Turbostratic Carbon Powder

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Abstract

Fine particulate carbon was created by grinding Solvent Extracted Carbon Ore (SECO), lightly oxidizing it, and heat treating it to various temperatures to produce a dense micropowder. Eight SECO samples were prepared and analyzed via proximate (dry) and elemental analyses, as well as with XRD and SEM. The electrical resistivity of some of the samples was also assessed. Dr. Cooper's group at Lawrence Livermore National Laboratory (LLNL), as well as Dr. Steven Chuang's group at the Department of Chemical Engineering, University of Akron, Akron, OH, were supplied with these SECO samples for testing in their Direct Carbon Fuel Cells (DCFC). It was found that heat-treated samples demonstrated a continuous increase in carbon, and a decrease in volatile matter, nitrogen, hydrogen and sulfur as the heat-treated temperature was raised. The oxygen content jumped significantly following oxidation, and then decreased dramatically upon heat treatment. XRD results indicated some degree of order in the structure of these heat-treated SECO samples, where a higher degree of order was observed for the samples heated at higher temperatures. Carbon foams consisting of coal tar pitch (CTP) combined with one of the oxidized SECO samples were made and assessed for their properties. The SECO/CTP foam sample showed properties consistent with other similar foam samples. The SECO heat-treated to 800 °C and above proved to be a more conductive carbon for use in the fuel cell, and showed favorable performance in LLNL's fuel cell. The University of Akron also reported favorable results for the oxidized SECO. The low ash, low sulfur, and highly conductive character of these samples makes them very attractive for application in the direct carbon fuel cell.

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Executive Summary

This program is focused on the production of a low cost coal-derived turbostratic carbon powder for structural and other applications, and investigates the suitability of such a material for use in a carbon fuel cell, such as that which has been demonstrated by Lawrence Livermore National Laboratory (LLNL). The Direct Carbon Conversion (DCC) process, pioneered by a LLNL team led by Dr. John Cooper, employs a unique fuel cell to produce electric power. The overall cell reaction is combustion of carbon with oxygen (from ambient air) forming carbon dioxide and electricity. Cooper's results at LLNL suggest that the ideal carbon feed for a carbon fuel cell should be a turbostratic carbon with microscopic domain size of 30 nm or smaller, with an ash value less than 0.5%, low VM, a sulfur level between 0.25% to 1.0%, low graphitization, and high electrical conductivity.

Solvent-extracted carbon ore (SECO) was initially suggested due to its low ash and sulfur content, as well as its presumed high friability enabling the production via grinding of extremely small particles. Fine particulate carbon was created by grinding Pilot Plant and lab-scale SECO, where the lab-scale SECO was lightly oxidized and coked to 400 °C, 600 °C 800 °C, 1000 °C, to produce a dense micropowder. The SECO samples were analyzed via proximate (dry) and elemental analyses, as well as with XRD and SEM. The electrical resistivity of some of the samples was also assessed. For testing purposes, Dr. Cooper's group was supplied with these SECO samples as well as cokes prepared from condensed byproducts from other CPCPC projects. Dr. Steven Chuang's group at the Department of Chemical Engineering, University of Akron, Akron, OH, is also working on the development of a carbon fuel cell and collaborated in evaluating the SECO samples produced by WVU.

Additionally, carbon foams consisting of coal tar pitch (CTP) combined with one of the oxidized SECO samples were made and assessed for their properties. The SECO/CTP foam sample was successfully created and showed properties consistent with other similar foam samples with a crush test value of 12.3 MPa.

WVU Coal Extraction Pilot Plant SECO samples SECO-4 and SECO-6/7 were found to be low in ash and sulfur. However, both samples had a large VM content (~40%) and difficulties were encountered during testing in the fuel cell. Thus, the lab-scale SECO batch was made, where heat treatment resulted in a dramatic decrease of volatile matter. Heat-treated samples also demonstrate a continuous increase in carbon, and a decrease in nitrogen, hydrogen and sulfur. The XRD results show some degree of order in the structure, and data indicate higher order for the samples heated at higher temperatures. All the samples thus produced have domain sizes less than 30 nm, indicating turbostratic carbon. No unusual surface effects due to heat-treatment are evident in the SEM images of the SECO. Resistivity measurements indicated that the higher the heat-treatment temperature, the lower the resistivity of the SECO becomes. Thus, the 800°C or above heat-treated SECO proved to be a more conductive carbon for use in the fuel cell, and showed favorable performance in LLNL’s fuel cell. The University of Akron also reported favorable results for the oxidized SECO.

Introduction

This program is focused on the production of a low cost coal-derived turbostratic carbon powder for structural and other applications. This material will differ from carbon black in that it will be a turbostratic carbon with low surface area, as opposed to carbon black, which is usually a high surface area amorphous material. The turbostratic carbon powder, when combined with an appropriate binder pitch, can be used as part of a carbon composite manufacturing process. In addition, such a material can be suitable for use in a carbon fuel cell, such as that which has been demonstrated by Lawrence Livermore National Laboratory (LLNL). The Direct Carbon Conversion (DCC) process, pioneered by a LLNL team led by Dr. John Cooper, employs a unique fuel cell to produce electric power. The overall cell reaction is combustion of carbon with oxygen (from ambient air) forming carbon dioxide and electricity. A schematic diagram of the direct carbon fuel cell concept is shown in Figure 1.

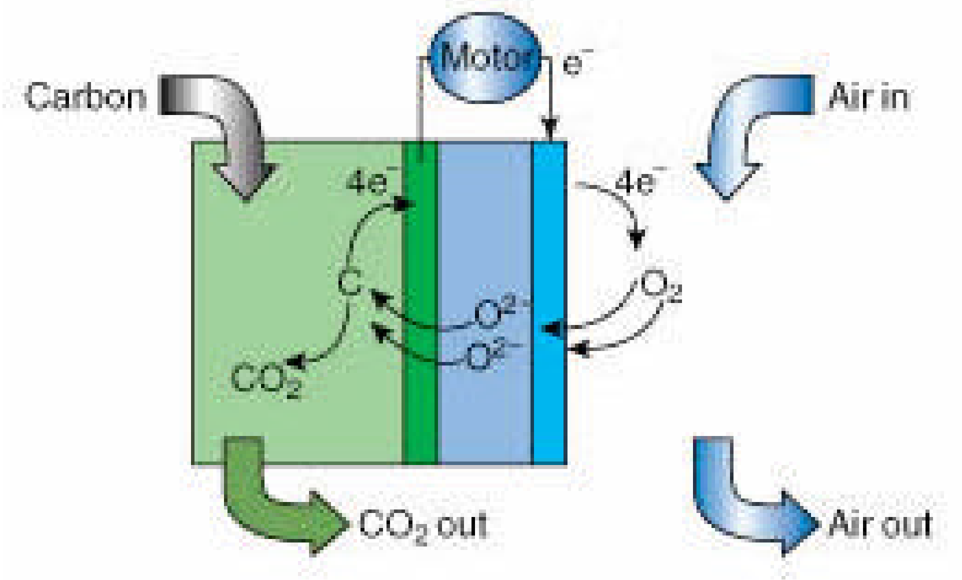

Figure 1. Direct Carbon Conversion Concept (by LLNL)

The reactions involved in this process are as follows[1]:

$C + 2CO_3^{2-} = 3CO_2 + 4e^-$	Anode
$O_2 + 2CO_2 + 4e^- = 2CO_3^{2-}$	Cathode
$C + O_2 = CO_2$	Overall Reaction ?G ~32.8 MJ/kg; E = 1.02 V at T=800 °C

LLNL results to date have shown that the fuel cell reaction yields 80 percent of the carbon-oxygen combustion energy as electricity[1]. It provides up to 1 kilowatt of power per square meter of cell surface area, which is comparable to other electrochemical systems. Laboratory cells tested at LLNL consist of a nickel anode current collector, a ceramic matrix for containing the molten electrolyte, a nickel screen for reacting the oxygen from the air, and a thermocouple (see Figure 2). The solid carbon fuel is fed as a fine powder to the cell. Currently the cell is operated in the batch mode but continuous operation is possible.

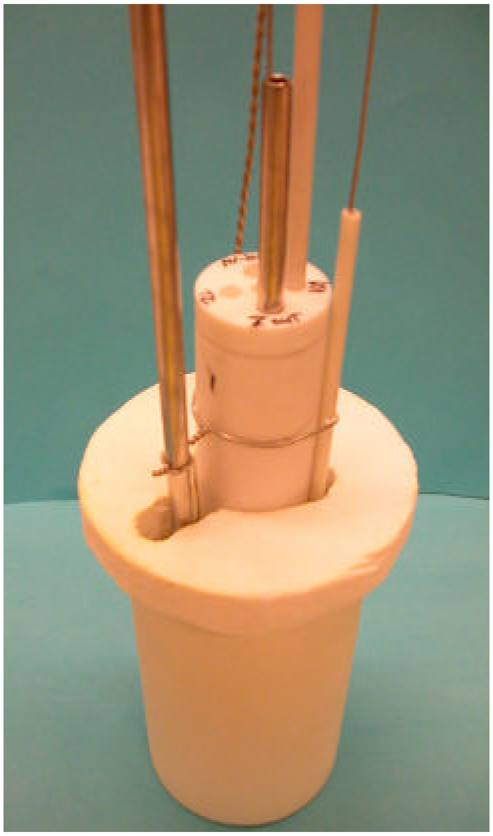

Figure 2. LLNL Fuel Cell (1.8cm^2)

Cooper's results at LLNL suggest that the ideal carbon feed for a carbon fuel cell should be a turbostratic carbon with microscopic domain size of 30 nm or smaller.[1] It also must have an ash value less than 0.5% to prevent excessive contamination of the molten salt electrolyte. Ranging degrees of volatile matter (VM) are of interest, but low VM is preferred. The sulfur level should be between 0.25% to 1.0% so that the metal electrodes are not chemically attacked and degraded excessively. Cooper's results also indicate that the electrochemical performance of the carbon improves with less graphitization as that increases reactivity of the carbon to oxidation. Performance is also enhanced with carbon of increased electrical conductivity which improves performance due to enhanced charge transport.[1]

In this project, the use of solvent-extracted carbon ore (SECO) was evaluated as a feedstock for the direct carbon fuel cell. The SECO was initially suggested due to its low ash and sulfur content, as well as its presumed high friability enabling the production via grinding of extremely small particles. Fine particulate carbon was created by grinding SECO, lightly oxidizing it, and heat-treating it to produce a dense micropowder. Eight SECO samples were prepared and analyzed via proximate (dry) and elemental analyses, as well as with XRD and SEM. The electrical resistivity of some of the samples was also assessed. For testing purposes, Dr. Cooper’s group was supplied with these SECO samples as well as cokes prepared from condensed byproducts from other CPCPC projects. Dr. Steven Chuang’s group at the Department of Chemical Engineering, University of Akron, Akron, OH, is also working on the development of a carbon fuel cell and collaborated in evaluating the SECO samples produced by WVU.

Additionally, carbon foams consisting of coal tar pitch (CTP) combined with one of the oxidized, heat-treated SECO samples were made and assessed for their properties.

Experimental

A reconfiguration of the Coal Extraction Pilot Plant at West Virginia University was completed in the Fall of 2003. Figure 3 shows an overall view of the pilot plant as it currently is configured. In a typical extraction run, approximately 30 pounds of SECO can be produced.

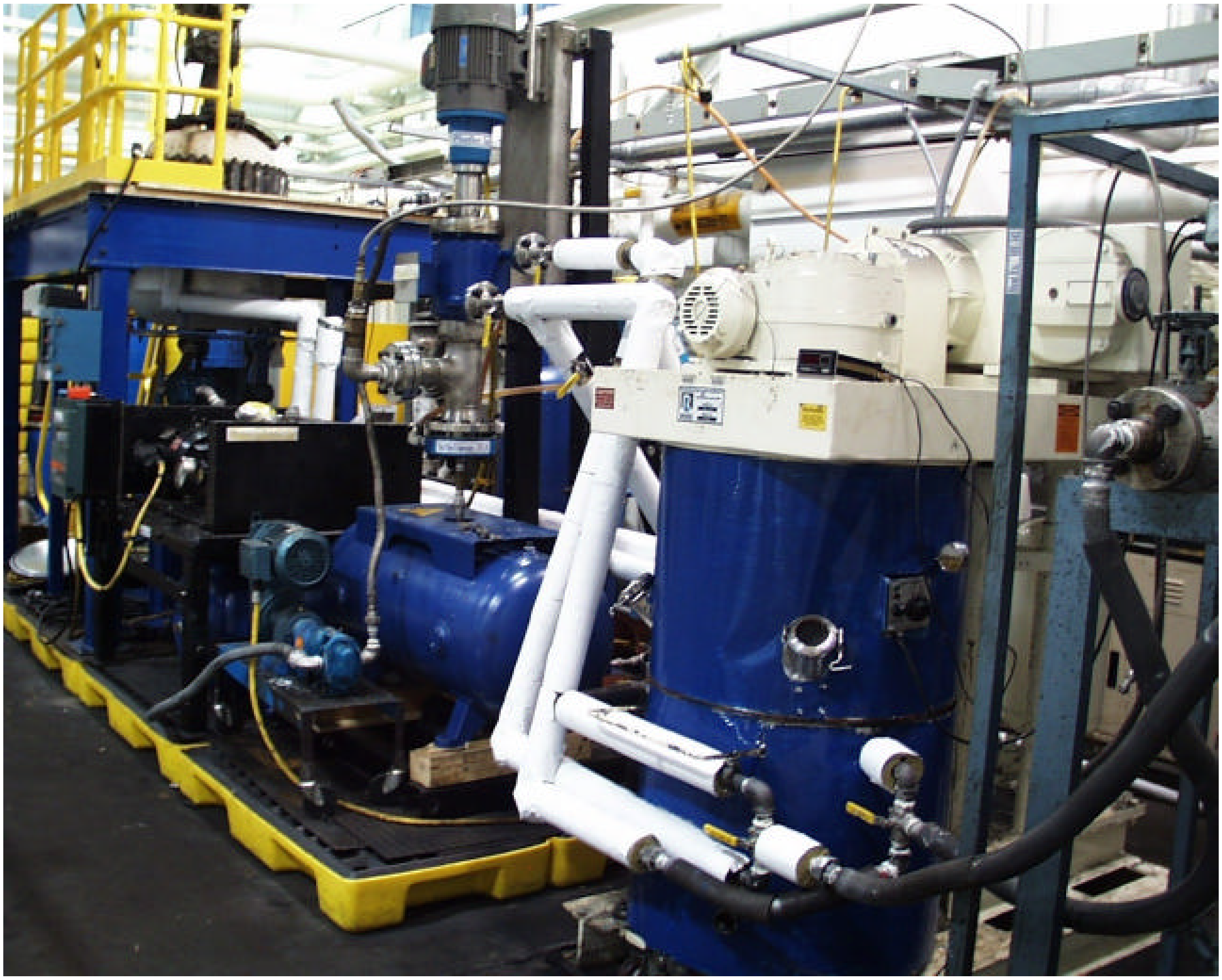

Figure 3. Solvent Extraction Pilot Plant Facility at WVU

Samples from two runs at the pilot plant, SECO-4 and SECO-6/7, were obtained for use in this project. These SECO samples were produced from a raw Kingwood coal mined in Preston Co., WV. The proximate and elemental analyses for the raw coal are shown in Table 1. The SECO samples were ground to ~200 mesh and were also characterized via proximate and elemental analysis. Results of these tests are shown in Table 1 as well.

Table 1. Proximate and Elemental Analysis Results of raw Kingwood Coal and SECO samples 4, 6/7 made in the WVU Pilot Plant

a) Proximate:	Moisture (wt %)	Volatile Matter (wt %)	Fixed Carbon (wt %)	Ash (wt %)
Raw Kingwood Coal	6.86	30.07	55.03	8.04
SECO-4	0.66	36.73	62.11	0.50
SECO-6/7	2.10	40.60	56.79	0.51
b) Elemental Composition:	C	N	H	S
Raw Kingwood Coal	77.44	1.18	4.95	1.58
SECO-4	83.36	2.78	5.73	0.62
SECO-6/7	80.01	3.86	6.04	0.54

As can be seen from the analysis, these two SECO samples are similar in composition. It should also be noted that the samples are very low in ash and contain only 0.5% sulfur, a considerable reduction from the ash and sulfur in the raw coal. Roughly 30 grams of SECO-4, 6/7 were sent to LLNL for testing and evaluation.

For more efficient operation of the direct carbon fuel cell, it is advantageous to grind the SECO to the micron range. To this end, an ultra sonic grinder (Ultrasonic Processor for Industrial Application, Model UIP2000) was obtained and tested for this purpose. The device was found to reduce the size of smaller particles to about 3 microns. However it was ineffective at reducing large particles of SECO. Thus, conventional grinding, via the ball mill, was still necessary. Figure 4 shows an electronmicrograph of

SECO ground using conventional ball milling equipment. It can be seen that particle sizes on the order of 1µm can easily be obtained.

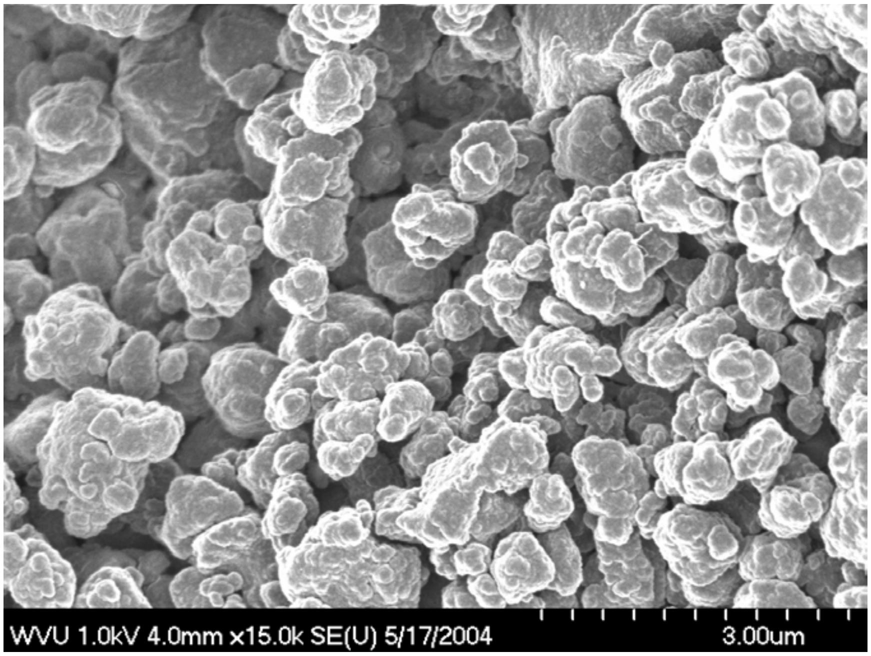

Figure 4. SEM image of ground SECO using a conventional ball mill

Feedback from Dr. Cooper indicated that the high volatile matter content (~40%) created difficulties in testing and determining accurate cell efficiency since some of the fuel left the cell as unburned volatiles. To rectify this problem, another batch of SECO from the same coal was prepared on the lab scale.^{2} A block flow-sheet of the process is shown as Figure 5.

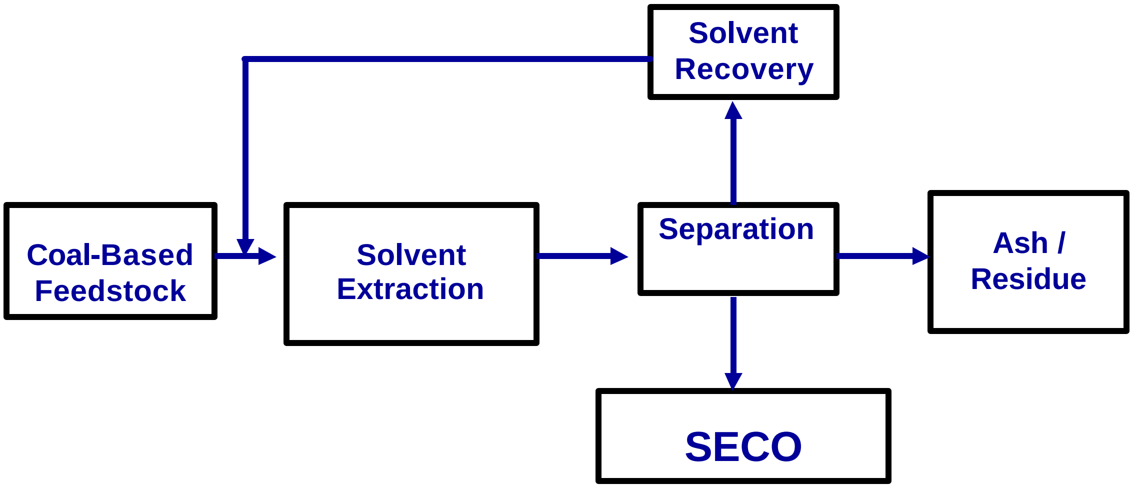

Figure 5. Flowchart of the solvent extraction process for SECO

Kingwood Coal, first air-dried for two days, was ground and vacuum-dried overnight at about 120°C. N-methyl pyrrolidone (NMP) was heated to about 200 °C after which coal was mixed in a ten-to-one solvent-to-coal ratio (10mL of NMP = 1g of coal) while being continuously stirred under nitrogen. This solution was left to reflux for 30 minutes. Following extraction, the solution was cooled and vacuum-filtered. Final evaporation of the NMP in a rotary evaporator precipitated the desired SECO as a solid product.

The resultant SECO was then heat-treated to varying temperatures to drive off some of the volatile matter. However, upon heat-treatment, the raw SECO was found to foam violently and boil out of the vessel. To prevent this, the sample was first partially oxidized at 250 °C for 4 hours in air so that cross-linking could be effected. Following oxidation, the sample was again heat treated, this time with no foaming noted. Four different levels of heat treatment were performed: 4 hours each at 400 °C, 600 °C, 800 °C, 1000°C in an inert atmosphere. These samples were then subjected to elemental and proximate analysis along with evaluation by x-ray diffraction (XRD) and SEM.

The electrical resistivity of the compacted heat treated SECO powders was also assessed by placing the powder in a nonconductive piston/cylinder assembly with electrodes on each end. Figure 6 shows photographs of the resistivity cell.

Figure 6. Electrical resistivity cell (WVU)

The resistance measurements were made by placing a known amount of sample in the cell. Electrical contact was made with the sample by means of two polished brass discs placed on either side of the sample bed. The diameter and the height (i.e. distance between the electrodes) of the sample bed was measured. A Keithley Model 2700 DVM was connected to the electrodes and resistance measurements were made as various amounts of weight were placed on the piston so that the powders could be compacted. The weight was increased until no further change in resistance was noted. Unfortunately, for highly conductive powders (like graphite and heat-treated SECO), the lower limit of the ohm meter was reached and further measurements were not possible. Hence reliable data were obtained only for the SECO samples heated to 600 °C and 800 °C. The 1000 °C sample had a resistivity too low for accurate measurement.

Three additional coke samples were prepared by Dr. Stansberry from a related CPCPC project. These samples were cokes made from the by-product coal liquids driven off during preparation of hydrogenated coal extracts. During processing, one of the steps is a partial distillation to raise the softening point of the desired product. This step produces a volatile stream of by-product hydrocarbons which when coked produces a low-sulfur, low-ash carbon. These recovered liquids were then coked at a rate of 3 °C per minute pausing at 300 °C for 2 hours and then raised the temperature at the same rate to 700 °C. This temperature was held for 2 hours before cooling to room temperature. The entire operation was performed in a covered crucible which was itself immersed in coke breeze to prevent burning. These samples were sent to LLNL. Samples were also sent to Dr. Chuang's group at the University of Akron.

A calcined carbon foam was produced from one of the previously mentioned SECO samples and tested for density, porosity and crush strength. The SECO powder that was oxidized and heat treated to 800 °C was ground to less than 200 mesh and mixed with CTP, which was also ground to under 200 mesh, in a ratio of seven parts coke to 3 parts CTP. The mixed powder was then put into a mold and heated under nitrogen to 500 °C at a rate of 5 °C per minute for 5 hours. Then the sample was calcined at 1000 °C for 5 hours. For comparison, a similar sample was made of only CTP without the addition of the coke. It was heat treated by the identical protocol.

Results and Discussion

Elemental and proximate analyses of the raw coal used in this project, Kingwood coal mined in Preston Co., WV, and WVU Coal Extraction Pilot Plant SECO samples SECO-4 and SECO-6/7 are listed in Table 1. Both SECO samples were low in ash and sulfur. However, due to the samples' large VM content (~40%), difficulties during the testing of the DCFC were experienced and an accurate determination of the cell efficiency was not possible since some of the fuel left the cell as unburned volatiles.

With the goal of decreasing VM and ash content, another lab-scale SECO batch was made (also from Kingwood Coal).[2] To prevent foaming during heat treatment, the samples were first partially oxidized at 250°C for 4 hours in air so that cross-linking could be effected. Following oxidation, the samples were again heat treated, this time with no foaming noted. Four different levels of heat treatment were performed: 4 hours each at 400 °C, 600 °C, 800 °C, and 1000 ºC in an inert atmosphere. The extract and oxidized heat-treated samples were subjected to elemental and proximate analysis. These results are shown in Table 2.

Table 2. Proximate (Dry) and Elemental Analysis of Raw and Heat-Treated SECO (Kingwood) made in the laboratory

Proximate Analysis			VM wt%	FC wt%	Ash wt%
Kingwood Coal			33	58	8.92
SECO Extract			32	67.82	0.18
Oxidized SECO Extract			32.17	67.54	0.29
Oxidized SECO, HT 400°C			28.87	70.90	0.24
Oxidized SECO, HT 600°C			9.05	90.66	0.29
Oxidized SECO, HT 800°C			3.84	95.90	0.27
Oxidized SECO, HT 1000°C			1.78	97.82	0.40
Elemental Analysis	**C**	**N**	**H**	**S**	**O**
Kingwood Coal	77.44	1.18	4.95	1.58	ND
SECO Extract	85.80	1.78	5.83	0.64	3.91
Oxidized SECO Extract	75.50	1.90	3.77	0.65	11.70
Oxidized SECO, HT 400°C	80.37	1.88	3.50	0.64	8.60
Oxidized SECO, HT 600°C	84.24	1.69	2.19	0.61	2.52
Oxidized SECO, HT 800°C	88.75	1.35	0.80	0.56	1.51
Oxidized SECO, HT 1000°C	91.00	1.48	0.34	0.44	ND

Table 3. XRD Analysis of Bench Samples

Sample	2? (002)	2 ? (110)	d(002) (nm)	d(110) (nm)	La (nm)	Lc (nm)
Raw SECO	22.9	81.43	0.3884	0.11819	2.4151	1.126
Ox/SECO	23.21	81.4	0.3832	0.11822	2.7072	1.0678
Ox/SECO 400°C	23.79	81.24	0.374	0.11842	2.3795	1.4925
Ox/SECO 600°C	23.94	81.45	0.3717	0.11816	2.7219	1.832
Ox/SECO 800°C	24.08	79.34	0.3696	0.12077	3.3361	2.2706
Ox/SECO 1000°C	ND	ND	ND	ND	ND	ND
Graphite Sample	26.33	73.83	0.3385	0.12835	27.851	45.118

XRD results can be seen in Table 3 and Figure 7, and a typical SEM image is show in Figure 8.

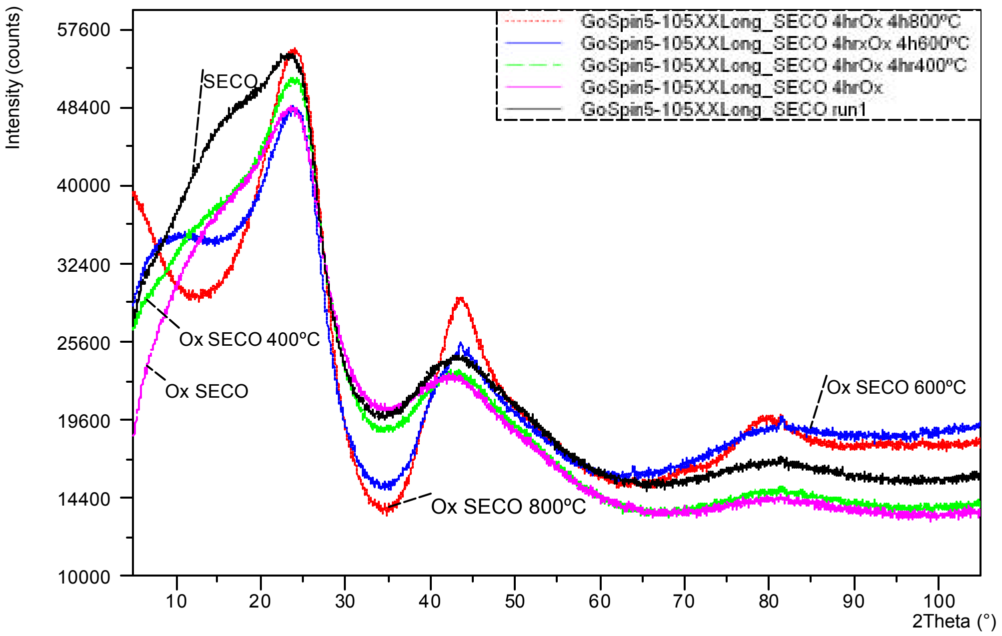

Figure 7. XRD analysis of bench samples

One may note, from the proximate analysis, the heat treatment results in a dramatic decrease in volatile matter. Heat-treated samples also demonstrate a continuous increase in carbon, and a decrease in nitrogen, hydrogen and sulfur. The oxygen content jumps significantly following oxidation, as is expected, and then decreases dramatically upon heat treatment. The XRD results show that the d (002) spacing consistently decreases upon heating to higher temperatures indicating some degree of order in the structure. In addition, the crystallographic domain sizes Lc and La increase steadily as a result of heat treatment. For reference, a sample of graphite was also tested in the XRD for comparison. These data indicate higher order for the samples heated at higher temperature. As can be seen, all the carbons thus produced have domain sizes less than 30 nm, indicating turbostratic, carbons.[4,5]

From the SEM image for the SECO treated at 800 °C, shown as Figure 8, one may see that any unusual effect of heat-treatment is not evident on the surface. The image resembles that of a friable glassy carbon, and is representative of the other images taken of the Oxidized SECO samples treated at different temperatures.

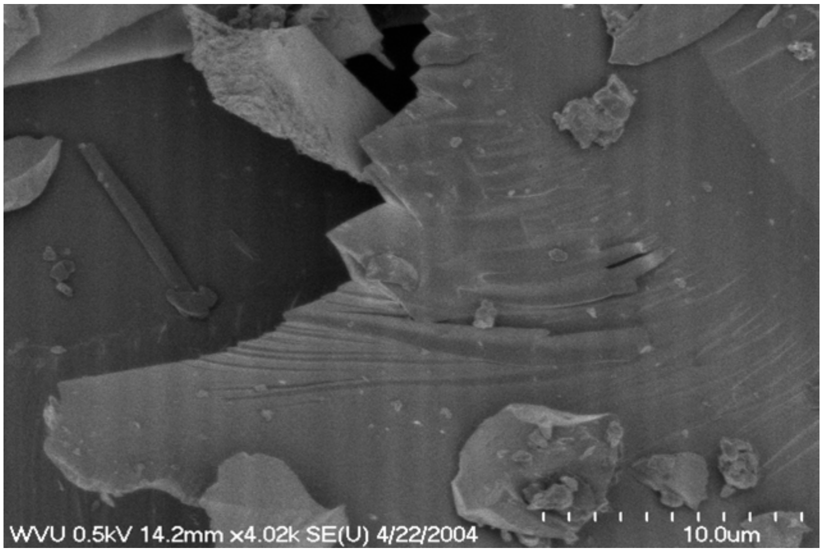

Figure 8. SEM image of oxidized SECO coked to 800 ºC

The electrical resistivity of the compacted heat treated SECO powders was assessed by placing the powder in a nonconductive piston with electrodes on each end. The resistivity was measured with respect to packing weight loaded on top of the piston. The results are listed in Table 4, as well as in Figures 9 and 10. From the results listed in Table 4, one can clearly see that the higher the heat-treatment temperature, the lower the resistivity of the SECO becomes. Thus the 800 °C sample represents one with low VM and resistivity and should perform well in the carbon fuel cell.

Table 4. Electrical Resistivity Measurements of some heat treated SECO samples

Sample Name	Resistivity (Ocm)
Oxidized, Heat-Treated to 400 °C SECO	Too high for accurate measurement
Oxidized, Heat-Treated to 600 °C SECO	6.1×10^6
Oxidized, Heat-Treated to 800 °C SECO	29
Oxidized, Heat-Treated to 1000 °C SECO	Too low for accurate measurement

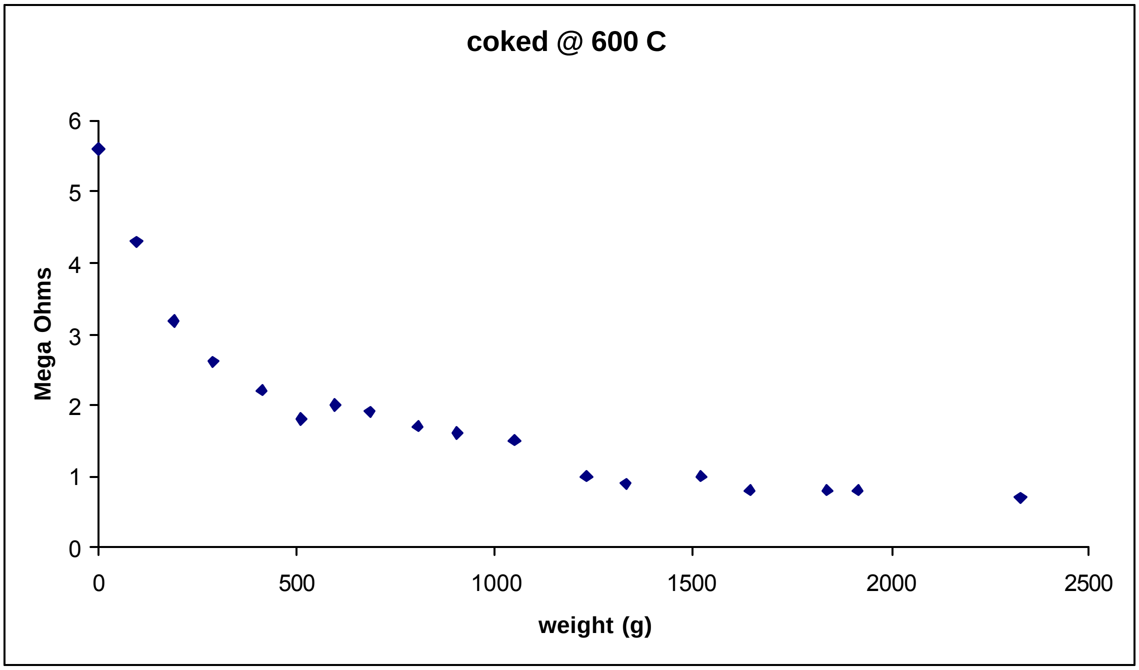

Figure 9. Electrical resistance measurements versus weight loading for Oxidized SECO heat treated to 600 °C

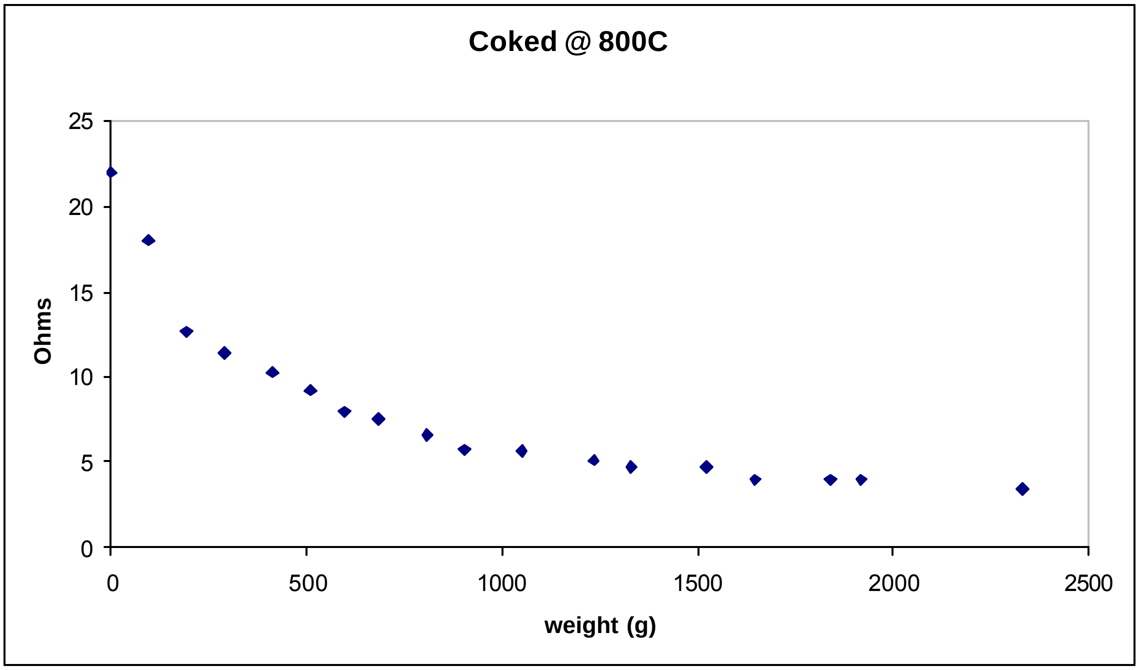

Figure 10. Electrical resistance measurements versus weight loading for Oxidized SECO heat treated to 800 °C

Dr. Cooper's group at LLNL was only able to test two of the samples sent to them by WVU. These two samples were the Oxidized, 400 °C heat-treated SECO and the oxidized, 800°C heat-treated SECO. In the case of the 400°C fuel, the cell voltage was reported to be poor due to sulfur interference with the cathode activation. However, the 800°C sample showed very favorable performance. These results are shown in Figures 11 and 12.

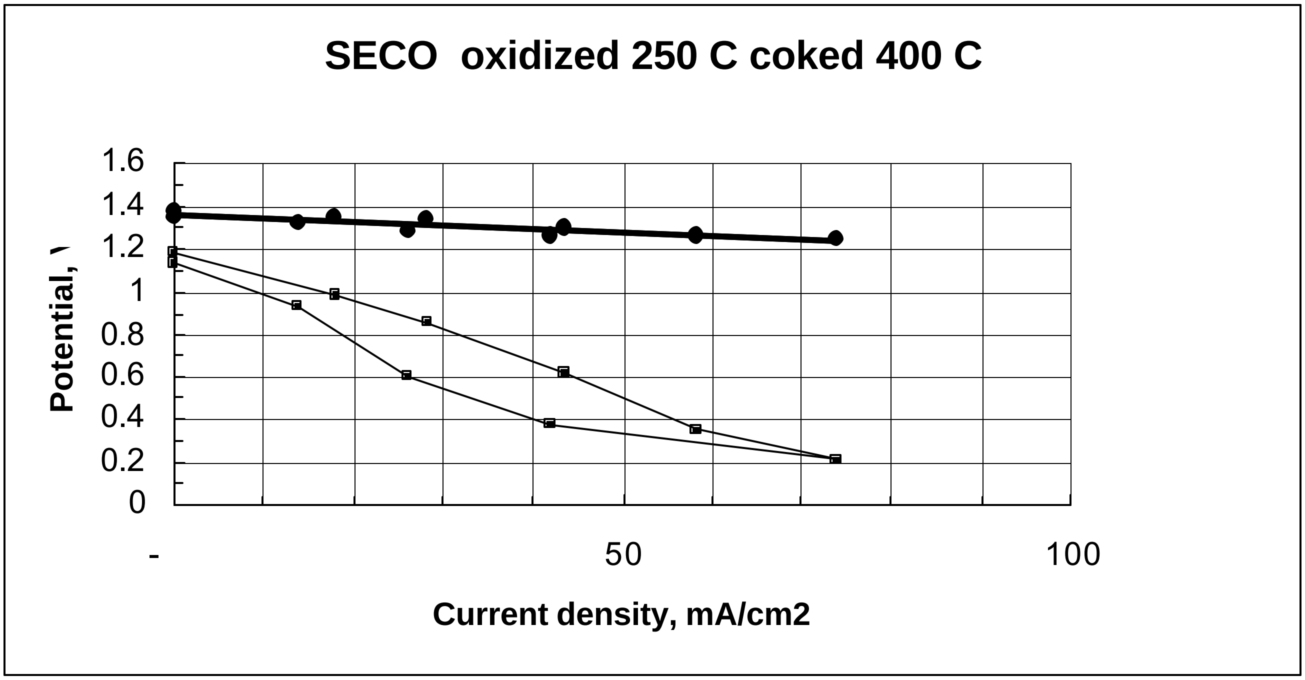

Figure 11. Polarization of Coked Solvent-Derived Sample
In this cell, we attempted to activate cathode in presence of C. The polarization of the carbon (upper trace) was favorable

Vcarbon ° Ecarbon - Eref (Au/ 5/7 air, 2/7 CO2)

Cell voltage was poor because S interfered with cathode activation

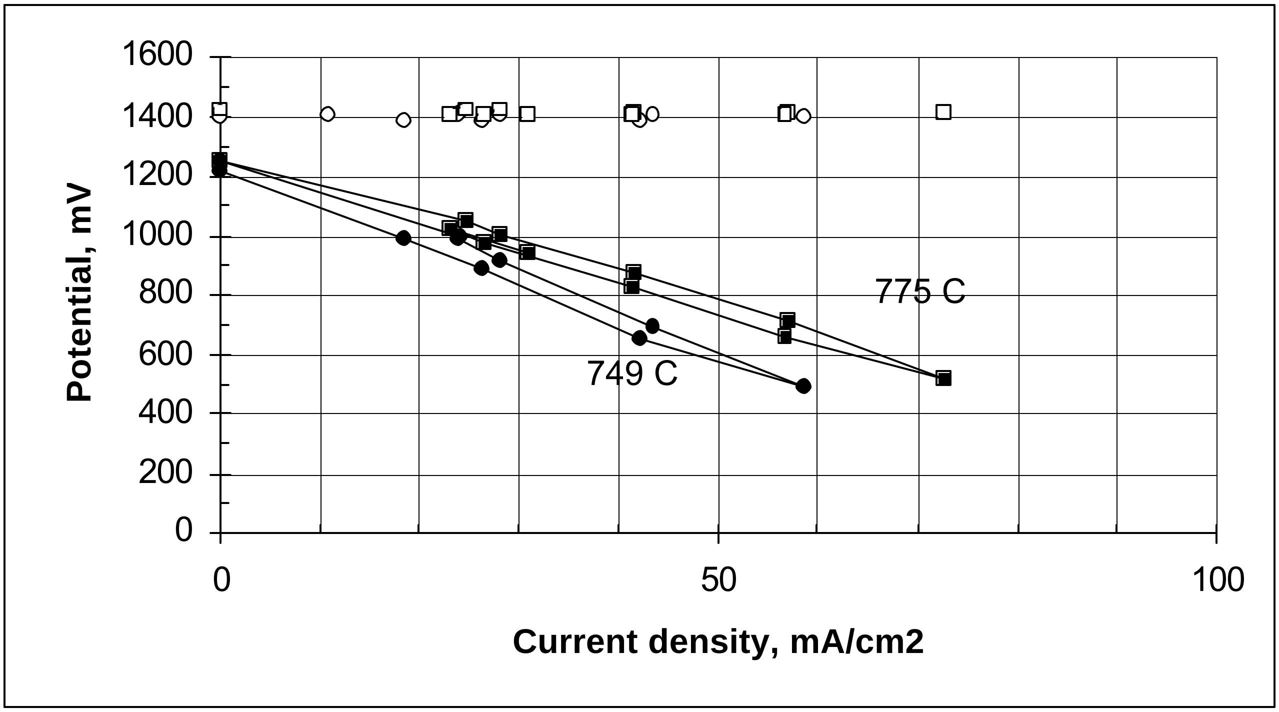

Figure 12. Pre-activation of Nickel Cathode Maximizes Performance of Carbon/Air Cell

- **Sample: WVU low-ash SECO; oxidized 250 °C, coked at 800 °C**
- **Cathode: activated prior to adding carbon to cell**
- **Favorable performance: 0.8 V at ~ 50 mA/cm2**

Dr. Chuang's assessment of the lab-scale SECO was as follows:

> "Your clean coal performed at the same level as Ohio no. 5 coal without producing much ash. The problem is the current density of our fuel cell remains to be very low. The low current density is not due to your clean coal. It is due to the problem inherent to our fuel cell. We are working to improve our fuel cell. Once I get good results, I will let you know immediately."[3]

Finally, Figure 13 shows a photograph of two foam samples produced during this work. The one on the left is a foam made from only CTP and the one on the right is the foam made from the blend of 30% CTP and 70% oxidized SECO which was heat-treated to 800°C. As can be seen, the CTP-only foam was very porous, of low density and of low strength. In contrast, the SECO/CTP was very dense with much smaller pores and with much greater strength.

Figure 13. Carbon foam produced from SECO powder and CTP (right), and from CTP alone treated under the same conditions (left)

A crush test was conducted on the SECO/CTP foam sample and resulted in a value of 12.3 MPa. The density of the foam was found to be 0.87 g/cm^3 and the porosity was found to be 47%. These results are comparable to other carbon foams produced from a variety of feedstocks at WVU.

Conclusions

Elemental and proximate analyses of WVU Coal Extraction Pilot Plant SECO samples, SECO-4 and SECO-6/7, showed that the samples were low in ash and sulfur. However, a significant reduction in the VM content was needed in order to minimize difficulties during the testing of the DCFC. With the goal of decreasing the VM, and minimizing ash, a lab-scale SECO batch was successfully made from Kingwood Coal. This SECO was partially oxidized to prevent foaming during the heat-treatment process which was conducted at four different temperature levels (400, 600, 800, 1000 °C). Heat treatment resulted in a dramatic decrease in volatile matter. Heat-treated samples also demonstrated a continuous increase in carbon, and a decrease in nitrogen, hydrogen and sulfur. The oxygen content jumped significantly following oxidation, as was expected, and then decreased dramatically upon heat treatment.

XRD results indicated some degree of order in the structure of these heat-treated SECO samples. A higher degree of order as indicated by both the d-spacing and L values was observed for the samples heated at higher temperatures. All the carbons thus produced had domain sizes less than 30 nm, indicating turbostratic carbons.

Electrical resistivity measurements showed that the higher the heat-treatment temperature, the lower the resistivity of the SECO becomes. Thus, with the oxidized SECO heat-treated to 800°C or above, a more conductive carbon is obtained. Unfortunately, an accurate value for the resistivity of the 1000 °C heat-treated sample was not possible with the equipment available in the laboratory.

A SECO/CTP foam sample was successfully created and showed properties consistent with other similar foam samples.

The oxidized, 800 °C heat-treated SECO showed favorable performance in LLNL's fuel cell. The lab-scale SECO was also shown to perform well according to Dr. Chuang's findings. The low ash character of these samples makes them very attractive for application in the direct carbon fuel cell. Further work is this direction will be pursued in the future.

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LIST OF ACRONYMS AND ABBREVIATIONS

LLNL:	Lawrence Livermore National Laboratory
DCC:	Direct Carbon Conversion
VM:	Volatile Matter
SECO:	Solvent-Extracted Carbon Ore
XRD:	X-ray diffraction
SEM:	Scanning Electron Microscopy
VM:	Volatile Matter
CTP:	Coal Tar Pitch
NMP:	N-methyl pyrrolidone
DCFC:	Direct Carbon Fuel Cell
WVU:	West Virginia University