QUARTERLY RESEARCH REPORT

(Reporting Period: 01/01/96-03/31/96)

ON

Slag Characterization and Removal Using Pulse Detonation for Coal Gasification

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The research activities performed for the project in the first quarter of 1996 (reporting period: January 1, 1996 to March 31, 1996) are summarized below:

On February 26, 1996, Dr. Ziaul Huque, Dr. Daniel Mei and Muhammad R. Ali from Prairie View A&M University (PVAMU), Dr. Wilson and Mr. Scott from University of Texas (UTA) and Dr. Lou Hunter from Lockheed- Martin Co. visited the Aerospace Research Center at the campus of UTA to check the set-up of the detonation engine chamber and discuss preliminary design concepts for the fixture to be attached at the exit of the detonation tube. A meeting was also attended at the same place and same date by all the persons involved. The meeting discussion concentrated on fixture design and the techniques to be developed to attach the slag samples around a representative boiler tube. The meeting concluded that the experiments will focus on breaking the bonds within the slag itself by using detonation wave according to test plan to be generated to finish the project on schedule.

Based on the decision of the meeting the followings were performed in this quarter:

- PVAMU contacted MTI for slag samples and received three different types of samples, collected from three different locations of an actual boiler at the Northern States Power Company in the form of powder and solid state. The solid form of slags were collected from Reheater Inlet Unit and the powder form was collected from the Economizer Unit.

- According to the suggestions provided by MTI a high viscosity epoxy resin was decided to be used because it will not diffuse into the slag to strengthen the bonding within the slag.

- An extensive market survey was performed and different types of high viscosity resin samples were purchased and tested with slag samples. Finally J-B weld was chosen because it satisfied our requirements.

- To build up slag deposit around the representative tube, 0.635 inches diameter stainless steel rod was decided to be used for first batch sample testing.
• Some stand-still picture of the detonation tube were taken along with video of the total set-up of the detonation engine tube. Based on those pictures and video designs of the fixture were made.

• Four fixture designs were reviewed and the designs were finalized to facilitate the testing in consultation with the Aerospace Research Center Laboratory of UTA personnel (Fixture designs are attached in Appendix C).

• The fixture materials were evaluated based on portability and prices. High strength aluminium was selected over stainless steel because it is cheaper and lighter.

• The fabrication of the fixture were completed as per design.

• Attachment of slag samples on stainless steel rod were also partially completed.

**Description of the fixture:**

The fixture consists of three sets of parts. Two flange base plates, two slag loader plates and the representative steel rods attached with slag samples. The flange base plates will be attached at flange of the detonation tube and the slag loader plates will be fitted at the flange base. The steel rod attached with slag samples will be inserted between the slag loader plates.

**Slag samples preparation and static load test results with the conical rod:**

The surface of the representative steel rods were to be cleaned by coarse sand paper and the slag sample pieces (approximately 1.5 in. x 1 in.) were attached by the J-B weld. The samples were allowed to be dried overnight before putting to use. A static load was applied to the slag samples on the top of a steel cylindrical cone to measure the hardness of the slag and also to characterize the failure modes of different samples under the same load configuration. Test result will be discussed after the sample testing is complete.
Future Work:

- Both powder and solid form of slag will be attached with the stainless steel rod.

- The fixture will be taken to the UTA Aerospace Research Center Laboratory and fitted at the exit of the detonation engine tube.

- After consulting with Dr. Wilson, Dr. Benson and Mr. Scott the test will be performed between end of May and June, 1996 according to the following test plan.

Test Plan:

Fuel Types: Propane, Methane and Hydrogen

Out of these three we want to use the one which gives the strongest and the most stable waves in all three categories (deflagration, weak detonation and C-J).

Slag Type: Three

Slag location: Three axial position

Slag orientation: Two

Pulse strength: Three (detonation, weak detonation and C-J)

Pulse frequency: Two (two Hertz and single Pulse)

Slag Characterization Study:

The slag characterization study was done by MTI and provided to PVAMU. The report is attached.
1.0 Introduction

Microbeam Technologies Incorporated is working with Prairie View to develop and demonstrate a new method to remove deposits from coal-fired utility boilers. MTI is providing background information on fuel properties, ash formation, ash deposition, and ash removal. In addition, MTI is providing deposits collected from a full scale utility boiler. Ash deposits on fireside heat exchange surfaces of power plants significantly decrease plant efficiency and are aggravated by variability in coal quality. Deposit formation is related to coal quality (chemical and physical characteristics of the inorganic material), system operating conditions, and system design (1). Variations in coal quality can significantly influence ash deposition on heat transfer surfaces resulting in decreased plant performance and availability. Deposits in the radiant and convective pass heat transfer surfaces of the boiler require sootblowing and loadshedding for removal, both of which decrease plant efficiency and availability. Ash accumulations on heat transfer surfaces require annual or semi-annual shutdowns for cleaning which result in cleaning costs and lost revenues from being off-line. In addition, maintaining slag flow in wet bottom boilers and cyclone-fired boilers can require co-firing of other fuels and outages to remove frozen slag resulting in decreased efficiency and availability.

During this reporting period MTI performed analysis of deposits collected from full-scale utility boilers. Deposit samples were obtained from Basin Electric and from Northern States Power (NSP). The analyses were conducted using scanning electron microscopy/microprobe techniques as described in the past quarterly report. The chemical and physical properties of the deposits were determined. The results for samples collected from NSP’s Riverside plant are reported here.

2.0 Results

2.1 Boiler Description

The deposit samples used for testing were collected from two units. Deposit samples, MTI93-6, MTI93-7, and MTI93-8, are from a 580 Mw gross Babcock and Wilcox opposed wall fired unit. This unit is Basin Electric Laramie River Station, Unit 1. Deposit samples, MTI96-54, MTI96-55, and MTI96-56 are from a Babcock and Wilcox cyclone fired boiler. The unit is Northern States Power Company’s Riverside Unit #8.

2.2 Coal Analysis

Detailed analysis of the minerals was conducted using computer controlled scanning electron microscopy (CCSEM). The abundance of minerals in the coals are listed in Table 1. Appendix B summarizes the results of the sizes and abundance of mineral grains.
Table 1. Analysis Summary of Basin Electric Laramie River Station Coal and of NSP Rochelle Coal.

<table>
<thead>
<tr>
<th>CCSEM Analysis (wt.% mineral basis)</th>
<th>Laramie River Station Coal</th>
<th>Rochelle Coal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz</td>
<td>46.8</td>
<td>35.7</td>
</tr>
<tr>
<td>Iron Oxide</td>
<td>.6</td>
<td>.2</td>
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<tr>
<td>Rutile</td>
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<td>1.1</td>
</tr>
<tr>
<td>Alumina</td>
<td>.5</td>
<td>.0</td>
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<td>Calcite</td>
<td>4.1</td>
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<tr>
<td>Dolomite</td>
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<td>0.0</td>
</tr>
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<td>Kaolinite</td>
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</tr>
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<td>Montmorillonite</td>
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<td>4.2</td>
</tr>
<tr>
<td>K Al-Silicate</td>
<td>.2</td>
<td>1.5</td>
</tr>
<tr>
<td>Fe Al-Silicate</td>
<td>.1</td>
<td>0.0</td>
</tr>
<tr>
<td>Ca Al-Silicate</td>
<td>.7</td>
<td>.7</td>
</tr>
<tr>
<td>Aluminosilicate</td>
<td>.9</td>
<td>2.1</td>
</tr>
<tr>
<td>Mixed Al-Silica</td>
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<td>3.3</td>
</tr>
<tr>
<td>Ca Silicate</td>
<td>.1</td>
<td>0.0</td>
</tr>
<tr>
<td>Pyrite</td>
<td>20.8</td>
<td>2.7</td>
</tr>
<tr>
<td>Pyrrhotite</td>
<td>.1</td>
<td>0.0</td>
</tr>
<tr>
<td>Oxidized Pyrrhotite</td>
<td>.2</td>
<td>0.0</td>
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<tr>
<td>Gypsum</td>
<td>.0</td>
<td>3.3</td>
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<tr>
<td>Barite</td>
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<td>.2</td>
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<tr>
<td>Apatite</td>
<td>.0</td>
<td>.1</td>
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<tr>
<td>Ca Al-P</td>
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<td>13.0</td>
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<td>Gypsum/Barite</td>
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<td>1.1</td>
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<td>Gypsum/Al-Silica</td>
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<tr>
<td>Si-Rich</td>
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<td>2.4</td>
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<tr>
<td>Ca-Rich</td>
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<td>0.0</td>
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<td>Unknown</td>
<td>1.8</td>
<td>5.2</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Bulk Ash Analysis Oxides (wt.%)</th>
<th>Laramie River Station Coal</th>
<th>Rochelle Coal</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>30.78</td>
<td>33.43</td>
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<tr>
<td>Al₂O₃</td>
<td>15.77</td>
<td>16.86</td>
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<td>Fe₂O₃</td>
<td>6.62</td>
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<tr>
<td>TiO₂</td>
<td>1.33</td>
<td>1.35</td>
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<tr>
<td>P₂O₅</td>
<td>0.78</td>
<td>0.94</td>
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<tr>
<td>CaO</td>
<td>24.55</td>
<td>23.74</td>
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<tr>
<td>MgO</td>
<td>6.11</td>
<td>5.68</td>
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<tr>
<td>Na₂O</td>
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<tr>
<td>K₂O</td>
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<td>0.12</td>
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<tr>
<td>SO₃</td>
<td>13.07</td>
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<tr>
<td>% Ash</td>
<td>6.31</td>
<td>5.26</td>
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2.3 Deposit Analysis

2.3.1 Deposit Descriptions

**Basin Electric Deposits.**

- MTI 93-7 is a convective pass deposit from the rear of the reheater inlet-2. This deposit has a medium to low porosity and is primarily sulfate-based.
- MTI 93-8 is a convective pass deposit from the front of the reheater inlet. This deposit has a medium to low porosity and is primarily sulfate-based.
- MTI 93-6 is an economizer deposit. This deposit is highly porous and is sulfate-based.

**Northern States Power Deposits.**

- MTI 96-55 is a reheater deposit.
- MTI 96-56 is an economizer deposit.
- MTI 96-54 is an air heater inlet deposit.

2.3.2 Morphological Analysis and Porosity Calculations

The deposits were mounted in epoxy resin, allowed to harden, cross-sectioned and polished for analysis in the scanning electron microscope. The results for the NSP samples are reported in this quarterly. Results for the Basin Electric samples will be reported in the next quarterly. The SEM was used to obtain images at two magnifications for each of the samples.

The reheater deposit (MTI96-55) cross-section is illustrated in Figure 1a and 1b. Figure 1a is a low magnification (100 X) backscattered electron image that illustrates the overall microstructure. Figure 1b is a higher magnification image (500 X) that shows bonding between individual ash particles. The larger particles are bonded together by surface coatings. Detailed examination of the coating layers indicates that the matrix material that bonds the larger 10 to 20 micrometer particles together is rich in calcium sulfur likely in the form of calcium sulfate.

The economizer deposit (MTI96-56) is shown in Figures 2a and 2b. Figure 2a is low magnification image showing a very dense region of the deposit and a porous region. The very dense region is likely a deposit fragment from another part of the boiler that was trapped in the porous deposit. The more porous appearing material is much more representative of an economizer deposit. A higher magnification image, Figure 2b, shows that the deposited particles are very small (less than 10 micrometers). These deposits, if not removed, can develop very high strengths because of the high surface area of the deposited particles. Particle sintering (densification) due to heat treatment and reaction with gas phase SO$_2$ and SO$_3$ will be rapid.
Figure 2a. Backscattered Electron Image of polished cross-section of sample MTI 96-56 (Riverside #8 Economizer) at 110X.

Figure 2b. Backscattered Electron Image of polished cross-section of sample MTI 96-56 (Riverside #8 Economizer) at 500X.
The airheater inlet deposit (MTI96-54) is shown in Figures 3a and 3b. Figure 3a is a low magnification image illustrating the overall morphology of the deposit. The deposit is made up primarily of particles greater in size than 5 micrometers. The particles appear to be bonded by surface coatings rich in sulfates. A high magnification image of the deposit cross-section is shown in Figure 3b.

Figure 3a. Backscattered Electron Image of polished cross-section of sample MTI 96-54 (Riverside #8 Airheater Inlet) at 100X.
Figure 3b. Backscattered Electron Image of polished cross-section of sample MTI 96-54 (Riverside #8 Reheater) at 500X.

Measurements were conducted to determine the area of the epoxy and deposit in the cross-section. The numbers are not an actual porosity they represent the percent of the plug surface that is epoxy. The percent epoxy provides an indication of the void space between particles. The percent epoxy was determined from the gray scale histogram of the backscattered electron image. The results obtained for the NSP Riverside deposits are shown in Table 2. The highest area percent epoxy was found for the air heater followed by the economizer. The lowest percent epoxy was found for the reheater deposit. This deposit was subjected to higher temperatures and resulting in more sintering and in lower porosity.

Table 2. Area percent epoxy in the deposit.

<table>
<thead>
<tr>
<th>MTI #</th>
<th>Description</th>
<th>Area % Epoxy</th>
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<tbody>
<tr>
<td>MTI 96-55</td>
<td>Reheater</td>
<td>29.4</td>
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<tr>
<td>MTI 96-56</td>
<td>Economizer</td>
<td>38.6</td>
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<tr>
<td>MTI 96-54</td>
<td>Airheater</td>
<td>44.4</td>
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</table>
2.3.3 SEMPC Analysis - Deposit Phase and Compositional Analysis

The distribution of phases were determined in the deposits by the scanning electron microscopy point count routine. The composition of the deposit as well as the formation of various phases in the deposit significantly affect the ability of the deposit to develop strength and be resistant to soot blowing. For example, in some cases the deposits highly enriched in crystalline phases can be susceptible to crack propagation and weakening of the deposit allowing them to be easily removed by soot blowing. While in other deposits the formation of a highly sulfated deposit may cause significant bonding to occur in the deposit causing it to be difficult to remove. The SEMPC analysis of the deposits are summarized in Tables 3 and 4. Table 3 shows the major phases present in the samples. The most abundant phase is the unclassified phase which is largely made up of glassy fly ash particles or partially sulfated materials. The compositions of the unclassified phases are known however, the compositions do not fit into classification criteria designated for the phases. The next most abundant phase in the reheater and economizer samples is anhydrite (CaSO₄). This material is the primary bonding material in the deposits. The air heater samples contains some partially sulfated materials that is designated unclassified that is contributing to the bonding. The bulk and amorphous phase composition are summarized in Table 4. (The compositional information in Table 4 is the elemental composition of the material expressed as equivalent oxide. This convention is used by the coal research and industrial community when reporting the composition of coal ash and related ash-derived materials.) All of the samples have relatively high levels of SO₃ indicating a sulfate based deposit. The amorphous phase is the composition of the silicate based material that makes up the amorphous material. The sulfates are not considered. The physical properties of deposits at high temperature is highly dependent upon the composition and abundance of the amorphous phase in the deposit.

Table 3. Riverside #8 Deposits - SEMPC Analyses - Phases by Region

<table>
<thead>
<tr>
<th>Phase</th>
<th>Airheater Inlet MTI 96-54</th>
<th>Reheater MTI 96-55</th>
<th>Economizer MTI 96-56</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gehlenite</td>
<td>2.7</td>
<td>.0</td>
<td>.0</td>
</tr>
<tr>
<td>Anorthite</td>
<td>.7</td>
<td>.7</td>
<td>.7</td>
</tr>
<tr>
<td>Albite</td>
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<td>.3</td>
<td>.7</td>
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<td>Pyroxene</td>
<td>.3</td>
<td>.0</td>
<td>.3</td>
</tr>
<tr>
<td>Haynne</td>
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<td>Calcium Aluminate</td>
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<td>1.0</td>
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<td>Anhydrite</td>
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<tr>
<td>Sulfated Dolomite</td>
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<td>.0</td>
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<td>Unclassified</td>
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<tr>
<td>Pure Kaolinite (amorp)</td>
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<td>.0</td>
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<tr>
<td>Kaolinite Derived</td>
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<td>Illite</td>
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<td>.0</td>
<td>.3</td>
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<tr>
<td>Montmorillonite (amorp)</td>
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<td>.7</td>
</tr>
<tr>
<td>Calcium Derived</td>
<td>.0</td>
<td>.0</td>
<td>.3</td>
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3.0 Summary and Conclusions

Samples of deposits were obtained from Northern States Power for testing at Prairie View. The deposits were characterized to determine some of the chemical and physical properties that effect the strength of the deposit as well as the resistance to removal. The deposits range is porosity (area percent epoxy) from 44.4% for the weakest to 29.4% for the strongest. The critical porosity value for removal full scale utility boilers by a soot blower is about 25% porosity (Wain and others (1992)). The deposits are primarily sulfate based with varying levels of sulfation. The microstructure indicated coating on the particles that are likely contributing to the strength development. These chemical and physical properties of the deposits influence the ability of conventional sootblowers to remove the deposits.

4.0 Future Work

- Characterize fragments of deposits removed to determine where the deposit bonds were broken.
- Make relationships between deposit physical and chemical properties to removability.

### Table 4. Riverside #8 Deposits - SEMPC Analyses - Chemical Composition

<table>
<thead>
<tr>
<th></th>
<th>Airheater Inlet MTI 96-54</th>
<th>Reheater MTI 96-55</th>
<th>Economizer MTI 96-56</th>
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<tr>
<td></td>
<td>Bulk</td>
<td>Amorp</td>
<td>Bulk</td>
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<tr>
<td>SiO₂</td>
<td>16.5</td>
<td>24.5</td>
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<td>7.4</td>
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<td>TiO₂</td>
<td>.9</td>
<td>1.4</td>
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<td>P₂O₅</td>
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<td>Na₂O</td>
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<td>ClO</td>
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Reference


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# Appendix A: Chemical Formulas for Minerals in CCSEM Analysis

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<thead>
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<th>Mineral</th>
<th>Formula</th>
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<tr>
<td>Quartz</td>
<td>SiO$_2$</td>
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<tr>
<td>Iron Oxide</td>
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</tr>
<tr>
<td>Rutile</td>
<td>TiO$_2$</td>
</tr>
<tr>
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<td>Al$_2$O$_3$</td>
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<td>CaCO$_3$</td>
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<td>Kaolinite</td>
<td>Al$_2$SiO$_5$(OH)$_2$</td>
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<tr>
<td>Montmorillonite</td>
<td>(1-x)Al$_2$O$_3$ \cdot x(MgO,Na$_2$O)\cdot 4SiO$_2$·H$_2$O</td>
</tr>
<tr>
<td>K Al-Silicate</td>
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APPENDIX B: CCSEM Analysis Data

CCSEM Analysis Results for Basin Electric, Laramie River Station Coal Sample

Particle Size Bins, \( \mu \)

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## CCSEM Analysis Results for NSP, Rochelle Coal Sample

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Totals: 23.2 34.2 13.9 18.2 8.4 2.0 100.0
APPENDIX C:

DESIGN OF FIXTURE
Figure c1: Schematic of Flange Base Plate
Figure c2: Details Drawing of Slag Loader Plate

- .635 ± .005
- .255 ± .005
- 5.000
Figure c3: Representative Steel Rod

- Diameter: 0.635
- Length: 6.500
- Threads: (1/4)x20
Figure c4: Schematic of Slag Loader Plate
Figure c5: Schematic of Static Load Test Set-up

- static load
- conical steel rod
- cone
- slag sample
- Table