Graduate Theses, Dissertations, and Problem Reports

2005

Carbon fuels for the direct carbon fuel cell

Abha Saddawi
West Virginia University

Follow this and additional works at: <https://researchrepository.wvu.edu/etd>

Recommended Citation

Saddawi, Abha, "Carbon fuels for the direct carbon fuel cell" (2005). *Graduate Theses, Dissertations, and Problem Reports*. 1679.

<https://researchrepository.wvu.edu/etd/1679>

This Thesis is protected by copyright and/or related rights. It has been brought to you by the The Research Repository @ WVU with permission from the rights-holder(s). You are free to use this Thesis in any way that is permitted by the copyright and related rights legislation that applies to your use. For other uses you must obtain permission from the rights-holder(s) directly, unless additional rights are indicated by a Creative Commons license in the record and/ or on the work itself. This Thesis has been accepted for inclusion in WVU Graduate Theses, Dissertations, and Problem Reports collection by an authorized administrator of The Research Repository @ WVU. For more information, please contact researchrepository@mail.wvu.edu.

Carbon Fuels for the Direct Carbon Fuel Cell

Abha Saddawi

**Thesis submitted to the
College of Engineering and Mineral Resources
at West Virginia University
in partial fulfillment of the requirements
for the degree of**

**Master of Science
in
Chemical Engineering**

**John W. Zondlo, Ph.D., Chair
Charter D. Stinespring, Ph.D.
Liviu Magean, M.S.**

Department of Chemical Engineering

**Morgantown, West Virginia
2005**

Keywords: Coal, Carbon Products, Direct Carbon Fuel Cells

Copyright 2005 Abha Saddawi

Abstract

Carbon Fuels for the Direct Carbon Fuel Cell

Abha Saddawi

This research focuses on using the U.S.' abundant coal reserves to create carbon fuels for the production of energy in a very efficient and environmentally friendly manner via the use of a Direct Carbon Fuel Cell (DCFC). This cell utilizes a process by which carbon is electrochemically converted directly to electricity, allowing theoretical cell efficiency to approach 80%. Using carbon made from the solvent extraction of coal eliminates most of the volatiles, ash, and sulfur from the fuel while most of the remaining NO_x, sulfur, and fly-ash byproducts are captured by the cell itself. The aim of this project is to investigate coal-derived carbon fuel for a DCFC made by Scientific Applications & Research Associates (SARA). This DCFC is unique in that the fuel also acts as the cell's anode, necessitating that the coal-derived carbon fuel be electrically conductive. The cell also requires the fuel to be in solid cylindrical form. A method to produce solid cylindrical carbon fuel rods was developed, and fuel rods were made with varying amounts of petroleum coke, coal tar binder pitch, and either one of two coal-derived fuels (Cenfuel and Solvent Extracted Carbon Ore, or SECO). These fuel rods were then analyzed for their chemical composition, density, and electrical resistivity. The least amount of binder necessary to hold the fuel rod together was found to be 10% by weight. The resistivity of the rods made with SECO, was lower than for those made with any other material, having reached a low value of 80 to $\mu\Omega\text{-m}$ for the 100% SECO rod. The SECO rods also had consistently lower ash and sulfur content. SARA test results indicate that coal-derived rods perform significantly better than their graphite counterparts due to increased electrochemical activity in the DCFC. This suggests that SECO could be an excellent fuel for the DCFC.

Dedicated to My Parents

Dr. Salma and Dr. Shafa Saddawi

Acknowledgements

I would like extend my deepest thanks and gratitude to my advisor, Dr. John Zondlo, for his continuous guidance, support, and encouragement. Many thanks to my committee members, Dr. Charter D. Stinespring, and Mr. Liviu Magean for being generous with their time, for all of their invaluable assistance and recommendations. I would also like to thank Mr. Jason C. Hissam for countless hours of aid in the laboratory, and for his moral support. Thanks to Mr. Elliot Kennel, the program coordinator for the Carbon Products Program at WVU.

I would like to express my gratitude to Dr. Dady Dadyburjor, thank you for believing in me. Thank you to Mr. Jim Hall for truly artful equipment fabrication. Also, thanks to Mr. Liviu Magean for fabulous laboratory analysis aid. My most sincere thanks to Mrs. Linda Rogers, and Mrs. Bonita Helmick for all of their non-research related help.

I would like to acknowledge the U.S. Department of Energy for providing support for this research (Project ID No. DE-FC26-03NT41873).

Finally, I would like to express my most heartfelt thanks to my wonderful parents, whose loving guidance and support made this thesis possible.

Table of Contents

Abstract	ii
Acknowledgements	iv
Table of Contents	v
List of Tables	vii
List of Figures	ix
List of Figures	ix
Chapter 1 Introduction	1
1.1 Scope of Research	2
Chapter 2 Background	4
2.1 Coal Defined	4
2.1.1 Coal Formation	4
2.1.2 Coal Classification	5
2.1.3 Structure of Coal	6
2.2 Carbonization	8
2.2.1 Coke	8
2.2.2 Pitch	8
2.3 WVU Solvent Coal Extraction	8
2.4 The Direct Carbon Fuel Cell	9
2.4.1 Brief DCFC History	9
2.4.2 SARA DCFC Design	10
2.4.3 Fuel Specifications Based on SARA's Cell Design	12
Chapter 3 Experimental	13
3.1 Fuel Ingredients	13
3.1.1 Petroleum Coke (pet-coke)	13
3.1.2 Binder Pitch	14
3.1.3 Cenfuel	14
3.1.4 Solvent Extracted Carbon Ore (SECO)	14
3.2 Rod Production	14
3.2.1 Stage One - The Disc	15
3.2.2 Stage Two - Making a Longer Rod	16
3.2.3 Stage Three – Clamshell Mold	18
3.3 Mixing the Fuel Ingredients	23
3.4 Softening Point Determination	24
3.5 Heating Regimen	24
3.6 Binder Pitch Levels	25
3.7 SECO Production	25
3.8 Rod Resistivity	26
3.9 Rod Density	29
3.10 Cenfuel, Pet-Coke Rods	29
3.11 SECO, Pet-Coke Rods	30
3.12 Proximate Analysis	30
3.13 Elemental Analysis	31
Chapter 4 Results and Discussion	33
4.1 Elemental and Proximate Analyses of Fuel Ingredients	33

4.2 Pitch Softening Point	34
4.3 Rod Production	34
4.4 Cenfuel and Pet-Coke Rods	36
4.4.1 Elemental and Proximate Analysis	36
4.4.2 Density and Resistivity	37
4.5 Density Verification.....	40
4.6 SECO and Pet-Coke Rods	42
4.6.1 Elemental and Proximate Analysis	42
4.6.2 Density and Resistivity	43
4.7 Results From SARA's DCFC	46
Chapter 5 Conclusions and Recommendations.....	49
5.1 Conclusions.....	49
5.1.1 Summary	50
5.2 Recommendations for Future Work.....	51
Bibliography	52
Appendix A	54
Experimental Raw Data	54

List of Tables

Table 2. 1 The ASTM System for Classifying Coals by Rank	6
Table 4. 1 Elemental and Proximate Analyses of Fuel Ingredients	33
Table 4. 2 Elemental Analysis and Ash Content for Cenfuel/Pet-Coke Rods (all rods contain 15% binder pitch by weight).	36
Table 4. 3 Resistivities and Densities of Cenfuel/Pet-Coke Rods	37
Table 4. 4 Density and Resistivity values Rods made from 75% Cenfuel and 25% Petroleum Coke	41
Table 4. 5 Density and Resistivity values for Rods made from 50% Cenfuel and 50% Petroleum Coke	41
Table 4. 6 Elemental Analysis and Ash Content for SECO/Pet-Coke Rods (all rods contain 15% binder pitch by weight)	43
Table 4. 7 Resistivities and Densities of SECO/Pet-Coke Rods	43
Table A. 1 Elemental and Proximate Analyses of Fuel Ingredients	55
Table A. 2 Elemental Analyses and Ash Content of Batch 1 Cenfuel/Pet-Coke Rods	55
Table A. 3 Elemental and Proximate Analyses of Batch 2 Cenfuel/Pet-Coke Rods	56
Table A. 4 Elemental Analyses and Ash Content of SECO/Pet-Coke Rods	58
Table A. 5 Voltage and Current Readings for Batch 1, 25% Cenfuel/75% Pet-Coke Rod	59
Table A. 6 Voltage and Current Readings for Batch 1, 50% Cenfuel/50% Pet-Coke Rod	59
Table A. 7 Voltage and Current Readings for Batch 1, 75% Cenfuel/25% Pet-Coke Rod	60
Table A. 8 Voltage and Current Readings for Batch 1, 80% Cenfuel/20% Pet-Coke Rod	60
Table A. 9 Re-Tested Voltage and Current Readings for Batch 1, 75% Cenfuel/25% Pet- Coke Rod	61
Table A. 10 Re-Tested Voltage and Current Readings for Batch 1, 75% Cenfuel/25% Pet- Coke Rod	62
Table A. 11 Re-Tested Voltage and Current Readings for Batch 1, 50% Cenfuel/50% Pet- Coke Rod	62
Table A. 12 Re-Tested Voltage and Current Readings for Batch 1, 50% Cenfuel/50% Pet- Coke Rod	63
Table A. 13 Voltage and Current Readings for Batch 2, 80% Cenfuel/20% Pet-Coke Rod	63
Table A. 14 Voltage and Current Readings for Batch 2, 100% Cenfuel/0% Pet-Coke Rod	64
Table A. 15 Voltage and Current Readings for Batch 2, 25% Cenfuel/75% Pet-Coke Rod	64
Table A. 16 Voltage and Current Readings for Batch 2, 0% Cenfuel/100% Pet-Coke Rod	65

Table A. 17 Voltage and Current Readings for Reproducibility Verification, 75% Cenfuel/25% Pet-Coke Rod	65
Table A. 18 Voltage and Current Readings for Reproducibility Verification, 75% Cenfuel/25% Pet-Coke Rod	66
Table A. 19 Voltage and Current Readings for Reproducibility Verification, 75% Cenfuel/25% Pet-Coke Rod	66
Table A. 20 Voltage and Current Readings for Reproducibility Verification, 75% Cenfuel/25% Pet-Coke Rod	67
Table A. 21 Voltage and Current Readings for Reproducibility Verification, 50% Cenfuel/50% Pet-Coke Rod	67
Table A. 22 Voltage and Current Readings for Reproducibility Verification, 50% Cenfuel/50% Pet-Coke Rod	68
Table A. 23 Voltage and Current Readings for Reproducibility Verification, 50% Cenfuel/50% Pet-Coke Rod	68
Table A. 24 Voltage and Current Readings for Reproducibility Verification, 50% Cenfuel/50% Pet-Coke Rod	69
Table A. 25 Voltage and Current Readings for 100% SECO/0%Pet-Coke Rod	69
Table A. 26 Voltage and Current Readings for 75% SECO/25%Pet-Coke Rod	70
Table A. 27 Voltage and Current Readings for 50% SECO/50%Pet-Coke Rod	70
Table A. 28 Voltage and Current Readings for 25% SECO/75%Pet-Coke Rod	71

List of Figures

Figure 2. 1 Proposed Structure of Coal as Put Forth by Wiser Showing Main Structural Groups in Coal	7
Figure 2. 2 SARA DCFC Showing Molten Hydroxide Electrolyte Bath and Carbon Fuel/Anode Rod	11
Figure 3. 1 Earliest Model of a Fuel Rod Shaping Mold.....	15
Figure 3. 2 Hydraulic Piston and Longer Rod Making Mold #1	17
Figure 3. 3 Clamshell Mold Setup Showing Mold and Piston Assembly	19
Figure 3. 4 Clamshell Mold in the Hydraulic Press.....	21
Figure 3. 5 Carbon Fuel Rod Within An Opened Mold	22
Figure 3. 6 The Finished Green Carbon Fuel Rod.....	23
Figure 3. 7 Rod Resistivity-Testing Apparatus.....	27
Figure 3. 8 Schematic of Rod Resistivity-Testing Apparatus.....	28
Figure 3. 9 Leco TGA 701 Thermo-Gravimetric analyzer	31
Figure 3. 10 Thermoquest Inc. Flash EA-1112 CHNS-O Elemental Analyzer.....	32
Figure 4. 1 Temperature of Clamshell Mold's Inner Chamber during Heat-Up Regimen	35
Figure 4. 2 Temperature of Clamshell Mold's Inner Chamber during Cool-Down	35
Figure 4. 3 Cenfuel/Pet-Coke Rods: Resistivity vs. Composition.....	38
Figure 4. 4 Cenfuel/Pet-Coke Rods: Resistivity vs. Density	39
Figure 4. 5 Cenfuel/Pet-Coke Rods: Density versus Composition.....	40
Figure 4. 6 SECO/Pet-Coke Rods: Resistivity vs. Composition	44
Figure 4. 7 SECO/Pet-Coke Rods: Resistivity vs. Density	45
Figure 4. 8 SECO/Pet-Coke Rods: Density vs. Composition	46
Figure 4. 9 SARA DCFC Performance with Coal-Derived Fuel vs. Graphite	47
Figure 4. 10 Anode Polarization Curves for Cenfuel/pet-coke Fuel Rods at 600 °C in NaOH+LiOH melt, 0.5l/min airflow humidified at 70°C	48

Chapter 1

Introduction

The future energy needs of the United States, as well as the well-being of the environment, are currently much debated topics. Natural gas power plants generate most of this country's electricity today because they require less capital cost than do coal-fired power plants. It is expected, however, that gas prices will soon skyrocket, far surpassing those of coal, making the use of coal a more economically viable option. There are also, of course, rising concerns with the availability of gas supplies in the future, again, directing focus upon the use of coal. Another reason why new energy ideas are focusing on coal is that it makes up 90% of this country's fossil energy resources, and it is, thus, the most abundant domestic fuel. [13]

Today, coal-fired power plants generate about 55% of this nation's electricity, which consumes 85% of the total U.S. coal production. [13] Some drawbacks to generating electricity with coal-fired plants are that they are quite inefficient as well as environmentally non-friendly. It has been estimated that coal-fired power plants obtain a maximum efficiency of ca. 35%. [6] In a typical coal power plant, the heat from combustion is used to boil water, which makes steam that powers a steam turbine-generator. "Only a third of the energy value of coal is actually converted into electricity...the rest is lost as waste heat." [5] "Out of the entire electric industry, coal-fired power plants contribute 96% of sulfur dioxide emissions, 93% of nitrogen oxide emissions, 88% of carbon dioxide emissions, and 99% of mercury emissions." [17]

The present research focuses on using the U.S.' abundant coal reserves to create carbon fuels for the production of energy in a much more efficient and environmentally friendly manner via the use of a Direct Carbon Fuel Cell (DCFC).

The DCFC utilizes a process by which carbon is electrochemically converted directly to electricity. Because of the thermodynamics involved, theoretical cell efficiency can be as high as 80%, a sharp contrast to conventional coal-fired power plants. The cell is environmentally friendly because it could use carbon made from the solvent extraction of coal, which eliminates most of the volatiles, ash, and sulfur from the fuel. Due to cell design, most remaining NO_x, sulfur, and fly-ash byproducts are captured. Also, the cell produces a concentrated carbon dioxide stream, which is easier and cheaper to capture.

The first fuel cell operating upon this principle was made in the late 1800's by Dr. William Jacques. Many attempts to develop a DCFC have been made. In the mid 1990's Scientific Applications & Research Associates (SARA), Inc. developed a DCFC based on a molten metal hydroxide electrolyte. By 2004, SARA had developed an operational cell and contacted West Virginia University (WVU) for help in providing a solid carbon fuel that would also work as the DCFC's anode. This research is dedicated to that end.

1.1 Scope of Research

The aim of this project is to investigate the use of coal-derived feed-stocks as the carbon fuel for SARA's DCFC. The SARA design of the DCFC requires the fuel to be solid as well as act as the cell's anode. Thus, one requirement of the fuel created in this study was that it must be electrically conductive. Due to the cell's structure, the fuel

must also be a durable rod-shaped solid. The fuel should be low in sulfur to meet environmental constraints, and it should also have a low ash content since ash poisons the electrolyte. Finally, the fuel must be low in cost to satisfy overall economic requirements.

The project goals were as follows:

- To develop a method for creating solid cylindrical carbon fuel rods.
- To discover the least amount of binder necessary to provide the fuel rod with structural integrity.
- To discover the most economical yet still effective mixture of petroleum coke and de-ashed coke.
- To characterize rod properties including density, resistivity, composition.
- To determine if a WVU coal-derived carbon can be used as a substitute for petroleum coke.
- To test the various carbon rods for their performance in the DCFC.

Chapter 2

Background

In this chapter, the basic coal and coal technology concepts employed in this research are discussed. This discussion also provides a brief history of the DCFC and a review of its fundamentals.

2.1 Coal Defined

Coal is a complex heterogeneous substance. It varies in appearance and properties depending on its type and source, and ranges from “a soft, moist, brownish material to a very hard, glossy, black solid.” [15] It is an organic sedimentary rock containing varying amounts of carbon, hydrogen, nitrogen, oxygen, sulfur, and trace amounts of other elements. [19] Because of coal’s complexity and non-homogeneity, it has presented researchers with great difficulty with respect to its characterization and structural correlation. [18]

2.1.1 Coal Formation

Coal is formed from plant matter that is converted over tens or hundreds of millions of years through a process known as coalification. The first step in coal formation occurs when dead plant matter is attacked by anaerobic bacteria and disintegrates into a carbon rich residual material. This residual material accumulates as more and more plants disintegrate, resulting in the compression of the bottom layers. This compressed plant matter, along with residual organic debris, sulfur, and whatever

inorganic species were present in the area is called peat. Following the formation of peat, some form of environmental change causing the deposition and growth of sedimentary layers on top of the peat is necessary for the continuation of the coalification process. The weight of the sediments slowly compacts the peat and forces it deeper into the ground, where it is exposed to elevated temperatures fueled by subterranean heat sources. Over time, this increase in pressure and temperature transforms the peat into brown coal or lignite. With continued exposure to pressure and heat, more mature, and thus better grades, of coal are created. Anthracite is the oldest coal and thus, also the highest ranking. [15], [8]. Coal varies not only by maturity, but also by the unique ingredients and conditions that formed it. Thus, a system of coal classification is necessary.

2.1.2 Coal Classification

Coal deposits occur throughout the whole planet and thus, many classification systems have been developed worldwide. Unfortunately, the terms used to describe the same coals are not used uniformly in these systems. [19] In the United States, the coal classification system was created by the American Society for Testing and Materials (ASTM). The ASTM classification system is based on coal rank, indicating degree of geological maturity, as stated earlier. Volatile matter, and fixed carbon amounts, obtained via proximate analysis, as well as heating values are used as indicators of rank. [15] The ASTM system for classifying coals by rank is shown in Table 2.1

Table 2. 1 The ASTM System for Classifying Coals by Rank

Class	Group	Fixed Carbon^a	Volatile Matter^a	Heating Value^b
Anthracitic	metaanthracite	>98	<2	
	anthracite	92-98	2-8	
	semianthracite	86-92	8-14	
Bituminous	low-volatile	78-86	14-22	
	medium-volatile	69-78	22-31	
	high-volatile A	<69	>31	>14,000
	high-volatile B			13,000-14,000
Subbituminous	high-volatile C			10,500-13,000
	subbituminous A			10,500-11,500
	subbituminous B			9500-10,500
Lignitic	subbituminous C			8300-9500
	lignite A			6300-8300
	lignite B			<6300

^aThe fixed carbon and volatile matter, reported as percentages, are determined on a dry, mineral-matter-free basis. The mineral matter is calculated from the ash content by the Parr formula: mineral matter = 1.08 [percent ash + 0.55 (percent sulfur)].

^bThe heating value, reported in British thermal units per pound, is expressed on a moist, mineral-matter-free basis. The moisture content is the bed moisture or equilibrium moisture of the coal after equilibration with a nominally 100% relative humidity atmosphere. Some overlap occurs in the heating-value range of subbituminous A and high-volatile C coals. Coals with heating values between 10,500 and 11,500 are classified as high-volatile C bituminous if they display caking properties and as subbituminous A if they do not. [15]

2.1.3 Structure of Coal

Determining an overall chemical structure for coal has been problematic. How does one assign a structural correlation to a complex heterogeneous substance? Although coal is composed of compounds that have varying molecular weights and compositions, there are some structural features that are common to all bituminous coals and to some subbituminous ones as well. It has been pointed out that the “carbon skeleton of coals can be viewed as consisting of hydroaromatic structures.” [20] The higher the rank of the coal, the higher its aromaticity. A number of attempts have been made to represent coal

structure using molecular weight, size, and linkage between aromatic units, elemental composition, aromatic-to-aliphatic carbon ratio, as well as experiments with chemical and thermal reactions. [18] Perhaps the most popular model of bituminous coal, however, was proposed by Wiser, which can be seen in Figure 2.1 [18]. This model captures the main structural groups as well as the incorporation of hetero-atoms in the structure.

Figure 2. 1 Proposed Structure of Coal as Put Forth by Wiser Showing Main Structural Groups in Coal

2.2 Carbonization

Coal carbonization is accomplished by heating coal in an oxygen-free environment to produce a solid, porous, carbonaceous residue, called coke, and evolve volatile products such as tar which can be later treated to produce pitch. [21]

2.2.1 Coke

Almost all of the coke produced by the carbonization process is used in blast furnaces for smelting iron ore. In general, coke has an 85-90% carbon content. Coke can also be obtained by the delayed coking of petroleum. Some forms of petroleum coke (pet-coke) are used in the aluminum industry to extract aluminum from aluminum oxide ore. In this thesis research, pet-coke is used as an ingredient in the carbon fuel created for the DCFC. [7]

2.2.2 Pitch

Most coal-derived carbon pitches are made as a byproduct from the production of coke. Pitches can also be made by the solvent extraction of coal under mild conditions. Pitch can also be derived as a by-product of petroleum refining. There are many end uses for pitch, one of which is as a binder in the production of electrodes where it holds coke particles together. [4]

2.3 WVU Solvent Coal Extraction

Solvent extracted carbon ore (SECO) is a low-ash extract material produced at West Virginia University by the solvent extraction of raw coal using standard pure solvents. It was found that N-methylpyrrolidone (NMP) was a superior solvent for the

reflux extraction of coal at the normal boiling point of the solvent (around 202 °C). [4]
SECO has many uses such as a being a carbon-product precursor, a clean fuel, or as an ingredient in some of the solid fuel rods created for the DCFC in this research.

2.4 The Direct Carbon Fuel Cell

The Direct Carbon Fuel Cell (DCFC) utilizes the direct electrochemical conversion of carbon to electricity. What follows, is a brief history of the DCFC, and a description of how the technology is now practiced at SARA.

2.4.1 Brief DCFC History

The first attempts at making a Direct Carbon Fuel Cell can be traced back to the late 1800's with Dr. William Jacques, an electrochemist from Boston. His cell consisted of a steel pot of molten sodium hydroxide (caustic soda). He bubbled air through the pot and placed in it a carbon rod. He later received a patent for his work titled "Method of Converting Chemical Energy of Carbon into Electrical Energy." Although many at that time doubted the feasibility of this process, it was later discovered that Dr. Jacques was actually on the right track.

The next breakthrough for the Direct Carbon Fuel Cell came in the early 1970's and arose mostly due to the oil crisis that was occurring at that time. Dr. Robert Weaver, who worked for the Stanford Research Institute, proved that the electrochemical oxidation of carbon was indeed feasible. Dr. Weaver and his group built a number of half cells and tested many different carbons in molten sodium carbonate. Some of their findings indicated that a coal derived anode is far more active electrochemically than is

graphite. Then in the 1980's oil prices dropped and DOE funding for carbon fuel cell research was suspended.

In 1995 SARA began its work on the DCFC. They chose to pursue a molten metal hydroxide electrolyte and a solid anode type of a cell, similar to the original of Dr. Jacques. SARA's initial runs were conducted mostly with graphite anodes, and they soon discovered that coal derived ones gave superior performance.

In parallel with the work being done at SARA, alternate designs for the DCFC are being researched at the Lawrence Livermore National Laboratory, and at CellTech Power.

2.4.2 SARA DCFC Design

The SARA type of fuel cell is unique, because the carbon fuel also acts as the cell's anode (i.e. it is used to withdraw electrons from the cell). Figure 2.2 depicts the SARA DCFC design.

Figure 2. 2 SARA DCFC Showing Molten Hydroxide Electrolyte Bath and Carbon Fuel/Anode Rod

The main reactions that drive the molten hydroxide DCFC are as follows:

Humidified air is bubbled in through the bubbler element, as seen in Figure 2.2, and reacts with water at the cathode and makes hydroxide. The hydroxide then diffuses to the carbon anode surface, where they meet to make CO_2 and water, and of course,

produce electrons. The process may be thought of as a controlled combustion of carbon, but electricity is produced directly, without the parasitic energy losses involved in ordinary combustion in a coal-fired power plant. This allows the cell thermodynamic efficiency to reach nearly 80%. [10] SARA's DCFC delivers ca. 1 volt DC.

2.4.3 Fuel Specifications Based on SARA's Cell Design

Because of SARA's Cell design, the fuel acts as the anode. Thus, the solid carbon fuel created at WVU must be electrically conductive. The fuel, which is rod-like in shape, must also be a durable solid to withstand handling. The fuel should be environmentally friendly by being low in sulfur, it should also have a low ash content as ash poisons the electrolyte, and, most importantly, the fuel should be inexpensive.

Chapter 3

Experimental

This chapter deals with the feed materials used to make the solid carbon fuel rods for the DCFC, and the experimental techniques employed. It also describes the methods used to analyze the feed materials as well as the carbon rods.

3.1 Fuel Ingredients

The Direct Carbon Fuel Cell designed by SARA is unique in that the solid carbon fuel rod acts not only as the fuel, but also as the cell's anode. This presents a number of challenges in tailoring the perfect fuel: it must be a cylindrical solid to fit the cell's geometry, it must also be durable for practical reasons, to successfully fulfill its role as the cell's anode, the solid carbon fuel rod must be sufficiently conductive, it must have a low ash and sulfur content, and finally, it must be inexpensive. Based on these constraints, a number of materials were chosen to be incorporated into the fuel mixture, which include Petroleum Coke, Binder Pitch, Cenfuel, and Solvent Extracted Carbon Ore.

3.1.1 Petroleum Coke (pet-coke)

Certain types of pet-coke are anisotropic in nature and therefore should have a low electrical resistivity. It was initially chosen as a main fuel ingredient for that reason. The price of anode grade pet-coke is around \$200/Ton.

3.1.2 Binder Pitch

Binder pitch was chosen to be utilized as a “glue” to hold all the ingredients together. When coked, it has desirable conductive properties which may add to the electrical conductivity of the fuel. The binder pitch in this research is Koppers Coal Tar Pitch with a 110 °C softening point. Binder pitch costs about \$300/Ton.

3.1.3 Cenfuel

Cenfuel is essentially a coal that has been treated with hydrofluoric acid, which dramatically reduces its ash content. The Cenfuel for this research was obtained from SARA. It is made by CarbonXT (formerly named Cenfuel).

3.1.4 Solvent Extracted Carbon Ore (SECO)

The SECO utilized in this research is produced in the WVU labs. It is derived from raw Kingwood coal, mined in Preston Co., WV. A description of the lab production of SECO follows in Section 3.7. Like Cenfuel, it was chosen as a substitute for the pet-coke because it is a much greener material. The price of coked SECO is highly dependent on the efficiency of solvent recovery and in previous estimates was about \$200/Ton. [4]

3.2 Rod Production

In this section the evolution of methods and equipment used to produce the solid carbon fuel rods is discussed.

3.2.1 Stage One - The Disc

The first attempt at rod production involved making a small disc $\frac{3}{4}$ -inch in diameter and $\frac{1}{2}$ -inch in height. This type of disc was created to see whether the fuel ingredients could be cold, dry pressed in a mold. The bottom plug of the steel mold is inserted into the inner chamber, followed by the fuel mixture as shown in Figure 3.1. A steel piston is then inserted, and the cell pressed with a hydraulic press to 1000 lb_f.

Figure 3. 1 Earliest Model of a Fuel Rod Shaping Mold

After the fuel mixture is pressed, the bottom plug is removed and the disc sample is extracted. Three such discs were made, and although successful they were rather brittle. It was decided that some form of heating was necessary for the following scaled-up technique.

3.2.2 Stage Two - Making a Longer Rod

SARA's cell specifications call for a rod of 5 to 10 inches long and $\frac{3}{4}$ -inch in diameter. Thus a new mold and press set up was developed. A hydraulic piston was set in a steel frame as can be seen in Figure 3.2.

Figure 3. 2 Hydraulic Piston and Longer Rod Making Mold #1

Similar to the set up for the disc mold, the fuel ingredients are mixed and added to a stainless steel cylindrical mold. The mold was heated, then secured into place, and pressed with the hydraulic piston. The heating regimen was a trial and error process that was based initially on the softening point of the binder pitch. Mixing and heating methods, as well as softening point determination are discussed in Sections 3.3, 3.5, 3.4 respectively.

The setup shown in Figure 3.2 was problematic for two reasons. First, the fuel mixture adhered to the inner walls of the mold so it could not be removed from the mold intact. Varying types of lubricants were applied to the inner chamber of the cylinder

prior to inserting the fuel mixture with little success. Second, the pressure necessary to create a fuel rod approached 3000 psi (1300 lbs), and proved too high for the steel frame, which buckled on the bottom. This set up was deemed unsuccessful and also unsafe.

3.2.3 Stage Three – Clamshell Mold

In the third, and final setup, the hydraulic piston in a metal frame was abandoned for the more stable Carver press (hydraulic unit model # 3912) which was used to press the initial disc mold. The cylindrical shaped mold was replaced by a new clamshell mold, depicted in Figure 3.3. The mold is 16 in. long and the inner diameter is 3/4 in. The mold body and piston are made out of 304-stainless steel.

Figure 3. 3 Clamshell Mold Setup Showing Mold and Piston Assembly

This new clamshell mold is capable of being opened for easier rod extraction. The inside of the mold is sprayed with three layers of SPRAYON Dry Graphite Lube lubricant (product # S00204). Each layer is allowed to dry completely prior to the application of the next, to ensure proper coating. The two clamshell pieces are then aligned, and the bolts are tightened. The bottom disc plug is inserted and a layer of aluminum tape, wrapped around the bottom of the mold body, keeps it in place. The pre-mixed fuel ingredients are then sprinkled into the mold from the top orifice and are packed with a piston by hand after ca. every two grams. 95 grams in total are stuffed into the mold and packed in this fashion. After the fuel mix is in the mold, the top separator disc is inserted and the mold is heated. The top separator disc was not initially employed, but it was found that the warm fuel would stick to the piston without it; because of the limited vertical space available in the Carver press, it is necessary to use incrementally longer pistons (short piston to begin with, then as the fuel is compressed more and more, the piston is replaced by a longer one) which is difficult if the fuel is stuck to the piston. Figure 3.4 is a picture of the mold with a piston, inside the press. The packed mold is heated in an oven to 210 °C for one hour prior to pressing in the Carver press. This enables the binder pitch to soften and wet the coke particles in the mixture.

Figure 3. 4 Clamshell Mold in the Hydraulic Press

The fuel mixture is pressed to 5000 lbs from the top. The pressure is released, the aluminum tape is removed and the mold is inverted and pressed again. It is then allowed to cool under pressure for 30 minutes. After cooling, the pressure is released, the bolts are loosened and removed, and the two sides of the mold are separated. Figure 3.5 shows an open mold with a carbon fuel rod in it.

Figure 3. 5 Carbon Fuel Rod Within An Opened Mold

Once the mold is open, the rod is gently removed by applying pressure to the entire surface area of either the top or bottom, with a piston by hand. The finished carbon fuel rod is shown in Figure 3.6.

Figure 3. 6 The Finished Green Carbon Fuel Rod

The mold and all other parts are then thoroughly cleaned with solvent.

3.3 Mixing the Fuel Ingredients

Prior to mixing, the Cenfuel, pet-coke, and SECO are ground using a Retsch (model SK 100) grinder to particles equal to or less than 0.12 mm in size. The binder pitch is ground using a conventional Kitchen Aid coffee grinder (model # BCG100ER1). Dry ice is inserted into the coffee grinder along with the pitch to prevent the frictional and motor heat from softening the pitch.

Once all ingredients have been ground, they are weighed out, combined in pre-determined quantities for a total of 100 grams, and then blended. To ensure a homogeneous sample, the fuel ingredients are blended in a Osterizer kitchen blender (model # 6630) for two minutes. Then the contents in the pitcher are hand blended and

all fuel ingredients are scraped off of the sides. This process is repeated a total of four times. The material is recovered from the blender and 95 grams are weighed out to be inserted into the mold.

3.4 Softening Point Determination

In order to determine the heating regimen that will ensure the best adhesion of the fuel ingredients, it is essential to first find the softening point of the binder pitch. The softening point is tested according to ASTM D3104 via a Mettler FP80HT central processor and a Mettler dropping cell. [1] The sample holder was filled and heated until the sample melted. Once the sample cooled, the sample holder was placed into the dropping cell. The device was set to heat at a rate of 2 °C/min. When the sample begins to flow and breaks the light beam below the sample holder, the softening point is automatically recorded. If the pitch has a softening point near 100 °C, it is well suited for binding purposes. It was found that the pitch sample used here had a softening point of 110 °C.

3.5 Heating Regimen

Initially, molds containing a fuel sample of 15% (by weight) binder pitch and 85% pet-coke, were heated to 180 °C at a rate of 10 °C/min., 70 °C above the softening point of the pitch, and soaked at this temperature for one hour. Molds heated to this temperature did not produce successful rods. Thus, the heating temperature was raised to 210 °C, also at 10 °C/min. and soaked there for 1 hour.

To ensure proper softening of the pitch, the core temperature of the fuel ingredients was monitored during the entire heating and cool down process with a thermocouple that was inserted into the middle of the mold's inner chamber.

210 °C with 1 hour of soaking provided a high enough core temperature (about 170 °C) for the pitch to completely soften and coat all of the surrounding particles. Molds heated according to this regimen yielded successful rods.

3.6 Binder Pitch Levels

While experimenting with an efficient method of creating a solid carbon fuel rod, higher percentages, by weight, of binder pitch were used to facilitate rod production. Once a rod making method was established, it was necessary to determine the lowest amount of binder pitch that could be successfully used to minimize cost.

Rods were made with varying binder pitch levels to determine the lowest amount that would consistently yield a successful carbon fuel rod. Weight percent ranges from 5 to 25% were tested. The lowest amount of pitch blended with the other fuel ingredients that yielded a successful rod was found to be 10%. However, it proved difficult to work with that percentage as successful rods could not be consistently created. Thus, 15% binder pitch, by weight, was used for all subsequent rods.

3.7 SECO Production

Solvent extraction is a technique by which most of the ash and other environmentally harmful impurities found in coal are removed to obtain a purer source of carbon, for use in various carbon products. Many solvents can be used for the solvent extraction of raw coal. N-methyl Pyrrolidone (NMP) is used in this laboratory scale

research, because it was proven to be a superior solvent by prior research conducted at WVU. The solvent and coal are mixed in at 10 to 1 ratio (i.e. for ten grams of coal, 100mL of solvent are utilized).

The solvent is placed in a flat bottom flask connected to a reflux condenser and electrically heated and stirred via a Fisher Scientific Isotemp hot plate. Once the solvent is heated, raw Kingwood coal is added. The extraction is carried out under total reflux conditions. The mixture is heated to 202 °C, the boiling point temperature of NMP under a nitrogen blanket while continuously stirring. Once the mixture begins to boil, the temperature is reduced slightly to prevent violent boiling, and kept there for 30 min.

The heated mixture is then vacuum filtered to remove all solid impurities such as ash and un-dissolved coal with G6 Fisher brand Glass Fiber Filter Circles, retaining particle sizes greater than 1.6 μm . The solids are discarded and the filtrate is then placed in a rotovap under reduced pressure to remove and recover the solvent.

The solid remainder is then put in a vacuum oven at 180 °C over night under nitrogen to drive off volatiles and any remaining solvent. Once completely dry, the material is coked by heating in an inert atmosphere to 500 °C at a rate of 10 °C/hour. Upon reaching 500 °C, the heating-rate is increased to 25 °C/h to a temperature of 1000°C and soaked there for 10 hours. The SECO is then ready for grinding.

The Cenfuel was coked as received by the same procedure described for the SECO.

3.8 Rod Resistivity

Because the solid carbon fuel rods act as the anode in the SARA Direct Carbon Fuel Cell, it is vital for the fuel rods to conduct electricity. To measure the resistivity of

the rods, ASTM C 611 “Standard Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature” was followed. To that end, a four point system was assembled consisting of a power supply, current meter, variable resistor, volt meter, and a carbon rod holder that has two copper plates on both ends for electrical contact with the rod. The assembled system is shown in Figure 3.7.

Figure 3. 7 Rod Resistivity-Testing Apparatus

The current is measured by a digital amp meter (Keithley Model 2000) that is in series with the carbon fuel rod. The voltage drop across the rod is measured with a second digital volt meter (Keithley Model 2000) that is connected to the rod via two pointed contact pins over a measured distance. The circuit schematic is shown in Figure 3.8. The variable resistor allows for precise control of the current in the circuit.

Figure 3. 8 Schematic of Rod Resistivity-Testing Apparatus

The resistivities are calculated by the following formula:

$$\rho = \left(\frac{V}{I} \right) \left(\frac{A}{L} \right) (10^3)$$

Where ρ is resistivity ($\mu\Omega\text{-m}$)

I is current (amps)

V is voltage (volts)

A is the rod's cross-sectional area (mm^2)

L is length between contact points on the rod (mm)

3.9 Rod Density

Because the resistivity of the carbon fuel rods is a strong function of rod density, experiments were conducted to determine the reproducibility with respect to rod density, of the rod making technique. Two sets of three rods were created, one set with 75% Cenfuel and 25% pet coke (combined with 15% by weight of binder pitch), and a second set made with a 50% Cenfuel 50% pet-coke mixture (again, combined with 15% by weight of binder pitch). The density of these six rods is tested by dividing each rod's weight by its volume.

3.10 Cenfuel, Pet-Coke Rods

Initially, rods were made with binder pitch and pet-coke only. Pet-coke is expensive and relatively high in both sulfur and ash. It is expected to have a low electrical resistivity, although this varies depending on the grade of the pet-coke. It is therefore vital to determine the least amount of petroleum coke necessary to keep the electrical conductivity of the carbon fuel rods at a satisfactory level while blending into the rod some other greener carbon source.

While Cenfuel has a rather high amount of ash, its low sulfur content makes it an attractive fuel additive. Rods of the following compositions were created to that end:

0% Cenfuel, 100% Petroleum Coke
25% Cenfuel, 75% Petroleum Coke
50% Cenfuel, 50% Petroleum Coke
80% Cenfuel, 20% Petroleum Coke
100% Cenfuel, 0% Petroleum Coke

Note that all rods have 15% pitch by weight.

3.11 SECO, Pet-Coke Rods

Just as with Cenfuel, rods with varying amounts of SECO and petroleum coke were created to determine the lowest amount of pet-coke that can be tolerated while keeping electrical conductivity high.

The rods that were fabricated are as follows:

0% SECO, 100% Petroleum Coke
25% SECO, 75% Petroleum Coke
50% SECO, 50% Petroleum Coke
75% SECO, 25% Petroleum Coke
100% SECO, 0% Petroleum Coke

Again, all rods have 15% pitch by weight.

3.12 Proximate Analysis

The proximate analysis is a set of procedures that determine moisture, volatile matter, ash content, and fixed carbon percentages in various coal and carbon materials. [14] In the present research, a TGA 701 Thermo-Gravimetric analyzer by LECO is used according to the ASTM D 3174 method for ash determination. This instrument, shown in Figure 3.9, measures the weight loss of the sample as a function of temperature and time in a set atmosphere. About 2 grams of sample are placed in a silica crucible located on a rotating wheel which passes a balance as it rotates at a set frequency. Temperature profiles are monitored and recorded with the aid of an attached computer.

Figure 3. 9 Leco TGA 701 Thermo-Gravimetric analyzer

3.13 Elemental Analysis

The elemental analysis, sometimes referred to as ultimate analysis, is used to determine the elemental composition of the sample with respect to carbon, hydrogen, sulfur, and nitrogen. [14]

In the present research, the elemental analysis is carried out using a Thermoquest Inc. Flash EA-1112 CHNS-O elemental analyzer, shown in Figure 3.10. This instrument utilizes chromatography to determine the elemental content of a sample. The instrument is calibrated using a standard compound with known elemental composition. The sample

is weighed in a tin container and dropped into the reactor, which is maintained at 900 °C, where the sample combusts in the presence of excess oxygen in excess. The oxides that are produced are separated by the gas chromatograph and their relative amounts are determined by a thermal conductivity detector with reference to the standard.

Figure 3. 10 Thermoquest Inc. Flash EA-1112 CHNS-O Elemental Analyzer

Chapter 4

Results and Discussion

In this chapter, the results obtained in creating a carbon fuel for the direct carbon fuel cell are presented and discussed.

4.1 Elemental and Proximate Analyses of Fuel Ingredients

Both elemental and proximate analyses were conducted for the raw Kingwood coal, coked SECO, coked Cenfuel, pet-coke, and as-received coal tar pitch. Table 4.1 shows the elemental and proximate analyses results for these materials.

Table 4. 1 Elemental and Proximate Analyses of Fuel Ingredients

Elemental Analyses	Nitrogen	Carbon	Hydrogen	Sulfur
Kingwood Coal	1.18	77.4	4.95	1.58
SECO (coked)	1.78	85.8	5.83	0.64
Cenfuel (coked)	1.99	89.9	2.50	0.49
Pet-Coke	1.33	94.8	0.02	2.03
Coal Tar Pitch	1.47	91.2	4.86	0.00
Proximate Analyses		Volatile Matter (as received)	Fixed Carbon (as received)	Ash (as received)
Kingwood Coal		33	58	8.92
SECO		32	67.82	0.18
Cenfuel		30.74	66.57	1.65
Pet-Coke		1.11	97.28	0.56
Coal Tar Pitch		44.74	54.72	0.11

As can be seen from Table 4.1, both SECO and Cenfuel have very low sulfur contents. However, SECO dramatically outperforms Cenfuel in terms of ash content and is therefore a better candidate for use as a fuel ingredient in the carbon fuel rods.

4.2 Pitch Softening Point

The softening point of the Koppers Coal Tar Pitch used in the carbon fuel rods, tested according to ASTM D3104 via a Mettler FP80HT central processor and a Mettler dropping cell, was found to be 110 °C. It is therefore deemed suitable for use as a binder in the rods.

4.3 Rod Production

It was found that the combination of a clamshell mold, piston, and stable hydraulic press is the most successful rod producing setup (detailed description found in Section 3.2.3).

The best heating regimen of the fuel ingredient-full mold involved raising the temperature of the oven containing the filled mold to 210 °C at a rate of 10 °C/min., and soaking it at that temperature for 1 hour. The core temperature of the fuel ingredients was monitored during the entire heating and cool down process with a thermocouple that was inserted into the middle of the mold's inner chamber. When the mold is heated to 210 °C for one hour, the core temperature of the fuel reaches about 175 °C, and is enough to completely soften the pitch, as is shown in Figure 4.1.

Figure 4. 1 Temperature of Clamshell Mold's Inner Chamber during Heat-Up Regimen

The rod must be pressed before the pitch and other fuel ingredients cool down and harden. Figure 4.2 demonstrates the temperature of the inner chamber during cool down following removal from the oven, and indicates that the pressing procedure must be completed within approximately 15 minutes of rod removal from the heating oven.

Figure 4. 2 Temperature of Clamshell Mold's Inner Chamber during Cool-Down

4.4 Cenfuel and Pet-Coke Rods

To determine the least amount of petroleum coke necessary to keep the electrical conductivity of the carbon fuel rods at a satisfactory level while blending it with the greener Cenfuel, rods of the following compositions were created.

0% Cenfuel, 100% Petroleum Coke
25% Cenfuel, 75% Petroleum Coke
50% Cenfuel, 50% Petroleum Coke
80% Cenfuel, 20% Petroleum Coke
100% Cenfuel, 0% Petroleum Coke

All the rods were made with 15% pitch by weight.

4.4.1 Elemental and Proximate Analysis

Table 4.2 shows the elemental analysis and ash content for these Cenfuel/pet-coke rods.

Table 4. 2 Elemental Analysis and Ash Content for Cenfuel/Pet-Coke Rods (all rods contain 15% binder pitch by weight).

Coke Blend	Nitrogen%	Carbon%	Hydrogen%	Sulfur%	Ash
0% Cenfuel,100% Pet-Coke	1.22	97.53	0.01	2.01	0.67
25% Cenfuel, 75% Pet-Coke	1.57	93.61	0.05	1.67	0.80
50% Cenfuel, 50% Pet-Coke	1.68	97.21	0.08	1.33	1.06
75% Cenfuel, 25% Pet-Coke	1.37	88.61	0.14	0.94	1.12
80% Cenfuel, 20% Pet-Coke	1.36	93.08	0.12	0.96	1.46
100% Cenfuel, 0% Pet-Coke	1.23	92.19	0.21	0.37	1.59

It is evident from this table that as the amount of Cenfuel increases in the rod, the ash increases and the sulfur content dramatically decreases, as is to be expected from the values of the individual fuel ingredients shown in Table 4.1.

4.4.2 Density and Resistivity

The density and the resistivity of the Cenfuel/pet-coke rods were tested according to the methods described in Sections 3.8 and 3.9. Table 4.3 shows the densities and resistivities of the Cenfuel/pet-coke rods.

Table 4. 3 Resistivities and Densities of Cenfuel/Pet-Coke Rods

Rod	Resistivity ($\mu\Omega\text{-m}$)	Density (g/cc)
0% Cenfuel,100% Pet-Coke	202	1.52
25% Cenfuel, 75% Pet-Coke	317	1.41
50% Cenfuel, 50% Pet-Coke	298	1.23
75% Cenfuel, 25% Pet-Coke	235	1.25
80% Cenfuel, 20% Pet-Coke	140	1.40
100% Cenfuel, 0% Pet-Coke	109	1.36

Plots of Cenfuel concentration against rod resistivity were prepared to determine whether a correlation exists between these two parameters. The data are presented in Figure 4.3

Figure 4. 3 Cenfuel/Pet-Coke Rods: Resistivity vs. Composition

It is not clear that there is a direct correlation between the Cenfuel content in a rod and its resistivity. However, the data suggests a decreasing resistivity with an increase in Cenfuel content. A plot of the resistivity of the rods against their densities also shows no evident correlation between these values, as can be seen in Figure 4.4. This is perhaps due to the small range of density variation within this group of rods. It is also feasible that the low resistivity of the Cenfuel/pet-coke rods, regardless of content ratio, is due to the low resistivity of the binder pitch which is present throughout all the rods. These results are interesting in that the resistivity of the 100% pet-coke rod was higher than that of the 100% Cenfuel rod, which was thought initially to have much higher resistivity. Thus it appears that pet-coke is not needed at all as an additive to enhance resistivity. It is suspected that the grade of pet-coke acquired for these tests was not quality anode or

needle coke. This might explain the high resistivity of the rods made with higher levels of pet-coke.

Figure 4. 4 Cenfuel/Pet-Coke Rods: Resistivity vs. Density

Figure 4. 5 Cenfuel/Pet-Coke Rods: Density versus Composition

Figure 4.5 is a plot of Cenfuel concentration versus rod density, and shows that although there is density variation within this group of Cenfuel/pet-coke rods, it is indeed slight. The same can be said for the resistivities of these rods. Apparently independent from Cenfuel concentration, rod resistivity remains rather constant from one rod to another. It is suspected that the variation in properties is due to slight differences encountered in the fabrication steps for the rods.

4.5 Density Verification

Because there was apparent variation in rod densities, and since the resistivity of the carbon fuel rods is believed to be a function of rod density, experiments were conducted to determine the reproducibility, with respect to rod density and resistivity, of the rod making technique. Two sets of rods were created, one set with 75% Cenfuel and 25% pet coke (combined with 15% by weight of binder pitch), and a second set made

with a 50% Cenfuel and 50% pet-coke mixture (again, combined with 15% by weight of binder pitch). Tables 4.4 and 4.5 show the density and resistivity results for these rods.

Table 4. 4 Density and Resistivity values Rods made from 75% Cenfuel and 25% Petroleum Coke

Rod #	Density (g/cc)	Resistivity ($\mu\Omega\text{-m}$)
1	1.236	213.7
2	1.239	256.7
3	1.218	273.1
Average	$1.231 \pm 1\%$	$247.8 \pm 10\%$

Table 4. 5 Density and Resistivity values for Rods made from 50% Cenfuel and 50% Petroleum Coke

Rod #	Density (g/cc)	Resistivity ($\mu\Omega\text{-m}$)
1	1.220	300.1
2	1.218	327.5
3	1.221	345.8
4	1.259	319.8
Average	$1.229 \pm 2\%$	$323.3 \pm 7\%$

These tables show that although there is 1-2% variation in density and 7-10% variation in resistivity, generally, the numbers are consistent and that data are acceptable. Hence it appears that the fabrication technique developed here is capable of yielding rods with reproducible properties.

4.6 SECO and Pet-Coke Rods

Just as with Cenfuel, the following rods with varying amounts of SECO and Petroleum Coke were created to determine the lowest amount of Pet-Coke that can be tolerated while keeping electrical conductivity high.

0% SECO, 100% Petroleum Coke
25% SECO, 75% Petroleum Coke
50% SECO, 50% Petroleum Coke
75% SECO, 25% Petroleum Coke
100% SECO, 0% Petroleum Coke

All rods contain 15% binder pitch by weight.

4.6.1 Elemental and Proximate Analysis

Table 4.6 shows the elemental and proximate analysis for these SECO/pet-coke rods. As with the Cenfuel/pet-coke rods, with increasing SECO content in the rods, the sulfur decreases. However, the overall sulfur content is lower with the SECO rods than it is with the Cenfuel counterpart. The overall ash content for these rods is also lower than that observed with ones containing Cenfuel. This is due to the low ash and sulfur contents of both fuel materials.

Table 4. 6 Elemental Analysis and Ash Content for SECO/Pet-Coke Rods (all rods contain 15% binder pitch by weight)

Coke Blend	Nitrogen%	Carbon%	Hydrogen%	Sulfur%	Ash
0% SECO, 100% Pet-Coke	1.22	97.53	0.01	2.01	0.67
25% SECO, 75% Pet-Coke	1.10	83.7	0.00	0.77	0.62
50% SECO, 50% Pet-Coke	1.26	87.6	0.00	0.45	0.54
75% SECO, 25% Pet-Coke	1.22	88.3	0.00	0.24	0.62
100% SECO, 0% Pet-Coke	1.46	90.8	0.01	0.23	0.54

4.6.2 Density and Resistivity

The density and the resistivity of these rods were tested according to the methods described in Sections 3.8 and 3.9. Table 4.7 shows the densities and resistivities of the SECO/pet-coke rods.

Table 4. 7 Resistivities and Densities of SECO/Pet-Coke Rods

Rod	Resistivity ($\mu\Omega$-m)	Density (g/cc)
0% SECO, 100% Pet-Coke	202	1.52
25% SECO, 75% Pet-Coke	136	1.53
50% SECO, 50% Pet-Coke	140	1.52
75% SECO, 25% Pet-Coke	78.6	1.43
100% SECO, 0% Pet-Coke	81.2	1.31

Figure 4.6 is a plot of rod composition versus resistivity. It appears that the resistivity of the rods goes down as the SECO content increases.

Figure 4. 6 SECO/Pet-Coke Rods: Resistivity vs. Composition

Figure 4.7 is a plot of the resistivity of the rods against their densities, and as with Figure 4.4, shows no evident correlation between these values, perhaps due to the small range of density variation from one rod to another.

Figure 4. 7 SECO/Pet-Coke Rods: Resistivity vs. Density

Figure 4.8 is a plot of the amount of SECO present versus the density of the rod.

It appears as though the density of the rods goes down with an increase in SECO.

Figure 4. 8 SECO/Pet-Coke Rods: Density vs. Composition

The overall resistivity for the SECO rods is less than that of the pet-coke ones. It is therefore a good candidate for low ash/low sulfur fuel.

4.7 Results From SARA's DCFC

SARA's research shows that coal-derived material works better than graphite as a fuel in the DCFC which is evident in Figure 4.9.

Figure 4. 9 SARA DCFC Performance with Coal-Derived Fuel vs. Graphite

The first 100 hours of Figure 4.9 portrays cell performance using a coal and petroleum coke derived anode, and for the remaining 70 hours, the same data are shown for a graphite anode. The overall cell voltage drops when the switch from the coal and pet-coke to the graphite anode is made. Therefore it is clear that in this DCFC, coal performs more favorably than graphite.

Figure 4.10 shows the polarization curves (half-cell voltage with respect to a reference electrode vs. current) for the Cenfuel/pet-coke rods at 600 °C in NaOH+LiOH melt at a humidified airflow of 0.5 liters per minute at 70 °C.

Figure 4. 10 Anode Polarization Curves for Cenfuel/pet-coke Fuel Rods at 600 °C in NaOH+LiOH melt, 0.5l/min airflow humidified at 70°C

As can be seen from the anode potential, the higher the Cenfuel content, the better the anode, which is indicated by a much more negative half cell voltage. Although the graphite anode has a lower resistivity, its electrochemical activity in the cell pales to that of the Cenfuel anodes. The anode potentials of all the Cenfuel rods in Figure 4.10 are quite different; this supports the notion that the pet-coke used in the rods is not the main cause of lowered resistivity. It is more likely that the binder pitch and/ or the Cenfuel are the major resistance-lowering factors. The behavior differentiating between the Cenfuel/pet-coke rods can be attributed to Cenfuel's high electrochemical reactivity. To date, SARA has only tested these three rods and the data pertaining to the performance of all other rods in the DCFC is pending.

Chapter 5

Conclusions and Recommendations

5.1 Conclusions

- ◆ A method has been developed for creating solid cylindrical carbon fuel rods.
- ◆ The least amount of binder necessary to hold the fuel rod together has been determined to be 10%, although 15% allowed for easier fabrication.
- ◆ The chemical composition of all the feed materials, as well as of all the rods has been measured.
- ◆ Cenfuel was found to have low sulfur (0.64%) and a relatively high ash (1.65%) content.
- ◆ Conversely, pet-coke had a high sulfur content (2.03%) but had less ash (0.56%).
- ◆ SECO contained the least amount of ash (0.18%) and also contained very little sulfur (0.64%).
- ◆ The densities and resistivities of all the rods have been evaluated.
- ◆ The Cenfuel/pet-coke rods had progressively lower sulfur and higher ash as their Cenfuel content increased.
- ◆ The resistivity of the Cenfuel/pet-coke rods showed some variation with composition but on the whole remained in the range of 109 to 317 $\mu\Omega\cdot\text{m}$. The 100% Cenfuel rod had a lower resistivity than the 100% pet-coke rod. A similar random variation was seen in the density of the rods. This fluctuation is probably due to inconsistency in the fabrication procedure.

- ◆ Careful studies focused on the method of the fabrication procedure showed that when care is taken, the resistivity and the density of the rods varies between $\pm 10\%$ and $\pm 2\%$ respectively.
- ◆ The SECO/pet-coke rods had progressively lower sulfur and approximately constant ash as the amount of SECO was increased. These values of ash and sulfur were consistently lower than those found for the Cenfuel/pet-coke rods.
- ◆ The variation in resistivity for the SECO/pet-coke rods was similar for the Cenfuel/pet-coke rods, with values ranging from 80 to 200 $\mu\Omega\cdot\text{m}$. However, these values are lower than those for the Cenfuel/pet-coke rods indicating that SECO is an ideal candidate material for the DCFC rods. The density of both Cenfuel and SECO/pet-coke rods was comparable.
- ◆ Preliminary test results from SARA for the Cenfuel/pet-coke rods indicated excellent electrochemical performance as the Cenfuel concentration in the rods increased. The coal-derived rods perform significantly better than their graphite counterpart even though the graphite has much lower electrical resistivity ($\sim 6 \mu\Omega\cdot\text{m}$ typically). It appears that the addition of coal-derived material increases the electrochemical activity of the fuel rod.

5.1.1 Summary

The apparent resistivity of the pet-coke provided by SARA is higher than was expected as compared to anode coke. The SECO not only had low sulfur content but outperformed both the 100% Cenfuel and 100% pet-coke rods in terms of lower ash content and lower electrical resistivity. Preliminary SARA tests indicate that the rods

containing coal-derived material have excellent electrochemical activity in their DCFC. This suggests that SECO could be an excellent fuel for the DCFC.

5.2 Recommendations for Future Work

- The effect of binder pitch content on the resistivity of the rods should be studied more closely.
- Additional samples of pet-coke and needle coke with known degrees of anisotropy should be tested if further lower resistivity is required.
- The technique used herein for the fabrication of the rods is tedious and time-consuming. An alternate and preferably continuous process should be developed.
- Further electrochemical testing of the SECO and Cenfuel/pet-coke rods should be conducted to assess their behavior in the DCFC.

Bibliography

1. ASTM C 611 “Standard Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature”, Annual Book of ASTM Standards, 1998
2. ASTM D 3104, “Standard Test Method for Softening Point of Pitches (Mettler Softening Point Method)”, Annual Book of ASTM Standards, Volume 04.04, Philadelphia, Pa., 1995
3. ASTM D 3174, “Standard Test Method for Ash in the Analysis Sample of Coal and Coke from Coal”, Annual Book of ASTM Standards, Volume 05.05, Philadelphia, Pa., 1995
4. Bhole, M., “The Effect of Solvents and Processing Conditions on the Solvent Extraction of Coal”, Thesis (MS), West Virginia University, 2002
5. Department of Energy: <http://www.fe.doe.gov/programs/powersystems/gasification/>
6. Endress and Houser:
<http://www.endress.com/eh/home.nsf/?Open&DirectURL=C9C410E0B81B8525C12568AC0032BDAB>
7. Fitzer, E., Manocha L.M., “Carbon Reinforcements and Carbon/Carbon Composites”, Springer, Germany, 1998
8. Larsen, J.W., “Panel Discussion on the Chemical and Physical Characterization of Coal”, Scientific problems of coal utilization, Technical Information Center DOE, 1978, Proceedings of a conference at West Virginia University, Morgantown, WV May 23-25, 1977, pp. 76-97
9. Larminie, J., Dicks, A., “Fuel Cell Systems Explained”, Wiley, England, 2003
10. LLNL: http://www-cms.llnl.gov/s-t/carbon_con.html
11. Marsh, H., “Introduction to Carbon Science”, Butterworths & Co. (Publishers) Ltd, 1989
12. Personal communications between Scientific Applications & Research Associates Incorporated, 6300 Gateway Dr. Cypress, Ca. 90630 and Dr. John W. Zondlo, Chemical Engineering Department at West Virginia University, Morgantown, WV. 26505.

13. Ruth, L.A., "The U.S. Department of Energy's Combustion 2000 Program: Clean, Efficient Electricity From Coal", *Energy Concerns Management*, Vol. 38, No. 10-13, pp. 1249-1257, 1997
14. Savage, G., "Carbon-Carbon Composites", Chapman & Hall, London, 1993
15. Schobert, H., "The Energy Source of the Past and Future", American Chemical Society, Washington, DC, 1987
16. Seeley W. Mudd Series: "Coal Preparation", edited by: Leonard, J.W., Mitchell D.R., Third Edition, The American Institute of Mining, Metallurgical, and Petroleum Engineers, INC, New York, 1968
17. Sierra Club: <http://www.sierraclub.org/cleanair/factsheets/power.asp#return3>
18. Smith, K.L., Smoot, L.D., Fletcher, T.H., Pugmire, R.J., "The Structure and Reaction Processes of Coal", Plenum Press, New York, 1994
19. Speight, J.G., "Handbook of Coal Analysis", Wiley, Hoboken, NJ, 2005
20. Sternberg, H.W., "Mechanism of Coal Liquefaction: A Challenge to the Organic Chemist", Scientific problems of coal utilization, Technical Information Center DOE, 1978, Proceedings of a conference at West Virginia University, Morgantown, WV May 23-25, 1977, pp. 46-63
21. Tsai, S.C., "Fundamentals of Coal Beneficiation and Utilization", Elsevier Scientific Publishing Company, The Netherlands, 1982

Appendix A

Experimental Raw Data

Table A. 1 Elemental and Proximate Analyses of Fuel Ingredients

	Nitrogen %	Carbon %	Hydrogen %	Sulfur %	Ash
Kingwood Coal	1.18	77.4	4.95	1.58	8.92
SECO (coked)	1.78	85.8	5.83	0.64	0.18
Cenfuel (coked)	1.9908	89.5789	2.4907	0.4772	1.59
Cenfuel (coked)	2.0216	90.1902	2.5050	0.4880	1.70
Cenfuel (coked)	1.9563	89.9680	2.4651	0.5065	N/A
Pet-Coke (coked)	1.3175	94.5684	0.0426	2.0246	0.60
Pet-Coke (coked)	1.3369	94.5395	0.0099	1.9670	0.52
Pet-Coke (coked)	1.3428	95.3102	0.0052	2.0860	N/A
Coal Tar Pitch	1.5393	90.7660	4.8803	0	0.07
Coal Tar Pitch	1.4035	90.8906	4.8439	0	0.15
Coal Tar Pitch	1.4584	91.8091	4.8562	0	N/A

Table A. 2 Elemental Analyses and Ash Content of Batch 1 Cenfuel/Pet-Coke Rods

	Nitrogen %	Carbon %	Hydrogen %	Sulfur %	Ash
85% - 100% Pet-Coke 0% Cenfuel 15% - Coal Tar Pitch	1.2472	95.9146	0.0093	1.9819	0.67
85% - 100% Pet-Coke 0% Cenfuel 15% - Coal Tar Pitch	1.1860	97.1302	0.0058	1.9065	0.67
85% - 100% Pet-Coke 0% Cenfuel 15% - Coal Tar Pitch	1.2286	99.5301	0.0128	2.1355	N/A
85% - 75% Pet-Coke 25% Cenfuel 15% - Coal Tar Pitch	1.6494	93.0353	0.0518	1.7079	0.89
85% - 75% Pet-Coke 25% Cenfuel 15% - Coal Tar Pitch	1.5425	92.6647	0.0366	1.5262	0.89
85% - 75% Pet-Coke 25% Cenfuel 15% - Coal Tar Pitch	1.5246	95.1420	0.0466	1.7672	N/A
85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	1.9970	97.5578	0.0817	1.3548	1.16

Table A.2 Continued

85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	1.5253	95.5530	0.0649	1.2724	1.16
85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	1.5294	98.5063	0.0858	1.3666	N/A
85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	1.4055	87.9137	0.1220	0.9739	1.63
85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	1.3589	89.1275	0.1443	0.9236	1.44
85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	1.3326	88.7941	0.1399	0.9237	N/A
85% - 20% Pet-Coke 80% Cenfuel 15% - Coal Tar Pitch	1.3085	91.5534	0.1195	0.9458	1.47
85% - 20% Pet-Coke 80% Cenfuel 15% - Coal Tar Pitch	1.4204	94.3435	0.1341	0.9607	1.45
85% - 20% Pet-Coke 80% Cenfuel 15% - Coal Tar Pitch	1.3444	93.3528	0.1146	0.9673	N/A

Table A. 3 Elemental and Proximate Analyses of Batch 2 Cenfuel/Pet-Coke Rods

	Nitrogen %	Carbon %	Hydrogen %	Sulfur %	Ash
85% - 75% Pet-Coke 25% Cenfuel 15% - Coal Tar Pitch	1.1867	94.5781	0.2812	1.3411	0.81
85% - 75% Pet-Coke 25% Cenfuel 15% - Coal Tar Pitch	1.1314	93.1951	0.1923	1.4429	0.78
85% - 75% Pet-Coke 25% Cenfuel 15% - Coal Tar Pitch	1.3080	95.5657	0.0552	1.6788	N/A
85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	1.2901	93.3862	0.0774	1.1884	1.07
85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	1.1721	91.5288	0.1559	1.3414	1.05

Table A.3 Continued

85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	1.2225	94.5843	0.1323	1.1903	N/A
85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	1.3212	95.2392	0.1575	0.8732	1.12
85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	1.2953	95.8547	0.1404	0.8981	1.10
85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	1.2923	93.5509	0.1692	0.8168	N/A
85% - 20% Pet-Coke 80% Cenfuel 15% - Coal Tar Pitch	1.0376	74.4752	0.2968	0.4633	1.40
85% - 20% Pet-Coke 80% Cenfuel 15% - Coal Tar Pitch	1.3955	103.5974	0.3488	0.7412	1.46
85% - 20% Pet-Coke 80% Cenfuel 15% - Coal Tar Pitch	1.4367	103.5974	0.1830	0.7581	N/A
85% - 20% Pet-Coke 80% Cenfuel 15% - Coal Tar Pitch	1.2374	90.6845	0.1948	0.6586	N/A
85% - 0% Pet-Coke 100% Cenfuel 15% - Coal Tar Pitch	1.2940	96.9514	0.1912	0.4358	1.57
85% - 0% Pet-Coke 100% Cenfuel 15% - Coal Tar Pitch	1.2088	86.2800	0.2289	0.3013	1.60
85% - 0% Pet-Coke 100% Cenfuel 15% - Coal Tar Pitch	1.1973	93.3290	0.2016	0.3696	N/A

Table A. 4 Elemental Analyses and Ash Content of SECO/Pet-Coke Rods

	Nitrogen %	Carbon %	Hydrogen %	Sulfur %	Ash
85% - 100% Pet-Coke 0% SECO 15% - Coal Tar Pitch	1.2472	95.9146	0.0093	1.9819	0.67
85% - 100% Pet-coke 0% SECO 15% - Coal Tar Pitch	1.1860	97.1302	0.0058	1.9065	0.67
85% - 100% Pet-Coke 0% SECO 15% - Coal Tar Pitch	1.2286	99.5301	0.0128	2.1355	N/A
85% - 75% Pet-Coke 25% SECO 15% - Coal Tar Pitch	1.0884	79.8815	0.0000	0.7468	0.60
85% - 75% Pet-Coke 25% SECO 15% - Coal Tar Pitch	1.1514	88.6096	0.0000	0.8800	0.64
85% - 75% Pet-Coke 25% SECO 15% - Coal Tar Pitch	1.0576	82.7182	0.0000	0.6760	N/A
85% - 50% Pet-Coke 50% SECO 15% - Coal Tar Pitch	1.2192	86.3309	0.0000	0.4259	0.53
85% - 50% Pet-Coke 50% SECO 15% - Coal Tar Pitch	1.2588	87.1490	0.0000	0.3390	0.54
85% - 50% Pet-Coke 50% SECO 15% - Coal Tar Pitch	1.3057	89.2812	0.0000	0.5823	N/A
85% - 25% Pet-Coke 75% SECO 15% - Coal Tar Pitch	1.3013	90.8888	0.0000	0.2094	0.62
85% - 25% Pet-Coke 75% SECO 15% - Coal Tar Pitch	1.1052	77.8926	0.0000	0.1465	0.62
85% - 25% Pet-Coke 75% SECO 15% - Coal Tar Pitch	1.2431	96.1598	0.0000	0.3732	N/A

Table A.4 continued

85% - 0% Pet-Coke 100% SECO 15% - Coal Tar Pitch	1.5280	93.2002	0.0000	0.0000	0.49
85% - 0% Pet-Coke 100% SECO 15% - Coal Tar Pitch	1.3776	85.3010	0.0066	0.0000	0.59
85% - 0% Pet-Coke 100% SECO 15% - Coal Tar Pitch	1.4760	93.8355	0.03469	0.0687	N/A

Table A. 5 Voltage and Current Readings for Batch 1, 25% Cenfuel/75% Pet-Coke Rod

85% - 75% Pet-Coke 25% Cenfuel 15% - Coal Tar Pitch	L = 14.35 cm Avg. Density = 1.41 g/cc Avg. Diameter = 1.944 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{-m}$)
0.0861	0.7328	243.0
0.0798	0.6792	243.0
0.0726	0.6182	242.9
0.0676	0.5756	242.9
0.0636	0.5408	243.2
0.0598	0.5088	243.1
0.0575	0.4891	243.2
0.0861	0.7328	243.0

Table A. 6 Voltage and Current Readings for Batch 1, 50% Cenfuel/50% Pet-Coke Rod

85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	L = 14.35 cm Avg. Density = 1.23 g/cc Avg. Diameter = 1.915 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.1189	0.7308	326.6
0.1070	0.6579	326.4

Table A.6 continued

0.0968	0.5948	326.6
0.0899	0.5523	326.7
0.0852	0.5238	326.5
0.0804	0.4938	326.8
0.0794	0.4882	326.4
0.1189	0.7308	326.6

Table A. 7 Voltage and Current Readings for Batch 1, 75% Cenfuel/25% Pet-Coke Rod

85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	L = 14.35 cm Avg. Density =1.25 g/cc Avg. Diameter =1.858 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0589	0.7343	151.6
0.0543	0.6771	151.5
0.0497	0.6197	151.5
0.0461	0.5748	151.5
0.0427	0.5315	151.8
0.0393	0.4898	151.6
0.0589	0.7343	151.6

Table A. 8 Voltage and Current Readings for Batch 1, 80% Cenfuel/20% Pet-Coke Rod

85% - 20% Pet-Coke 80% Cenfuel 15% - Coal Tar Pitch	L = 14.94 cm Avg. Density = 1.40 g/cc Avg. Diameter = 1.834 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0685	0.7337	165.1
0.0642	0.6876	165.1
0.0601	0.6441	165.0
0.0547	0.5859	165.1
0.0516	0.5522	165.2
0.0484	0.5184	165.1
0.0457	0.4895	165.1
0.0685	0.7337	165.1

Table A. 9 Re-Tested Voltage and Current Readings for Batch 1, 75% Cenfuel/25% Pet-Coke Rod

85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	L = 14.65 cm Avg. Density = 1.25 g/cc Avg. Diameter = 1.783 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0868	0.7337	201.6
0.0822	0.6942	201.8
0.0763	0.6447	201.7
0.0724	0.6121	201.6
0.0672	0.5674	201.9
0.0638	0.5392	201.7
0.0614	0.5185	201.8
0.0579	0.4895	201.6
0.0690	0.5826	201.8
0.0782	0.6604	201.8
0.0868	0.7336	201.7

Table A. 10 Re-Tested Voltage and Current Readings for Batch 1, 75% Cenfuel/25% Pet-Coke Rod

85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	L = 15.25 cm Avg. Density = 1.25 g/cc Avg. Diameter = 1.792 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.1042	0.7324	235.3
0.0987	0.6932	235.5
0.0928	0.6515	235.5
0.0870	0.6112	235.4
0.0745	0.5638	218.5
0.0711	0.5229	224.9
0.0695	0.4989	230.4
0.0829	0.5817	235.7
0.0981	0.6887	235.6
0.1043	0.7323	235.5

Table A. 11 Re-Tested Voltage and Current Readings for Batch 1, 50% Cenfuel/50% Pet-Coke Rod

85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	L = 13.34 cm Avg. Density = 1.23 g/cc Avg. Diameter = 1.871 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.1078	0.7322	303.4
0.0989	0.6715	303.6
0.0938	0.6360	304.0
0.0872	0.5912	304.0
0.0814	0.5522	303.8
0.0767	0.5200	304.0
0.0725	0.4920	303.7
0.0721	0.4888	304.0
0.0802	0.5438	304.0
0.0896	0.6076	303.9
0.0984	0.6672	304.0
0.1080	0.7320	304.1

Table A. 12 Re-Tested Voltage and Current Readings for Batch 1, 50% Cenfuel/50% Pet-Coke Rod

85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	L = 14.75 cm Avg. Density = 1.23 g/cc Avg. Diameter = 1.871 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.1171	0.7318	298.1
0.1130	0.7060	298.2
0.1043	0.6512	298.4
0.0984	0.6143	298.4
0.0931	0.5815	298.3
0.0888	0.5546	298.3
0.0854	0.5329	298.6
0.0814	0.5080	298.5
0.0783	0.4887	298.5
0.0871	0.5436	298.5
0.0952	0.5939	298.6
0.1069	0.6670	298.6
0.1172	0.7317	298.4

Table A. 13 Voltage and Current Readings for Batch 2, 80% Cenfuel/20% Pet-Coke Rod

85% - 20% Pet-Coke 80% Cenfuel 15% - Coal Tar Pitch	L = 13.45 cm Avg. Density = 1.40 g/cc Avg. Diameter = 1.853 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0515	0.7358	140.3
0.0475	0.6787	140.3
0.0450	0.6426	140.4
0.0422	0.6035	140.2
0.0404	0.5774	140.3
0.0380	0.5430	140.3
0.0355	0.5073	140.3
0.0343	0.4905	140.2
0.0376	0.5369	140.4
0.0403	0.5749	140.5
0.0437	0.6239	140.4
0.0515	0.7358	140.3
0.0514	0.7357	140.1

Table A. 14 Voltage and Current Readings for Batch 2, 100% Cenfuel/0% Pet-Coke Rod

85% - 0% Pet-Coke 100% Cenfuel 15% - Coal Tar Pitch	L = 7.16 cm Avg. Density = 1.36 g/cc Avg. Diameter = 1.799 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0228	0.7379	109.6
0.0214	0.6936	109.5
0.0202	0.6553	109.4
0.0192	0.6216	109.6
0.0178	0.5761	109.6
0.0166	0.5389	109.3
0.0158	0.5109	109.7
0.0152	0.4914	109.8
0.0166	0.5363	109.8
0.0177	0.5731	109.6
0.0195	0.6330	109.3
0.0228	0.7379	109.6
0.0228	0.7379	109.6

Table A. 15 Voltage and Current Readings for Batch 2, 25% Cenfuel/75% Pet-Coke Rod

85% - 75% Pet-Coke 25% Cenfuel 15% - Coal Tar Pitch	L = 10.16 cm Avg. Density = 1.41 g/cc Avg. Diameter = 1.962 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0789	0.7336	320.0
0.0756	0.7032	319.9
0.0711	0.6600	320.6
0.0667	0.6259	317.1
0.0634	0.5952	317.0
0.0598	0.5617	316.8
0.0572	0.5365	317.3
0.0550	0.5156	317.4
0.0550	0.5156	317.4
0.0522	0.4896	317.3
0.0564	0.5289	317.3
0.0599	0.5617	317.3
0.0647	0.6073	317.0
0.0709	0.6644	317.5
0.0783	0.7337	317.6
0.0783	0.7337	317.6

Table A. 16 Voltage and Current Readings for Batch 2, 0% Cenfuel/100% Pet-Coke Rod

85% - 100% Pet-Coke 0% Cenfuel 15% - Coal Tar Pitch	L = 12.51 cm Avg. Density = 1.52 g/cc Avg. Diameter = 1.968 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0481	0.5407	202.7
0.0481	0.5408	202.7
0.0564	0.6340	202.7
0.0622	0.6990	202.8
0.0653	0.7342	202.7

Table A. 17 Voltage and Current Readings for Reproducibility Verification, 75% Cenfuel/25% Pet-Coke Rod

85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	L = 8.956 cm Avg. Density = 1.236 g/cc Avg. Diameter = 1.793 cm	Rod 2
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0536	0.7081	213.5
0.0568	0.7496	213.6
0.0601	0.7933	213.7
0.0644	0.8494	213.7
0.0704	0.9289	213.7
0.0743	0.9796	213.7
0.0815	1.0746	213.7
0.0885	1.1672	213.7
0.0991	1.3069	213.7
0.1033	1.3625	213.7
0.0710	0.9359	213.8
0.0643	0.8478	213.8
0.0568	0.7485	213.8
0.0537	0.7075	213.8

Table A. 18 Voltage and Current Readings for Reproducibility Verification, 75% Cenfuel/25% Pet-Coke Rod

85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	L = 12.692 cm Avg. Density = 1.239 g/cc Avg. Diameter = 1.791 cm	Rod 3
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0912	0.7058	256.6
0.0966	0.7470	256.6
0.1037	0.8020	256.6
0.1094	0.8460	256.6
0.1196	0.9247	256.7
0.1292	0.9990	256.7
0.1385	1.0705	256.8
0.1503	1.1616	256.8
0.1579	1.2202	256.8
0.1250	0.9661	256.8
0.1121	0.8658	256.8
0.0993	0.7677	256.8

Table A. 19 Voltage and Current Readings for Reproducibility Verification, 75% Cenfuel/25% Pet-Coke Rod

85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	L = 12.682 cm Avg. Density = 1.218 g/cc Avg. Diameter = 1.804 cm	Rod 4
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0954	0.7038	273.1
0.0996	0.7348	273.1
0.1090	0.8045	273.1
0.1168	0.8622	273.1
0.1281	0.9451	273.1
0.1430	1.0550	273.1
0.1601	1.1816	273.1
0.1710	1.2615	273.1
0.1207	0.8907	273.1
0.1088	0.8031	273.1

Table A. 20 Voltage and Current Readings for Reproducibility Verification, 75% Cenfuel/25% Pet-Coke Rod

85% - 25% Pet-Coke 75% Cenfuel 15% - Coal Tar Pitch	L = 12.78 cm Avg. Density = 1.218 g/cc Avg. Diameter = 1.804 cm	Rod 4 Repeat
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.1030	0.7491	275.0
0.1102	0.8012	275.0
0.1259	0.9158	275.0
0.1455	1.0582	275.0
0.1703	1.2390	275.0
0.1280	0.9314	275.0

Table A. 21 Voltage and Current Readings for Reproducibility Verification, 50% Cenfuel/50% Pet-Coke Rod

85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	L = 8.964 cm Avg. Density = 1.22 g/cc Avg. Diameter = 1.860 cm	Rod 5
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0698	0.7669	299.5
0.0728	0.7376	299.5
0.0788	0.7964	300.0
0.0866	0.8747	300.1
0.0958	0.9683	300.1
0.1051	1.0619	300.1
0.1140	1.1518	300.1
0.0925	0.9338	300.1
0.0800	0.8085	300.1
0.0700	0.7066	300.1

Table A. 22 Voltage and Current Readings for Reproducibility Verification, 50% Cenfuel/50% Pet-Coke Rod

85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	L = 14.255 cm Avg. Density = 1.218 g/cc Avg. Diameter = 1.860 cm	Rod 6
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.1209	0.7038	327.3
0.1314	0.7649	327.3
0.1494	0.8698	327.4
0.1680	0.9777	327.5
0.1906	1.1094	327.5
0.2124	1.2358	327.5
0.1496	0.8703	327.5
0.1280	0.7449	327.6

Table A. 23 Voltage and Current Readings for Reproducibility Verification, 50% Cenfuel/50% Pet-Coke Rod

85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	L = 12.774 cm Avg. Density = 1.221 g/cc Avg. Diameter = 1.863 cm	Rod 7
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.1141	0.7042	345.6
0.1262	0.7792	345.7
0.1408	0.8692	345.7
0.1575	0.9720	345.8
0.1727	1.0663	345.8
0.1937	1.1953	345.8
0.1561	0.9630	345.8
0.1356	0.8867	345.8
0.1206	0.7444	345.8

Table A. 24 Voltage and Current Readings for Reproducibility Verification, 50% Cenfuel/50% Pet-Coke Rod

85% - 50% Pet-Coke 50% Cenfuel 15% - Coal Tar Pitch	L = 12.779 cm Avg. Density = 1.259 g/cc Avg. Diameter = 1.852 cm	Rod 8
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.1130	0.7454	319.7
0.1282	0.8450	319.7
0.1437	0.9476	319.8
0.1607	1.0588	319.8
0.1758	1.1587	319.9
0.1465	0.9652	319.9
0.1263	0.8319	319.9
0.1137	0.7492	320.0

Table A. 25 Voltage and Current Readings for 100% SECO/0%Pet-Coke Rod

85% - 0% Pet-Coke 100% SECO 15% - Coal Tar Pitch	L = 9.9644cm Avg. Density = 1.31 g/cc Avg. Diameter = 1.878 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0211	0.722	81.24
0.0227	0.778	81.11
0.0248	0.848	81.30
0.0268	0.919	81.07
0.0295	0.009	81.27
0.0315	1.077	81.31
0.0340	1.162	81.34
0.0364	1.243	81.41
0.0267	0.912	81.38
0.0234	0.802	81.11

Table A. 26 Voltage and Current Readings for 75% SECO/25%Pet-Coke Rod

85% - 25% Pet-Coke 75% SECO 15% - Coal Tar Pitch	L = 8.3274 cm Avg. Density = 1.43 g/cc Avg. Diameter = 1.924 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0158	0.702	78.58
0.0173	0.768	78.65
0.0189	0.840	78.55
0.0207	0.920	78.55
0.0227	1.006	78.78
0.0243	1.080	78.55
0.0265	1.175	78.74
0.0290	1.289	78.55
0.0217	0.962	78.75
0.0190	0.842	78.78

Table A. 27 Voltage and Current Readings for 50% SECO/50%Pet-Coke Rod

85% - 50% Pet-Coke 50% SECO 15% - Coal Tar Pitch	L = 9.9492 cm Avg. Density = 1.52 g/cc Avg. Diameter = 1.915 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0351	0.724	140.3
0.0376	0.776	140.3
0.0402	0.830	140.2
0.0431	0.885	141.0
0.0459	0.949	140.0
0.0497	1.027	140.1
0.0533	1.100	140.3
0.0594	1.227	140.1
0.0503	1.038	140.3
0.0453	0.935	140.3
0.0398	0.821	140.3

Table A. 28 Voltage and Current Readings for 25% SECO/75%Pet-Coke Rod

85% - 75% Pet-Coke 25% SECO 15% - Coal Tar Pitch	L = 9.9416 cm Avg. Density = 1.53 g/cc Avg. Diameter = 1.939 cm	
Voltage	Current	Resistivity ($\mu\Omega\text{m}$)
0.0321	0.698	136.6
0.0343	0.748	136.2
0.0379	0.826	136.3
0.0409	0.891	136.3
0.0444	0.967	136.4
0.0480	1.045	136.4
0.0520	1.132	136.4
0.0576	1.254	136.4

Note, density data was taken and recorded for all rods. The raw data for these values has been lost.