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Integrated Waste Treatment Unit (IWTU) Input Coal Analyses and Off-Gas Filter (OGF) Content Analyses

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PREFACE OR ACKNOWLEDGEMENTS

The authors would like to thank CWI (CH2M-WG Idaho) personnel for sampling the Fluidized Bed Steam Reformer (FBSR) feedstock supersacks of coal. The authors would like to thank Ronnie Rutherford of the Savannah River National Laboratory (SRNL) Analytic R&D Programs and Materials Characterization for performing the coal heat treatments and sieve analyses and Whitney Riley and David Best of the Process Science Analytic Laboratory (PSAL) for the analyses of the coal ash. The authors would also like to thank Tommy B. Edwards of the Environmental & Chemical Process Technology (E&CPT) for the statistical analysis of the coal particle size data.

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EXECUTIVE SUMMARY

A full engineering scale Fluidized Bed Steam Reformer (FBSR) system is being used at the Idaho Nuclear Technology and Engineering Center (INTEC) to stabilize acidic Low Activity Waste (LAW) known as Sodium Bearing Waste (SBW). The INTEC facility, known as the Integrated Waste Treatment Unit (IWTU), underwent an Operational Readiness Review (ORR) and a Technology Readiness Assessment (TRA) in March 2014. The IWTU began non-radioactive simulant processing in late 2014 and by January, 2015 ; the IWTU had processed 62,000 gallons of simulant. The facility is currently in a planned outage for inspection of the equipment and will resume processing simulated waste feed before commencing to process 900,000 gallons of radioactive SBW. The SBW acidic waste will be made into a granular FBSR product (carbonate based) for disposal in the Waste Isolation Pilot Plant (WIPP).

In the FBSR process calcined coal is used to create a CO_2 fugacity to force the waste species to convert to carbonate species. The quality of the coal, which is a feed input, is important because the reactivity, moisture, and volatiles (C,H,N,O, and S) in the coal impact the reactions and control of the mineralizing process in the primary steam reforming vessel, the Denitration and Mineralizing Reformer (DMR). Too much moisture in the coal can require that additional coal be used. However since moisture in the coal is only a small fraction of the moisture from the fluidizing steam this can be self-correcting. If the coal reactivity or heating value is too low then the coal feedrate needs to be adjusted to achieve the desired heat generation. Too little coal and autothermal heat generation in the DMR cannot be sustained and/or the carbon dioxide fugacity will be too low to create the desired carbonate mineral species. Too much coal and excess S and hydroxide species can form. Excess sulfur from coal that (1) is too rich in sulfur or (2) from overfeeding coal can promote wall scale and contribute to corrosion in process piping and materials, in excessive off-gas absorbent loading, and in undesired process emissions.

The ash content of the coal is important as the ash adds to the DMR and other vessel products which affect the final waste product mass and composition. The amount and composition of the ash also affects the reaction kinetics. Thus ash content and composition contributes to the mass balance. In addition, sodium, potassium, calcium, sulfur, and maybe silica and alumina in the ash may contribute to wall-scale formation. Sodium, potassium, and alumina in the ash will be overwhelmed by the sodium, potassium, and alumina from the feed but the impact from the other ash components needs to be quantified. A maximum coal particle size is specified so the feed system does not plug and a minimum particle size is specified to prevent excess elutriation from the DMR to the Process Gas Filter (PGF).

A vendor specification was used to procure the calcined coal for IWTU processing. While the vendor supplied a composite analysis for the 22 tons of coal (Appendix A), this study compares independent analyses of the coal performed at the Savannah River National Laboratory (SRNL) and at the National Energy Technology Laboratory (NETL). Three supersacks[a] were sampled at three different heights within the sack in order to determine within bag variability and between bag variability of the coal. These analyses were also compared to the vendor's composite analyses and to the coal specification. These analyses were also compared to historic data on Bestac coal analyses that had been performed at Hazen Research Inc. (HRI) between 2004-2011.

[a] A "supersack" is a name used in the specialty coal/materials trade which refers to essentially a large bag containing coal, coke, or charcoal. The bags in this instance contain 1,100 pounds of a calcined coal received from a vendor in China that is used as a feedstock for the DMR.

The following was noted regarding the chemical composition of coal as defined in terms of its Proximate[a] and Ultimate[b] (elemental) analyses:

- The results of the ASTM D4749 sieve analyses when assessed against the IWTU coal specification demonstrates that the mean particle size is ~8mm versus the preferred size of 10mm. However, most of the coal is within the range of sizes indicated by the coal specification. There is very little within bag size variability on a per supersack basis. There is little size variation between supersacks but 9.78-11.07% of the coal is <6mm versus the 2.42% claimed by the IWTU coal vendor.
- The Proximate ASTM D7582/D3172analyses performed at SRNL were performed at 750°C per the ASTM procedure using furnaces. Duplicate Proximate analyses were performed at NETL at 950°C (volatile) and 750°C (ash) by ASTM D7582 using a Thermogravimetric Analysis (TGA). All moisture measurements were performed at 107°C.
- All of the moisture measurements performed at SRNL and NETL were within the coal specification and close to the preferred value in the specification. The within supersack variability was also small. SRNL moisture analyses were biased lower than the NETL and vendor analyses. The NETL moisture analyses were biased ~0.5% lower than the coal vendor analyses.
- The SRNL Proximate analyses of the coal samples indicated that the volatile losses at 750°C in argon were biased low compared to the coal specification values: the average measured value was lower than the preferred value. Two supersacks had low within bag variability, while the third had high within bag variability. However, the NETL Proximate values indicated that one supersack was below the minimum value of volatiles in the coal specification but the other two supersacks were were within the coal specification values. The average volatiles were 10.88% as measured by NETL which is just above the minimum volatile value of 10% in the coal specification. The preferred volatile value is 15% and both the SRNL and NETL data confirmed that the volatiles were below the preferred value in the coal specification. Both the SRNL and NETL volatile measurements were significantly lower than that of the coal vendors analyses.
- The SRNL Proximate analyses of the coal samples indicated that the ash contents at 750°C in air were within the coal specification values and the average measured value was ~1 wt% higher than the preferred value. The within bag variability was low. The NETL Proximate analyses were biased high in ash content by ~1 wt% compared to the preferred. The SRNL analyses agreed with the coal vendors analyses.
- The fixed carbon by difference is within the values calculated from the specification by difference since a fixed carbon range is not actually specified.
- The SRNL and NETL ash content CaO concentration in wt% is within the specification range but the average concentration is slightly larger than the preferred value instead of lower than the preferred value as indicated in the specification. The vendor analysis of CaO was lower than the SRNL or NETL analyses but since both SRNL and NETL's values were within the coal specification so was the vendor analyses.

[a] The Proximate analysis of coal was developed as a simple means of determining the distribution of products obtained when the coal sample is heated under specified conditions. As defined by ASTM D 121 (Terminology of Coal and Coke), proximate analysis separates the products into four groups: (1) moisture, (2) volatile matter, consisting of gases and vapors driven off during pyrolysis, (3) fixed carbon, the nonvolatile fraction of coal, and (4) ash, the inorganic residue remaining after combustion

[b] The Ultimate analysis of coal was developed to determine the amount of potential energy in coal that can be converted into actual heating ability. Elemental or ultimate analysis encompasses the quantitative determination of carbon, hydrogen, nitrogen, sulfur and oxygen within the coal. Additionally, specific physical and mechanical properties of coal and particular carbonization properties are determined.

- The SRNL and NETL combined $Na_2O + K_2O$ content of the IWTU Bestac coal is also within the specification and way below the preferred value which is excellent. The SRNL and NETL analyses were in agreement that the $Na_2O + K_2O$ content was lower than the specification but the vendor analyses for these oxides were biased high but still within the specification.
- The SRNL SiO_2 content of the IWTU Bestac coal is biased about 5 wt% higher than the maximum preferred value in the coal specification while the NETL analyses are biased low compared to the specification. In addition, the within supersack and between supersack variability is high. The SRNL and coal vendor analyses are in agreement that the SiO_2 content is biased higher than the preferred value of SiO_2 but are within the specification limits.
- NETL performed Ultimate ASTM D3176 analyses and the sulfur content was within the coal specification.
- The NETL average IWTU Bestac coal heating value measured was 13,403 Btu/lb while the coal specification preferred value was >12,500 Btu/lb. The coal vendor analyses indicated an average heating value of 12,060 Btu/lb which was within the lower bounds of the coal specification but not at or above the preferred value.

The SRNL Proximate analyses in this study was compared to the averages of the moisture content, volatiles, fixed carbon and ash to the historic average of Bestac coal analyses used in previous pilot scale testing performed. The IWTU coal is ~1 wt% lower in moisture, ~7 wt% lower in volatiles, and ~8.7 wt% higher in fixed carbon. When the ash analysis for the IWTU coal was compared to the average values for Bestac coal used in pilot scale demonstrations of the FBSR technology from 2004 through 2011 the IWTU Bestac coal is higher in SiO_2 content by ~ 5 wt%, lower in Al_2O_3 by ~5 wt%, higher in CaO content by about 0.5 wt%, lower in Fe_2O_3 by ~ 0.5 wt%, and lower in Na_2O+K_2O by ~ 2 wt%.

Two X-ray Diffraction (XRD) analyses were performed at SRNL on the IWTU Bestac raw coal samples, a chunk of coal was ground up and analyzed and the <400 mesh fines from the sieve analyses were also analyzed for comparison. An XRD analysis of the IWTU Bestac coal ash was also performed. The ground chunk of coal showed the presence of excess quartz (SiO_2) while the fines showed the presence of excess quartz and excess kaolinite ($Al_2Si_2O_5(OH)_4$). Both of these phases are the sources of excess SiO_2 in the IWTU Bestac coal.

In general the IWTU Bestac coal met most of the vendor coal specifications except for % volatiles in the coal and % SiO_2 in the coal ash. The coal ash is simultaneously high in CaO compared to the "preferred value" in the coal specification but the CaO ash content is within the coal specification. Any of these three findings could cause operational difficulties in the form of coal reactivity and unwanted process chemistry. The coal has a considerable (>10 wt%) fines fraction (<6mm) although this was not a requirement in the specification.

In addition, Proximate and Ultimate analyses and XRD were also performed on the coke used in the Carbon Reduction Reformer (CRR) and the wood based charcoal used to startup the DMR SRNL performed the Proximate analyses of the wood based charcoal at 750°C and the coke at 950°C. NETL performed the Ultimate analyses at 950°C for both. The ash for the wood based charcoal was analyzed and the coke produced insufficient ash to be analyzed. This completed the analyses of all the different types of coal, charcoal, coke that is used in the IWTU.

In October 2014, coal and water were being fed to the IWTU during startup in advance of feeding SBW simulant. During an outage on October 8, 2014 solids were found in the Off Gas Filter (OGF) which were sampled and sent to SRNL for analyses. The particle size distributions for the OGF samples represent an aggregate over months of IWTU startup with coal, water and steam, but without SBW simulant. The samples were pulled on different shifts from different positions in the OGF and do not represent steady state operations. The particle size distribution has maximum particle sizes at ~ 10 microns ~ 70-110 microns. One sample had a large maxima in the at ~120 microns that is likely bauxite carryover from the CRR since the CRR bed elutriates during operation and has to be replenished periodically. As part of this analysis bauxite from the CRR bed was also analyzed.

The Proximate analysis of the OGF solids indicates that the processed material is higher in moisture content than the CRR coke which is carried over to the OGF. The CRR operates at ~950°C which should eliminate any moisture from the coke carried over unless the coal is adsorbing moisture from the steam used in the process. For example, just downstream of the CRR water is injected (flash evaporates) in the Off Gas Cooler (OGC) to reduce the off-gas temperature from approximately ~950°C to ~165°C. This introduces a substantial amount of steam to the off-gas system prior to particle filtration in the OGF. Excess moisture in the OGF solids may be making the coke carryover adhere to itself since it is such a fine particulate.

The OGF solids are a combination of unreacted coal/coke, coal/coke ash from the CRR, and carryover of the bauxite bed from the CRR. The OGF solids are ~ 80 wt% unreacted coke from CRR elutriation (this percentage includes moisture, volatiles and fixed carbon), 7.2 wt% coke ash, and 12.8 wt% bauxite from the CRR. Since the OGF solids are so high in unburned carbon, an effort should be made to try to reduce the amount of coke being carried over from the CRR to the OGF.

The following recommendations are made:

- Coal analyses should be performed on incoming batches of IWTU coal to determine compliance with the coal vendor specification as excess SiO_2 in the coal ash could cause operational issues and excess volume in the waste canisters.
- Process gas velocities should be minimized to prevent solids carryover to the PGF and OGF from the DMR and the CRR, respectively.
- Coal feed rate to the DMR should be monitored and minimized to that needed to denitrate the SBW waste when simulated and/or radioactive waste is processed to minimize disposal volumes
- Overfeeding of coal to the DMR and/or CRR should be monitored and minimized to that needed to denitrate the SBW waste when simulated and/or radioactive waste is processed.
- Investigation as to the source of moisture in the OGF solids should be determined. This would include an evaluation of how hygroscopic the OGF solids are, and the potential for water absorption prior to sampling, versus the potential for water absorption from the water injection cooling process in the OGC.

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
CAA	Clean Air Act
CP	Carbonate Product
CRR	Carbon Reduction Reformer
CWI	CH2M-WG Idaho
DOE	Department of Energy
DMR	Denitration & Mineralization Reformer
E&CPT	Environmental & Chemical Process Technology
EM	Environmental Management
FBSR	Fluidized Bed Steam Reformer
HRI	Hazen Research Inc.
HWC	Hazardous Waste Combustor
ICDD	International Centre for Diffraction Data
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
INL	Idaho National Laboratory
INTEC	Idaho Nuclear Technology & Engineering Center
IWTU	Integrated Waste Treatment Unit
LAW	Low Activity Waste
MACT	Maximum Achievable Control Technology
NAS	Na-Al-Si (sodium alumino-silicate)
NETL	National Energy Technology Laboratory
NQA	Nuclear Quality Assurance
OGC	Off-Gas Cooler
OGF	Off-Gas Filter
ORR	Operational Readiness Review
OST	Operations Support Team
PSAL	Process Science Analytic Laboratory
PGF	Process Gas Filter
QAP	Quality Assurance Program
SAIC-STAR	Science Applications International Corporation- Science and Technology Applications Research
SBW	Sodium Bearing Waste
SRNL	Savannah River National Laboratory
TGA	Thermo-gravimetric Analyses
TRA	Technology Readiness Assessment
TTT	THOR® Treatment Technologies
WIPP	Waste Isolation Pilot Plant
WSRC	Westinghouse Savannah River Company
XRD	X-Ray Diffraction

1.0 Introduction

A full engineering scale Fluidized Bed Steam Reformer (FBSR) system is being used at the Idaho Nuclear Technology and Engineering Center (INTEC) to stabilize acidic Low Activity Waste (LAW) known as Sodium Bearing Waste (SBW). The INTEC facility, known as the Integrated Waste Treatment Unit (IWTU), underwent an Operational Readiness Review (ORR) and a Technology Readiness Assessment (TRA) in March 2014. The IWTU began non-radioactive simulant processing in late 2014 and by the end of January, 2015 the IWTU had processed over 60,000 gallons of simulant. The facility is currently in a planned outage for inspection of equipment and will resume processing simulated feed before commencing to process 900,000 gallons of radioactive SBW. The SBW acidic waste will be made into a granular FBSR product (carbonate based) for disposal in the Waste Isolation Pilot Plant (WIPP) [1,2].

1.1 Raw Coal Specification and the Need for Sampling and Analysis

In the FBSR process calcined coal is used to create a CO_2 fugacity to force the waste species to convert to carbonate species. The carbonate and aluminosilicate FBSR flow sheets were demonstrated with simulated SBW at the Science Applications International Corporation- Science and Technology Applications Research (SAIC-STAR) facility in Idaho Falls, Idaho. The pilot-scale tests were performed on the INL SBW [1,2,3] by a team of SAIC-STAR, Idaho National Laboratory (INL), and THOR® Treatment Technologies (TTT) team in 2003-2004. In 2006, the carbonate and aluminosilicate FBSR flow sheets were demonstrated at the engineering scale by TTT at the Hazen Research Inc. (HRI) in Golden, Colorado [4]. The differences in the pilot scale and engineering scale facilities and operation are summarized in Table 1.

Table 1. Comparison of Pilot and Engineering-scale FBSR for Producing Carbonate Products

Facility	Scale	Radioactive or Non-Radioactive?	FBSR Column Diameter	Externally or Autothermaly Heated?	Single Reformer Flowsheet	Reductant of Choice	Catalyst?	Waste
SAIC-STAR 2003-2004	Pilot	Non-Radioactive	6”	external and autothermal with coal	Single	BB* charcoal	No	INTEC SBW
TTT ESTD 2006	Engineering		15”	autothermal with coal	Dual	BB* charcoal and General Carbon (GC-CRB) coal	Yes	INTEC SBW

* Berger Brothers

Wall scale formed on the internal surfaces of the Denitration and Mineralization Reformer (DMR) during the INTEC Carbonate Product (CP) campaigns while INTEC SBW was being processed into the solid carbonate product. Scale formation was observed at the end of the CP-1 scoping tests (January 14, 2006), after a shutdown of the CP-1 production tests on January 21, 2006, and after the final shutdown of the CP-2 production tests (June 2006) [5]. The CP-1 scale was 1-6 mm (1/16-1/4”) thick on the DMR wall but heavier on the downcomer and corrosion coupons. After the CP-2 production test was concluded wall scale deposits of 1-6 mm were also discovered in the DMR on the inside metal and refractory surfaces.

TTT reported that the internal surfaces of the DMR, from the normal bed high level to just below the fluidizing gas distributors, were coated with a thin layer of hard deposits. Deposit thickness was reported to range from ~1/4" on the bottom edge of the corrosion coupons to ~ 1/16" on the downcomer pipe. Deposit thickness on the refractory walls appeared to be 1/16" to 1/8" in the lower portions of the bed with thinning deposits up the walls with deposits extending up to the enlarged diameter section in the freeboard. TTT also reported that the deposits could be manually broken off the refractory and metal surfaces to which they adhered and the deposits were determined to be water soluble. The deposits were reported to be gray in color and have the appearance of ripples as if they formed from a flowing film. The deposits had the appearance of tree bark. The CP-2 scale was similar in appearance to the CP-1 scale [5]. It should be noted that wall scale was not formed during the subsequent SBW aluminosilicate mineralizing runs.

Three causes of the CP-1/CP-2 deposits were identified [6] which included:

- The use of Mulcoa 70® as a bed additive which reacted with the potassium in the SBW
- The use of PureOX which is mostly Fe_2O_3 as a bed additive
- The use of a high sulfur containing coal (Berger Bros. P6) which caused $SO_4^=$ salts to form at 640°C and complex with other alkali salts in the SBW to form low melting eutectic phases that can cause agglomerations and/or initiate attack on any silicates or refractory present

Neither Mulcoa 70® nor PureOX are being used in IWTU and the DMR is not refractory lined as the DMR at HRI was. In addition, a low sulfur containing coal, Bestac coal, is being used at the IWTU and the coal procurement specification requires a range of S between 0-0.7 wt%. These precautions should prevent any scale formation in the IWTU. However, as a preventative measure the calcined Bestac coal, which is a low sulfur containing coal, should be rigorously and routinely tested for variability in sulfate content.

The high sulfur containing coal used in the CP-1/CP-2 campaigns at HRI was General Carbon (GC-CRB) coal (Table 1) which contained 3.71 wt% S.[6] while the Bestac coal used by TTT in 2007 and 2009 contained only 0.16-0.32 wt% S. The procurement specification for the IWTU Bestac coal is shown in Table 2. The vendor coal analyses are given in Appendix A.

Table 2. IWTU Procurement Specification for Calcined Bestac Coal

Characteristic	How Measured	As-Received Specifications		
		Min	Preferred	Max
Reactivity	TGA temperature at oxidation reaction initiation (in air) [°C] – per Mfg's instructions	300	350	400
Reactivity	TGA temperature for oxidation extent of reaction = 95% (in air) [°C] – per Mfg's instructions	None	650	700
Volatiles	Proximate Analysis [wt%] – ASTM D3172	10%	15%	20%
Ash Content	Proximate Analysis [wt%] – ASTM D3172	0%	5%	10%
Total Sulfur	Ultimate Analysis [wt%] – ASTM D3176	0%	0.35%	0.7%
HHV	High Heating Value [BTU/lb] – ASTM D5875	11,500	> 12,500	None
Particle Size	Sieve Analysis – ASTM D4749	6 mm	10 mm	12 mm
Ash Analysis	Ash Analysis: – ASTM D2795			
	Ash CaO [wt%]	0%	< 2%	5%
	Ash K_2O and Na_2O (combined) [wt%]	0%	< 2%	3%
	Ash SiO_2 [wt%]	0%	< 60%	65%
Moisture[1]	Proximate Analysis [wt%] – ASTM D3172	0%	< 5%	9%

[1] The moisture content has a strong inversely proportional impact on the net heating value. Although a moisture content of 9% is acceptable, if lower moisture coal can be obtained, the net heating value will proportionately increase, resulting in a reduction in the total coal usage.

In addition, the TTT coal specification [6] states "since there can be significant variability in coal composition, even in the same shipment, it is recommended that frequent sampling and analyses be used to confirm that the coal used in the DMR meets these specifications. TTT recommends that the supplier provide separate analyses for every 10,000 lb (10 bulk bags) by using a composite sample of shipped coal. Grab samples from the delivered coal should be obtained and analyzed by CH2M-WG Idaho (CWI) to (at least one per every 5 bulk bags) confirm the supplier's analysis. The procurement and delivery of the coal should be performed such that there is sufficient time to reorder coal in the event lab analyses of the delivered coal shows non-compliance with the coal specification."

In this document, the volatile content, ash content, sulfur content, particle size analysis, ash analysis, moisture analysis, and phase identification by X-ray diffraction (XRD) for 9 samples of IWTU Bestac coal from the first IWTU procurement of coal are documented. The 9 samples came from 3 different supersacks of coal on different pallets and from the top-middle-bottom of each of the three bags. This was done to examine the bag-to-bag variability in addition to the between-bag variability of the IWTU Bestac coal. The coke used in the CRR and the wood based charcoal used to start up the DMR were also analyzed but particle size was not determined.

1.2 Off-Gas Filter (OGF) Content Analyses

The IWTU process flowsheet is shown in Figure 1-1. The THOR® FBSR mineralizing technology uses reformers to pyrolyze organics, if any are present, in the presence of a fluidization media of steam. Steam reforming, as a chemical conversion process, has been used for >100 years on gaseous fuels. Mineralizing FBSR's can be externally heated or internally heated or a combination of the two heating methods. Externally heated FBSR's are normally limited to a diameter in the 6-8" range while coal or another reductant such as sugar can be used to assist in the denitration reactions. Coal is used to auto-thermally heat larger reformers (>8" diameter). FBSR flowsheets can be single reformer or dual reformer. Organics not pyrolyzed in the DMR and excess H_2 are oxidized in the second reformer known as the Carbon Reduction Reformer (CRR). The CRR is a fluidized bed that uses petroleum coke as its fuel source and operates at a higher temperature (950°C) and is more oxidizing than the DMR. The DMR is also a fluidized bed but it uses calcined coal as its fuel source and operates at ~650°C for making carbonate products at ~725-750°C to make primarily sodium carbonate minerals with admixed NAS minerals. A CRR or another thermal oxidizer (e.g. natural gas burner system) can be used in the second stage of off-gas treatment, but the IWTU has a CRR.

Off-gases from the CRR are cooled by direct water injection in an off-gas cooler vessel (OGC) and are then filtered in the off-gas filter (OGF). The off-gas then goes through a set of HEPA filters so that the primary emissions released to the atmosphere from the process are carbon dioxide and water vapor (there are no liquid effluents from the process). The samples analyzed in this report are from material that elutriated from the CRR and was present in the holdup material in OGF vessel. The samples were taken after the OGF cooled and represent an accumulation of material during a variety operational parameters or set points.

The off-gas from the DMR contains fine particulates that pass through an internal cyclone system which returns relatively larger and/or heavier particles back to the DMR. Particles that are small and/or light enough pass through the internal cyclones and travel via a pipe duct system where they are removed via Inconel® sintered metal candle filters in the Process Gas Filter (PGF) vessel. An eductor system removes the flyash material from the bottom of the PGF vessel

The FBSR process is not combustion and is Clean Air Act (CAA) compliant. The FBSR technology has also been shown, during pilot-scale and engineering scale testing to be Hazardous Waste Combustor (HWC) Maximum Achievable Control Technology (MACT) compliant for Hg, Cl, CO, total hydrocarbons, and heavy metals [7,8]. A significant benefit of the FBSR process is that it produces zero-liquid releases. All water is released as water vapor.

In October 2014, coal and water were being fed to the IWTU during startup in advance of feeding SBW simulant. During an outage on October 8, 2014 solids were found in the OGF which were sampled and sent to SRNL for analyses. Similar analyses to the analyses performed on the raw coal were performed in order to compare the coke fines in the OGF to the raw input coal. The solids in the OGF were sampled during two different shifts from two different parts of the OGF. The samples were identified by the shift that did the sampling and the shift identifications do not compare to any specific operating conditions.

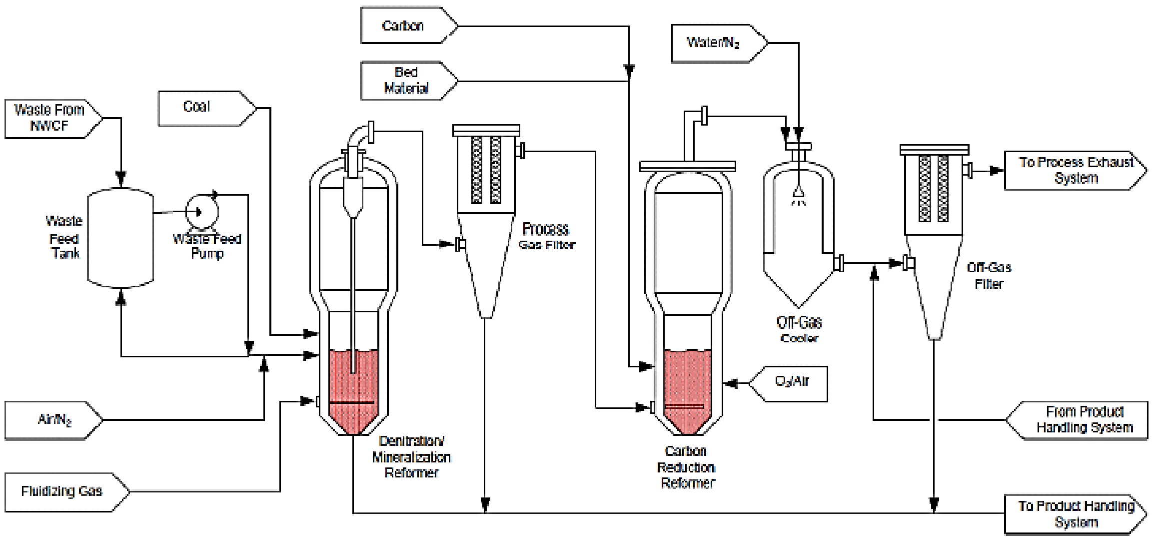


Figure 1-1. IWTU FBSR Process Flowsheet.

2.0 Experimental Procedure

2.1 Coal Proximate and Ultimate Analyses Including Ash Compositions

The coal Proximate analysis (ASTM D3172 which in turn references D3173, D3174, D7582) was performed to get the coal moisture content at 107±3°C, the % volatile matter, the % ash, and the % fixed carbon at 750°C. The composition of the coal ash resulting from the Proximate analysis in oxides was determined by ASTM C1463 since ASTM D2795 and its companion procedure for sulfur analysis (ASTM C1757) have been withdrawn from ASTM. ASTM C1463, which is for glass dissolution is similar to ASTM D2795 for ash dissolution, but the ASTM C1463 dissolution allows for the sulfur to be determined from the dissolution residue by Inductively Coupled Atomic Emission Spectroscopy (ICP-AES) instead of by precipitation (the withdrawn ASTM C1757). The ash was analyzed for Al, Ca, Fe, K, Mg, Mn, Na, Ni, P, S, Si, and Ti by ICP-AES at the SRNL PSAL. The bauxite bed was analysed in the same manner. The elemental concentrations measured were converted and reported on an oxide basis as the ash is composed of oxides. The oxide sum was calculated to demonstrate that ~100% of the components in the ash were accounted for. Additional Proximate analyses were performed by the National Energy Technology Laboratory (NETL) using ASTM D7582 for comparison. SRNL did not perform the Ultimate analyses but did report the SO_3 content of the coal ash from the Proxmiate analyses which underestimates the total sulfur in the coal and so it that data is not compared to the coal specification. NETL also performed the Ultimate coal analyses using ASTM D2013. Sulfur (ASTM

D4239) and chlorine (ASTM D6721) analysis was performed to determine the content in the coal. NETL also reported SO_3 analyses on the coal ash from the Ultimate analyses.

Proximate and Ultimate analyses were also performed on the coke used in the CRR and the wood based charcoal used to startup the DMR. The wood based charcoal produced ash that was analyzed in the same manner as the IWTU Bestac coal. The CRR coke did not produce any quantifiable amount of ash.

2.2 X-Ray Diffraction Analyses

Representative as-received IWTU Bestac coal samples were ground for 5 minutes in an agate mortar and pestle using ethanol as a lubricant to facilitate grinding. The IWTU Bestac coal was also analyzed by XRD after ashing in air at 750°C. In addition, the coke from the CRR and the wood based charcoal was analyzed by XRD and the ash from the wood based charcoal was analyzed. The coke used in the CRR produced insufficient ash to perform XRD. The bauxite was also analyzed by XRD.

Ground coal or ash powder was smeared and fixed to a square glass slide using a 1:10 collodion/amyl acetate mixture. X-ray diffraction data were collected on a Bruker theta-2theta D8 Advance X-ray Diffractometer. The instrument was step scanned over a 5-70° 2Θ range with a 0.02° step size and a dwell time of 1s for a total measurement time of ~60 min. A detailed compilation of all the instrument parameters is included in Table 3. Compound search-match identification was performed with Jade™ 2010 software from Materials Data Inc. using the inorganic powder diffraction file PDF4™ powder diffraction database from the International Centre for Diffraction Data (ICDD). A typical layout for the x-ray diffractometer is shown in Figure 2-1 , where DS is the divergence slit, AS is an antiscatter slit (either diffracted side or detector antiscatter), SS is a soller slit (either divergence [primary] and/or diffracted [secondary] sides), and RS is the receiving slit.

Table 3. Instrument Parameters

Radiation Source	CuKα
X-ray Source Power	45kV,40mA
Wavelength	1.5405982 Å
Goniometer	Bruker D8
Divergence Soller Slit	None
Divergence Slit	1º
Divergence Antiscatter	1º
Specimen Rotation	No
Diffracted Beam Antiscatter	1º
Diffracted Beam Soller	1º
Secondary Monochromator	Curved pyrolytic graphite
Receiving Slit	0.15º
Detector	NaI Scintillation
2θ Range	5º - 70º
Step Size	0.02º (2θ)
Time per Step	1 s

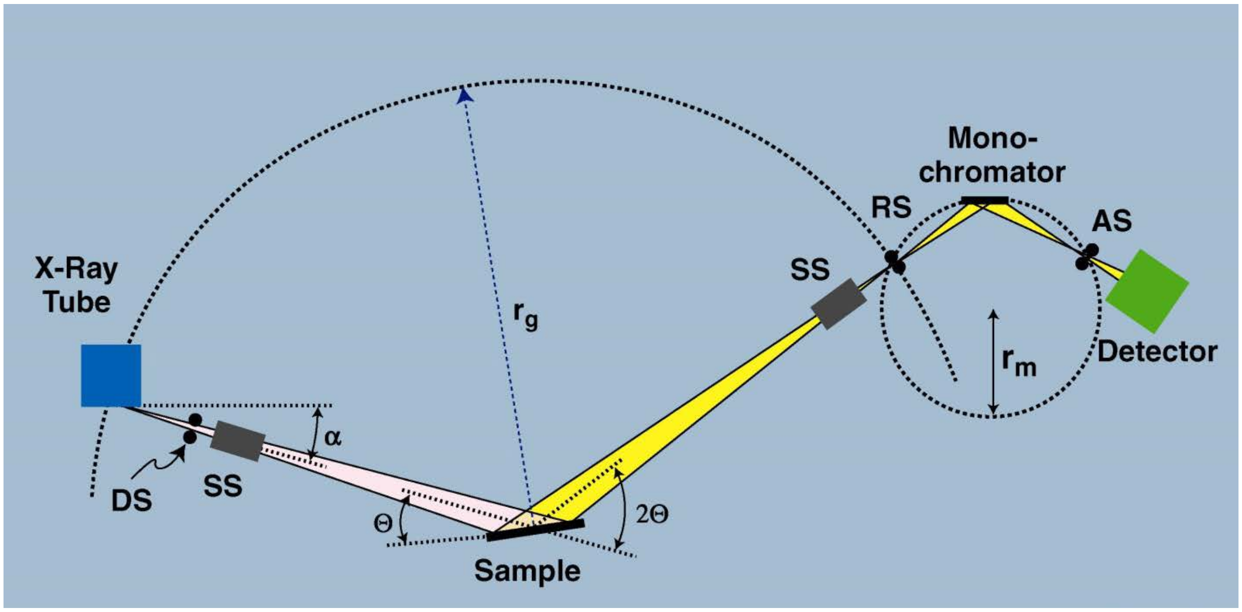


Figure 2-1. Typical X-ray Diffractometer Configurations

2.3 Particle Size Analysis

Particle size analysis was measured by ASTM D4749. The Microtrac S3500 particle size analyzer uses a wet sample delivery controller (recirculator) to disperse the sample uniformly in a fluid and deliver the sample to the analyzer. This wet sample delivery controller in its basic form consists of a reservoir where the sample is introduced, a fluid pump, a valve to the drain system, and the necessary tubing connections to the analyzer. The flow through the analyzer sample cell is always from the bottom to the top. The analyzer consists of the sample cell and three lasers (improves resolution) and two silicon photodiode array detectors. Figure 2-2 depicts the top-down view showing the positions of the lasers and detectors.

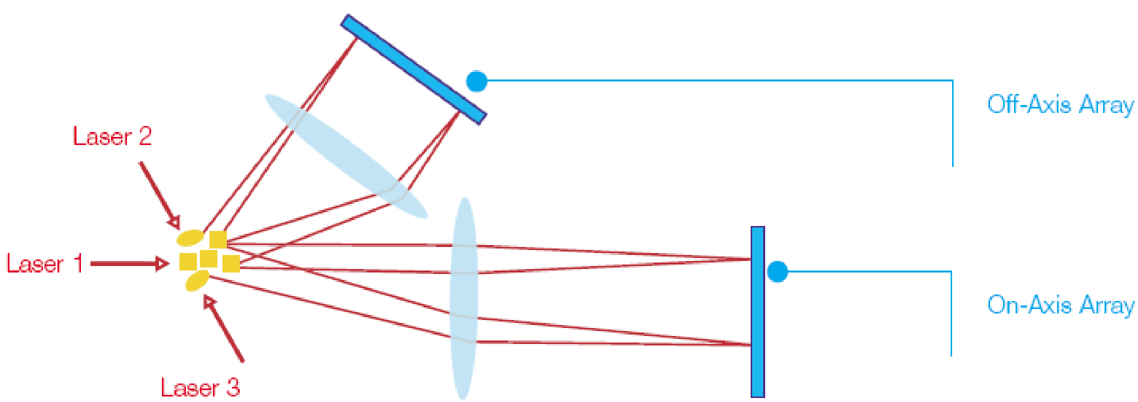


Figure 2-2. Top-down view showing the optical configuration of the Microtrac S3500

A laser beam is projected through the sample cell that contains a stream of moving particles suspended in a liquid. Light rays that strike a particle are scattered (Mie scattering, where the particle radius ≈ laser wavelength.). The scattered light forms an angular pattern which is measured by the two photodiode arrays. Electrical signals proportional to the measured light intensities are then processed by the computer using modified Mie calculations for non-spherical particles to form a multichannel histogram of the particle size distribution.

The required mass to obtain an average sample loading index on the Microtrac S3500 varies with particle size, i.e., the finer the particles size distribution, the smaller the mass needed. Approximately, 0.5 g of the representative as-received coal samples was used for the analysis. A complete list of all the instrument operating parameters can be found in Table 4.

Table 4. S3500 Instrument Parameters for Particle Size Measurements

Transparency	Absorbing
Particle Shape	Irregular
Particle Refractive Index	NA
Number of Channels	100
Progression	Geom 8 Root
Residuals	Disabled
Filter	Enabled
Fluid	Water

2.4 Sample Selection and Analyses

The samples of calcined coal used in the DMR were analyzed from three different supersacks of IWTU Bestac coal that had been sampled on September 5th, 2014 from different pallets of supersacks. There are only two supersacks per pallet. The samples were labelled as shown in Table 5.

Table 5. Alphabetic Sample Codes for Raw Coal Analyses

Bag ID	Top/Middle Bottom	Alphabetic Code
1	Top	F
	Middle	H
	Bottom	A
2	Top	D
	Middle	E
	Bottom	I
3	Top	G
	Middle	B
	Bottom	C

The same analyses that were performed on the raw coal were performed on the coal deposits from the OGF. Two samples of CRR bed material, bauxite, were analyzed as well. This was done in order to compare the raw coal and bauxite to the processed coal and bauxite mixture found in the OGF.

2.5 Quality Assurance

The SRNL work was performed in accordance with a Quality Assurance Program (QAP) that meets the Quality Assurance criteria specified in DOE O. 414.1, *Quality Assurance*, 10 CFR 830, *Nuclear Safety Management*, Subpart A, "*Quality Assurance Requirements*", paragraph 830.122 and also meets the requirements of ASME Nuclear Quality Assurance (NQA)-1-2004, *Quality Assurance Requirements for Nuclear Facility Applications* including NQA-1a-2005 and NQA-1b-2007 Addenda, or later version

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

3.0 Results and Discussion

3.1 Raw Coal Sieve Analyses

The results of the ASTM D4749 sieve analyses are given in Table 6 and plotted graphically in Figure 3-1. When the data in Figure 3-1 is examined against the IWTU coal specification given in Table 2, it can be seen that the preferred size of 10mm is not met as the Gaussian distribution indicates that the mean particle size is ~ 8mm. However, most of the coal is within the range of the coal specification as shown by the gray shaded region of Figure 3-1.

The percent oversize, percent undersize and the percentage in the correct specification size range is given in Table 7. There is a tail of fine particle sizes outside the specification range. A statistical analysis was performed in two different manners on the coal particle data to derive the percentage of coal <6mm and > 12mm. A statistical interpolation of cumulative weight percents on each sieve was calculated and the measurement of the area under the curves in Figure 3-1 in the ranges of <6mm, 6-12mm, and >12mm were performed. The IWTU coal specification is given in Table 2 and the SRNL data is compared to the specification and to the particle size analysis performed by the IWTU vendor (Appendix A). The coal specification given in Table 2 does not specify an acceptable percentage of <6mm and/or greater than 12mm coal. Table 2 only specifies 6mm as the minimum and 12 mm as the maximum coal size. The IWTU vendor claimed 2.24% of the coal was <6mm and 1.32% was greater than 12mm (Appendix A). The analysis at SRNL indicates that 9.78-11.07% of the coal is <6mm while 0.79-3.98% was >12mm (Table 7). There is little variability of size within or between bags.

The lack of variability of size within a supersack or between supersacks is also shown by each of the three figures which are overlays of the data from Table 6 on a per supersack basis. There is also little between supersack variability as can be seen by comparing the top, middle, and bottom figures of Figure 3-1.

3.2 Raw Coal Proximate Analyses (Moisture, Volatiles, Fixed Carbon, Ash Content)

The results of the Proximate analyses of the nine coal samples from the three supersacks as measured by SRNL are given in Table 8 and the data measured by NETL is given for comparison in Table 9. The results of the moisture loss at 107°C by the two different laboratories are plotted in Figure 3-2. Measurements at SRNL were made in duplicate (Table 8) but the average of the duplicates is plotted in Figure 3-2. All of the moisture measurements were within the coal specification given in Table 2 and indicated by the shaded region in Figure 3-2. The average measured value (solid line in Figure 3-2) is virtually the same as the preferred value (dashed line is superimposed in Figure 3-2 but not visible) of moisture from the specification. The within supersack variability is indicted by the data within an ellipse and the between bag variability is indicted by comparing the ellipses to each other. There is little variability in moisture within a supersack or between supersacks.

The SRNL performed the volatile loss at 750°C in argon and the data are given in Table 8 and plotted in Figure 3-3. The NETL performed thee volatile loss at 950°C and the data are given in Table 9 and plotted in Figure 3-3. The NETL values at 950°C fall mostly within the specification although a few are biases low. SRNL's values are all biased low due to the low temperature at which the volatile loss was conducted. Most of the measurements are biased low compared to the preferred value given in the coal specification in Table 2 and indicated by the shaded region in Figure 3-3. The average measured value (solid line in Figure 3-3) is lower than the preferred value (dashed line in Figure 3-3) for volatiles from the Table 2 specification. The within supersack variability is indicted by the data within an ellipse and the between bag variability is indicted by comparing the ellipses to each other. There is wide variability within some supersacks.

The results of the ash content measurement at 750°C as measured by SRNL and NETL are given in Table 8 and Table 9 and plotted in Figure 3-3. Most of the measurements were within the coal specification values given in Table 2 and indicated by the shaded region in Figure 3-4. The average measured value (solid line in Figure 3-4) is ~1.5 wt% higher than the preferred value (dashed line in Figure 3-4) for volatiles from the Table 2 specification. The within supersack variability is indicted by the data within an ellipse and the between bag variability is indicted by comparing the ellipses to each other. While supersack #2 and #3 have low within bag variability, supersack #1 has high within bag variability.

The fixed carbon by difference is plotted in Figure 3-5 and is within the values calculated from the specification by difference since Table 2 does not specify a fixed carbon range.

Table 8 and Table 9 also compares the averages of the moisture content, volatiles, fixed carbon and ash from these 9 samples of Bestac coal to the historic average of Bestac coal analyses used by TTT at HRI (Appendix B) and to the IWTU vendor analysis (Appendix A). The IWTU coal is <1 wt% lower in moisture, ~4-5 wt% lower in volatiles, and ~10 wt% higher in fixed carbon. The ash content is about the same.

Table 6. ASTM D4749 Coal Sieve Size by Supersack and by Position Within a Supersack

Short ID	Long ID	ASTM Sieve Designation	Opening of Sieve (mm)	Percent Retained on Each Sieve (%)
Bag F	Bag 1 TOP	½ "	12.5	0.29
		3/8 "	9.5	15.58
		5/16 "	8	39.77
		¼ "	6.3	29.05
		No. 3 1/2	5.6	9.43
		No. 4	4.75	4.33
		No. 8	2.36	1.09
		No. 16	1.18	0.08
		No. 30	0.6	0.06
		No. 50	0.3	0.05
		No. 100	0.15	0.05
		No. 200	0.075	0.06
		No. 400	0.038	0.07
		< No. 400	<0.038	0.1
Bag H	Bag 1 MID	½ "	12.5	0.38
		3/8 "	9.5	19.88
		5/16 "	8	39.79
		¼ "	6.3	27.11
		No. 3 1/2	5.6	6.84
		No. 4	4.75	3.57
		No. 8	2.36	1.76
		No. 16	1.18	0.15
		No. 30	0.6	0.09
		No. 50	0.3	0.07
		No. 100	0.15	0.06
		No. 200	0.075	0.07
		No. 400	0.038	0.1
		< No. 400	<0.038	0.12
Bag A	Bag 1 BOT	½ "	12.5	0.39
		3/8 "	9.5	21.11
		5/16 "	8	40.28
		¼ "	6.3	25.38
		No. 3 1/2	5.6	6.59
		No. 4	4.75	4.02
		No. 8	2.36	1.64
		No. 16	1.18	0.15
		No. 30	0.6	0.08
		No. 50	0.3	0.06
		No. 100	0.15	0.06
		No. 200	0.075	0.06
		No. 400	0.038	0.08
		< No. 400	<0.038	0.11

Short ID	Long ID	ASTM Sieve Size	Opening of Sieve (mm)	Percent Retained on Each Sieve (%)
Bag D	Bag 2 TOP	½ “	12.5	0.43
		3/8 “	9.5	19.75
		5/16 “	8	36.76
		¼ “	6.3	27.49
		No. 3 1/2	5.6	7.92
		No. 4	4.75	4.91
		No. 8	2.36	2.16
		No. 16	1.18	0.16
		No. 30	0.6	0.09
		No. 50	0.3	0.06
		No. 100	0.15	0.05
		No. 200	0.075	0.06
		No. 400	0.038	0.08
		< No. 400	<0.038	0.1
Bag E	Bag 2 MID	½ “	12.5	0.2
		3/8 “	9.5	20.98
		5/16 “	8	40.91
		¼ “	6.3	26.46
		No. 3 1/2	5.6	5.95
		No. 4	4.75	3.61
		No. 8	2.36	1.26
		No. 16	1.18	0.15
		No. 30	0.6	0.09
		No. 50	0.3	0.07
		No. 100	0.15	0.06
		No. 200	0.075	0.06
		No. 400	0.038	0.09
		< No. 400	<0.038	0.09
Bag I	Bag 2 BOT	½ “	12.5	0.18
		3/8 “	9.5	20.65
		5/16 “	8	38.93
		¼ “	6.3	26.21
		No. 3 1/2	5.6	6.58
		No. 4	4.75	4.56
		No. 8	2.36	2.07
		No. 16	1.18	0.24
		No. 30	0.6	0.13
		No. 50	0.3	0.09
		No. 100	0.15	0.07
		No. 200	0.075	0.08
		No. 400	0.038	0.09
		< No. 400	<0.038	0.11

Short ID	Long ID	ASTM Sieve Size	Opening of Sieve (mm)	Percent Retained on Each Sieve (%)
Bag G	Bag 3 TOP	½ "	12.5	0.33
		3/8 "	9.5	21.55
		5/16 "	8	38.64
		¼ "	6.3	25.47
		No. 3 1/2	5.6	7.37
		No. 4	4.75	4.22
		No. 8	2.36	1.78
		No. 16	1.18	0.16
		No. 30	0.6	0.1
		No. 50	0.3	0.07
		No. 100	0.15	0.06
		No. 200	0.075	0.06
		No. 400	0.038	0.07
		< No. 400	<0.038	0.1
Bag B	Bag 3 MID	½ "	12.5	0.25
		3/8 "	9.5	29.76
		5/16 "	8	41.68
		¼ "	6.3	20.51
		No. 3 1/2	5.6	4.41
		No. 4	4.75	1.8
		No. 8	2.36	0.86
		No. 16	1.18	0.16
		No. 30	0.6	0.11
		No. 50	0.3	0.08
		No. 100	0.15	0.07
		No. 200	0.075	0.08
		No. 400	0.038	0.12
		< No. 400	<0.038	0.13
Bag C	Bag 3 BOT	½ "	12.5	0.74
		3/8 "	9.5	26.41
		5/16 "	8	41.43
		¼ "	6.3	21.59
		No. 3 1/2	5.6	4.84
		No. 4	4.75	2.98
		No. 8	2.36	1.5
		No. 16	1.18	0.12
		No. 30	0.6	0.07
		No. 50	0.3	0.06
		No. 100	0.15	0.05
		No. 200	0.075	0.06
		No. 400	0.038	0.07
		< No. 400	<0.038	0.09

Table 7. IWTU Bestac Coal Particle Size Percentages by Bag and Position Within a Bag

Short ID	Long ID	Percent <6 mm		Percent 6mm-12mm		Percent >12mm	
		STAT	AREA	STAT	AREA	STAT	AREA
Bag F	Bag 1 TOP	11.28	12.50	85.84	86.46	2.88	1.04
Bag H	Bag 1 MID	9.9	11.95	86.40	87.37	3.70	0.68
Bag A	Bag 1 BOT	10.03	11.00	86.08	88.5	3.90	0.50
Bag D	Bag 2 TOP	12.20	13.50	84.10	85.77	3.70	0.73
Bag E	Bag 2 MID	8.88	9.51	87.40	89.73	3.72	0.76
Bag I	Bag 2 BOT	11.20	12.69	85.17	86.92	3.63	0.39
Bag G	Bag 3 TOP	10.83	12.68	85.23	86.62	3.94	0.70
Bag B	Bag 3 MID	5.93	7.17	88.88	92.41	5.19	0.42
Bag C	Bag 3 BOT	7.77	8.61	87.10	89.48	5.13	1.91
This study	Average	9.78	11.07	86.24	88.14	3.98	0.79
IWTU Vendor (Appendix A)	Average	10.43		87.19		2.385	

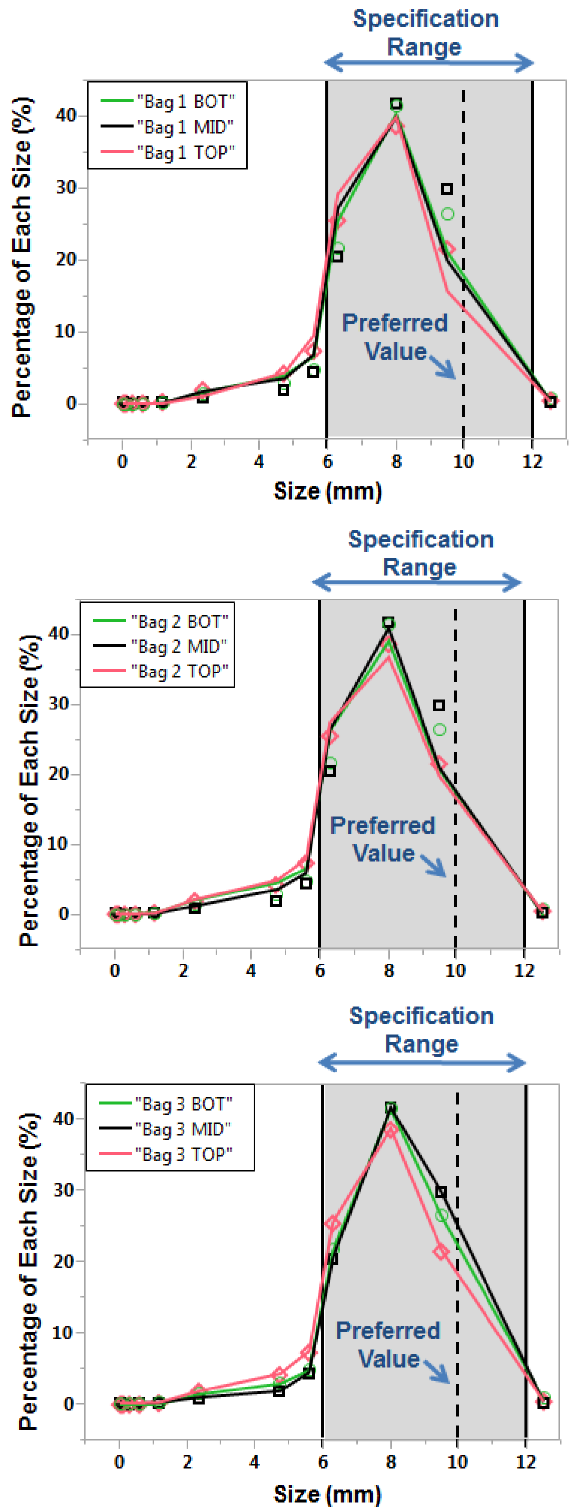


Figure 3-1. Size Distribution in mm of IWTU Bestac Coal in Supersacks.

Shaded region is coal specification from Table 2. Preferred Value is also from coal specification in Table 2.

Table 8. SRNL ASTM D3172 Proximate Coal Analyses by Supersack and by Position

Short ID	Long ID	Duplicate Moisture % 107°C in vac	Average Moisture % 107°C in vac	Volatiles % at 750°C in Ar (dry)	Ash % in air at 750°C (dry)	Fixed Carbon % by Difference (dry)
Bag F	Bag 1 TOP	5.05 5.53	5.29	11.27	4.76	83.97
Bag H	Bag 1 MID	4.73 5.07	4.90	7.92	10.21	81.87
Bag A	Bag 1 BOT	4.49 5.9	5.20	6.15	7.74	86.11
Bag D	Bag 2 TOP	3.78 4.61	4.20	7.22	5.27	87.51
Bag E	Bag 2 MID	4.99 4.33	4.66	10.22	5.77	84.01
Bag I	Bag 2 BOT	4.94 4.66	4.8	6.83	5.00	88.17
Bag G	Bag 3 TOP	4.3 5.75	5.03	2.75	6.24	91.01
Bag B	Bag 3 MID	5.13 4.82	4.98	9.23	7.46	83.31
Bag C	Bag 3 BOT	3.82 5.32	4.57	11.48	4.24	84.28
Average 9 Samples		4.85	4.85	7.92	6.30	85.58
Bestac Avg 2004, 2006, 2011		Not Applicable	5.86	15.11	6.80	78.09
IWTU Vendor (Appendix A)			5.80	14.5	6.4	79.10

*Note that dry basis means the volatiles + ash + fixed carbon sum to 100%

NETL had additional proximate analysis (ASTM D7582) performed on the 9 samples of IWTU Bestac calcined coal (samples A-I). The samples were prepared according to ASTM D2013. This analysis was done using macro thermogravimetric analysis (TGA). Moisture was analyzed at 107^{o}C in nitrogen. Volatile matter was analyzed up to a maximum temperature of 950^{o}C under nitrogen. The ash content was analyzed at 750^{o}C in an oxygen atmosphere. These conditions follow the testing methods given by ASTM D7582-12 for coal samples. The average values across all samples are moisture 5.47wt%, volatile 10.88wt%, ash 7.40wt% and fixed carbon 81.72wt%. The results of this analysis are presented in Table 9. The volatile, ash and fixed carbon content are presented on a dry basis.

Table 9. NETL ASTM D7582, ASTM D5865 Proximate Coal Analyses and Heating Value by Supersack and by Position

Short ID	Long ID	Moisture % At 107°C in nitrogen	Volatiles % at 950°C (dry)	Ash % at 750°C (dry)	Fixed Carbon % Difference (dry)	Heating Value [Btu/lb]
Bag F	Bag 1 TOP	6.10	11.61	7.20	81.19	13497
Bag H	Bag 1 MID	5.75	11.95	7.14	80.91	13425
Bag A	Bag 1 BOT	5.67	12.07	6.16	81.77	13544
Bag D	Bag 2 TOP	5.66	11.02	7.55	81.43	13351
Bag E	Bag 2 MID	5.51	10.63	7.60	81.77	13380
Bag I	Bag 2 BOT	5.23	11.51	6.87	81.62	13482
Bag G	Bag 3 TOP	5.45	9.42	8.96	81.62	13161
Bag B	Bag 3 MID	5.41	9.25	7.15	83.60	13468
Bag C	Bag 3 BOT	4.42	10.49	7.96	81.55	13323
Average 9 Samples	Not Applicable	5.47	10.88	7.40	81.72	13403
Bestac Avg 2004, 2006, 2011		5.86	15.11	6.80	72.23	13442
IWTU Vendor (Appendix A)		5.80	14.5	6.4	73.3	12060

The NETL proximate analyses moisture percentage average is 5.47 wt% (Table 9) while the SRNL average moisture values are 4.85 wt% (Table 8). While the SRNL values are biased low, both sets of values fall within the IWTU coal specification given in Table 2. The NETL ashing was performed at 750°C as was the SRNL ashing (per ASTM D7582) but the volatile analyses were done at different temperatures. The moisture and ash content between the two laboratories compared favorably but not the volatiles and fixed carbon by difference in the Proximate Analyses.

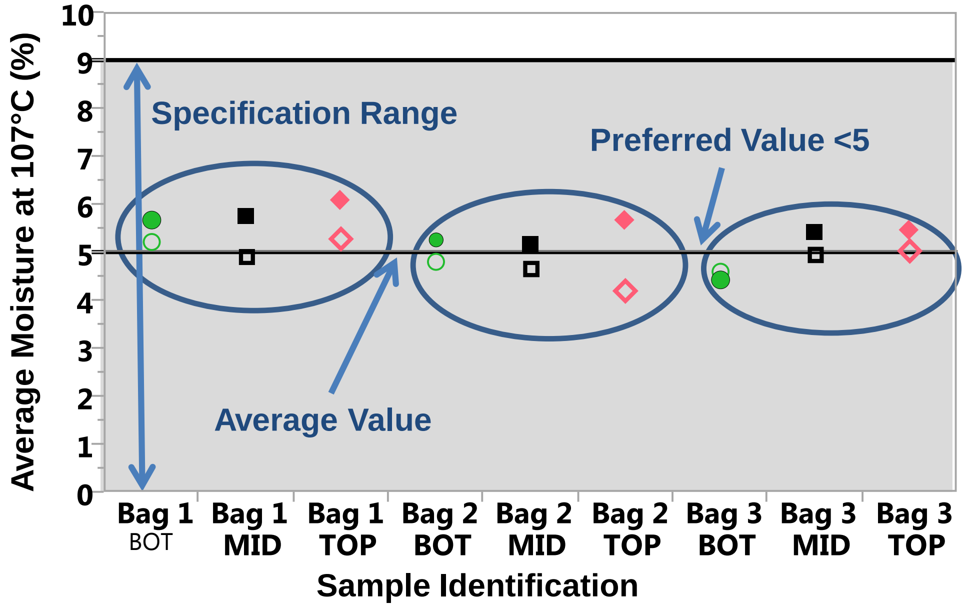


Figure 3-2. Moisture content of coal from Proximate Analysis at 107°C (SRNL data are the open circles and NETL data are the solid circles).
Coal specification range is indicated by shading. Preferred specification value is indicated by a dashed line and the average measured value is indicated by the solid line. Within bag variability is indicted by the data within an ellipse and the between bag variability is indicted by comparing the ellipses to each other.

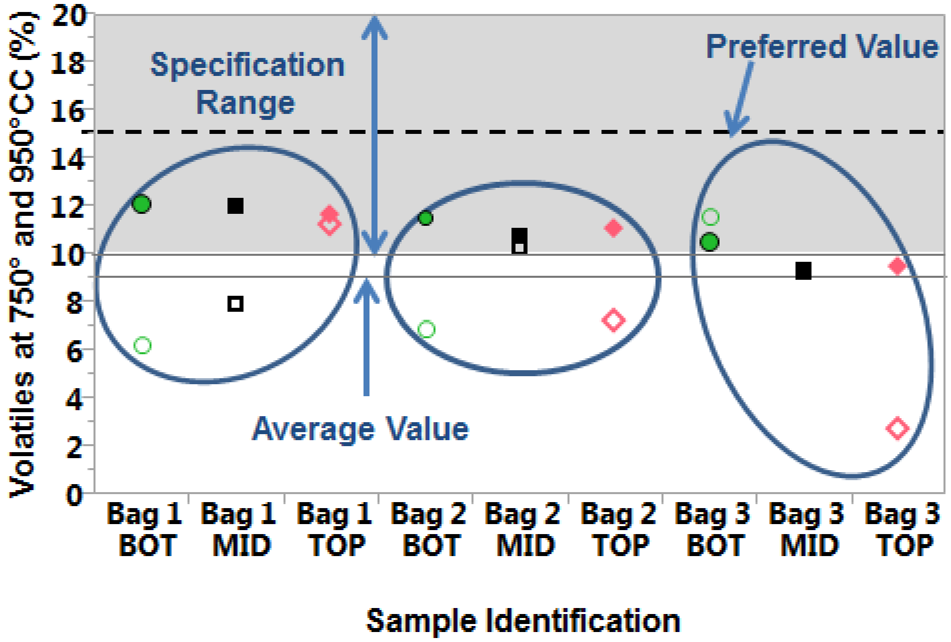


Figure 3-3. Volatile content of coal from Proximate Analysis at 750°C and 950°C (SRNL data are the open circles and NETL data are the solid circles).
Coal specification range is indicated by shading. Preferred specification value is indicated by a dashed line and the average measured value is indicated by the solid line. Within bag variability is indicted by the data within an ellipse and the between bag variability is indicted by comparing the ellipses to each other.

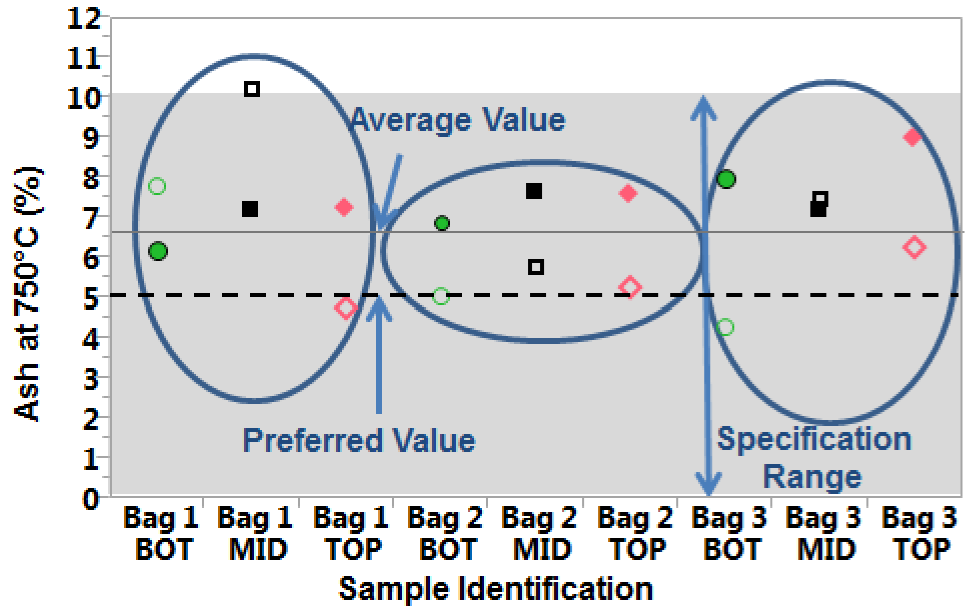


Figure 3-4. Ash content of coal from Proximate Analysis at 750°C (SRNL SRNL data are the open circles and NETL data are the solid circles).

Coal specification range is indicated by shading. Preferred specification value is indicated by a dashed line and the average measured value is indicated by the solid line. Within bag variability is indicted by the data within an ellipse and the between bag variability is indicted by comparing the ellipses to each other.

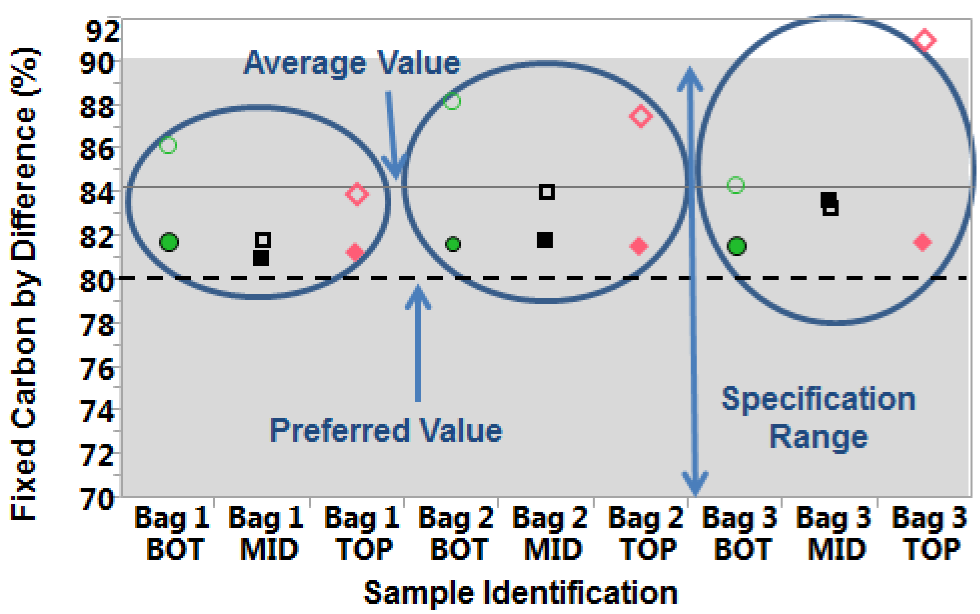


Figure 3-5. Fixed carbon content of coal from Proximate Analysis by Difference (SRNL data are the open circles and NETL data are the solid circles).

Coal specification range is indicated by shading. Preferred specification value is indicated by a dashed line and the average measured value is indicated by the solid line. Within bag variability is indicted by the data within an ellipse and the between bag variability is indicted by comparing the ellipses to each other.

3.3 Raw Coal Ultimate Analysis (C, H, N, O, S and Cl)

Ultimate analysis (ASTM D5373) was performed by CONSOL Energy Inc. on the 9 samples of IWTU Bestac calcined coal (samples A-I). The samples were prepared according to ASTM D2013. The content of carbon, hydrogen, nitrogen and oxygen (by difference) were analyzed for each sample. The average values across all samples are C 84.28wt%, H 1.67wt%, N 1.15wt% and O 5.30wt%. Sulfur (ASTM D4239) and chlorine (ASTM D6721) analysis was performed to determine the content in the coal. The average values of these species are S 0.21wt% and Cl 0.005wt%. In addition to the ash, these species make up the starting mass of the coal samples. The results of this analysis are presented in Table 10. The ash, carbon, hydrogen, nitrogen, oxygen, sulfur and chlorine content are presented on a dry basis. The dry sulfur analyses in Table 10 are within the IWTU coal specification given in Table 2.

Table 10. NETL ASTM D5373, ASTM D4239, ASTM D6721 Ultimate, Sulfur and Chlorine Coal Analyses.

Short ID	Long ID	Ash % (dry)	Carbon % (dry)	Hydrogen % (dry)	Nitrogen % (dry)	Oxygen % Difference (dry)	Sulfur % (dry)	Chlorine % (dry)
Bag F	Bag 1 TOP	7.20	83.52	1.70	1.19	6.18	0.20	0.0055
Bag H	Bag 1 MID	7.14	85.18	1.72	1.14	4.62	0.20	0.0048
Bag A	Bag 1 BOT	6.16	84.80	1.80	1.14	5.87	0.22	0.0063
Bag D	Bag 2 TOP	7.55	83.97	1.71	1.13	5.43	0.21	0.0047
Bag E	Bag 2 MID	7.60	84.31	1.67	1.14	5.04	0.24	0.0045
Bag I	Bag 2 BOT	6.87	84.73	1.75	1.16	5.29	0.20	0.0047
Bag G	Bag 3 TOP	8.96	82.82	1.46	1.09	5.46	0.20	0.0054
Bag B	Bag 3 MID	7.15	84.93	1.53	1.15	5.04	0.20	0.0043
Bag C	Bag 3 BOT	7.96	84.22	1.67	1.18	4.76	0.20	0.0051
Average		7.40	84.28	1.67	1.15	5.30	0.21	0.005

3.4 Raw Coal Ash Analyses

The last chemical analyses performed were the analyses of the coal ash on an oxide basis. The data is given in Table 11 and is shown to sum to 100±5 wt% on an oxide basis. Each ash sample was analyzed in duplicate and the duplicates are plotted in Figure 3-6 and Figure 3-6 to Figure 3-7 will be discussed first as these elements are identified as important in the coal specification given in Table 2. Figure 3-8 to Figure 3-11 cover other oxides not specifically called out in the coal specification.

Figure 3-6a indicates that the CaO content of the IWTU Bestac coal ash is within the specification range given in Table 2 but the average concentration is slightly larger than the preferred value instead of lower than the preferred value as indicated in the specification. Figure 3-6b indicates that the Na_2O + K_2O content of the IWTU Bestac coal is also within the specification and way below the preferred value. Since the "preferred value" in Table 2 says at or below this value that is excellent. Figure 3-7 indicates that the SiO_2 content of the IWTU Bestac coal is biased about 5 wt% higher than the maximum preferred value in the coal specification. In addition, the within supersack and between supersack variability is high.

Other ash oxide variability, for oxides not included in the coal specification given in Table 2 are shown in Figure 3-8 to Figure 3-11. Figure 3-8 indicates the variability in Al_2O_3 while Figure 3-11a indicates the SO_3 content of the IWTU Bestac coal ash. The SO_3 content of the ash cannot be equated to the total S content analysis of an Ultimate analysis which is specified in Table 2 because not all the S in coal is represented by the SO_3 in the ash. There is also high variability in the TiO_2 content of the Bestac coal ash both within a supersack and between supersacks (Figure 3-11b).

Lastly, the minimum value, maximum value, and average value of the important oxides in the ash are given in Table 11. The values of the ash content in this study is compared to the average values measured by HRI for FBSR processing for Bestac coal from 2004, 2006, and 2011. Since this data is not available in any publications the actual values measured by HRI are given in Appendix B. It can be seen that the IWTU Bestac coal is higher in SiO_2 content by ~ 5 wt%, lower in Al_2O_3 by 5 wt%, higher in CaO content by about 0.5 wt%, lower in Fe_2O_3 by ~ 0.5 wt%, and lower in Na_2O+K_2O by ~ 2 wt%.

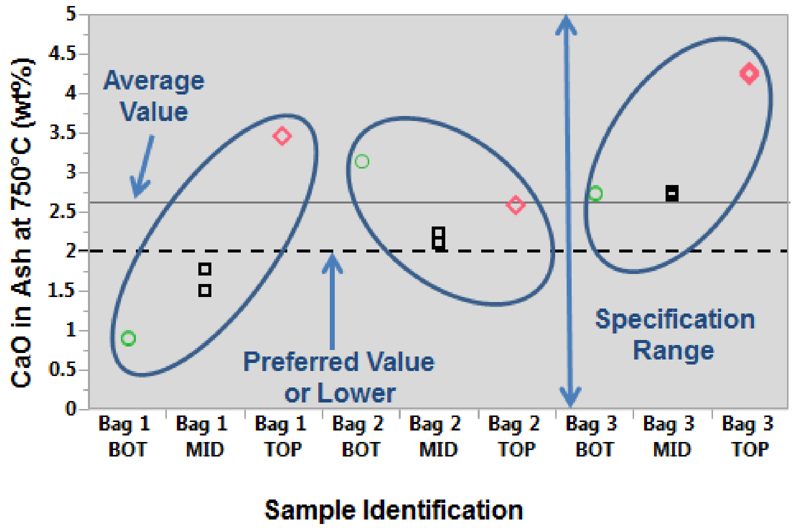
Additional ash analysis was performed by NETL on three samples of IWTU Bestac calcined coal (samples B, E & H) that had been ashed at 550°C under air for 8 hours. The samples were ground using a cutting mill and sieved to pass through a #50 mesh (< 0.3 mm). The analysis was done using inductively coupled plasma optical emission spectroscopy (ICP-OES). The ash samples were analyzed in duplicate and the average values are given. The results are presented in the oxide form and the species analyzed by ICP-OES are shown to sum to 90-92 wt% of the ash. When the sulfur content from ultimate analysis, in the form of SO_3, is added to the mass balance this value increases to ~99 wt%. The average values across all three samples are Al_2O_3 17.9wt%, CaO 3.07wt%, Fe_2O_3 13.98wt%, K_2O 0.34wt%, MgO 1.09wt%, MnO_2 0.398wt%, Na_2O 0.67wt%, P_2O_5 1.44wt%, SiO_2 52.16wt% and TiO_2 0.57wt%. The results of this analysis are shown in Table 12. Oxide species are given as wt% of oxide in ash.

Since the SRNL and NETL used different ashing temperatures, 750°C and 550°C respectively, the ash analyses are similar but not identical. Most notably the NETL analyses indicate a higher concentration of sulfate in the ash than the SRNL analyses. However, the SRNL analyses are more in line with the historic analyses of Bestac coal between 2004-2011 as given in Table 11 and Appendix B.

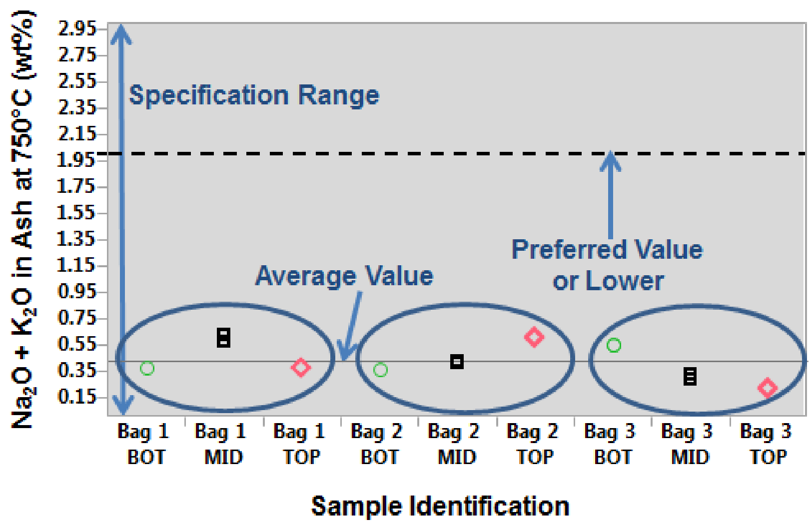
3.5 Raw IWTU Bestac Coal and Coal Ash Phase Analyses by X-Ray Diffraction

Two X-ray Diffraction (XRD) analyses were performed on the coal sample H (Bag #1 middle sample). A chunk of coal from sample H was ground up and analyzed and the <400 mesh fines from the sieve analyses of sample H were also analyzed for comparison. The ground chunk of coal showed the presence of excess quartz (SiO_2) as shown in Figure 3-12a. The fines showed the presence of excess quartz as well and the presence of excess kaolinite ($Al_2Si_2O_5(OH)_4$) as shown in Figure 3-12b. Both of these phases are sources of excess SiO_2 in the Bestac coal as discussed in the previous section.

Bestac (IWTU) coal from bag F was ashed in air at 750°C for 5 hours. X-ray diffraction analysis of the ashed coal indicated excess SiO_2 and excess Fe_2O_3.



(a)



(b)

Figure 3-6. Analysis of IWTU Bestac Coal Ash for CaO and Alkali (Na_2O+K_2O) ashed at 750°C.

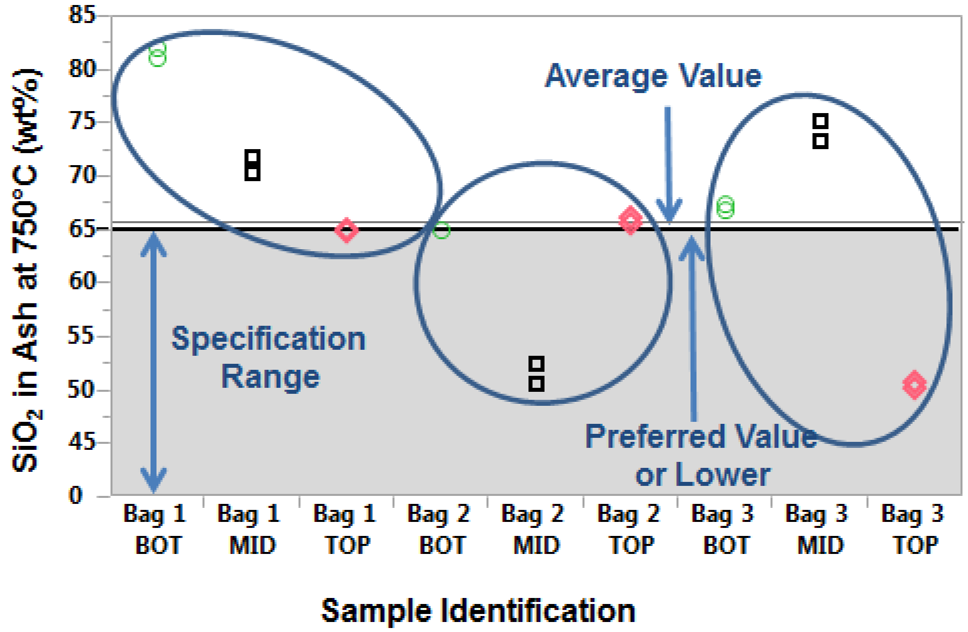


Figure 3-7. Analysis of IWTU Bestac Coal Ash for SiO_2 ashed at 750°C.

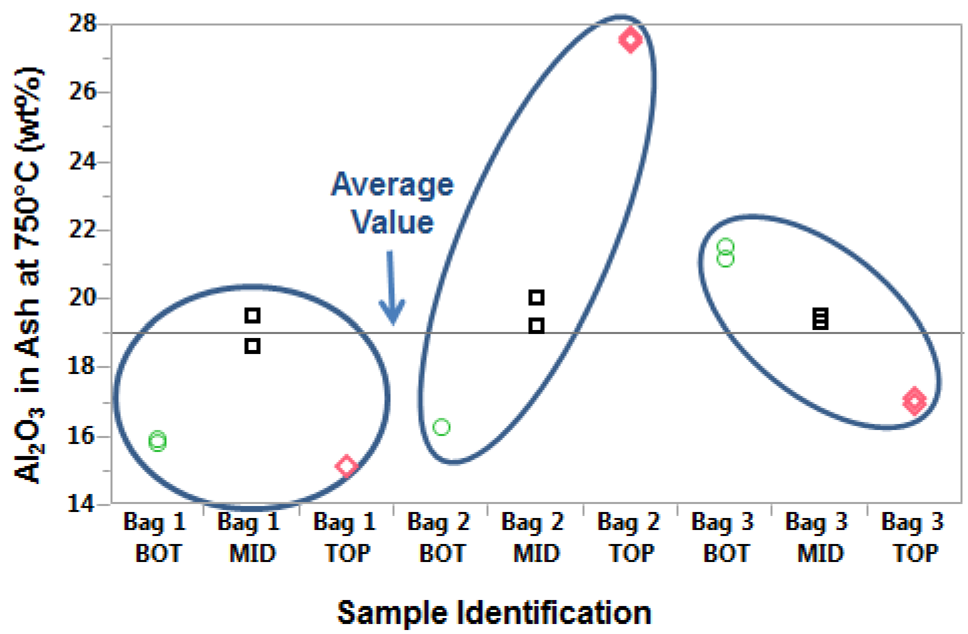


Figure 3-8. Analysis of IWTU Bestac Coal Ash for Al_2O_3 ashed at 750°C.

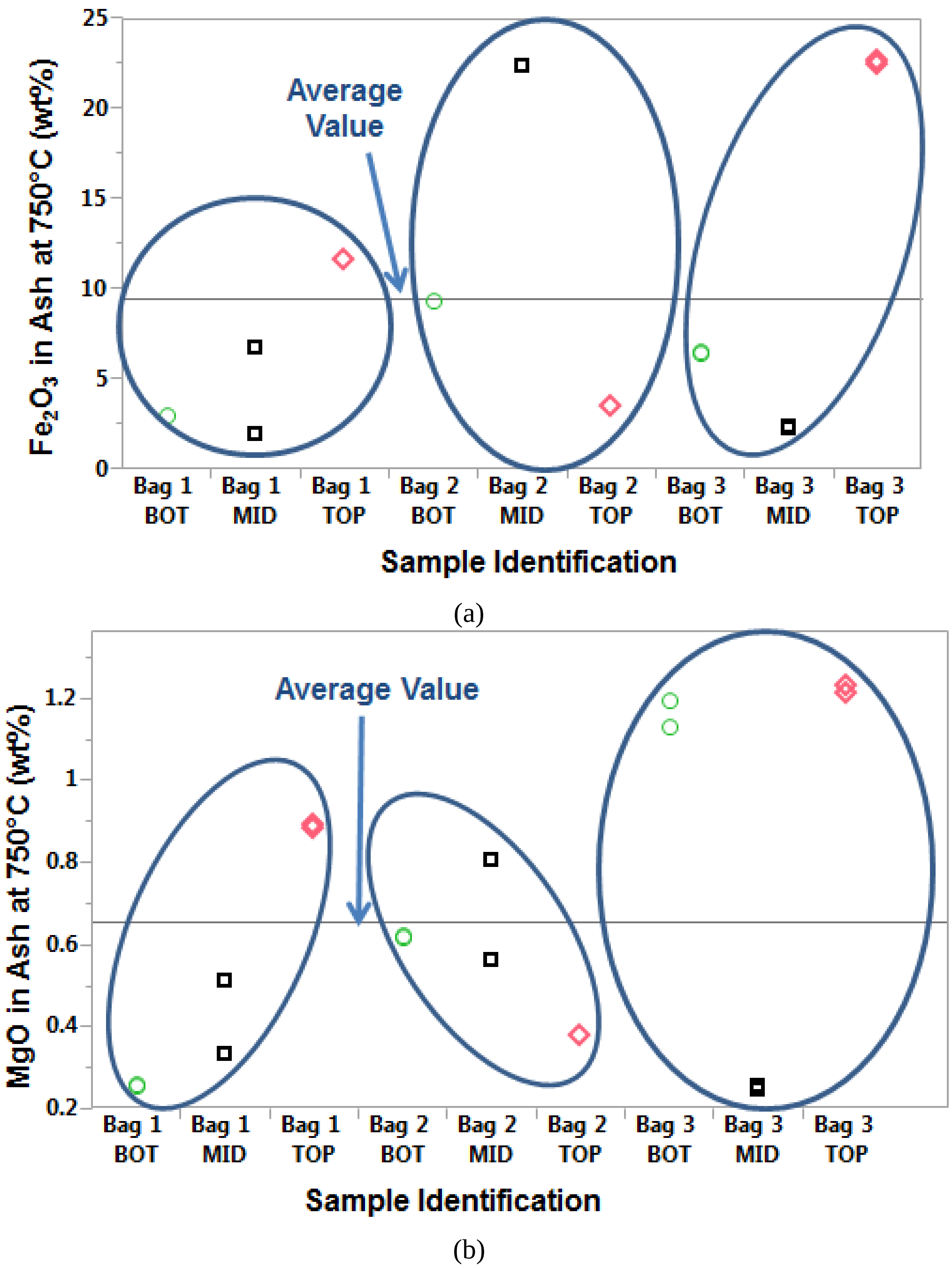
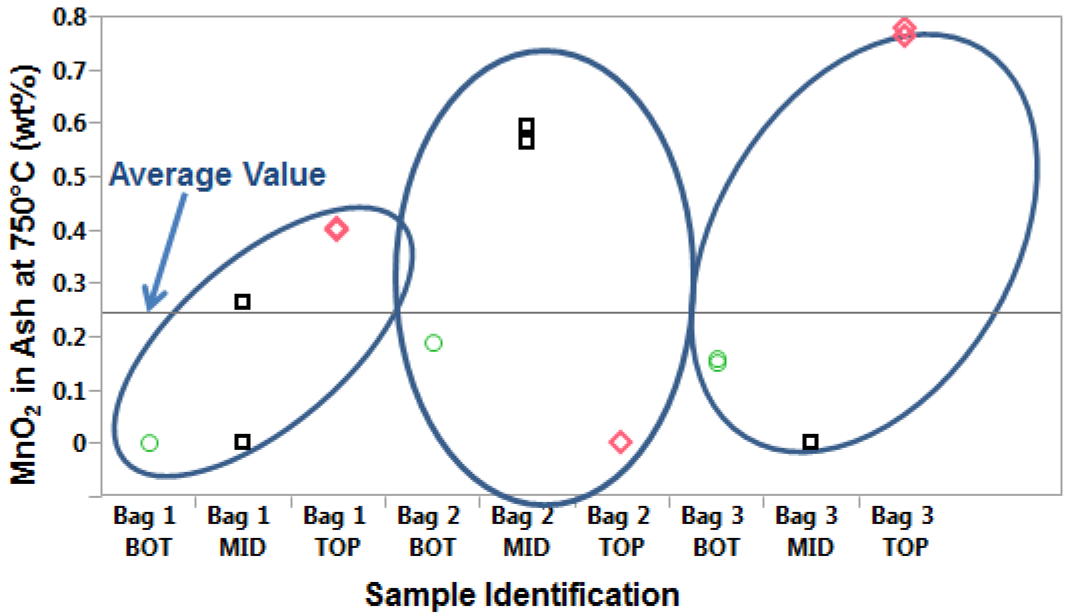
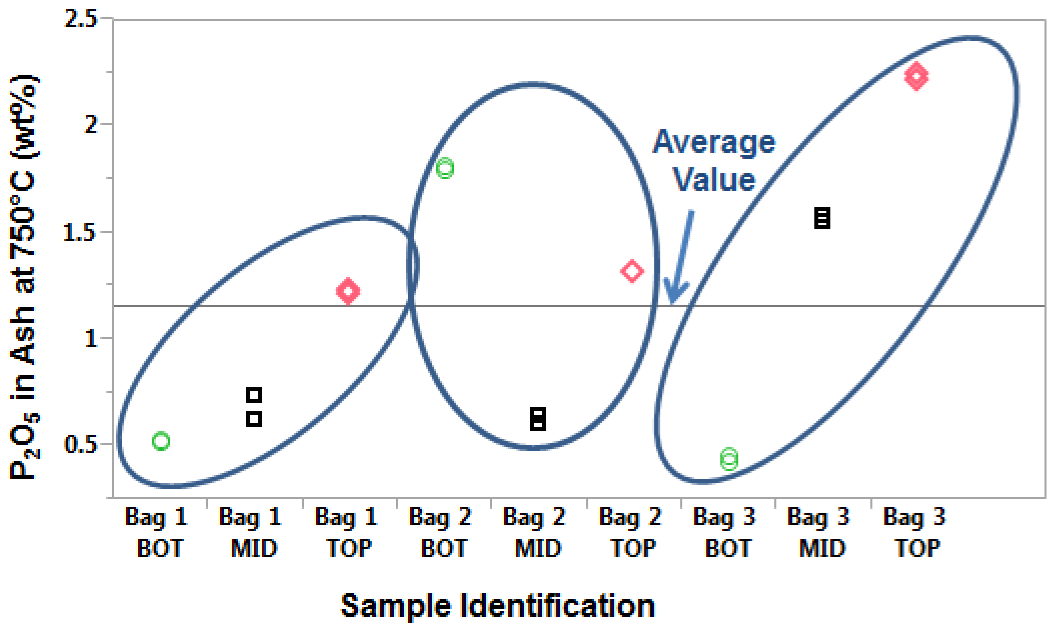


Figure 3-9. Analysis of IWTU Bestac Coal Ash for Fe_2O_3 and MgO ashed at 750°C



(a)



(b)

Figure 3-10. Analysis of IWTU Bestac Coal Ash for MnO_2 and P_2O_5 ashed at 750°C.

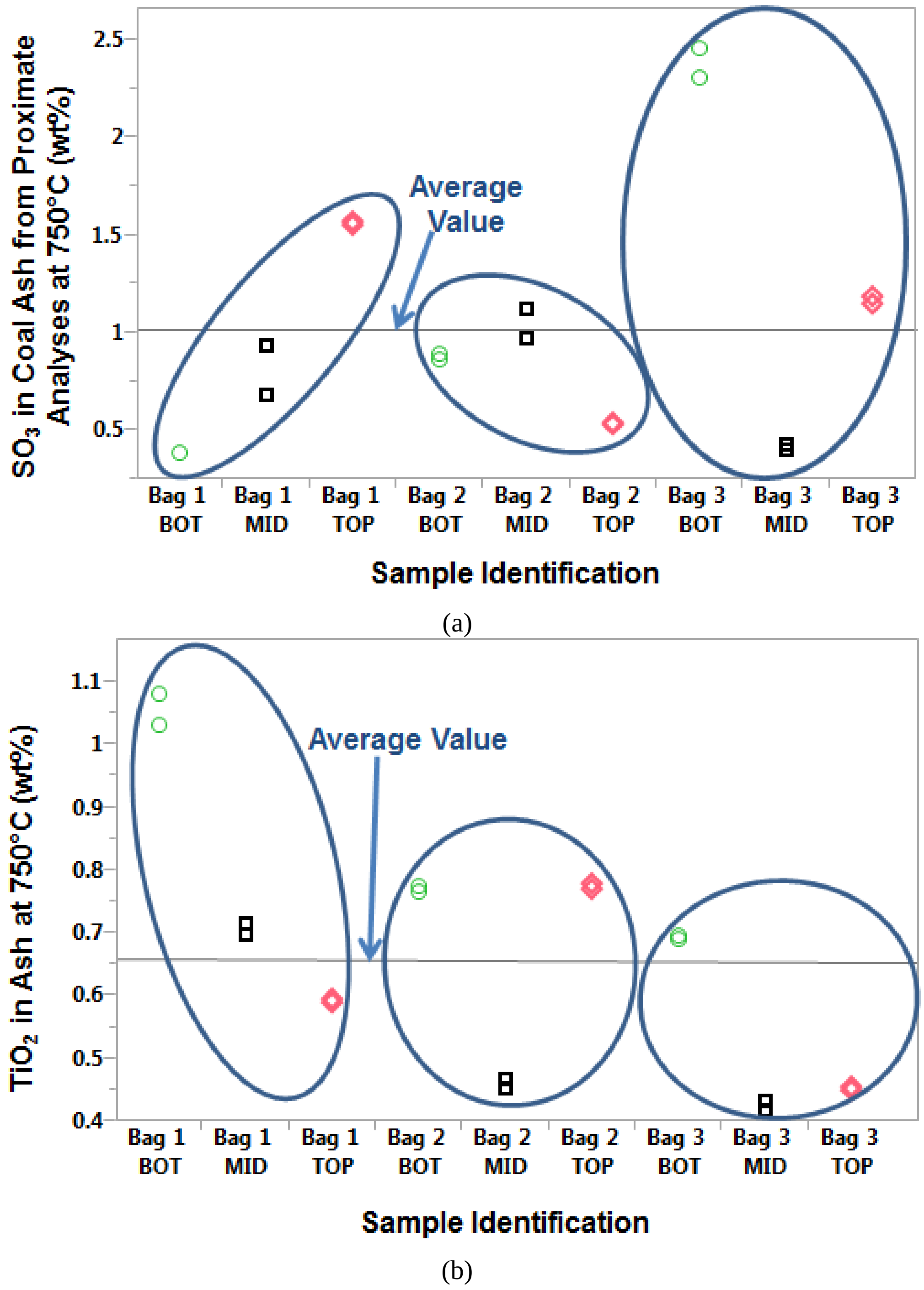


Figure 3-11. Analysis of IWTU Bestac Coal Ash for SO_3 and TiO_2 ashed at 750°C.

Table 11. Analysis of the Coal Ash from ASTM D3172 Proximate Coal Analyses at 750°C by Supersack and by Position within a Supersack

Short ID	Long ID	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	MnO_2	Na_2O	P_2O_5	SO_3	SiO_2	TiO_2	SUM	Na_2O+K_2O
Bag F	Bag 1 TOP	15.10	3.44	11.70	0.00	0.89	0.40	0.38	1.23	1.57	64.70	0.59	100.01	0.38
		15.10	3.45	11.70	0.00	0.88	0.40	0.37	1.22	1.55	65.00	0.59	100.26	0.373
Bag H	Bag 1 MID	19.50	1.51	2.03	0.39	0.33	0.00	0.19	0.63	0.68	71.70	0.70	97.65	0.579
		18.60	1.77	6.79	0.43	0.51	0.26	0.20	0.73	0.93	70.20	0.71	101.15	0.631
Bag A	Bag 1 BOT	15.90	0.92	2.87	0.15	0.26	0.00	0.23	0.52	0.38	82.00	1.03	104.25	0.38
		15.80	0.90	2.84	0.15	0.26	0.00	0.23	0.51	0.37	81.00	1.08	103.14	0.38
Bag D	Bag 2 TOP	27.50	2.59	3.53	0.25	0.38	0.00	0.37	1.32	0.53	65.50	0.78	102.73	0.611
		27.60	2.59	3.55	0.25	0.38	0.00	0.36	1.32	0.54	66.00	0.77	103.35	0.6
Bag E	Bag 2 MID	20.00	2.22	22.40	0.15	0.81	0.56	0.26	0.64	1.12	52.40	0.45	101.02	0.412
		19.20	2.10	22.40	0.14	0.56	0.60	0.28	0.61	0.97	50.50	0.47	97.83	0.42
Bag I	Bag 2 BOT	16.30	3.14	9.32	0.00	0.62	0.19	0.36	1.81	0.88	65.00	0.77	98.39	0.362
		16.30	3.15	9.32	0.00	0.62	0.19	0.36	1.79	0.86	65.00	0.77	98.35	0.36
Bag G	Bag 3 TOP	17.10	4.27	22.70	0.00	1.23	0.78	0.21	2.25	1.18	50.60	0.45	100.77	0.21
		16.90	4.22	22.50	0.00	1.21	0.76	0.22	2.22	1.15	50.10	0.45	99.74	0.22
Bag B	Bag 3 MID	19.50	2.74	2.26	0.00	0.25	0.00	0.32	1.58	0.40	75.10	0.42	102.57	0.32
		19.30	2.71	2.39	0.00	0.26	0.00	0.29	1.55	0.43	73.20	0.43	100.56	0.289
Bag C	Bag 3 BOT	21.50	2.75	6.43	0.15	1.13	0.15	0.40	0.42	2.30	67.40	0.69	103.31	0.544
		21.20	2.72	6.37	0.16	1.19	0.16	0.39	0.44	2.45	66.80	0.69	102.57	0.551
SRNL MIN		15.10	0.90	2.03	0.00	0.25	0.00	0.19	0.42	0.37	50.10	0.42	97.65	0.21
SRNL MAX		27.60	4.27	22.70	0.43	1.23	0.78	0.40	2.25	2.45	82.00	1.08	104.25	0.63
SRNL AVG		19.02	2.62	9.51	0.12	0.65	0.25	0.30	1.15	1.02	65.68	0.66	100.98	0.42
AVG Bestac 2004, 2006, 2011		23.64	1.98	8.75	0.56	0.25	NA	1.97	1.08	0.47	61.01	0.62	100.36	2.53
IWTU Vendor Analyses (Appendix A)			1.93								63.69			1.68

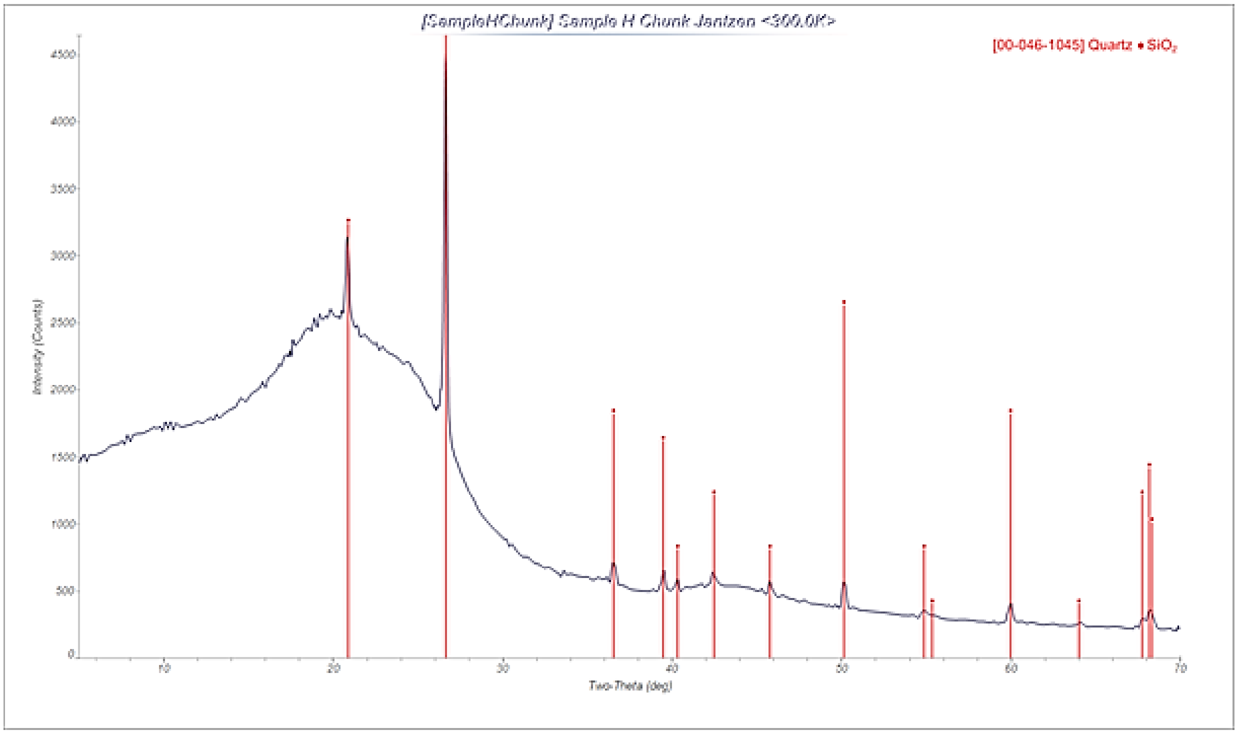
NA = Not Analyzed

Table 12. NETL ICP-OES Analysis of the Coal Ash at 550°C of IWTU Bestac calcined coal samples

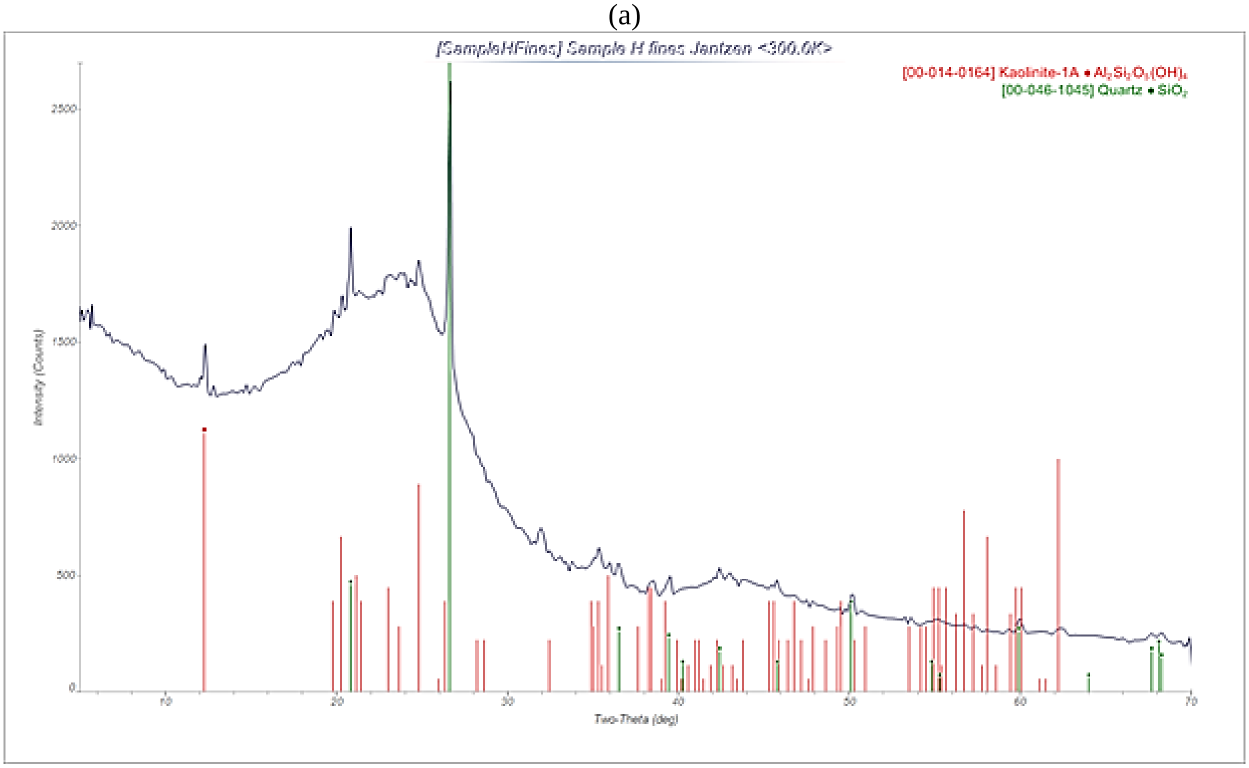
Short ID	Long ID	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	MnO_2	Na_2O	P_2O_5	SO_3	SiO_2	TiO_2	SUM	Na_2O+K_2O
Bag H	Bag 1 MID	17.28	3.00	15.93	<0.34	1.33	0.46	0.90	1.28	6.80	50.56	0.59	98.48	1.24
Bag E	Bag 2 MID	16.87	3.18	13.73	<0.34	1.05	0.43	0.68	1.51	7.65	52.47	0.57	98.48	1.02
Bag B	Bag 3 MID	19.54	3.03	12.27	<0.34	0.89	0.30	<0.43	1.52	7.80	53.45	0.53	99.08	0.77
NETL AVG		17.90	3.07	13.98	<0.34	1.09	0.40	0.67	1.44	7.08	52.16	0.57	98.68	1.01
IWTU Vendor Analyses (Appendix A)			1.93								63.69			1.68

***Detection limits: K_2O 0.34wt%, Na_2O 0.43wt%.**

****SO_3 content taken from ultimate analysis.**



(a)



(b)

Figure 3-12. X-ray Diffraction Analyses of Sample H. A ground chunk was analyzed and <400 mesh fines from the sieving analyses was analyzed.

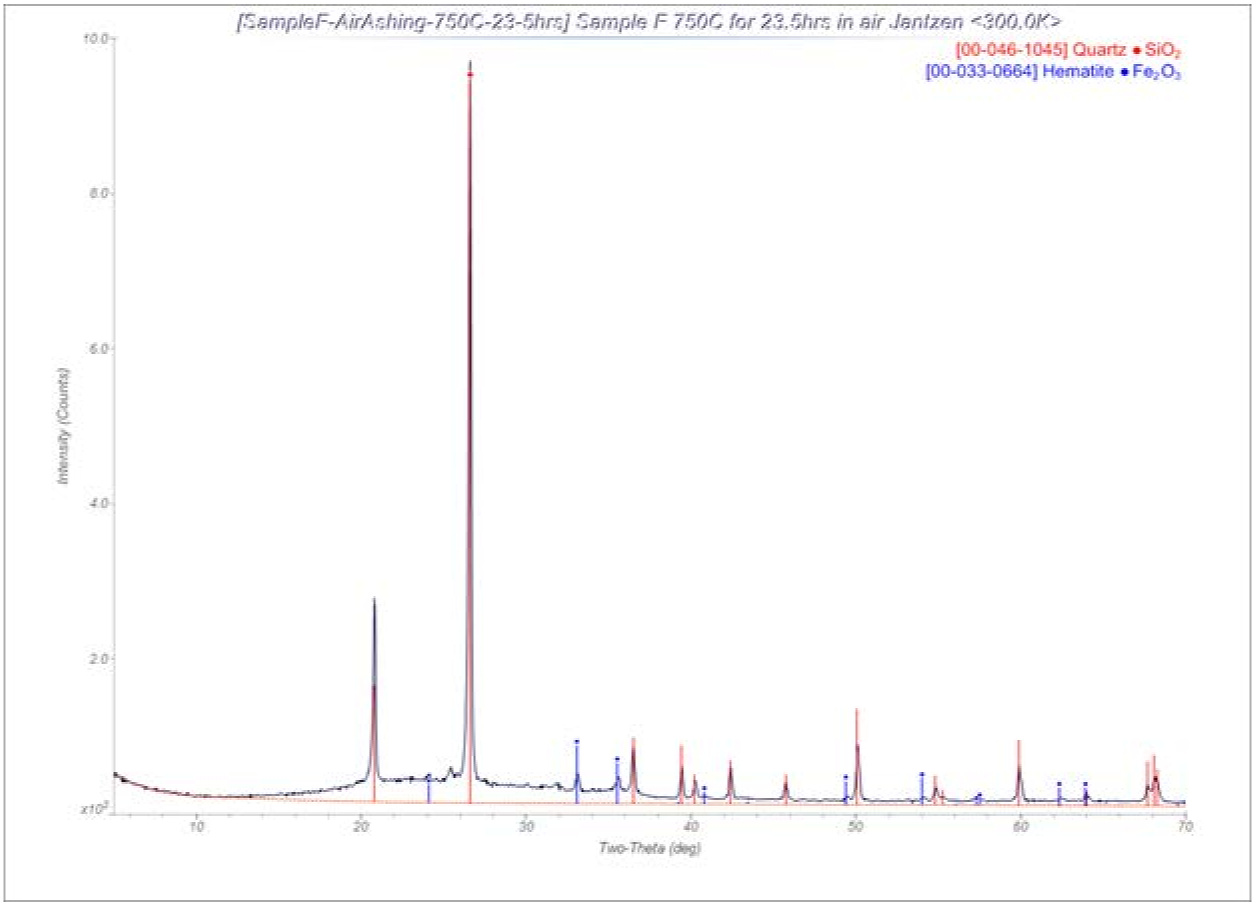


Figure 3-13. X-ray Diffraction X-ray Diffraction Analyses of IWTU Bestac Coal Sample F after ashing at 750°C in air for 5 hours.

3.6 Proximate Analyses, Ash Analyses, and XRD of Coke and Wood Based Charcoal

The Proximate Analyses of the wood based charcoal was performed at 750°C at SRNL in accordance with ASTM D7582 while the coke Proximate analyses were performed at 950°C in accordance with ASTM D7582. NETL performed the Ultimate analyses of the coke and wood based charcoal. Note the similarity of the values in Table 13 and Table 14 for the coke and wood based charcoal samples. The wood based charcoal was heated for the recommended time to generate the data in Table 13 but heat treatment continued for close to 100 hours and an interesting finding was that the sample continued to lose volatiles at 750°C.

Table 13. SRNL Proximate Analyses of Coke and Wood Based Charcoal (Dry Basis*)

Sample ID	Proximate Analysis Temperature	Moisture % 107°C	Volatiles %	Ash %	Fixed Carbon % by Difference
Wood Based Charcoal	750°C	7.12	13.94	16.64	69.42
Coke	950°C	0.41	0	0.10	99.90

*Note that dry basis means the volatiles + ash + fixed carbon sum to 100%

Table 14. NETL Ultimate Analyses of Coke and Wood Based Charcoal

Concentration (Wt%, Dry Basis)*									
Sample Description	As Det'd Moisture (Wt%)	Volatile Matter	Ash @750° C	Fixed Carbon (by diff)	Carbon	Hydrogen	Nitrogen	Sulfur	Oxygen (by diff)
Wood Based Charcoal	3.92	22.40	18.81	58.79	67.33	2.19	1.01	0.03	10.63
Coke	0.03	0.39	0.12	99.49	97.34	0.25	0.61	<0.02	1.68

*Note that dry basis means the volatiles + ash + fixed carbon sum to 100%

Table 15 gives the analyses of the wood based charcoal ash on an oxide basis as measured by SRNL PSAL. The coke had only 0.1-0.12% ash (Table 13 and Table 14) so there was an insufficient amount of ash to determine the oxide content. The wood based charcoal ash is extremely high in CaO and K_2O.

Table 15. SRNL Analysis of the Coal Ash from ASTM D3172 Proximate Coal Analyses at 750°C of Wood Based Charcoal Used to Start the DMR

Sample ID	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	MnO_2	Na_2O
Wood Based Charcoal	1.70	50.4	0.51	8.91	4.54	1.747	0.897
	1.68	51.1	0.53	8.85	4.53	1.809	0.872
	P_2O_5	SO_3	SiO_2	TiO_2	SUM	Na_2O+K_2O	
	1.99	1.30	4.43	0.047	76.42	9.81	
	2.04	1.41	4.34	0.041	77.30	9.72	

The XRD of the wood based charcoal and coke is given in Figure 3-14. The XRD of the wood based charcoal ash after being ashed at 750°C for 22.5 hours contained CaO, MgO, $Ca(OH)_2$ and traces of Na_2CO_3 (natrite).

The XRD of the coke used in the CRR showed only the presence of carbon as the major constituent (Figure 3-16). Since there was little to now ash from the coke no XRD analyses were performed on the CRR ash.

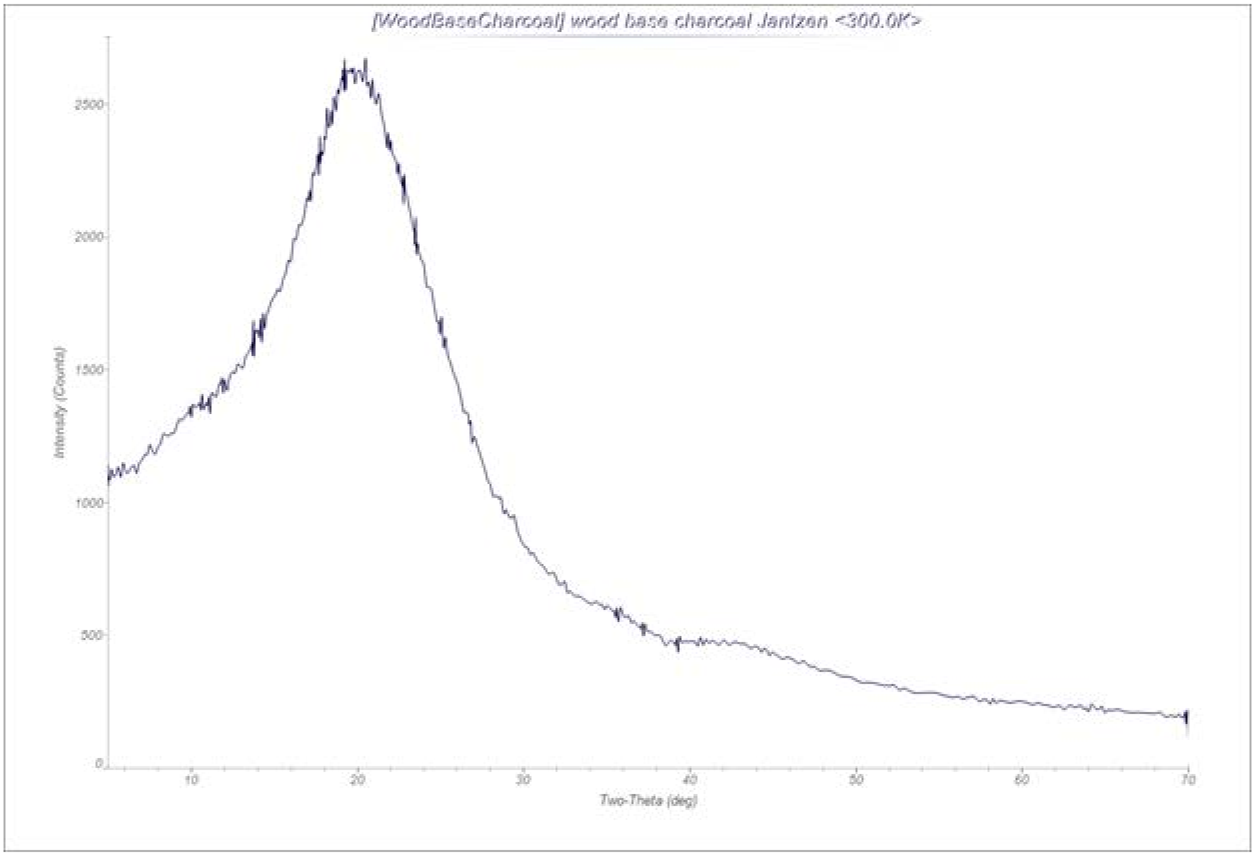


Figure 3-14. X-ray Diffraction Pattern of the Wood Based Charcoal is amorphous.

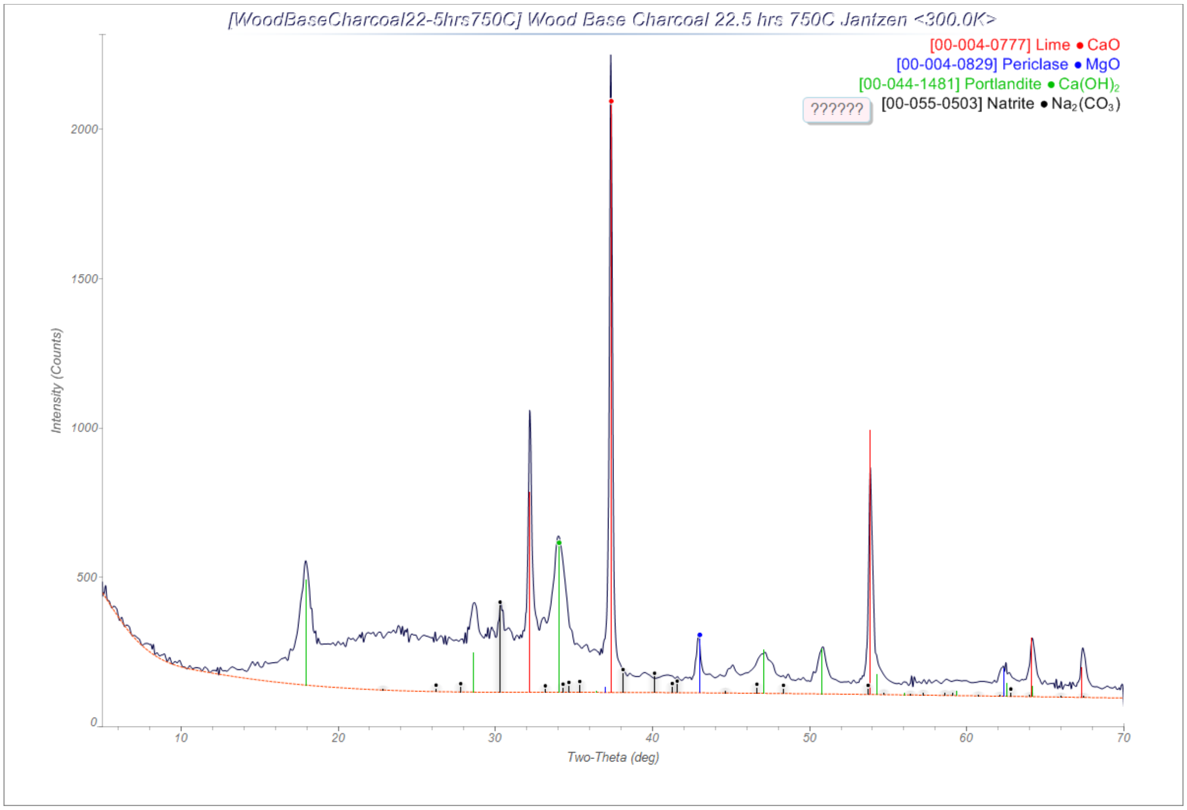


Figure 3-15. X-ray Diffraction analysis of the wood based charcoal ash after 22.5 hours at 750°C.

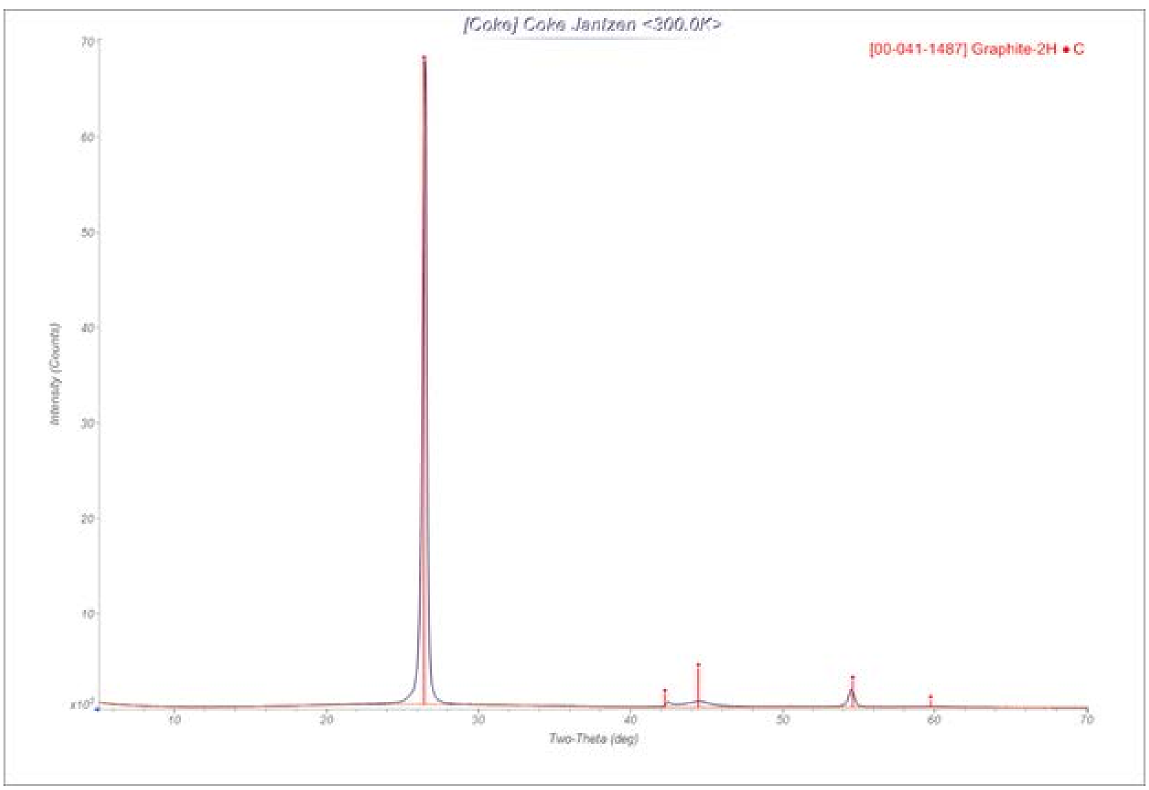
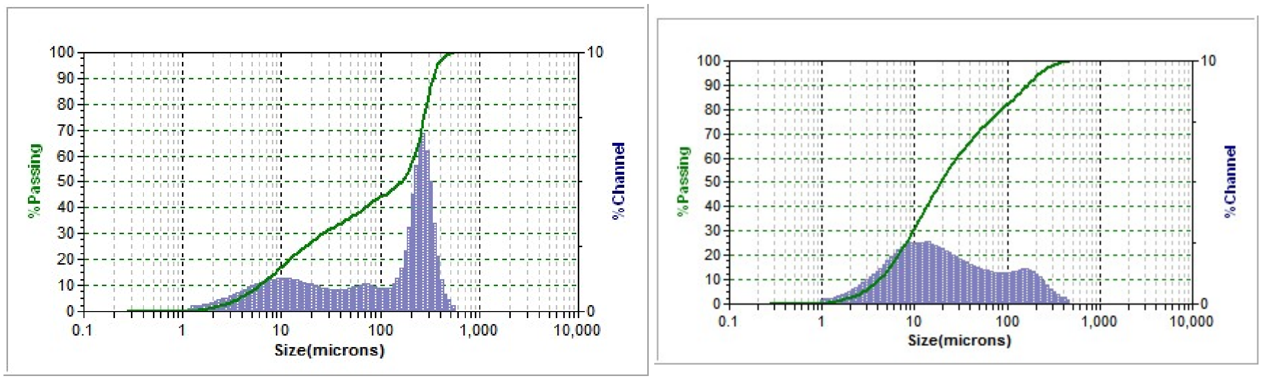


Figure 3-16. X-ray Diffraction analysis of the coke used in the CRR.

3.7 Off-Gas Filter (OGF) Solids Particle Size Analyses

The particle size distributions for the OGF samples represent an aggregate over months of IWTU startup with coal, water and steam, i.e. no SBW simulant. The samples were pulled on different shifts from different positions in the OGF and do not represent steady state operations. The particle size distribution pulled by the day shift are shown in Figure 3-17a and from the night shift in Figure 3-17b. The raw particle size distribution scans and statistics are given in Appendix C.

Since the particle size scans were taken from different parts of the OGF on the different shifts, it is clear that both samples have maximum particle sizes at ~ 10 microns and secondary maxima at 70-80 microns for the day shift sample (Figure 3-17a) and ~110 microns for the night shift sample (Figure 3-17b). The large maxima in the day shift (Figure 3-17a) at ~120 microns is likely bauxite carryover from the CRR since the CRR bed elutriates during operation and has to be replenished periodically. Moreover, the CRR coke contains only minimal ash (Table 13 and Table 14), i.e. 0.1-0.12 wt%.



(a) (b)

Figure 3-17. Particle Size Analyses of Day Shift and Night Shift OGF Solid Samples.

3.8 Off-Gas Filter (OGF) Solids Proximate Analyses

The Proximate analysis of the OGF solids Table 16 indicates that the processed material is higher in moisture than the coke fuel used in the CRR (see Proximate and Ultimate analyses in Table 13 and Table 14) which is the vessel prior to the OGF in the flowsheet (Figure 1-1). The CRR operates at ~900°C which should have eliminated any moisture from the coke carried over unless the coal is adsorbing moisture from the steam used in the process or the off-gas coolant water injected in the OGC. Excess moisture in the OGF solids may be making the coke carryover adhere to itself since it is such a fine particulate. There is an average of 76.98 wt% (100-23.02% ash) to 83.03% (100-16.97% ash) wt% coke carryover from the CRR into the OGF on an “ash-free basis” and 16.97-23.02% ash carryover from the CRR to the OGF. It is shown in Section 3.9 that the “ash carryover” is actually mostly CRR bauxite bed carryover and only minimal coke ash carryover since it is known from Table 13 and Table 14 that the ash content of the coke is only 0.1-0.12 wt%.

The volatiles and fixed carbon in the OGF solids (Table 16) are comparable to the raw coke analyses (Table 13 and Table 14) indicating that the coke carryover has not reacted very much. The ash content of the OGF solids (Table 16) is high compared to the coke (Table 13 and Table 14) but it is shown in Section 3.9 that the “ash” is elutriated bauxite from the CRR bed.

Table 16. ASTM D3172 Proximate Coal Analyses of OGF Solids Compared to Raw Coal

Short ID	Average Moisture % 107°C in vac	Volatiles % at 750°C in Ar	Ash % in air at 750°C	Fixed Carbon % by Difference	Total
Day Shift OGF	0.60	6.87	23.02	69.51	100.00
Night Shift OGF	1.99	7.43	16.97	73.61	100.00
Average OGF	1.30	7.15	20.00	71.56	100.00
Coke from CRR (NETL Ultimate Analyses)	0.03	0.39	0.12	99.49	100.00

*from Section 3.2

3.9 Off-Gas Filter (OGF) Solids Ash Analysis

The ash analysis of the OGF solids indicate high Al_2O_3 content from the bauxite bed carryover from the CRR (Table 17). The coke ash could not be analyzed due to the small amount of ash generated by the coke in the Proximate and Ultimate analyses. The SRNL wet chemical analysis of two different bauxite bed materials are given in Table 17 along with an analysis provided by INL microprobe analyses. When each analysis is normalized to 100 wt% the Al_2O_3 in the SRNL analyses are 73.09 wt% while the Al_2O_3 in the INL analysis is 73.58 wt%. Likewise, the SRNL TiO_2 is 3.44 wt% and 4.03 wt% respectively. Based on the averaged SRNL analyses of the amount of TiO_2 found in the OGF solids (an average of 2.4 wt% TiO_2 from Table 17) and the amount of TiO_2 in the CRR bauxite bed material (average 3.74 wt% TiO_2 from the averaged SRNL/INL analyses), approximately 64% of the ash solids are bauxite fines that are being carried over from the CRR to the OGF. Based on the amount of Al_2O_3 in the bauxite (average of the SRNL/INL analyses), all of the alumina in the OGF "ash" is from the 64% bauxite carryover (0.0.64*73.34 wt% Al_2O_3 ~46.94 wt% Al_2O_3) and about 6.23 wt% (0.64*9.73 wt% SiO_2 ~ 6.23 wt% SiO_2) of the SiO_2 is carryover from the CRR bauxite bed. The remaining SiO_2 in the OGF ash is likely ash from the coke. So the OGF solids are ~ 80 wt% coal (moisture, volatiles and fixed carbon from Table 16) and 20 wt% ash where the "ash content" is 12.8 wt% bauxite carryover (0.64*20 wt% ash) from the CRR and ~7.2 wt% ((26.08-6.23)*0.36 fraction of ash =7.15 wt%) coke ash.

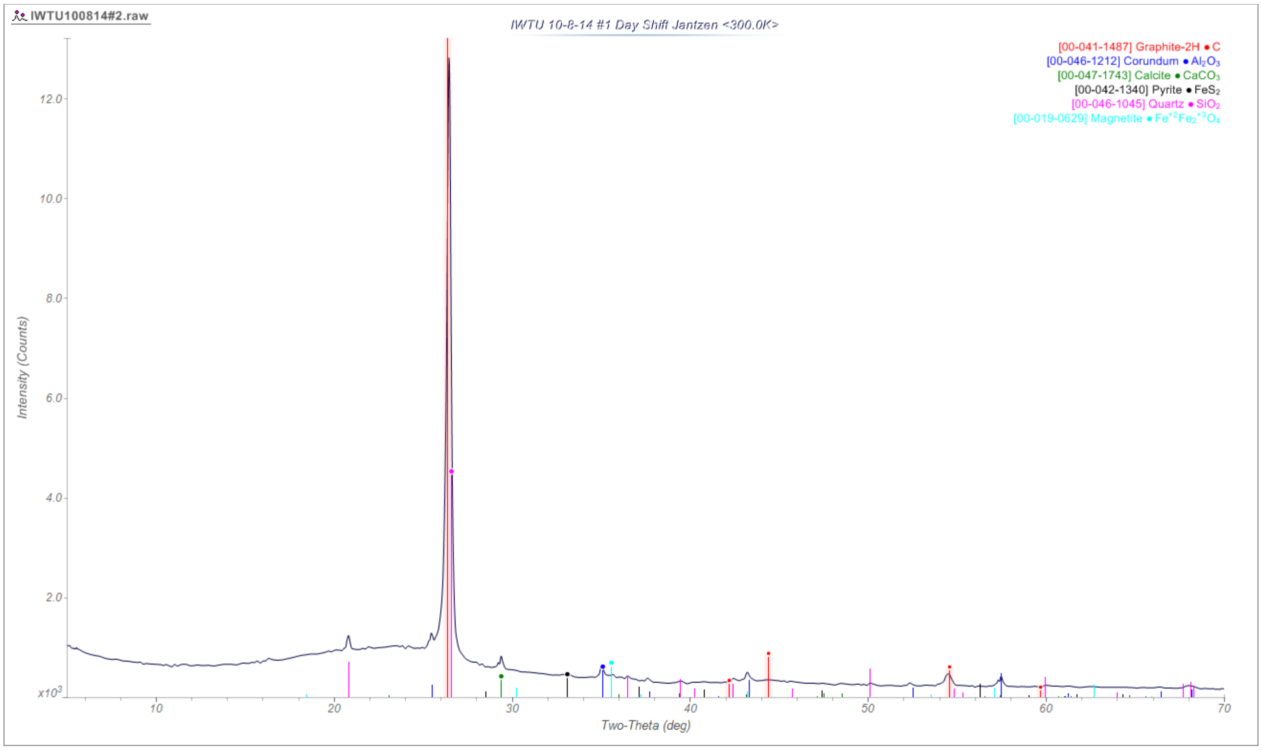
Table 17. Analysis of the Coal Ash from ASTM D3172 Proximate Coal Analyses of OGF Solids

Short ID	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	MnO_2	Na_2O	P_2O_5	SO_3	SiO_2	TiO_2	SUM
Night Shift OGF	50.69	5.08	11.38	1.33	0.75	0.54	0.26	0.38	2.93	20.67	2.74	96.75
	50.62	5.18	11.68	1.33	0.74	0.54	0.26	0.37	2.95	21.16	2.72	97.56
Day Shift OGF	34.97	8.88	10.52	2.06	1.16	0.56	0.34	0.65	4.65	31.24	2.07	97.10
	35.15	8.90	10.52	1.91	1.12	0.54	0.33	0.57	4.63	31.24	2.07	96.99
AVG OGF	42.86	7.01	11.03	1.66	0.94	0.55	0.30	0.49	3.79	26.08	2.40	97.11
Bauxite from CRR Bed*	76.18	0.76	6.38	1.20	NA	NA	0.36	0.32	NA	14.16	4.17	103.53
40/80 Bauxite from CRR Bed	69.90	0.27	13.35	0.07	0.06	0.35	0.25	0.17	0.00	7.28	3.47	95.17
40/80 Bauxite Black	69.40	0.27	13.35	0.07	0.06	0.37	0.50	0.18	0.00	7.48	3.41	95.09
SRNL/INL Average Normalized Bauxite Analyses	73.34	0.43	11.41	0.44	0.06	0.38	0.38	0.23	0.00	9.73	3.75	100.00

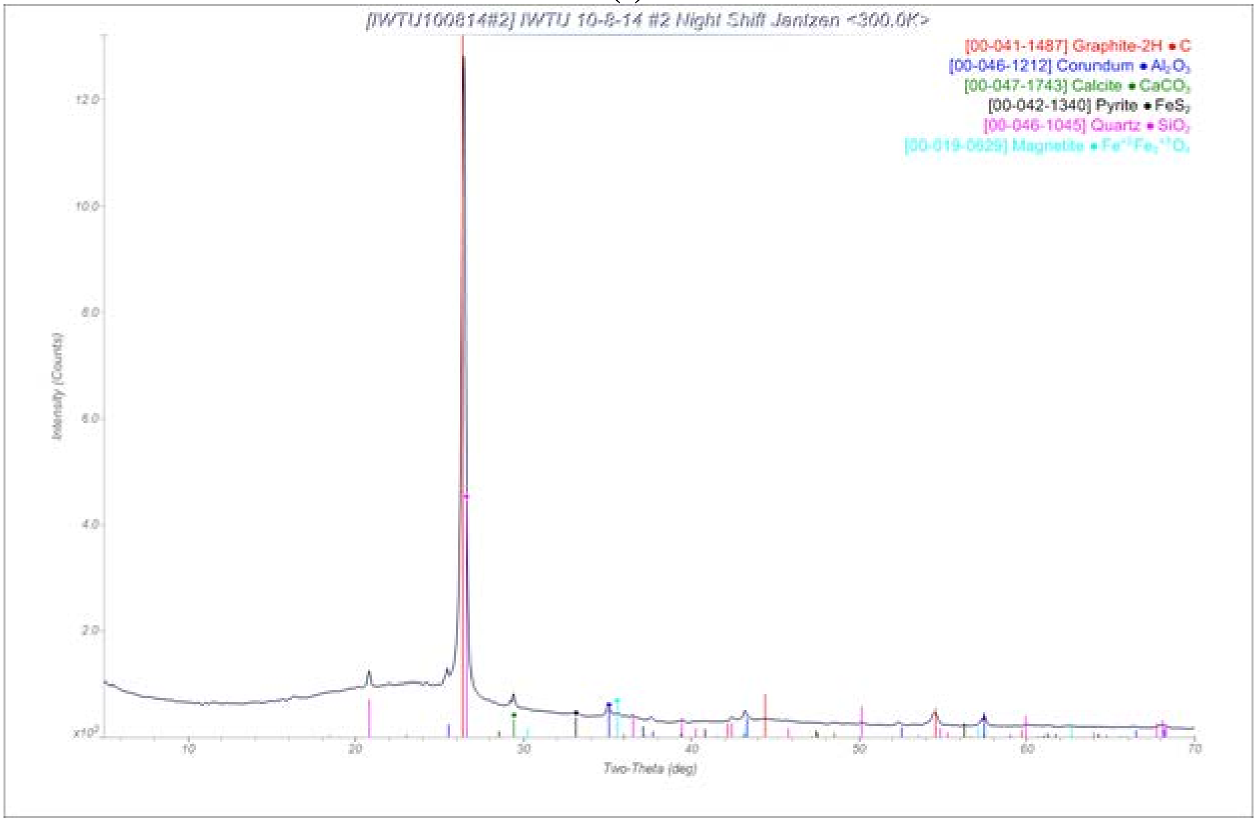
*courtesy of Soleberg of INL NA = Not Analyzed

3.10 Off-Gas Filter (OGF) Solids Phase Analyses by X-Ray Diffraction

X-ray Diffraction (XRD) analyses were performed on each of the OGF filter solid samples taken on October 8, 2014. The sample taken on day shift and the sample taken on night shift both showed C, Al_2O_3 (corundum), $CaCO_3$ (calcite), FeS_2 (pyrite), SiO_2 (quartz), and $Fe^{+2}Fe^{+3}O_4$ (magnetite) as shown in Figure 3-12. The primary component of the OGF solids is SiO_2 and some amorphous material. The amorphous material is likely coal ash (fly ash). The corundum, quartz and magnetite are likely breakdown of the bauxite bed as the bauxite bed contains Al_2O_3, SiO_2 and Fe_2O_3 in significant quantities while the graphite component comes from elutriation of unreacted coke, i.e. compare Figure 3-16 to Figure 3-18.



(a)



(b)

Figure 3-18. X-ray Diffraction Analyses of OGF solids sampled on day shift and night shift of October 8, 2014.

4.0 Conclusions

In general the IWTU Bestac coal met most of the vendor coal specifications except for % volatiles in the coal and % SiO_2 in the coal ash. The coal ash is simultaneously high in CaO compared to the "preferred value" in the coal specification but the CaO ash content is within the coal specification. Any of these three findings could cause operational difficulties in the form of coal reactivity and unwanted process chemistry. While there was no minimum percentage of coal <6mm specified in the coal specification, 9.78-11.07% of the coal is <6mm versus the 2.42% claimed by the IWTU coal vendor.

The OGF solids are a combination of unreacted coal/coke, coal/coke ash from the CRR, and carryover of the bauxite bed from the CRR. The OGF solids are ~ 80 wt% unreacted coke (this percentage includes moisture, volatiles and fixed carbon), 7.2 wt% coke ash, and 12.8 wt% bauxite from the CRR.

5.0 Recommendations, Path Forward or Future Work

1. Coal analyses should be performed on incoming batches of IWTU coal to determine compliance with the coal vendor specification as excess SiO_2 in the coal ash could cause operational issues.
2. Process gas velocities should be minimized to prevent solids carryover to the OGF from the DMR and the CRR.
3. Overfeeding of coal to the DMR and/or CRR should be monitored and minimized to that needed to denitrate the SBW waste when simulated and/or radioactive waste is processed.
4. The source of moisture in the OGF solids should be determined. This would include an evaluation of how hygroscopic the OGF solids are, and the potential for water absorption prior to sampling, versus the potential for water absorption from the water injection cooling process in the OGC.
5. Investigate the impact of reducing the amount of unburned coke carryover regarding bridging potential in the OGF. Would lessening the amount of coke elutriation reduce the potential for bridging?

6.0 Appendix A. IWTU Vendor Analysis of 22 Tons of Bestac Coal

Testing Report - Coal Data

Bestac International, Inc.
P.O. Box 585, Wayne, NJ 07470 USA
Tel: 973 633 6878 Fax: 973 633 0828

Jan 6, 2011

IWTU VENDOR DATA REVIEW STATUS
D - Received for Information Only.
CWI Vendor Data Coordinator: Becky Metcalf
Date: 01/06/11

Subcontract No. 0000739897
CH2M-WG IDAHO, LLC

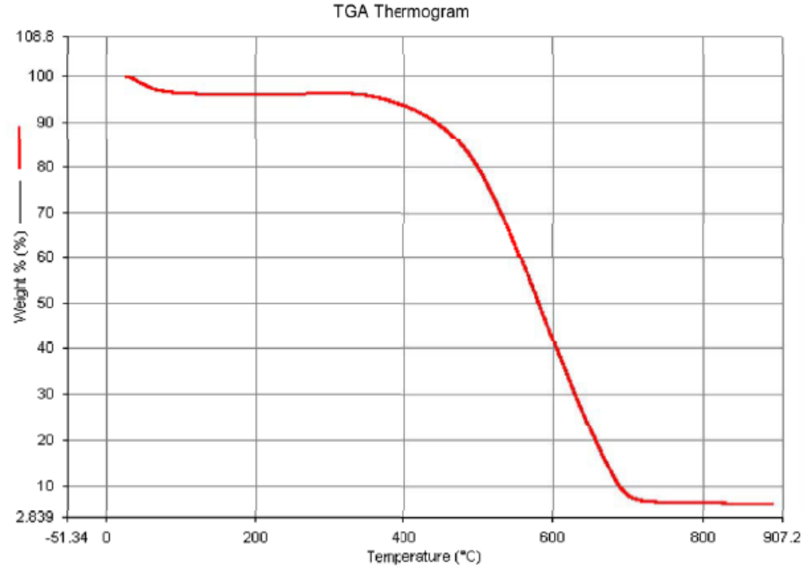
Coal Data Sheet before 1st shipment

Description of Goods	Calcined Coal 6-12mm, MT-612RC
Quantity and Package	1100 lbs net bag, total 40 bags 22 tons

This is a typical data based on the testing results for production quality control purpose. The numbers obtained are for reference only and should not be deemed a warranty for the specifications listed on the purchasing order.

Proximate analysis, Volatiles (wt%):	14.5%
Proximate analysis, Ash Content (wt%):	6.4%
Proximate analysis, Moisture (wt%):	5.8%
Ultimate analysis, Total sulfur (wt):	0.44%
Heating Value (BTU/lb):	12060
Particle size, 6-12mm:	<6mm: 2.24%; >12mm: 1.32%
Ash Analysis	Ash CaO [wt%]: 1.93% Ash K2O + Na2O (combined) [wt%]: 1.68% Ash SiO2 [wt%]: 63.89%

TGA Thermogram



BESTAC INTL INC.
P.O. BOX 585
WAYNE, NJ 07470

7.0 Appendix B. Historic Bestac Coal Analyses from TTT (2004, 2006,2011).

Oxide, wt %	2004 Bestac Analyses						2006 Bestac Analyses					2011 Bestac Analyses			Avg All
	4762	4763	4764	4765	4766	4767	5260	5282	5363	5383	5504	8142	8201	8245	
	3 bag composites						Single bag samples					Single bag samples			
SiO_2	60.45	63.2	61.08	61.48	60.91	61.66	61.01	60.52	65.01	61.33	61.59	56.2	60.85	58.82	61.01
Al_2O_3	20.42	21.06	20.98	23.9	20.93	22.55	25.13	25.2	24.8	24.89	25.11	25.17	24.97	25.83	23.64
Fe_2O_3	13.69	10.29	11.19	9.24	11.66	11.35	7.18	7.11	7.30	6.98	6.80	7.64	5.8	6.27	8.75
CaO	1.26	1.37	1.4	1.49	1.9	0.99	2.26	1.99	2.26	2.28	2.00	2.64	3.56	2.38	1.98
MgO	0.42	0.3	0.34	0.34	0.37	0.32	0.12	0.17	0.15	0.14	0.13	0.23	0.21	0.25	0.25
Na_2O	0.27	0.24	0.19	0.19	0.22	0.22	4.91	4.82	4.95	4.82	4.87	0.75	0.53	0.61	1.97
K_2O	0.98	1.04	1.07	0.94	1.04	0.96	0.27	0.23	0.16	0.18	0.15	0.34	0.27	0.19	0.56
TiO_2	0.6	0.73	0.63	0.69	0.64	0.69	0.57	0.57	0.61	0.58	0.63	0.58	0.64	0.57	0.62
P_2O_5	0.48	0.37	0.38	0.46	0.52	0.52	1.44	1.28	1.55	1.59	1.42	1.43	2.16	1.55	1.08
SO_3	0.47	0.68	0.33	0.67	1.16	0.29	0.25	0.39	0.39	0.40	0.40	0.37	0.49	0.34	0.47
Total	99.04	99.28	97.92	99.4	99.35	99.55	103.14	102.28	107.18	103.19	103.1	95.35	99.48	96.81	100.36

	St.Dev.	%RSD
SiO_2	1.97	3.23
Al_2O_3	1.99	8.41
Fe_2O_3	2.46	28.08
CaO	0.67	33.64
MgO	0.10	39.91
Na_2O	2.25	114.28
K_2O	0.41	72.58
TiO_2	0.05	8.19
P_2O_5	0.60	55.22
SO_3	0.23	49.36

8.0 Appendix C. Particle Size Analysis of OGF Samples

- Particle Size Analysis -

10.3.0	IWTU 10-8-14 #1 day shift Jantzen	
	10/14/2014 13:10	S3000/S3500 S3754

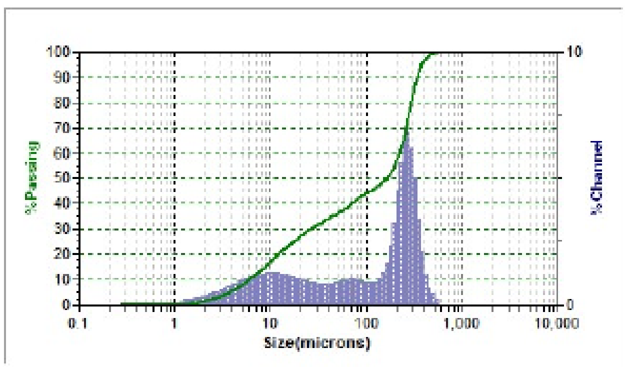
Distribution:	Volume	Run Time:	30 Sec	Fluid:	WATER		
Progression:	Geom 8 Root	Run Num:	Avg of 3	Fluid Ref. Index:	1.333	Loading Factor:	0.041
Upper Edge:	1408	Particle:	CRAWFORD ABS	Above Residual:	0	Transmission:	0.94
Lower Edge:	0.243	Transparency:	Absorbing	Below Residual:	0	RMS Residual:	0.26E-03
Residuals:	Disabled	Part. Ref. Index:	N/A			Flow:	40 %
Num. Channels:	100	Part. Shape:	Irregular	Cell ID:	0595	Usonic Power:	N/A
Analysis Mode:	S3000					Usonic Time:	N/A
Filter:	Enabled	DB Record:	4074	Recalc Status:		Serial Num:	S3754
Analysis Gain:	Default	Database:	C:\Program Files (x86)\Microtrac FLEX 10.3.0\Databases\psd-2014.MDB				

Data Item	Value
MV(um):	161.4
MN(um):	2.281
MA(um):	17.98
CS:	3.34E-01
SD:	150.2

Size(um)	%Tile
0.800	0.00

%Tile	Size(um)
10.00	5.94
16.00	9.22
25.00	17.30
40.00	69.83
50.00	164.7
60.00	222.4
70.00	258.6
75.00	275.5
90.00	340.0
95.00	381.0

Dia	Vol%	Width
235.8	73.0	288.30
7.73	27.0	11.20



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Size(um)	%Chan	% Pass	Size(um)	%Chan	% Pass	Size(um)	%Chan	% Pass	Size(um)	%Chan	% Pass
1408	0.00	100.00	74.00	1.04	40.70	3.89	0.73	5.47			
1291	0.00	100.00	67.86	1.02	39.66	3.57	0.66	4.74			
1184	0.00	100.00	62.23	0.97	38.64	3.27	0.60	4.08			
1086	0.00	100.00	57.06	0.93	37.67	2.999	0.53	3.48			
995.6	0.00	100.00	52.33	0.88	36.74	2.750	0.48	2.95			
913.0	0.00	100.00	47.98	0.84	35.86	2.522	0.43	2.47			
837.2	0.00	100.00	44.00	0.82	35.02	2.312	0.38	2.04			
767.7	0.00	100.00	40.35	0.82	34.20	2.121	0.34	1.66			
704.0	0.00	100.00	37.00	0.83	33.38	1.945	0.30	1.32			
645.6	0.00	100.00	33.93	0.84	32.55	1.783	0.26	1.02			
592.0	0.22	100.00	31.11	0.87	31.71	1.635	0.23	0.76			
542.9	0.44	99.78	28.53	0.91	30.84	1.499	0.20	0.53			
497.8	0.69	99.34	26.16	0.94	29.93	1.375	0.20	0.33			
456.5	1.23	98.65	23.99	0.99	28.99	1.261	0.13	0.13			
418.6	2.16	97.42	22.00	1.04	28.00	1.156	0.00	0.00			
383.9	3.38	95.27	20.17	1.08	26.96	1.060	0.00	0.00			
352.0	5.03	91.89	18.50	1.13	25.88	0.972	0.00	0.00			
322.8	6.20	86.86	16.96	1.18	24.75	0.892	0.00	0.00			
296.0	6.86	80.66	15.56	1.22	23.57	0.818	0.00	0.00			
271.4	6.68	73.80	14.27	1.24	22.35	0.750	0.00	0.00			
248.9	5.67	67.12	13.08	1.26	21.11	0.688	0.00	0.00			
228.2	4.53	61.45	12.00	1.27	19.85	0.630	0.00	0.00			
209.3	3.26	56.92	11.00	1.27	18.58	0.578	0.00	0.00			
191.9	2.32	53.66	10.09	1.26	17.31	0.530	0.00	0.00			
176.0	1.70	51.34	9.25	1.25	16.05	0.486	0.00	0.00			
161.4	1.29	49.64	8.48	1.22	14.80	0.446	0.00	0.00			
148.0	1.06	48.35	7.78	1.19	13.58	0.409	0.00	0.00			
135.7	0.92	47.29	7.13	1.16	12.39	0.375	0.00	0.00			
124.5	0.88	46.37	6.54	1.11	11.23	0.344	0.00	0.00			
114.1	0.87	45.49	6.00	1.06	10.12	0.315	0.00	0.00			
104.7	0.91	44.62	5.50	1.00	9.06	0.2890	0.00	0.00			
95.96	0.96	43.71	5.04	0.93	8.06	0.2650	0.00	0.00			
88.00	1.01	42.75	4.62	0.86	7.13						
80.70	1.04	41.74	4.24	0.80	6.27						

Warnings: NONE

- Particle Size Analysis -

Microtrac Inc. 10.3.0	IWTU 10-8-14 #2 night shift Jantzen	
	10/14/2014 13:17	S3000/S3500 S3754

Distribution:	Volume	Run Time:	30 Sec	Fluid:	WATER		
Progression:	Geom 8 Root	Run Num:	Avg of 3	Fluid Ref. Index:	1.333	Loading Factor:	0.023
Upper Edge:	1408	Particle:	CRAWFORD ABS	Above Residual:	0	Transmission:	0.95
Lower Edge:	0.243	Transparency:	Absorbing	Below Residual:	0	RMS Residual:	4.23E-03
Residuals:	Disabled	Part. Ref. Index:	N/A			Flow:	40 %
Num. Channels:	100	Part. Shape:	Irregular	Cell ID:	8595	Usonic Power:	N/A
Analysis Mode:	S3000					Usonic Time:	N/A
Filter:	Enabled	DB Record:	4070	Recalc Status:		Serial Num:	S3754
Analysis Gain:	Default	Database:	C:\Program Files (x86)\Microtrac FLEX 10.3.0\Databases\psd-2014.MDB				

Data Item	Value
MV(um):	54.09
MN(um):	1.971
MA(um):	10.07
CS:	5.96E-01
SD:	54.08

Size(um)	%Tile
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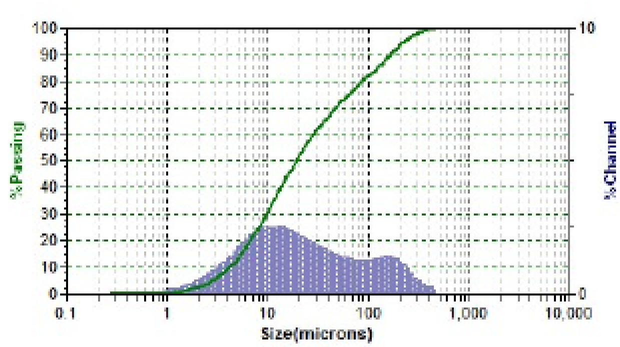
%Tile	Size(um)
10.00	4.12
16.00	5.66
25.00	8.08
40.00	13.53
50.00	19.19
60.00	28.61
70.00	48.80
75.00	62.82
90.00	166.0
95.00	228.5

Dia	Vol%	Width
19.19	100.0	108.20

Size(um)	%Chan	% Pass	Size(um)	%Chan	% Pass	Size(um)	%Chan	% Pass	Size(um)	%Chan	% Pass
1408	0.00	100.00	74.00	1.34	77.57	3.89	1.22	9.13			
1291	0.00	100.00	67.86	1.38	76.23	3.57	1.10	7.91			
1184	0.00	100.00	62.23	1.42	74.85	3.27	0.98	6.81			
1086	0.00	100.00	57.06	1.46	73.43	2.999	0.88	5.83			
995.8	0.00	100.00	52.33	1.52	71.97	2.750	0.77	4.95			
913.0	0.00	100.00	47.98	1.57	70.45	2.522	0.68	4.18			
837.2	0.00	100.00	44.00	1.64	68.88	2.312	0.59	3.50			
767.7	0.00	100.00	40.35	1.71	67.24	2.121	0.51	2.91			
704.0	0.00	100.00	37.00	1.79	65.53	1.945	0.44	2.40			
645.6	0.00	100.00	33.93	1.86	63.74	1.783	0.38	1.96			
592.0	0.00	100.00	31.11	1.94	61.88	1.635	0.33	1.58			
542.9	0.00	100.00	28.53	2.03	59.94	1.499	0.29	1.25			
497.8	0.16	100.00	26.16	2.10	57.91	1.375	0.26	0.96			
456.5	0.26	99.84	23.99	2.19	55.81	1.261	0.23	0.71			
418.6	0.30	99.58	22.00	2.27	53.62	1.156	0.22	0.48			
383.9	0.37	99.28	20.17	2.34	51.35	1.060	0.18	0.26			
352.0	0.49	98.91	18.50	2.41	49.01	0.972	0.07	0.10			
322.8	0.62	98.42	16.96	2.48	46.60	0.892	0.03	0.03			
296.0	0.77	97.80	15.56	2.55	44.12	0.818	0.00	0.00			
271.4	0.94	97.03	14.27	2.56	41.57	0.750	0.00	0.00			
248.9	1.11	96.09	13.08	2.53	39.01	0.688	0.00	0.00			
228.2	1.24	94.98	12.00	2.52	36.48	0.630	0.00	0.00			
209.3	1.35	93.74	11.00	2.54	33.96	0.578	0.00	0.00			
191.9	1.42	92.39	10.09	2.54	31.42	0.530	0.00	0.00			
176.0	1.43	90.97	9.25	2.50	28.88	0.486	0.00	0.00			
161.4	1.43	89.54	8.48	2.44	26.38	0.446	0.00	0.00			
148.0	1.39	88.11	7.78	2.34	23.94	0.409	0.00	0.00			
135.7	1.36	86.72	7.13	2.22	21.60	0.375	0.00	0.00			
124.5	1.32	85.36	6.54	2.08	19.38	0.344	0.00	0.00			
114.1	1.30	84.04	6.00	1.93	17.30	0.315	0.00	0.00			
104.7	1.29	82.74	5.50	1.78	15.37	0.2890	0.00	0.00			
95.96	1.28	81.45	5.04	1.63	13.59	0.2650	0.00	0.00			
88.00	1.29	80.17	4.62	1.48	11.96						
80.70	1.31	78.88	4.24	1.35	10.48						

Warnings: NONE



DiH2O

9.0 References

1 Soelberg, N.R., D.W. Marshall, S.O. Bates, and D.D. Taylor, **"Phase 2 THOR Steam Reforming Tests for Sodium Bearing Waste Treatment"**, INEEL/EXT-04-01493, Rev. 1, Idaho National Engineering and Environmental Laboratory, Idaho Falls, Idaho (2004).

2 Marshall, D.W., N.R. Soelberg, and K.M. Shaber, **"THORsm Bench-Scale Steam Reforming Demonstration"**, INEEL/EXT.03-00437, Idaho National Laboratory, Idaho Falls, Idaho (2003).

3 Olson, A.L., N.R. Soelberg, D.W. Marshall, and G.L. Anderson, **"Fluidized Bed Steam Reforming of INEEL SBW Using THORsm Mineralizing Technology"**, INEEL/EXT-04-02564 Idaho National Laboratory, Idaho Falls, Idaho (2004).

4 Crawford, C.L. and C.M. Jantzen, **"Durability Testing of Fluidized Bed Steam Reformer (FBSR) Waste Forms for Sodium Bearing Waste (SBW) at INL"**, U.S. DOE Report WSRC-STI-2007-00319, Savannah River National Laboratory, Aiken, South Carolina (2007).

5 THOR® Treatment Technologies, **"Pilot Plant Report for Treating Sodium-Bearing Waste Surrogates Carbonate Flowsheet,"** Idaho Cleanup Project, Integrated Waste Treatment Unit, Project Number 28276, Document Number RT-ESTD-002, Rev. 1 (August 2006).

6 THOR® Treatment Technologies, **"IWTU Coal Specification,"** Idaho Cleanup Project, Integrated Waste Treatment Unit, Project Number 28266, Document Number RT-00-005, Rev. 0 (March 2010).

7 Soelberg, N.R., D.W. Marshall, S.O. Bates, and D.D. Taylor, **"Phase 2 THOR Steam Reforming Tests for Sodium Bearing Waste Treatment"**, INEEL/EXT-04-01493, Rev. 1, Idaho National Engineering and Environmental Laboratory, Idaho Falls, Idaho (2004).

8 Marshall, D.W., N.R. Soelberg, and K.M. Shaber, **"THORsm Bench-Scale Steam Reforming Demonstration"**, INEEL/EXT.03-00437, Idaho National Laboratory, Idaho Falls, Idaho (2003).

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