Coal-based Products from the Co-coking Process
For use in Anodes for Aluminum Electrolysis

Final Report

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Abstract

Co-coking involves the simultaneous co-carbonization of coal and petroleum residua in order to produce coal-based liquids and coke. Co-coking is based around the delayed coking process used in many refinery operations. Previous work has been directed with the intention of producing a coal-based liquid, which may be subsequently refined into an advanced thermally stable jet fuel. During the course of the research interest was developed into the coke product, and its potential use in anodes for aluminum electrolysis and other specialty carbon products. Exploratory, small-scale experiments were conducted at Penn State to evaluate feedstocks, feed composition and reaction conditions. The interactions between the coal and the petroleum resid during co-coking develop interesting phenomena in the composition and morphology of the coke. To date the quantity of products produced in the co-coking studies has only allowed us to perform detailed characterization. In the present study Penn State has scaled-up the co-coking process by constructing a small pilot-scale delayed coker unit. This unit produces sufficient quantities of products to allow further processing and utilization. This will include the hydrotreating of the liquid products in a fixed-bed reactor. The coke has been characterized to determine what affect the co-coking conditions have on morphology and composition. The coke that was produced was then calcined and made into test anodes. The test anodes were compared with conventional anodes made with petroleum coke.

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1.0 Executive Summary

The objective of this study was to produce a coke product from the co-coking of coal and petroleum resid that could be used in the production of anodes for aluminum electrolysis. To this end, blends of cleaned coal and decant oil were fed into a pilot-scale delayed coker under various operating conditions. The resulting cokes were fully characterized using a variety of analytical techniques. Finally, the cokes were made into test anodes and compared with conventional petroleum coke-based anodes.

After a series of set backs with the construction and operational understanding of the pilot-scale delayed coker, a series of tests and a parametric study were performed. This study aimed to establish links between the changes in processing conditions and the quality of the resulting cokes. Traditional and non-traditional image analysis techniques, petrographic analysis and x-ray tomography, respectively, were used to measure some of the compositional and physical attributes of the green cokes. Furthermore, information on product yields, proximate and ultimate analysis were collected.

The resulting cokes were produced in bulk and calcined at 1400 °C. The calcined cokes were sent to Alcoa Inc. to be made into test anodes. The test anodes were sent to Koppers Industries Inc. to be baked. The baked anodes underwent a series of tests to determine their quality relative to a standard test anode, and industry target values.

Results show that in some aspects including density, strength, resistivity, reactivity, the coal-based cokes produced in this study met or exceeded the standards set by the industry. However, due to the presence of residual mineral matter from coal in the coke, the ash values for the coal-based anodes are high.

2.0 Introduction

Petroleum coke is the major constituent of anodes used in the aluminum electrolysis process. It is derived from the thermal cracking of petroleum residua in coking operations. The aluminum industry consumes approximately 1.6 million metric tonnes of petroleum coke annually. The inclusion of heavier oils in the refinery processing stream leads to a decreasing quality (higher sulfur and heavy metals content) of petroleum products, which subsequently leads to a reduction in the quality of the coke products. This is because the sulfur and ash from heavy metals tend to concentrate in the petroleum residua, and subsequently are higher in the coke products after thermal processing. Sulfur in the coke products leads to the formation of SO_2 emissions from the electrolysis cell, which are strongly enforced by the EPA. Heavy metals, such as nickel (Ni) and vanadium (V) are known to catalyze the oxidation of the carbon electrodes in aluminum electrolysis, increasing excess carbon consumption and reducing the useful life of the anode. It is therefore very important to keep the sulfur, Ni, and V concentrations low to improve the quality of the anodes. We believe that this can be achieved by using the products from the co-coking process.

The process of co-coking[1-3] involves the simultaneous co-carbonization of coal and petroleum resid in a delayed coking environment. A slurry of high volatile bituminous coal and decant oil is fed into a delayed coker drum at between 460-500 °C, at pressures between 10 and 150 psig. Under these conditions the coal and decant oil become very fluid and coke to form a homogenous solid product.

In order to produce a product that is suitable for use in anodes for aluminum electrolysis certain criteria must be met regarding the ash and sulfur content of the anodes. By utilizing coal and decant oil with low sulfur contents, it will be possible to keep the sulfur levels in the coke products to a minimum. In preliminary studies we have shown that compared with the processing of individual feeds, using a blend of coal and resid enhances sulfur removal. Additionally, the use of a cleaned coal product will minimize ash derived from the coal. Work already conducted for Penn State by Preptech Inc. has shown that using specific fractions from coal benificiation processes high volatile bituminous coal can be cleaned successfully to approximately 2% ash. One advantage to the co-coking process is that it produces a solid coke product that has less petroleum-derived coke in it. This in turn leads to a lower concentration of Ni and V. The lowering of the Ni and V content of the carbon products reduces catalytic oxidation and hence enhances their working life.

Strength is another factor in the quality of the anodes. The strength characteristics of anodes may be tailored by using a coke product with the appropriate degree of porosity, blending it with the correct amount of coal-tar binder pitch, and processing them under the correct conditions. Additionally, because of the potential interactions between the coal-derived coke and the coal tar pitch binder we believe that we can enhance the strength of the carbon product using the coke derived from the co-coking of coal and petroleum. The coke and the pitch still maintain the physical and chemical characteristics of their parent coals. These characteristics may enhance the wetting properties of the binder pitch through physical attraction. They may also under further processing conditions (e.g. baking) form strong chemical bonds via radical recombination reactions of the aromatic centers within the coal-derived structural units of the coke and binder. Improved wetting and strong chemical bonds may in turn lead to a stronger, more uniform and denser product, which in turn should have a longer lifetime.

Figure 1 shows examples of the type of coke produced under the co-coking conditions (465 °C for 2 hours under an ambient nitrogen atmosphere in batch micro-autoclaves). The first image is of the coal-derived coke processed alone. The second is of the coal-derived coke processed with decant oil. The third is the decant oil processed under co-coking conditions. Results show that during co-coking with coal and decant oil, that the coal has an optical texture with greater similarities to decant oil-derived coke than coal-derived coke. In actuality the co-coked product shows signs of homogeneity, which is very beneficial, in terms of strength, if it is to be used as a product in anode manufacturing.

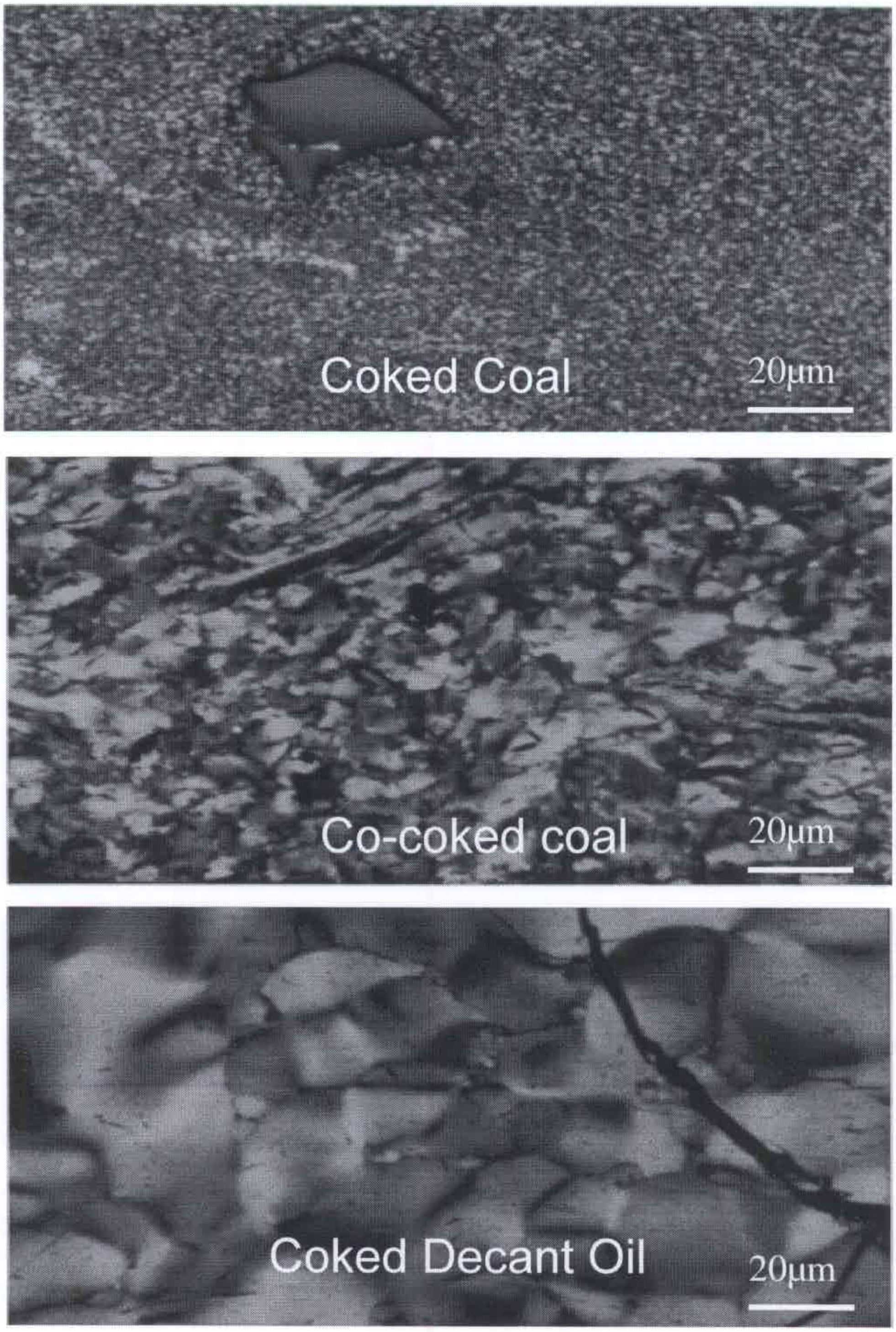

Figure 1. Photomicrographs of coke samples

3.0 Experimental

Table 1 gives a summary of the proximate and ultimate analysis, fluidity, particle size distribution and ash mineral composition of the deep cleaned Powellton coal. Preptech Inc. prepared the coal for this project from froth flotation samples taken from a coal cleaning plant. This operation cleans the coal to remove the majority of the mineral matter, and then Preptech further cleaned the sample using a more thorough separation technique. The resulting coal has an ash content of 1.9%. The major mineral composition in the ash derived from the coal is also listed in Table 1.

Over 70 % of the mineral matter in the coal is derived from clays (e.g. silicates and aluminates). The iron is predominantly iron sulfides (pyrite and pyrrhotite). The coal has a very high maximum fluidity. This is one reason why Powellton coal is desired as a metallurgical coking coal. Additionally, we believe that having a high fluidity in the coker operating temperature range will increase contact time between the coal and decant oil, and so ultimately improve the quality of the coke produced.

Tables 2 and 3 show the ultimate analyses data for the as received decant oil and the coal/oils slurry used in the initial experiments. The values for carbon, hydrogen, nitrogen and sulfur are very similar for both samples. The decant oil has no measurable ash content, but the slurry has a small contribution from the coal that was added at a 10 wt% fraction. The addition of the coal also increases the Conradson carbon number slightly from 7.13 in the as received oil to 8.49 in the slurry.

Table 1. Proximate and ultimate analysis, fluidity, particle size distribution and ash mineral composition of the deep cleaned Powellton coal.

Proximate Analysis [a]	
Fixed Carbon %	68.7
Volatile Matter %	29.4
Ash %	1.9
Ultimate Analysis [a]	
Carbon %	86.3
Hydrogen %	5.2
Nitrogen %	1.5
Sulfur %	0.9
Oxygen % (by difference)	6.1
Fluidity Data [b]	
Fluid Temperature Range (°C)	105
Maximum Fluidity (ddpm)	23,619
Softening Temperature (°C)	385
Particle Size Distribution [c]	
D[v,0.9]	152 μm
D[v,0.5]	61 μm
D[v,0.1]	10 μm
Ash Mineral Composition [d]	
Silicon Dioxide	45.16%
Aluminum Oxide	25.43%
Ferric Oxide	12.68%
Titanium Oxide	5.58%
Phosphorus Pentoxide	0.10%
Calcium Oxide	3.56%
Magnesium Oxide	1.51%
Sodium Oxide	0.34%
Potassium Oxide	2.51%
Sulfur Trioxide	3.49%

[a] Values reported on a dry basis

[b] Determined using a Geisler plastometer

[c] The D (v, 0.9), D (v, 0.5) and D (v, 0.1) values are the particle sizes where, respectively, 90, 50 and 10% of the particles, by volume, are less than the indicated size.

[d] Determined using ASTM D2795 and D3682

Table 2. Ultimate analysis of the as received decant oil.

Ultimate Analysis [a]	
Carbon %	90.7
Hydrogen %	10.3
Nitrogen %	0.2
Sulfur %	0.8
Ash content (wt%)[a]	0.0
Conradson carbon residue	7.13

[a]. Values reported on a dry basis

Table 3. Ultimate analysis of the slurry of 10% Powellton coal in decant oil.

Ultimate Analysis [a]	
Carbon %	89.9
Hydrogen %	9.8
Nitrogen %	0.4
Sulfur %	0.8
Ash content (wt%)[a]	0.2
Conradson carbon residue	8.49

[a]. Values reported on a dry basis

The green cokes in this study were produced in a small pilot-scale delayed coker at The Energy Institute at The Pennsylvania State University. The coke drum has internal dimensions of 7.5cm ID by 102.5 cm high, giving a volume of 4.5 liters. The unit is capable of operating under most delayed coking process conditions. The system pressure, temperature and flow rates are monitored by a number of computer-controlled devices, and data from these devices is recorded throughout the run. In this study the unit was run with and without continuous steam injection, so that the affect of steam on coke texture, coke morphology and product yields could be measured. The following operating conditions were set for the test runs: coke drum inlet temperature 465 °C, coke drum pressure 25 psig, resid feed rate 16.7 g/min, steam feed rate of 2vol% water to feed rate, and length of run 360 minutes. On the conclusion of the run the mass of the liquid condensate was measured. In addition, the height of the coke bed was measured prior to a core being drilled from the center of the bed. The remaining coke was removed and weighed.

Once testing of the system was complete attempts were made to assess the affect of processing conditions on the composition and characteristics of the co-coking products. Multiple runs have been made under the following conditions:

Wt% Coal	Wt% Oil	Inlet temp. (°C)	Coke drum pressure (psig)	Water feed rate*	Sample set
10	90	470	25	2%	A
10	90	470	50	2%	B
20	80	470	25	2%	C
10	90	470	25	1%	D

* The water feed rate is a percentage of the feedstock feed rate. For the purposes of this study the feedstock feed rate was set to 1 kg/hr. The water is used for steam injection.

The cores that were removed from the coke bed underwent extensive image analysis to determine the affect of the changes in run conditions of the texture, pore structure, pore volume and coke density. These measurements were obtained using optical microscopy and x-ray tomography techniques.

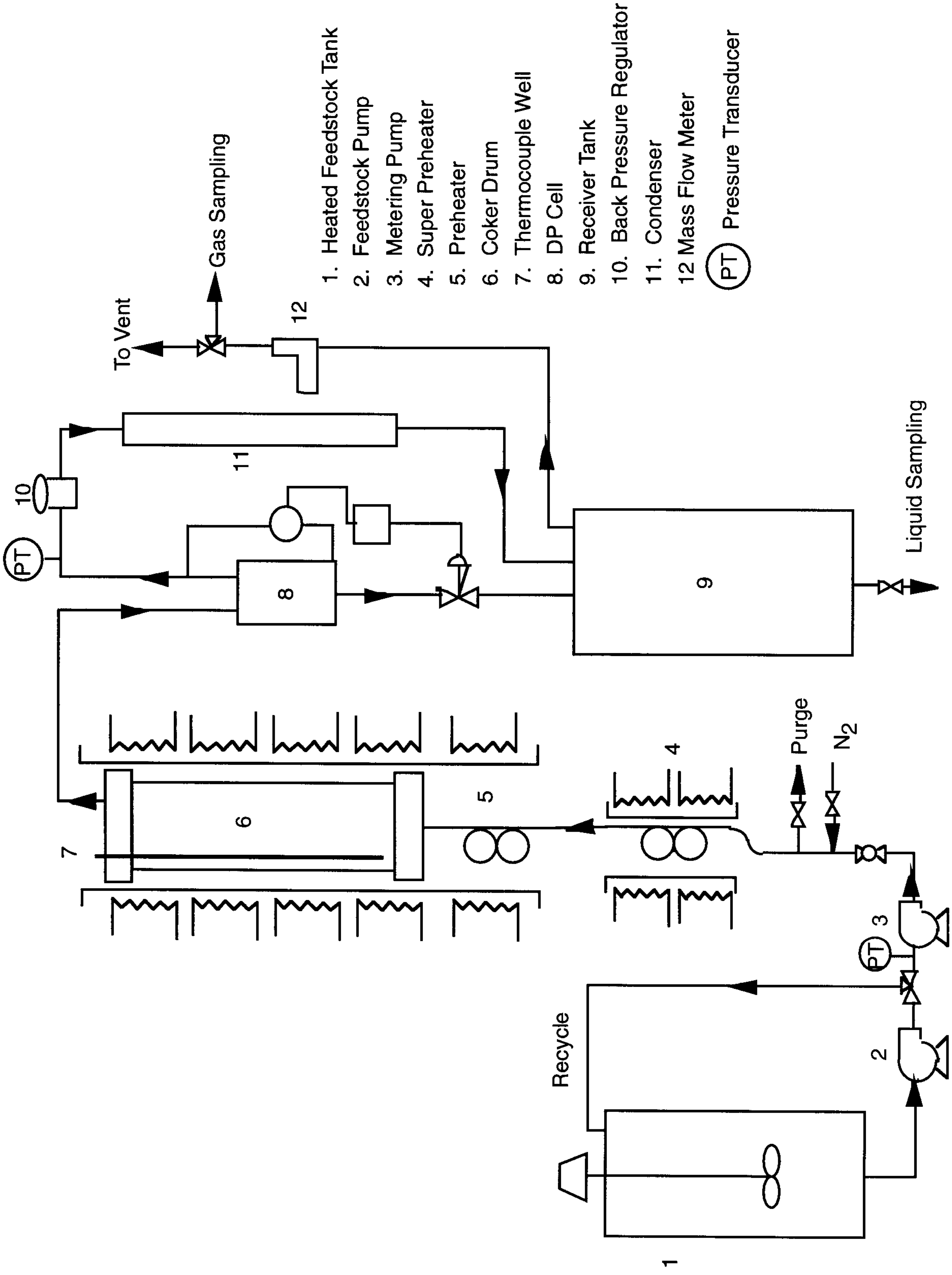

Figure 2. Schematic diagram of Penn State's delayed coker unit.

In general, cores of coke were taken off the centerline of the coke mass in the coker vessel and sometimes removed in 25 mm segments (if the coke was brittle) or as fairly large 40 - 70 mm lengths. Beginning from the bottom inlet and progressing upward in the direction of flow, cores were dense and compact, followed by a generally longer, less-dense middle section and topped by a competent but highly porous top section. Coke material was often found above the top of each core, but was too brittle for coring (no samples were provided).

It was important to include a complete bottom section for study, because it was found that coal-derived material was concentrated at the bottom of each core (at the inlet). Also, from earlier work it was determined that longitudinal sections were far more informative with regard to coke properties than cross-sectional areas. Therefore, when possible longitudinal sections of about 25 mm and oriented in the direction of flow were obtained. In general, subsections of cores were selected to include the bottom inlet area, a competent area from the center and top of each core for comparison. A handsaw was used to cut approximately 25 mm lengths of the core and each length was cut in half to expose a longitudinal surface. These segments were oriented so that the direction of flow would be known. Each core segment was glued into a plastic mold and impregnated with a cold setting epoxy resin by placing them under vacuum several times. Vacuum impregnation effectively forces epoxy to replace the connected air-filled voids in the coke, whereas those voids not connected to the exterior surface remain unfilled. After the epoxy hardened, the oriented surface of each specimen was ground and polished using a series of grit papers (400 and 600 grit) and alumina polishing slurries (0.3 and 0.05 micron).

Image analysis was performed using polarized-light microscopy (PM). All specimens were inspected using a Zeiss Universal research microscope using a 10X air (120 total magnification) or a 16X oil (250 total magnification) or 40X oil (625 total magnification) Antiflex objective with reflected, crossed-polarized white light. Photographs of selected areas of each segment were taken using 200 ASA Elite Chrome color 35 mm slide film. In addition, a point count analysis of the volume percentage distribution of carbon textures was performed on all core segments. This analysis was performed at 625X magnification in oil immersion by traversing the sample based upon a 0.2 x 0.2 mm grid parallel to the direction of flow and identifying the textural element under a crosshair held in a microscope eyepiece. In most cases, a total of 1000 counts were accumulated from each surface and the results (calculated to a volume percentage) are given in Table 9.

Nine different textural (or compositional) elements were used to characterize coke textures made from runs employing either 100% decant oil or blends of decant oil and Powellton coal. These textural elements, based on the size of isochromatic areas and/or shape, are sufficient to characterize the carbon derived from the individual components (decant oil and coal), but are unable to adequately identify the carbon material derived from their interaction. The different elements of this classification are described as follows.

Isotropic – a relatively low reflecting, dark gray carbon material derived from decant oil that displays little or no optical activity when the specimen is rotated under crossed-polarized light.

Mosaic – a higher reflecting carbon textural element derived from decant oil or decant oil/coal interaction that displays optical anisotropy and is characterized by isochromatic units <10 μm or between about 4 - 10 μm when coal is present.

Small Domain – is an anisotropic carbon texture derived from decant oil, which exhibits isochromatic units between 10 – 60 μm in diameter.

Domain – is an anisotropic carbon derived from decant oil having much larger isochromatic units of greater than 60 μm diameter.

Flow Domain – is an anisotropic texture exhibiting elongated isochromatic areas of greater than 60 μm in length and<10 μm wide.

Inertinite-derived – angular or rectangular fragments of isotropic carbon derived from inertinite coal macerals with sharp outlines or possessing remnant plant cell structure.

Vitrinite-derived Mosaic – aggregate areas composed of many anisotropic units of less than about 4 μm, which typically contain fragments of inertinite or mineral matter.

Vitrinite-derived Isotropic – recognizable fragments of vitrinite that have not developed thermoplastic properties either because they were not exposed to a high enough temperature or were oxidized.

Mineral Matter – the remnants of coal minerals that can be recognized including pyrite, clays and carbonates.

Analysis was conducted on each segment of coke and the results are recorded with respect to distance from the bottom inlet. Another approach to view this data is the sum of components that can be directly contributed to coal or decant oil. Because textural components derived from decant oil constitutes the majority of the coke material those components are normalized to 100% to assess the influence of the presence of coal. To compare the overall influence of operating conditions on textural components derived from decant oil, the average concentration of each component for each core is calculated from the segments analyzed.

The whole cores were analyzed using an OMNI-X x-ray CT scanner. The OMNI-X is a high-resolution industrial scanner. The system is mounted on a rigid optical grade structure that can move in any angle between 0-90. The positioning table can also move between tube and detector to change the magnification of the image on the image intensifier. The x-ray source can produce a cone-shaped beam that allows the collection of volumetric data. Currently, the system is capable of producing 1024x1024 pixels per image and enables a maximum resolution of 5-10 microns (with micro-focus x-ray tube) with energy levels of up to 225 kV. The system is also capable of collecting data in a varying number of views during scanning for the satisfactory image quality. For the present study coke samples were scanned with 90 kV and 375 micro A tube energy. The 2-D real resolution was 20 microns and slice thickness was 24 microns single slice scans. Approximately 40 scans were taken at intervals along the green coke core samples.

The remainder of the coke was removed from the coke drum and calcined in batches. The samples were heated from room temperature to 1400 °C at 10 °C/min and held at the maximum temperature for 30 minutes. The calcined coke was than sent to Alcoa Inc. for evaluation and to be made into test anodes. In order for the samples to meet their requirements Alcoa Inc. determined that the green mix for the anodes should be set at 16 wt% coal tar pitch. Once the green anodes were formed they were sent to Koppers Industries Inc. (KII) for baking and testing.

The anode cores were baked by KII using the following baking profile:

Baking: Anodes baked to ~1100°C under nitrogen purge.

0-600°C	10°C/hr.
600-1170°C	25°C/hr.
1170°C	14 hrs. hold

Each core was measured for baked apparent density, electrical resistivity, coefficient of thermal expansion, thermal conductivity, air permeability, crush strength, and flex strength. Alcoa Inc. had previously determined the green apparent density.

4.0 Results and Discussion

4.1 Production

Much of the early work centered around the redesign of the preheater set up, and the sample collection system. Initial results showed tests that 1/4" tubing was unsuitable, as the slurry formed blockages soon after start up. To that end, we began to use 3/8" tubing. However, this brought its own problems. The space velocity of the feed to the coker drum dropped considerably, and the residence times in the primary and secondary preheaters dropped from around 3 minutes with the 1/4" tubing to around 10 minutes in the 3/8" tubing. In some cases this had the affect of delaying the coking, and resulted in complete blockage of the tubing. To overcome the problem of coke build up we initiated the use of nitrogen blasts. These were very short bursts of 100 psig nitrogen into the tubing to aid in the removal of coke deposits. However, the nitrogen blasts also had the detrimental affect of blasting much of the feed out of the lines and up the walls of the coke drum. This in turn affected the yields. The removal of the primary preheater reduced the residence time of the sample prior to the coke drum inlet through the preheater zone to 3 minutes.

A secondary problem was centered on the injection of steam. It was discovered late on in the experiments that we were using too much steam. The water injection rate should have been set at 2 vol.% of the feed flow rate; however, it had been incorrectly set to 10 %. The water that had condensed in the product collection system was found to separate out the heavy molecular portions of the product, rather than remaining immiscible. The heavy waxes derived from the water/oil mixtures tended to concentrate in the valves, back pressure regulator and the mass flow controller. These problems forced us to re-plumb the collection apparatus to minimize the buildup of waxes in key components, and to date the results show that the efforts seem to be working well.

The measurement of sulfur content in the cokes produced during the experiments is shown in Table 4. These values indicate that there is very little difference between the sulfur content in cokes produced from decant oil alone or from the coal/oil slurry. The differences that are shown are probably within experimental error (although we have not confirmed this). What is interesting to note, however, is that there is definitely no concentration of the sulfur in the coke. In fact, it seems that we may be lowering the sulfur content, when compared to the starting feeds. This is not normally the case with coking operations. Usually sulfur tends to get concentrated in the coke fraction. We attribute our values to the use of steam injection. The steam may be stripping the sulfur from the reactive species as H_2S before it gets a chance to participate in the cross-linking type reactions that would lock it into the coke.

As expected the ash contents for the coke derived from the decant oil alone were zero (see Table 4). However, the ash contents for the cokes produced from the slurry were considerably higher than that of the feed. This indicates that we have concentrated the coal-derived material in the coke. Calculating the proportion of the coke that is coal-derived and that which is petroleum-derived would be very difficult. However, we may assume two things from these results. First, that we are performing liquefaction-type reactions with the coal. The coal liquids that are evolved from the reaction mixture are combined with the oil-derived volatiles in the products. Second, the coal may enhance the cracking of the oil through some surface, solid/liquid interactions. This increase the fraction of the oil-derived volatiles in the low boiling fractions and thus reduces the amount of sample in the higher boiling fractions that would normally undergo retrograde reactions to from coke. Further evidence for these assumptions is that the coke yield (as shown in Table 5) does not change significantly when slurry is used instead of decant oil.

Table 4. Examples of sulfur and volatile matter for cokes formed under various operating conditions from decant oil and a slurry of 10% Powellton coal in decant oil.

Sample	Coker inlet temperature (°C)	Primary preheater	Run time (minutes)	Sulfur (wt%) [a]	Ash content (wt%) [a]
Decant oil (as received)				0.8	0.0
Slurry (unreacted)				0.8	0.2
Decant oil	475	Yes	210	0.6	0.0
Decant oil	466	No	360	0.6	0.0
Decant oil	490	No	347	0.7	0.0
Slurry	475	Yes	148	0.5	0.8
Slurry	463	Yes	300	0.5	1.2
Slurry	465	No	360	0.6	1.1

[a]. Values reported on a dry basis

In Table 5 the two low coke yields of 8.1% and 6.7% for decant oil and slurry, respectively, are due to differences in the operating conditions. For the decant oil run there was evidence of unreacted pitch being combined with the coke. This explains why the corresponding volatile content of the coke sample is high. The pitch would normally convert to coke, and this is why the coke yield is low. We are unsure why the pitch was not converted. We had not seen this phenomenon before, and have not since either. For the slurry example the low yield was due to the short run time (147 minutes compared with the usual 300-360 minutes). We have noted that the rate of coke yield is dependant on time in the early stages of coke bed formation, and after 3-4 hours of operation a maximum coke yield is reached. This result is due to the coke bed enhancing further coke formation by promoting cracking reactions. The volatile content of the cokes are consistent, except for the experiment where we detected unreacted pitch.

In comparison with some industry standards for green sponge coke (see Table 6) the results from the analysis of the coke produced in this program are very favorable. As we do not use a water lance to remove the coke from our drum, the moisture content of the coke produced in our experiments is close to zero. Our coke has very comparable volatile matter content, and our sulfur content is much lower than that in the standard specifications. This is predominantly due to the fact that we start with low sulfur feeds, and we also see no concentration of sulfur in the coke products. The measured ash content of our coke is high, and thus our silicon and iron contents will be higher than those specified too. It was not our intention to perform an ash mineral analysis of the coke until after it was calcined. At which point some of the mineral matter will become volatilized.

After these initial testing runs the program was continued with parametric study focusing on the effect of operating conditions (temperature, flow rate, solids loading, steam injection, etc.) on the composition and morphology of the coke being formed. These tests were performed in at least triplicate in order to make a bulk sample for calcining. Table 5 includes some of the average data determined in each sample set.

Table 5. Examples of coke yield and volatile matter for cokes formed under various operating conditions from decant oil and a slurry of 10% Powellton coal in decant oil.

Sample	Coker inlet temperature (°C)	Primary preheater	Run time (minutes)	Coke yield (wt%)	Volatile matter (wt%) [a]
Decant oil	475	Yes		11.6	8.9
Decant oil	466	No	300	11.1	7.9
Decant oil	490	No	347	8.1	16.6*
Slurry	475	Yes	148	6.7	8.1
Slurry	463	Yes	300	10.7	8.2
Slurry	465	No	360	12.8	8.5

[a]. Values reported on a dry basis

* Coke was known to have unreacted pitch-like residue mixed in with it.

Table 6. Typical green sponge coke specifications [4].

Property	
Moisture	6-14%
Volatile matter	8-14%
Sulfur	1.0-4.0%
Silicon	0.02%
Iron	0.01%
Ash	0.25%

Table 7. Average product yields for co-coking runs with varying operating conditions.

Sample set	Coke yield (wt%)	Liquid yield (wt%)
A	18.5	61.6
B	29.3	51.8
C	24.0	65.2
D	20.2	65.4

Taking sample set A as the baseline set of runs the following observations can be made, with respect to the product yields:

1. With an increase in coke drum pressure, there was an increase in coke yield and a corresponding decrease in liquid yield. This is wholly consistent with results previously reported in the literature (2,5).
2. With an increase in coal in the feedstock, there was an increase in coke yield. This result is as expected. The ability to accurately determine how much of the coke is coal-derived has not yet been determined. The goal is to derive this value through yield calculations, with back up, concurring results from optical microscopy and other instrumental methods.
3. With the increase in coal to the feedstock, there was an increase in the liquid yield. This result would indicate that the volatiles, from coal, evolved during the coking process were resistant to thermal cracking, and so did not decompose to gaseous compounds.
4. A 50% decrease in water feed rate, and thus steam to the coker, resulted in an increase in coke and an increase in liquid yield. The coke increased because the lower steam rate resulted in less medium volatile compounds being swept from the coke drum. And so these medium volatiles subsequently coked. As there was an increase in liquid yield too, then the coking of the medium volatiles must have decomposed partially to intermediate lower boiling liquids, and produce gaseous products to a lesser extent.

4.2 Image Analysis of Green Cokes

4.2.1 Petrographic analysis of Green Cokes

Table 8 lists the samples analyzed using petrographic analysis techniques, and the experimental conditions under which the samples were produced.

Table 8. Conditions of Operation and Yield from Coker Runs

Coker Run Date	Feed Components	Feed Rate, g/min	Steam Injection Rate, ml/min	Drum Pressure psig	% Coke Yield	% Volatile Matter, daf	% Ash, dry
04/27/01	10% Powellton	16.5	na	25.2	9.1	13.64	1.57
06/04/01	10% Powellton	18.3	2.90	24.2	10.7	8.34	1.19
06/12/01	10% Powellton	17.7	2.80	26.3	12.8	8.58	1.10
06/20/01	100% Decant Oil	18.6	1.50	24.8	12.8	7.91	0.04
09/04/01	10% Powellton	17.4	0.30	24.7	19.3	7.79	0.85
09/11/01	10% Powellton	17.4	0.14	21.4	20.2	11.77	0.97
09/18/01	10% Powellton	15.8	0.30	50.2	31.3	24.89	0.48
09/25/01	20% Powellton	16.7	0.30	24.1	25.1	8.52	1.40

Regardless of operating conditions, in all cases coal-derived textural components are concentrated near the bottom inlet and decrease with increasing distance from the bottom (Tables 9 and 10). In contrast, the larger domain textures (domain and flow domain) derived from the decant-oil increase in concentration with distance from the bottom inlet. With respect to the one run (6-20-01) employing 100% decant oil (Table 11), operating conditions appeared to have had an influence on textures compared to earlier runs (2-26-01 and 3-7-01, Table 11). Generally, the coke of the 6-20-01 run was dominated by slightly smaller textures, but has a higher concentration of flow domain carbon owing to a greater porosity (which controls elongation of domains).

Table 9. Petrographic Analysis of Carbon Textures in Longitudinal Sections, Vol. %

Distance from Bottom of Core, mm	Vitrinite-derived Mosaic	Interinite-derived	Vitrinite-derived Isotropic	Mineral Matter	Pet. Isotropic	Pet. Mosaic	Pet. Small Domain	Pet. Domain	Pet. Flow Domai
Core 4-27-01; 10% Powellton Coal; with Primary Preheater									
0 - 34	9.4	4.6	0.1	0.3	0.0	26.8	54.2	2.8	1.8
Core 6-12-01; 10% Powellton Coal; 2.8 ml/min Steam rate; w/o Primary Preheater									
0 - 21	4.7	6.4	0.0	0.5	0.0	33.0	53.6	1.4	0.4
26 - 44	4.2	6.6	0.3	0.2	0.3	24.5	60.0	2.1	1.8
Core 6-20-01; 100% Decant Oil; 1.5 ml/min Steam rate; w/o Primary Preheater									
0 - 9	0.0	0.0	0.0	0.0	0.0	2.5	67.8	28.2	1.5
75 - 95	0.0	0.0	0.0	0.0	0.0	7.0	57.0	13.2	22.8
95 - 120 C*	0.0	0.0	0.0	0.0	0.0	8.2	55.8	16.6	19.4
Core 9-4-01; 10% Powellton Coal; 0.3 ml/min steam rate; 24.7 psig drum pressure									
0 - 14.5	13.9	11.7	0.0	0.3	0.0	28.5	37.4	6.4	1.8
102 - 127	5.4	7.1	0.0	0.1	0.3	8.8	54.1	19.3	4.9
280 - 305	1.2	4.4	0.0	0.0	0.0	5.3	68.1	18.1	2.9
356 - 381	0.2	3.8	0.0	0.2	0.0	13.6	49.8	14.0	18.4
Core 9-11-01; 10% Powellton Coal; 0.14 ml/min steam rate; 24.1 psig drum pressure									
0 - 26	5.1	9.1	0.1	0.1	3.3	38.7	41	1.9	0.7
52 - 72	0.0	6.2	0.2	0.8	79.0	11.8	1.8	0.2	0.0
127 - 150	1.3	4.2	0.2	0.1	4.5	19.7	54.3	12.8	2.9
Core 9-18-01; 10% Powellton Coal; 0.3 ml/min steam rate; 50. 2 psig drum pressure									
0 - 34	1.7	3.5	0.0	0.2	27.3	19.9	39.9	6.7	0.8
45 - 70	1.3	2.5	0.2	0.1	12.4	17.0	53.5	8.4	4.6
115 - 140	1.0	4.1	0.1	0.2	16.3	16.4	56.6	3.5	1.8
Core 9-25-01; 20% Powellton Coal; 0.3 ml/min steam rate; 24.1 psig drum pressure									
0 - 20	8.1	9.3	0.0	0.3	0.1	55.3	22.6	3.2	1.1
70 - 95	1.7	1.8	0.0	0.0	0.3	18.3	63.1	9.0	5.8
356 - 381	0.1	0.0	0.0	0.0	0.0	7.9	80.2	7.1	4.7

- C = Cross-section

Table 10. The Proportions of textures Derived from Coal and Decant Oil Compared with the Normalized Concentration of Decant Oil Textures, Vol. %

Distance from Bottom of Core, mm	%Coal-derived	%Petrol.-derived	% Isotropic	% Mosaic	% Small Domain	% Domain	% Flow Domain
Core 4-27-01; 10% Powellton Coal; with Primary Preheater							
0 - 34	14.4	85.6	0.0	31.3	63.3	3.3	2.1
Core 6-12-01; 10% Powellton Coal; 2.8 ml/min Steam rate; w/o Primary Preheater							
0 -21	11.6	88.4	0.0	37.3	60.6	1.6	0.5
26 - 44	11.3	88.7	0.3	27.6	67.7	2.4	2.0
Core 6-20-01; 100% Decant Oil; 1.5 ml/min Steam rate; w/o Primary Preheater							
0 - 9	0.0	100.0	0.0	2.5	67.8	28.2	1.5
75 - 95	0.0	100.0	0.0	7.0	57.0	13.2	22.8
95 - 120 C	0.0	100.0	0.0	8.2	55.8	16.6	19.4
Core 9-4-01; 10% Powellton Coal; 0.3 ml/min steam rate; 24.7 psig drum pressure							
0 - 14.5	25.9	74.1	0.0	38.5	50.5	8.6	2.4
102 - 127	12.6	87.4	0.3	10.1	61.9	22.1	5.6
280 - 305	5.6	94.4	0.0	5.6	72.1	19.2	3.1
356 - 381	4.2	95.8	0.0	14.2	52.0	14.6	19.2
Core 9-11-01; 10% Powellton Coal; 0.14 ml/min steam rate; 24.1 psig drum pressure							
0 - 26	14.4	85.6	3.9	45.2	47.9	2.2	0.8
52 - 72	7.2	92.8	85.1	12.7	2.0	0.2	0.0
127 - 150	5.8	94.2	4.8	20.9	57.6	13.6	3.1
Core 9-18-01; 10% Powellton Coal; 0.3 ml/min steam rate; 50. 2 psig drum pressure							
0 - 34	5.4	94.6	28.9	21.0	42.2	7.1	0.8
45 - 70	4.1	95.9	12.9	17.7	55.8	8.8	4.8
115 - 140	5.4	94.6	17.3	17.3	59.8	3.7	1.9
Core 9-25-01; 20% Powellton Coal; 0.3 ml/min steam rate; 24.1 psig drum pressure							
0 - 20	17.7	82.3	0.1	67.2	27.5	3.9	1.3
70 - 95	3.5	96.5	0.3	19.0	65.4	9.3	6.0
356 - 381	0.1	99.9	0.0	7.9	80.3	7.1	4.7

The similarity between cores derived from using 10% Powellton coal and obtained from 4-27-01 (with primary preheater) and 6-12-01 (without primary preheater) is striking. The competent portions of both cores were short (34 and 44 mm, respectively), there was almost no difference in the distribution of carbon textures (Table 9 and 10) and there was no significant difference in the distribution of carbon textures derived from decant oil (Table 11). This suggests that the removal of the primary preheater to improve operations has had no influence on coke properties.

Table 11. Comparison of Coke Textures Derived from Decant Oil from Coke Generated with and without Addition of Coal, Vol. %

Coker Run Date	Feed Components	Isotropic	Mosaic	Small Domain	Domain	Flow Domain
02/26/2001	100% Decant Oil	0.4	2.4	44.3	40.8	12.1
03/07/2001	100% Decant Oil	Trace	1.2	51.6	39.2	8.0
04/18/2001	10% Powellton	14.5	12.4	56.3	13.4	3.4
04/27/2001	10% Powellton	0.0	31.3	63.3	3.3	2.1
06/04/2001	10% Powellton	na	na	na	na	na
06/12/2001	10% Powellton	0.2	32.4	64.2	2.0	1.2
06/20/2001	100% Decant Oil	0.0	5.9	60.2	19.3	14.6
09/04/2001	10% Powellton	0.1	17.1	59.1	16.1	7.6
09/11/2001	10% Powellton	31.3	26.3	35.8	5.3	1.3
09/18/2001	10% Powellton	19.7	18.7	52.6	6.5	2.5
09/25/2001	20% Powellton	0.1	31.4	57.7	6.8	4.0

The influence of reducing steam injection rate is observed best by comparing coker runs from 6-12-01 and 9-4-01. Both runs involved 10% Powellton coal and both were terminated after 6 hours of operation, but the 9-4-01 run had a 90% reduction in steam rate. Both cokes have an average of 11-12 vol. % coal-derived components, but the distribution of decant oil-derived components was significantly different. As seen in

Table 11, an increase in the concentration of larger optical textures (domain and flow domain) at the expense of the smaller textures (mosaic and small domain) was observed. Another important distinction between these cokes that will not show up in the data, was the fact that the mosaic textures derived from coal were significantly enhanced in the 9-4-01 core. Also, coal textures were intimately mixed with those derived from the decant oil. This degree of enhancement and mixing was not observed in the 6-12-01 core, which may be a direct result of the higher steam injection rate when coal is present.

When the team injection rate was further reduced (0.14 ml/min, 9-11-01 core) operation of the coker appeared to be stable as nearly 370 mm of coke was generated in 6 hours of operation. However, a significant difference in the properties of the coke was observed. A considerable amount of isotropic carbon (without incorporated mesophase) derived from the decant oil filled the center section of the core (Table 9 and 10), whereas much less was observed near the bottom inlet and toward the top of the core. This amount of isotropic carbon contributed to a higher volatile matter content as shown in Table 8. The reason for the isotropic carbon cannot be necessarily blamed on the low steam rate, because in an earlier investigation that compared cokes from 100% decant oil with and without steam, a similar influence on coke properties was not observed. This suggests that other factors may have had an influence on the incomplete conversion of decant oil to an anisotropic carbon. Factors that may be involved in this unusual observation include the use of coal in the run, an unexpected loss of thermal control of the preheater, or there may have been changes in the chemical properties of the decant oil. The fact that mesophase spheres were not observed in the isotropic phase may support that latter two of these alternative factors.

Returning to the stable conditions and steam injection rate (0.3 ml/min) employed during the 9-4-01 run, problems were experienced in operation on 9-18-01. The run was terminated after about 3 hours as a result of excessive drum pressure (50.2 psig), although about 216 mm of coke was deposited. This run also produced a coke containing a great deal of isotropic carbon, except that there was a considerable amount of mesophase spheres trapped within the phase and the isotropic carbon was observed to be uniformly distributed throughout the core. Some of the isotropic carbon may result from how the run was terminated and may account for the high volatile matter content (Table 8). The high drum pressure that developed during the run may have had some influence on the formation of mesophase spheres in the isotropic phase. Furthermore, nearly all of the coal-derived material was significantly enhanced and intimately mixed with the decant oil derived carbon throughout the entire core. This too may be a benefit of the higher pressure.

Doubling the amount of coal to 20 wt.% at the low steam injection rate (0.3 ml/min, 9-25-01) did not adversely effect operations of the coker, as 381 mm of coke was generated during 6 hours of operation. However, the coke properties were significantly influenced, particularly near the bottom inlet, which was composed mostly of enhanced coal mosaic texture. Progressing in the direction of flow, coal textures showed less influence from contact with the decant oil and toward the top of the core much less coal-derived carbon was observed. This suggests that although most of the coal was isolated near the bottom of the core, it did not negatively effect coker operations.

4.2.2 X-ray Tomography of Green Carbon Products.

Figure 3 shows tomographs from the two green coke samples. Green coke core A (top) is from delayed coking with steam injection and green coke core B (bottom) is without steam injection. Tomographs 1, 2 and 3 represent green coke structures from the bottom, middle and top of the core sample, respectively. The white line on the photograph at the top is the area in the core where the tomograph was taken. Green coke B has a similar representation in tomographs 4-6.

It was observed that the bottom of core A had a much more open pore structure than core B. Therefore the steam injection significantly influenced the pore structure and coke deposition mechanisms in this region. This could be due to the steam keeping the medium volatile components from moving through this zone, and so stopping them from undergoing retrograde reactions leading to the formation of coke. The steam may also increase the volume of gas traveling through the pores, and hence could inhibit pore closure. Additionally, the steam may be cooling the surfaces within the coke structure and so may reduce the rate of coke formation. The green coke structures observed in the middle of both cores (tomographs 2 and 5) were very similar to each other indicating coking environments that are alike. The top two tomographs (3 and 6), however, are very different. This once again was due to the influence of steam. The image shown in tomograph 6 is of solidified froth. Froth is the first stage of coke formation on the leading edge of the coke bed as it rises up the drum. When the experiment was shut down the froth simply solidified in place. We do not see this in the core with steam injection (tomograph 3). This was because the overall coke bed height was 74 cm (bed height without steam was 30 cm), as the coring device was only 30 cm long we could not get all the core from the drum. However, the structure of the coke in the "frothy" zone of the core produced with steam injection was so friable that the coring bit would have destroyed it.

The data collected from the CT imaging was also used to estimate porosity (Figure 4) and density (Figure 5) with respect to coke bed height. The porosity measurements match the visual observations of the tomographs of each green coke core. That is, at the inlet to the coke drum the coke from steam injection has greater porosity than that of the sample with no steam injection. Both cores then produce a zone with near equal porosities. This was different from the PM analyses, which noted that the steam generated an overall higher porosity in the middle section. Finally, in the core with no steam injection there is a distinct rise in porosity in the zone of solidified froth.

Figure 5 shows a comparison of average densities and carbon matrix densities for the two green coke core samples. The average densities are shown as x-ray absorbtion intensities for each tomographic analysis taken along the core. Therefore this representation of density takes into account the pores in the green coke structure. The values for the carbon matrix densities are measured by ignoring the pores in the green coke structure. These results show that there is essentially no difference in the green coke density with respect to bed height for both green cokes. Hence the differences shown in the average density are due to the changes in the relative degree of porosity.

Volume scans can be used to develop 3-D images. By manipulating the image a map of the pores can be rendered, essentially ignoring the coke matrix. The image of the pores can then be used to measure the length and orientation of the pores. This data may be correlated with PM observations as to the overall size of areas with elongated domain, which would give an indication on what the quality of the coke would be after calcination and graphitization.

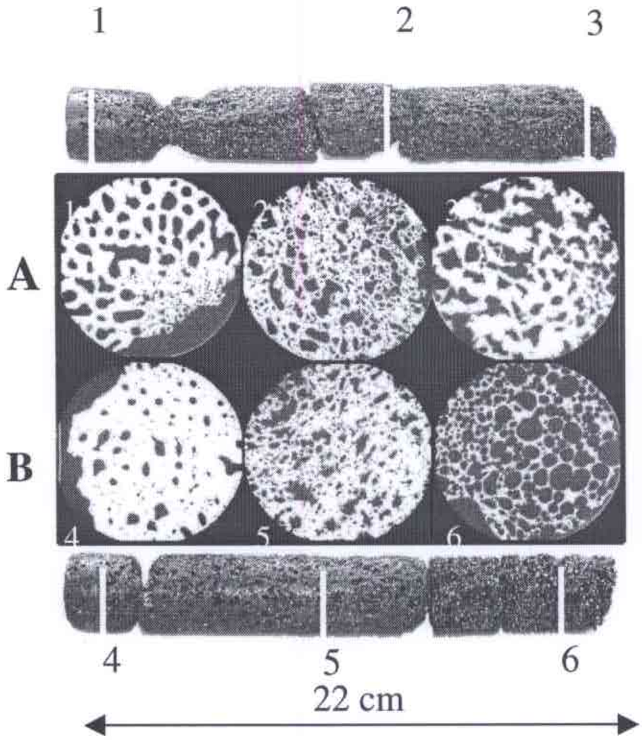

Figure 3. Tomographs of the green coke structures taken at intervals along cores from experiments with (A) and without (B) steam.

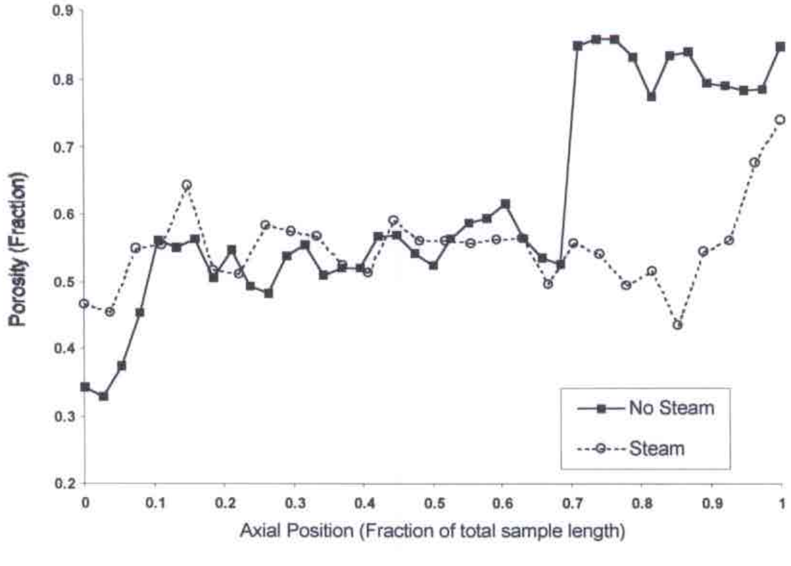

Figure 4. Porosity of the green coke samples with respect to bed height.

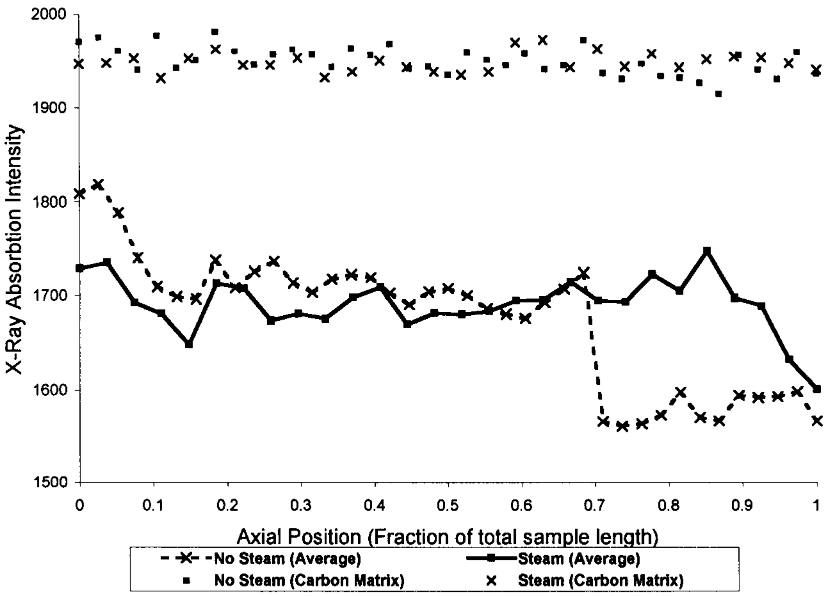

Figure 5. Average density and carbon density of coke samples with respect to bed height.

4.3 Analysis of Calcined Coke and Baked Anodes

Alcoa Inc performed the analysis of the baked anodes. Table 12 lists the concentration of some of the inorganic constituents of the calcined coke samples from the parametric co-coking study. The maximum values were obtained from Alcoa Inc. It can be seen that all of the Penn State samples exceed the limits set by Alcoa Inc. This is because of the residue clay minerals (Si, Na) and finely divided pyrite (Fe) that could not be cleaned full from coal when Preptech Inc. processed it.

Much of the mineral matter is disseminated and independent of the organic constituents of the coal, and this mineral matter can be mostly clean using froth flotation and other cleaning techniques. However, there is some mineral matter that is inherently bound within the coal structure. It is this mineral matter that is mainly contributing to the inorganic constituents in the Penn State samples.

On a more positive note, the sulfur concentrations in the Penn State samples are much lower than the maximum permissible level set by Alcoa Inc. Therefore, if samples from co-coking were ever to be used on an industrial-scale the SO_2 emissions would be lower than those from a conventional petroleum-based coke. In addition, the petroleum-derived minerals such as vanadium (V) were much lower than the maximum allowable limits. The vanadium was low due to the dilution factor brought about because of the coal-derived material in the coke. Vanadium can act as an oxidizing catalyst to the carbon during smelting, and therefore is detrimental to the product's use.

Table 12. Concentration of inorganic elements in calcined coke samples.

Sample No.	Ash (wt%)	Inorganic constituents				
		Si (ppm)	Fe (ppm)	Na (ppm)	V (ppm)	S (wt%)
Max. values	0.37	350	785	300	200	2.00
A	1.57	1900	1400	340	<50	0.66
B	1.46	1900	800	480	<50	0.63
C	1.82	3100	1600	260	<50	0.61
D	1.06	1200	600	190	<50	0.62

Table 13 lists some of the typical physical properties that are looked for in a test anode. The Penn State samples were compared with an Alcoa Inc. standard anode. The standard anode was made under the same conditions as the Penn State anodes, but was made with petroleum-derived coke.

There is little difference between the Alcoa Inc. standard anode and the Penn State anodes in the green and baked apparent density measurements, except for Sample B, which is the sample where 1% steam was used. The low steam rate must not have driven off the volatile matter like the 2% steam rate. This in turn may have led to a lighter, fluffier, less dense coke. The electrical resistivity of the Penn State samples was consistently lower than that of the Alcoa Inc. standard. This means that the Penn State samples would have used less electricity during the operation of the smelter. The air permeability for the Penn State samples varies quite a bit from that of the Alcoa Inc. standard. However, all the results are lower than that of the target value. A lower air permeability value is good because this means that there is less air infiltration into the sample, and thus reduces the level of reactivity with air. The Penn State samples are consistently stronger than the Alcoa Inc. standard. However, none of the samples meet the target values. The thermal conductivity values for the Penn State samples are closer to the target value than the Alcoa Inc. test anode.

Table 13. Physical properties measurements of baked test anodes

Sample No.	Green Apparent Density (g/cc)	Baked Apparent Density (g/cc)	Elec. Resist. (μΩm)	Air Perm. (nPm)	Crush Strength (MPa)	Flexural Strength Stress (MPa)	CTE Avg.Alpha (E^{-6}/°C @300°C)	Thermal Conduct. (W/mK)
Standard	1.614	1.535	70.8	1.25	24.41	5.59	4.351	2.43
A	1.623	1.543	58.5	1.08	29.00	7.47	4.657	3.08
B	1.568	1.491	66.8	1.42	29.41	6.32	4.769	2.87
C	1.648	1.558	53.7	1.09	31.58	9.05	4.849	3.45
D	1.624	1.529	57.0	1.76	30.94	6.60	4.637	3.04
Target values *		1.540	55.5	2.00	40-55	11.00	4.500	4.00

* These are values set by Alcoa Inc. or taken from *Anodes for the Aluminum Industry. 1st Ed.,* R+D Carbon Ltd., Switzerland.

Table 14 shows the data from reactivity measurements of the test anodes in air and CO_2. In reactivity measurements the goal is to attain as high a residue percentage as possible. The CO_2 reactivity residue percents for the Penn State anode fall short of the standard anode. Other than Sample D, the air reactivity resid values do not vary from the standard by too much.

Table 14. Reactivity measurements of baked anodes.

Sample No.	ISO CO_2 Reactivity				ISO Air Reactivity				Net carbon consumption kg/t Al
	% Wt. Loss	% Dust	% Attr.	% Resid.	% Wt. Loss	% Dust	% Attr.	% Resid.	
Standard	4.68	2.15	0.82	92.32	16.42	4.94	1.03	77.58	66.2
A	11.59	8.68	4.46	75.16	8.86	15.82	2.56	72.61	106.4
B	7.47	8.63	2.25	81.51	8.60	16.51	2.40	72.31	97.6
C					7.42	8.43	6.61	77.35	
D	5.91	3.45	2.41	88.17	9.20	22.43	3.75	64.49	102.5
Target values*	4-10	1-10		84-95	8-30	2-10		65-90	

** These are values taken from *Anodes for the Aluminum Industry. 1st Ed.*, R+D Carbon Ltd., Switzerland.

This data, along with the other physical property measurements, can be used to predict the net carbon consumption of the anodes. Equation 1 was developed by Fisher *et al* [6].

Equation 1. $NC = C + 334/CE + 1.2(BT - 960) - 1.7CR + 9.3AP + 8TC - 1.5AR$

Where:
NC = net carbon consumption in kg/t Al
C = cell factor in kg/t Al (set to 305 kg/t Al for this example)
CE = current efficiency (typical operations aim for a value of around 97%)
BT = bath temperature in °C (typical operations aim for a values ranging from 940-960 °C. for this example 960 °C was chosen)
CR = CO_2 reactivity residue in %
AP = Air permeability in nPm
TC = Thermal conductivity in W/mK
AR = Air reactivity residue in %

The net carbon consumption values are based on some general assumptions, and only serve to estimate differences between the standard anode and the Penn State samples. Net carbon consumption for the Penn State samples is higher than that of the standard anode. It is unclear whether the differences are significant at this stage.

5.0 Conclusions

In many aspects this study has been very successful. The majority of the goals set out for the study were attained. Moreover, it has been shown that a coal-based product can be produced from the co-coking of coal and petroleum resid that may ultimately have an application as a premium carbon product.

The objective of this study was to produce a coke product from the co-coking of coal and petroleum resid that could be used in the production of anodes for aluminum electrolysis. To this end, blends of cleaned coal and decant oil were fed into a pilot-scale delayed coker under various operating conditions. The resulting cokes were fully characterized using a variety of analytical techniques. Finally, the cokes were made into test anodes and compared with conventional petroleum coke-based anodes.

This study aimed to establish links between the changes in processing conditions and the quality of the resulting cokes. Traditional and non-traditional image analysis techniques, petrographic analysis and x-ray tomography, respectively, were used to measure some of the compositional and physical attributes of the green cokes. Furthermore, information on product yields, proximate and ultimate analysis were collected.

The resulting cokes were produced in bulk and calcined at 1400 °C. The calcined cokes were sent to Alcoa Inc. to be made into test anodes. The test anodes were sent to Koppers Industries Inc. to be baked. The baked anodes underwent a series of tests to determine their quality relative to a standard test anode, and industry target values.

Results show that in some aspects including density, strength, resistivity, reactivity, the coal-based cokes produced in this study met or exceeded the standards set by the industry. However, due to the presence of residual mineral matter from coal in the coke, the ash values for the coal-based anodes are high.

6.0 Acknowledgements

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7.0 References

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8.0 List of Acronyms and Abbreviations

Ni	nickel
V	vanadium
µm	micron
psig	pounds per square inch
g/min	grams per minute
vol%	volume percent
wt%	weight percent
kg/hr	kilograms per hour
CT	computed tomography
kV	kilo-volts
H_2S	hydrogen sulfide
ml/min	milliliters per minute
daf	dry ash free
Pet.	petroleum
PM	polarized-light microscopy
Si	silicon
Na	sodium
Fe	iron
SO_2	sulfur dioxide
g/cc	grams per centimeter cubed
µΩm	micro ohms per meter
nPm	nano perms
MPa	mega Pascals
CTE	coefficient of thermal expansion
Elec.	electrical
Resist.	resistivity
Perm.	permeability
Conduct.	conductivity
W/mK	watts per milli-Kelvin
CO_2	carbon dioxide
Attr.	attrition
Resid.	residue
kg/t Al	kilograms per tonne of Aluminum
NC	net carbon consumption
C	cell factor
CE	current efficiency
BT	bath temperature
CR	CO_2 reactivity residue
AP	Air permeability
TC	Thermal conductivity
AR	Air reactivity residue