	1	1.00	4,4	0	0	1	0	
F1-0.25-K6-S0-P0-C1-PL0-070204	Poly 6, ¼ inch	1	0.25	6,6	0	0	1	0	
F1-0.50-K6-S0-P0-C1-PL0-070204	Poly 6, ½ inch	1	0.50	6,6	0	0	1	0	
F1-1.00-K6-S0-P0-C1-PL0-070204	Poly 6, 1 inch	1	1.00	6,6	0	0	1	0	
F1-0.50-K4-S0-P0-C0-PL1-070204	PL 4, ½ inch	1	0.50	4,4	0	0	0	1	1
F1-0.50-K4-S0-P0-C0-PL1-070204	PL 4, ½ inch	1	0.50	4,4	0	0	0	1	2
F1-1.00-K4-S0-P0-C0-PL1-070204	PL 4, 1 inch	1	1.00	4,4	0	0	0	1	
F1-0.50-K6-S0-P0-C0-PL1-070204	PL 6, ½ inch	1	0.50	6,6	0	0	0	1	1
F1-0.50-K6-S0-P0-C0-PL1-070204	PL 6, ½ inch	1	0.50	6,6	0	0	0	1	2
F1-0.50-K6-S0-P0-C0-PL1-070204	PL 6, ½ inch	1	0.50	6,6	0	0	0	1	3
F1-0.50-K6-S0-P0-C0-PL1-070204	PL 6, ½ inch	1	0.50	6,6	0	0	0	1	4
F1-1.00-K6-S0-P0-C0-PL1-070204	PL 6, 1 inch	1	1.00	6,6	0	0	0	1	
F1-1.00-K0-S1-P0-C1-PL0-070204	Steel 1, 1 inch	1	1.00	0	1,1	0	1	0	
F1-1.00-K2-S1-P0-C1-PL0-070204	Steel 1, Kevlar 2, 1 inch	1	1.00	2,2	1,1	0	1	0	
F1-1.00-K4-S1-P0-C1-PL0-070204	Steel 1, Kevlar 4, 1 inch	1	1.00	4,4	1,1	0	1	0	
F1-0.50-K4-S0-P0-C1-PL0-070204	Disk	1	0.5	4,4	0	0	1	0	

Table 9. Rifle and Ammunition Summary

Rifle	Ammunition	Diameter	Muzzle Velocity	Weight	Kinetic Energy
Ruger 10/22	CCI Mini-Mag	22 cal.	1280 ft/sec	36 grains	0.178 kJ
30 Cal Carbine	AE FMJ	30 cal.	1990 ft/sec	110 grains	1.311 kJ
243 Winchester Model 70	Remington Core-Lokt	243 cal.	2960 ft/sec	100 grains	2.637 kJ

Figure 6 shows the basic setup of the target. Test fixtures did not comply with NIJ Standard 0100.04. In particular, our fixtures were smaller than those specified in the Standard, and we used a substitute for the clay specified.

Figure 6. The target setup consisted of a Kevlar/foam sample with a modeling clay backing.

Figure 7. A stable mount was used to ensure accuracy. PhD candidate Jim Bowers provided excellent marksmanship.

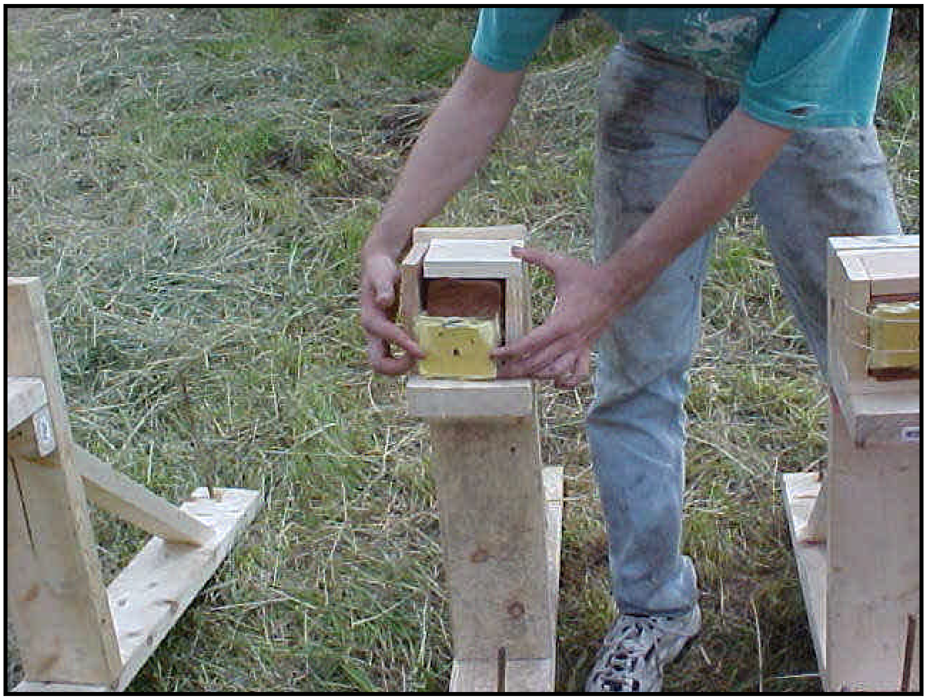

Figure 8. Overview of test setup showing clay behind layered sample.

Figure 9. Shots were taken at short range to ensure a high probably direct hits. The 30 cal Carbine was less accurate since it did not have a scope.

3.2 Ballistics Test Results

It is emphasized that our tests are not fully compliant with NIJ Standard 0101.04. However, the preliminary results showed that carbon foam was successful as part of a layered strategy for armor against the .22 Long Rifle, as shown in Figure 10. The bullet penetrated the front layer of Kevlar, and deformed the back layers. The crater behind the sample was about 0.8" deep.

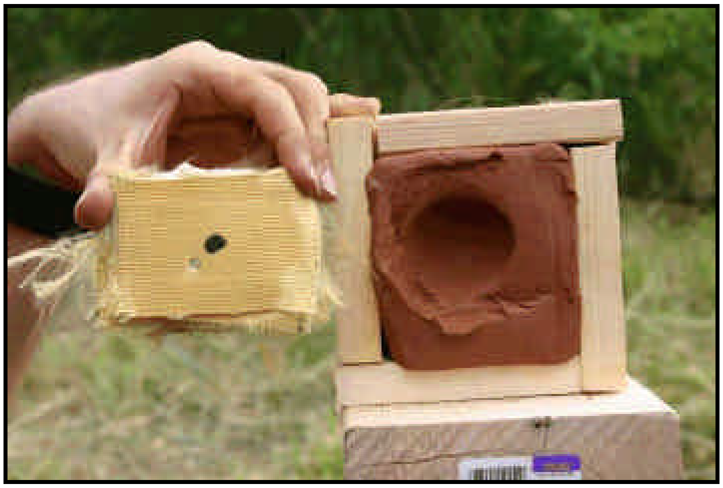

Figure 10. A single ½" thickness of carbon foam backed by Kevlar face sheets was successful at stopping a bullet from a .22 long rifle. The impact crater in the clay was about 0.8" deep and was considered a success.

Subsequently, our team was emboldened and carried out tests using the 243 Winchester Model 70, using sample F1-1.00-K6-S0-P30-C1-PL0-070204. No photographic record can be displayed as the target literally exploded on impact, scattering wood and clay over a large area and reducing the sample to a few stray wisps of airborne yellow fiber. Pieces of the bullet were found in the clay. Thus the foam did manage to reduce the speed of the bullet such that it could not make it entirely through the wood. Due to qualitative data, the impregnated foam did seem to be relatively successful, especially compared to the amount of foam used in subsequent tests.

We then tested the .243 against a thicker shield of foam. Two samples of F1-1.00-K4-S0-P0-C0-PL1-070204 foam were stacked together in front of the clay target. Again, the box, clay, and foam were scattered, but the foam was able to be recovered.

In order to test more layers of Kevlar, we stacked two layers of F1-1.00-K6-S0-P0-C0-PL1-070204-1 and F1-1.00-K6-S0-P0-C0-PL1-070204-2 which featured a total of 24 layers of Kevlar. As before, the box, clay, and foam were scattered, but the foam was able to be recovered.

Finally, four samples were mounted together using samples F1-0.50-K6-S0-P0-C0-PL1-070204-01, F1-0.50-K6-S0-P0-C0-PL1-070204-02, F1-0.50-K6-S0-P0-C0-PL1-070204-03 and F1-0.50-K6-S0-P0-C0-PL1-070204-04. This meant there were a total of 2" of foam and 48 layers of Kevlar. As before, the box, clay, and foam were scattered, but the foam was able to be recovered.

Tests using the 30 Cal Carbine were attempted despite the fact that it did not have a scope; hence it was more problematic to obtain a direct hit on the samples. In addition, the range could not be shortened owing to the problem of flying debris observed with the 243 Winchester Model 70. Trials using the 30 Cal Carbine using F1-0.50-K4-S0-P0-C0-PL1-070204-01 and F1-0.50-K4-S0-P0-C0-PL1-070204-02 did not produce satisfactory results due to inability to achieve a fair hit on the samples.

3.3 Discussion

The trials indicate that carbon foam can be a promising approach to providing armor protection. This first set of trials will be beneficial for future tests.

One of the key realizations is that the front layers of Kevlar are not effective in spreading the impulse load. Thus, the foam samples do not have enough time to deform and crush energy is not the primary means of dissipating kinetic energy. However, substantial crushing does occur, presumably as a result of the shock wave propagating through the foam. A cone of crushed foam was created in the samples we recovered, narrow at the surface and spreading outward as the bullet penetrated.

Other personnel armor discussed in the open literature has used an ultrahard ceramic to absorb the initial impact of the bullet, and to cause the energy to dissipate in the armor. It is hypothesized that this approach may be useful for carbon foam-based energy absorbers as well. Future tests may investigate this concept.

It was also found that the need to assure direct hits on the target makes larger target sizes very useful. Thus, the next series of tests will use much larger targets.

The use off fillers (either viscous fillers such as silicone, or non-viscous filler such as water) has also been discussed as a means of causing the kinetic energy to be better dispersed within the sample.

In some ways, protecting against a small, high velocity projectile is more difficult than protecting against larger threats such as grenades or debris because the kinetic energy and momentum per unit area is quite high.

Thus, other applications of armor (vehicle armor against grenades, land mines, etc) will be considered in the future.

4.0 CONCLUSION

Following the modifications to the SEPP facility as described above, intermittent production of variations of synthetic pitch materials is planned for purposes including binder pitch, coke precursor pitch, carbon foam precursor pitch and possibly others. Batch mode processes can be used to meet the near-term requirements for subcontractors Koppers and GrafTech to evaluate samples of binder pitch for anode production.

The plan described above is believed to be consistent with the existing Statement of Work and does not result in technical changes. However, some recategorizing of the budget will be needed in order to carry out the experiments planned. No increase in overall cost is expected. Additionally, more rapid than expected progress has occurred in the development of foaming and coking capabilities. Thus it is likely that the overall schedule will not be adversely impacted.

Table 10. Revised Schedule for High Pressure High Temperature Digestor.

Activity	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun
Design	X	X	X								
Entrainment Studies	X	X									
Thermal Modeling	X	X									
Inlet Manifold construction			X	X							
Outlet Manifold Construction			X	X							
Checkout and Testing				X	X						
Binder Pitch				X	X	X	X	X	X		
Coke precursor pitch						X	X	X	X		
Carbon Foam Precursor Pitch								X	X	X	X

5.0 REFERENCES

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[1] L. M. Clendenin, Masters Thesis, 2004.

[2] L. M. Clendenin, Masters Thesis, 2004.

[3] NIJ Standard–0101.04, Ballistic Resistance of Personal Body Armor. See also Howard L. Thomas, Ph.D., “An Overview of Industrial Fabric Ballistic Protection for Police and Military Personnel” Auburn University, Textile Engineering Department.