POWER SYSTEMS DEVELOPMENT FACILITY SUMMARY REPORT

GASIFICATION TEST CAMPAIGN TC23

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1.0 EXECUTIVE SUMMARY

In support of technology development to utilize coal for efficient, affordable, and environmentally clean power generation, the Power Systems Development Facility (PSDF) located in Wilsonville, Alabama, routinely demonstrates gasification technologies using various types of coals. The PSDF is an engineering scale demonstration of key features of advanced coal-fired power systems, including a KBR Transport Gasifier, a hot gas particulate control device, advanced syngas cleanup systems, and pressurized solids handling systems.

This report summarizes the results of test campaign TC23 of the PSDF gasification process. Test campaign TC23 was performed from August 7 to August 27, 2007. The test featured operation with high sodium lignite from the Freedom Mine in North Dakota which occurred after start up and initial operation with Powder River Basin (PRB) subbituminous coal.

The major objective for this test was to evaluate the process operational stability and performance using the high sodium lignite. This coal had been tested previously with the PSDF gasification process (TC13 in October of 2003, TC16 in August of 2004, and TC21 in December of 2006 and January 2007), and presented operational challenges in maintaining adequate solids circulation in the gasifier due to reactions of the sodium in the lignite with other lignite ash components to form low melting point compounds. To prevent the agglomerations experienced previously, a kaolin additive was fed with the lignite during this test campaign. Use of the kaolin proved successful in eliminating agglomeration within the gasifier; however, ash deposition in the primary gas cooler located downstream of the gasifier and subsequent loss of heat transfer became an operational issue as the test campaign progressed. The gasification process was operated for a total of 481 hours in TC23, with over 300 hours of operation with the high sodium lignite.

The PSDF gasification process, shown in Figure 1, consists of dry, pressurized coal feeders which utilize lock hopper systems and pneumatically convey coal into the KBR Transport Gasifier. The gasifier is a pressurized, circulating fluidized bed reactor consisting of a mixing zone, riser, a two stage solids separation unit, seal leg, standpipe, and J-leg. The particulate laden syngas exiting the gasifier is cooled in a primary syngas cooler and then filtered by a downstream high temperature, high pressure filter vessel, the particulate control device (PCD). The PCD removes virtually all the particulate from the gas stream. Gasification ash is removed from the gasifier and PCD using continuous ash depressurization systems which were developed at the PSDF by Southern Company.

A portion of the filtered syngas can be utilized in a slipstream syngas cleanup system to test various pollutant control technologies or it can be combusted in a piloted syngas burner coupled with a gas turbine. The syngas not utilized in the cleanup system or burner is further cooled in the secondary gas cooler. Some of this syngas is compressed and sent to the gasifier for aeration to aid in circulation. The remaining syngas passes through a pressure control valve and flows to an atmospheric syngas combustor, a heat recovery boiler, a baghouse, and then out a stack. Further information about the PSDF gasification process can be found on the PSDF web site, http://psdf.southernco.com.

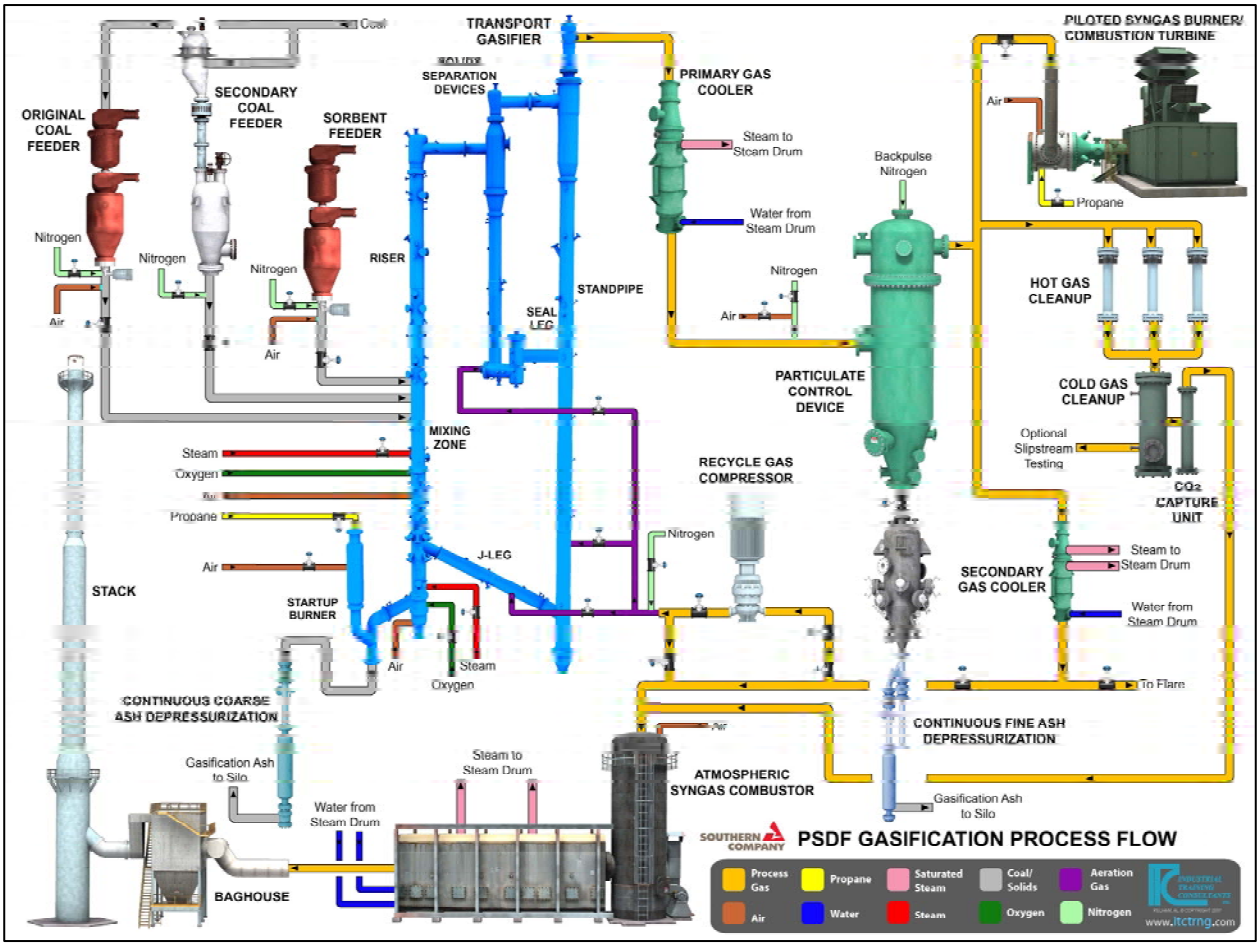

Figure 1. PSDF Gasification Process Flow Diagram.

TC23 began with gasifier operations on PRB coal in air blown mode from August 7 to August 14, 2007, to perform air compliance tests, conduct tracer gas experiments, and generate gasification ash to replace the initial startup bed material, sand, for the solids circulation medium in the Transport Gasifier. The gasifier operated on high sodium lignite in air blown mode from August 15 to August 27, 2007.

There were twenty eight steady state periods in TC23, including twelve periods on PRB and sixteen periods on lignite. Only two steady state periods did not include recycle gas operations. The original coal feeder was used for all of the steady periods. The steady state periods are defined based on maintaining gasifier operating conditions within defined ranges. The steady state operating periods and major operating parameters are shown in Appendix 1.

Gasifier operation was stable with the high sodium lignite, achieving carbon conversions up to 97.6 percent and projected syngas lower heating values of 125 to 140 Btu/SCF at the turbine inlet. Please note that the carbon conversions are based on the loss on ignition (LOI) of the fine ash collected by the PCD. The final carbon conversions will be based on the fine ash carbon

content. The carbon conversions will be revised when the fine solids analyses are received, typically though, the two methods produce similar results. The most significant concern arose from deposition in the primary gas cooler. Coal feeder system trips due to material plugging in the balance line, dispense vessel vent line and discharge line and interruptions in nitrogen flow arising from vendor supply problems caused some temporary operational setbacks.

The following report presents the operational data and preliminary results of gasification technology development at the PSDF during TC23, compiled in the sections listed below. Inspection results are also included.

- Coal feed systems — Discusses operations of the coal feeder systems and presents coal moisture values and particle sizes and their effect on coal feed system performance.
- Kaolin sorbent additive — Discusses operations of the sorbent feed system and details the use of kaolin sorbent material to improve gasifier operations with high sodium lignite.
- Transport Gasifier — Includes the major gasifier operating parameters and the gasifier performance as indicated by preliminary solids and gas analyses. Also includes the results of initial parametric testing and tracer gas testing. Operations of the primary gas cooler are included in this section.
- Sensor development — Discusses the ongoing testing of various instrumentation improvements.
- Particulate control device — Describes the hot gas filter particulate characteristics, filtration performance, and failsafe and filter element testing.
- Advanced syngas cleanup — Details various testing to support emissions control studies. New tests conducted in TC23 included water-gas shift reaction with a catalytic filter element, water-gas shift reaction in a fixed bed, H_2S direct oxidation, and CO_2 capture.
- Recycle syngas system — Describes operations of the recycle syngas system used to provide gasifier aeration and highlights a recent modification to the control logic.
- Air compliance test — Discusses air compliance test results.
- Slurry feeder system — Describes the slurry feeder system and summarizes its commissioning.

2.0 TECHNOLOGY DEVELOPMENT

2.1 Coal Feed Systems

The original coal feeder, a lock hopper system, ran during the entire test campaign (180 hours on PRB and 301 hours on high sodium lignite) at rates from 2,085 lb/hr up to 4,065 lb/hr and particle sizes varying from 243 to 864 microns (mass median diameter). The average, standard deviation, minimum, and maximum of the as-fed properties of the PRB subbituminous and lignite coals used during TC23 are given in Table 1. However, analyses for the last five days of lignite samples have not been received. Note, data received shows a step change in lignite Na_2O concentration around Hour 320. Prior to Hour 320 the lignite ash averaged 4.7 percent Na_2O and after Hour 320, the lignite ash averaged 9.7 percent Na_2O.

Nitrogen was used as coal transport gas for the majority of the test campaign; however, transport air was tested for about 20 hours. The feeder ran well throughout the test run; however, material

plugging the balance line, dispense vessel vent line, and discharge line caused some feed interruptions. The transport gas velocity and dispense vessel fluidization were varied during the run to optimize operations.

Table 1. PRB Subbituminous and North Dakota Freedom Mine High Sodium Lignite As-Fed Properties.

	PRB				High Sodium Lignite (Preliminary)[1]			
	Average Value	Minimum Value	Maximum Value	Standard Deviation	Average Value	Minimum Value	Maximum Value	Standard Deviation
Moisture, wt%	13.5	12.3	15.0	1.0	20.7	17.7	24.3	2.3
Carbon, wt%	58.9	57.8	59.9	0.8	51.3	49.0	54.0	1.6
Hydrogen, wt%	3.97	3.43	4.14	0.19	3.13	2.92	3.35	0.16
Nitrogen, wt%	1.04	1.02	1.07	0.02	0.94	0.88	0.98	0.03
Oxygen, wt%	16.1	15.4	16.8	0.6	15.3	14.6	16.5	0.5
Sulfur, wt%	0.29	0.22	0.36	0.04	0.74	0.66	0.95	0.07
Ash, wt%	6.2	5.5	7.5	0.6	7.9	7.3	8.5	0.3
Volatiles, wt%	35.6	34.9	36.3	0.5	32.7	31.3	33.8	1.0
Fixed Carbon, wt%	44.7	43.7	45.9	0.7	38.8	37.1	40.5	1.3
Higher Heating Value, Btu/lb	10,088	9,865	10,257	122	8,597	8,160	8,991	267
Lower Heating Value, Btu/lb	9,584	9,395	9,748	118	8,096	7,635	8,501	277
CaO, wt % in Ash	23.8	21.3	28.6	2.4	26.2	22.7	29.2	2.4
SiO_2, wt % in Ash	35.4	33.4	39.0	1.7	19.5	18.2	21.1	0.8
Al_2O_3, wt % in Ash	13.4	0.0	17.6	6.6	11.8	11.5	12.3	0.2
MgO, wt % in Ash	5.0	4.5	6.2	0.5	3.6	2.5	5.6	1.4
Na_2O, wt % in Ash	1.6	1.4	2.1	0.2	6.5	4.5	10.0	2.5

Notes:

1. Lignite samples from 8/22/07 14:00 to 8/27/07 14:00 have not yet been analyzed.

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Initially, material plugging in the discharge line caused the coal feeder to trip every four hours. The coal feed transport velocity was increased from 35 to 40 ft/s, and the trips due to the discharge line plugging were reduced to about once per day. The velocity was further increased to 45 ft/s and the trip frequency was again reduced.

Due to operational problems with the sorbent feed system, the coal feed system was utilized to feed kaolin simultaneously with the lignite during the initial operation of high sodium lignite (from Hour 180 to Hour 220). Additional problems were encountered with the discharge line plugging when feeding the lignite and kaolin simultaneously due to the size of the kaolin. Some of the large kaolin particles caused the discharge line to become plugged. The transport gas velocity was increased to 70 ft/s to reduce the frequency of trips. Additional testing is needed to conclusively evaluate the effect of changes in the dispense vessel fluidization on feeder operations.

The secondary coal feeder was operated briefly, but operation was stopped due to continued problems with the discharge line plugging. The discharge line will be modified in a future outage to improve the flow path.

Figure 2 gives the TC23 as-fed coal moisture, as weight percent, with the demarcation line showing lignite feed beginning at Hour 180. Note that the data shown reflects the lignite-kaolin mixture from Hour 180 to Hour 224. All of the PRB coal was processed in one mill. The moisture content in the processed as-fed PRB was typically below 15 percent, however, one sample was higher than 20 percent. The lignite was processed in two separate mills which resulted in a larger variation in moisture content. One mill was operating at a lower mass flow through the system resulting in a lower drying capacity and thus a higher moisture content. The lignite moisture content ranged from 16 and 24 percent. Generally, the lignite as-fed moisture content was higher than the PRB since the lignite as-received moisture content was higher.

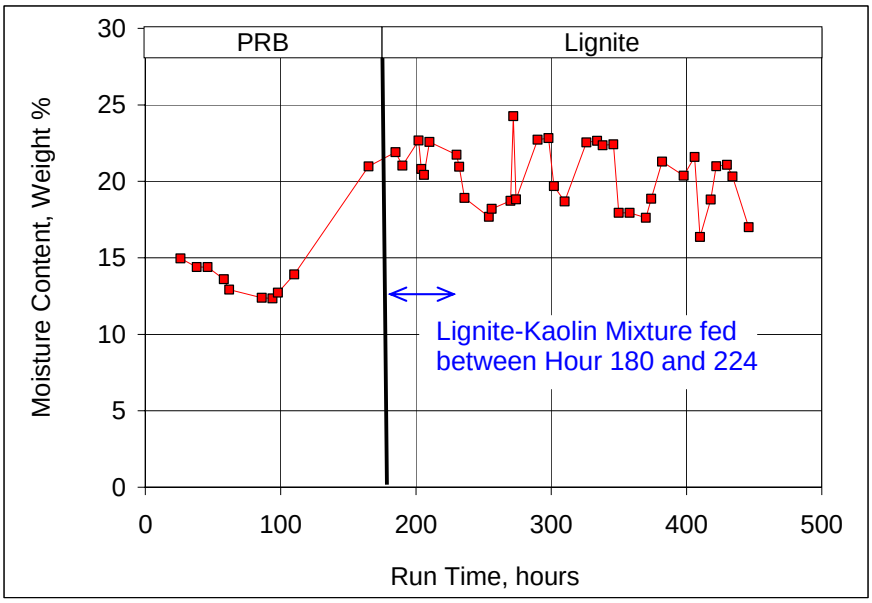


Figure 2. As-Fed Coal Moisture Content.

Figure 3 gives the sodium content in ash on a sodium oxide (Na_2O) basis, as weight percent. The sodium content for the PRB coal ash was about two percent. The sodium content remained low for about 50 hours after the transition from PRB to lignite but increased to about 4.7 percent at Hour 230. At Hour 310 the sodium content increased to about 10 percent and did not change to Hour 446. Data after Hour 446 has not been received.

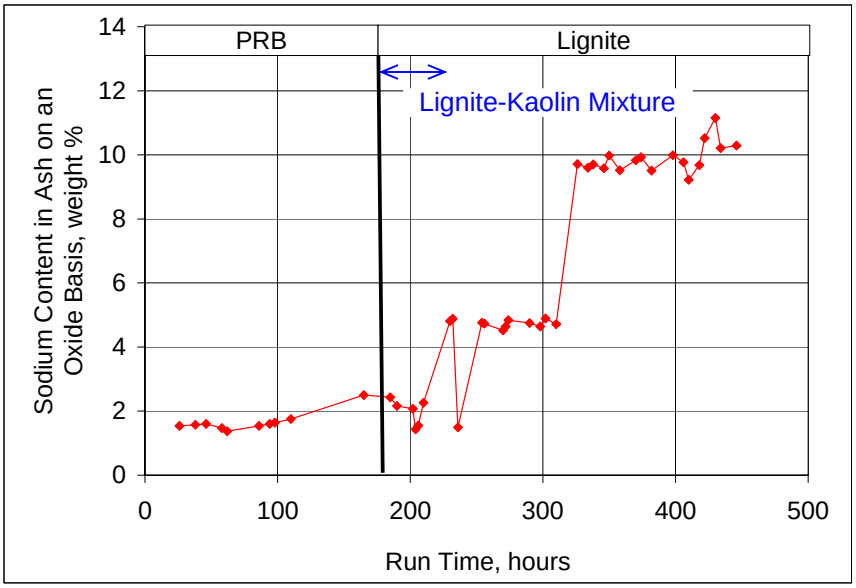


Figure 3. Sodium Content in Coal Ash on an Oxide Basis.

Figure 4 gives the TC23 coal particle sizes in both mass median diameter (MMD) and Sauter mean diameter (SMD). Again, data from Hour 180 to Hour 224 reflects the lignite kaolin mixture. The PRB coal particle size MMD varied from 243 to 437 microns with an average MMD of 348 microns and standard deviation of 45 microns, while the lignite coal particle size MMD varied from 271 to 864 microns with an average MMD of 505 microns and standard deviation of 134 microns. The PRB SMD varied between 129 and 254 microns with an average particle size of 194 microns and a standard deviation of 30 microns, while the lignite SMD varied between 141 and 373 microns with an average particle size of 238 microns and a standard deviation of 47 microns. The PRB particle sizes were slightly smaller and had less variability than the lignite particles sizes due to the differences in the two mill operations.

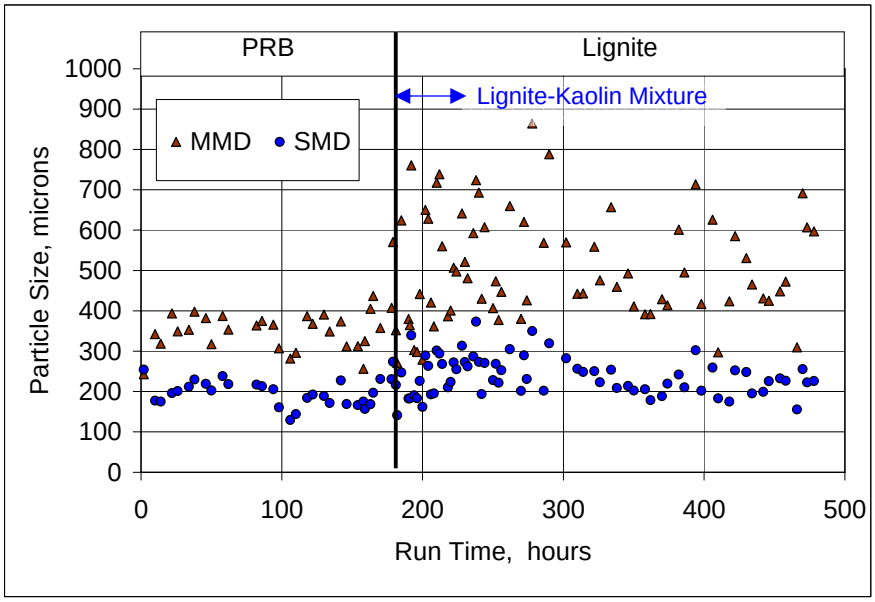


Figure 4. Coal Particle Sizes.

The percentage of coal particles above 1,180 microns and the percentage of coal particles below 45 microns are shown in Figure 5. The oversize particles above 1,180 microns were between 9 and 36 percent with an average of 16 percent. The coal fines less than 45 microns were between 1 and 20 percent with an average of 5 percent.

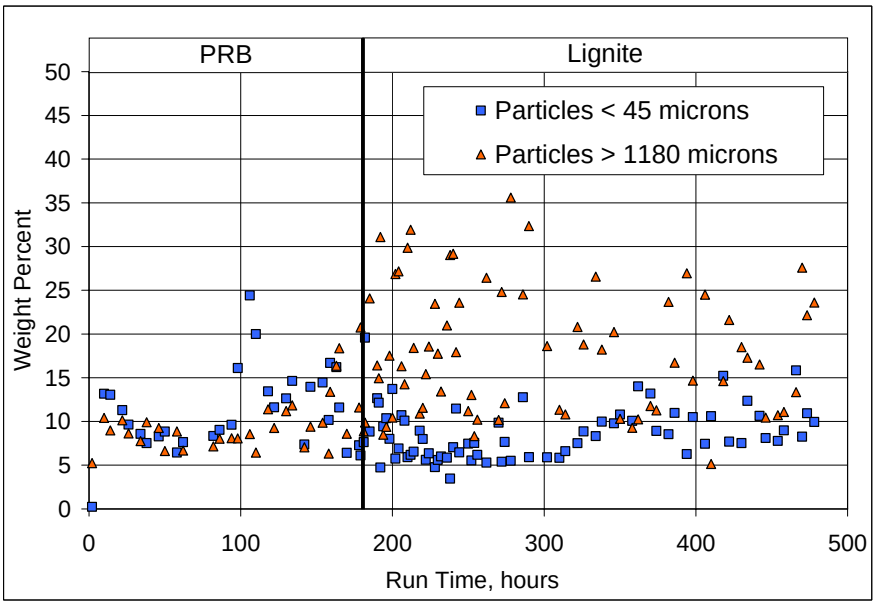


Figure 5. Percent Fine and Oversize Coal.

Based on TC23 operations, the coal feeder operating envelope for coal moisture content and particle size was further substantiated. Figure 6 shows the range of moisture and particle sizes

for acceptable feeder operation for PRB and high sodium lignite as compared to previous test runs.

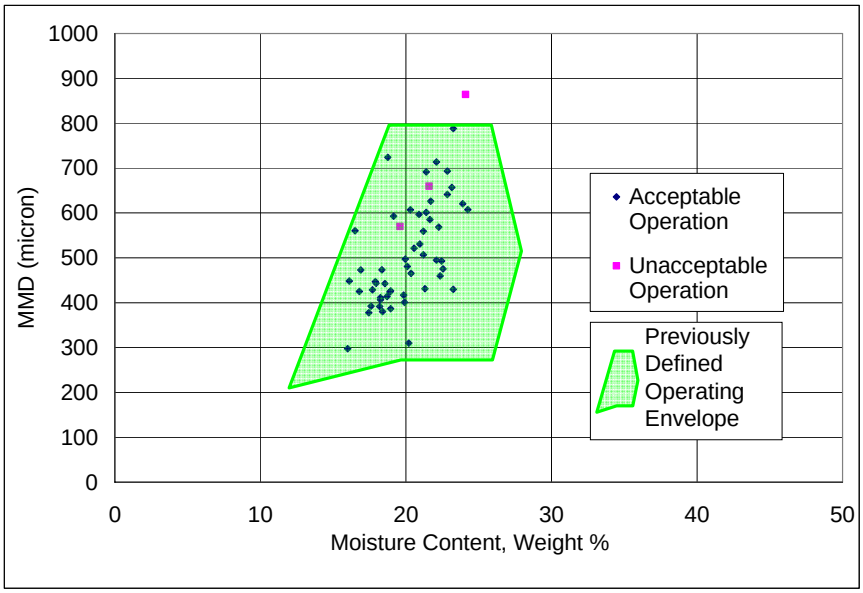


Figure 6. Coal Feeder Operating Envelope.

2.2 Sorbent Additive

Several sorbents including vermiculite, kaolin, kaolinite, dolomite, calcite, sand flour, and bauxite were evaluated as additives for testing with the high sodium lignite to address the agglomeration issues in the gasifier. Lab tests performed in a muffle furnace at the PSDF showed that the extent of sodium capture, based on energy-dispersive X-ray (EDX) analysis of the various reaction products, from greatest to least was: sand flour, kaolinite, kaolin, waste-derived kaolinite, Arkansas bauxite, vermiculite, and high-alumina bauxite. The finer kaolin and kaolinite showed a similar degree of consolidation as the sand flour; however, the larger kaolin particles, about 1,000 microns, did not show any signs of consolidation.

Initially, sand flour was selected as the additive since the amount of additive required is much lower than with kaolin or kaolinite due to the lower molecular weight. The existing sorbent feed system could not be used to feed the sand flour due to the small particle size. Thus, a slurry feed system was designed, procured, and installed to feed the sand flour.

In preparation for TC23, researchers at the Energy and Environmental Research Center (EERC), a non-profit technology branch of the University of North Dakota (UND), were consulted. Experiments in a test unit at similar gasification conditions showed kaolin and kaolinite was successful in eliminating agglomeration issues in the gasifier. The suggested chemical process was the reaction of kaolin with sodium to form the sodium aluminum silicates with a higher melting point than the maximum gasifier operating temperature, however due to the chemical properties of kaolin and kaolinite, a high kaolin or kaolinite to sodium ratio is required. It was decided to first test kaolin at the request of the North Dakota Lignite Industry Cost Share Participants.

The sorbent feed system, a lock hopper based system with a volumetric feeder that delivers material into a feed pipe that is then pneumatically conveyed into the gasifier, was used to feed kaolin into the Transport Gasifier during high sodium lignite operation. The kaolin tested, shown in Figure 7, was a minus 14-mesh, CK-46 Meta-kaolin purchased from CE Minerals. Meta-kaolin is a calcined kaoline clay with approximately 1.5 percent LOI and generally less than 1 percent quartz. The particle size distribution of the material is given in Figure 8. The sorbent feeder ran for 240 hours at rates from about 100 to 400 lb/hr. Due to a material shortage the kaolin feed rate was decreased below the required stoichiometric ratio for about 160 hours. Operating at the lower kaolin-to-sodium ratios was evident in the gasifier solids visual appearance as discussed in section 2.3.2.

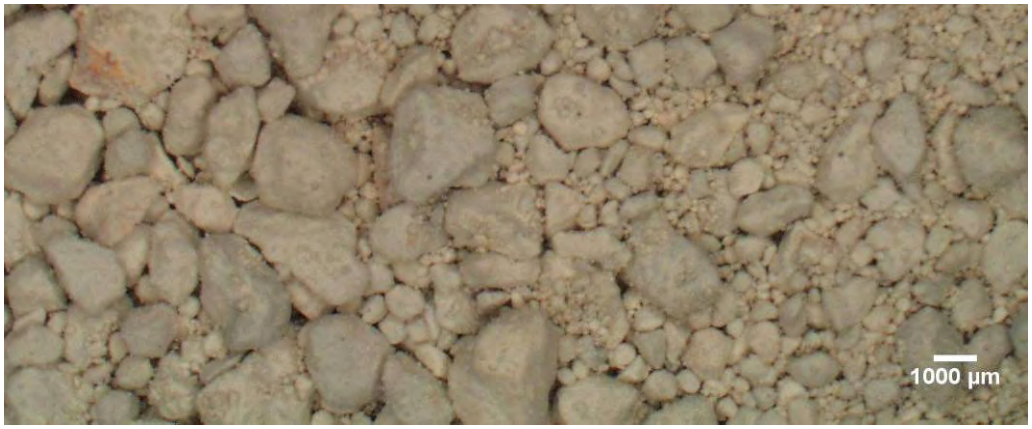


Figure 7. Raw Kaolin.

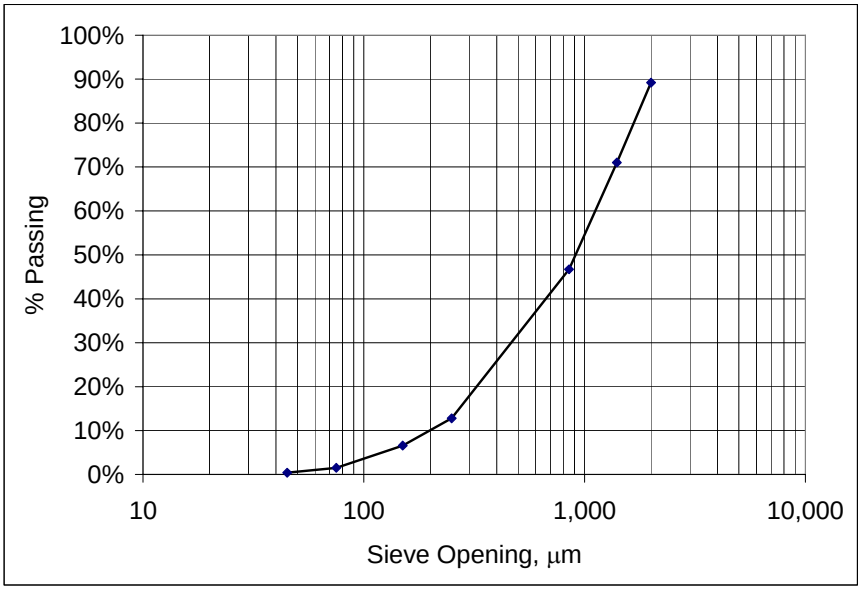


Figure 8. Kaolin Particle Size Distribution.

The raw kaolin was received in 2,000 pound super-sacks and was pneumatically conveyed to the surge hopper of the sorbent feed system utilizing a dense phase conveying system. Since this system was designed to handle a smaller particle size, the conveying line plugged periodically. Operational problems were encountered with the sorbent feed system when trying to establish operations initially. Since the sorbent feed system was not available, kaolin and lignite were fed simultaneously from the original coal feeder. After several days of troubleshooting, a failed level probe in the dispense vessel was discovered. The probe failure caused the dispense vessel and lock vessel to become overfilled, which resulted in plugging of the lock vessel and dispense vessel vent lines, rendering the sorbent feeder inoperable. After the probe was replaced, kaolin feed was successfully established using the sorbent feeder and was maintained for the remainder of the run about 250 hours.

2.3 Transport Gasifier

2.3.1 Gasifier Operating Parameters

Figure 9 gives the gasifier temperatures and pressures for the TC23 steady state periods. The mixing zone temperature varied between 1,725 and 1,795ºF for PRB and varied between 1,580 and 1,661ºF for lignite. Initially the gasifier was operated at lower temperatures with high sodium lignite as a conservative approach to minimize the chance for particle consolidation in the gasifier. The mixing zone temperature was gradually increased as the lignite testing progressed. The gasifier outlet pressure varied from 176 to 208 psig during PRB operation and varied from 130 to 220 psig during lignite operation. Again, as a conservative measure, initial operations were conducted at lower gasifier pressures to generate higher riser velocities. As the lignite testing progressed, the pressure was increased and the velocity was decreased.

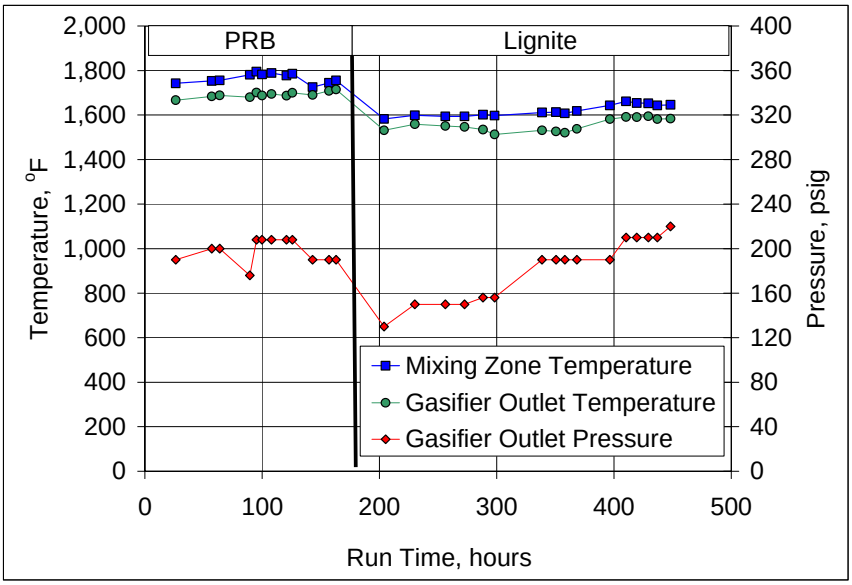


Figure 9. Gasifier Mixing Zone Temperatures and Outlet Temperatures and Pressures.

The major feed streams to the gasifier are shown in Figure 10. The coal feed rates were calculated from the feeder weigh cells, and the air, nitrogen, recycle gas, and steam flow rates

were taken from flow indicators. The PRB coal was fed at a rate of about 4,000 lb/hr, and was reduced to about 2,000 lb/hr prior to the transition to lignite feed. The lignite feed rate started at about 3,300 lb/hr and then was decreased to around 2,000 lb/hr around Hour 230 due to the kaolin shortage. The lignite feed rate was increased to around 4,000 lb/hr at Hour 273 and held there until the end of TC23.

The kaolin feed rates were calculated from the feeder weigh cells data, except for the Hour 204 test period, when it was estimated from the amount of kaolin conveyed into the coal surge bin. The kaolin feed rate was at about 300 lb/hr (0.09 pounds kaolin per pound of coal) for the first lignite steady period, and then decreased to about 120 lb/hr from Hour 230 to 368 (0.04 pounds kaolin per pound of coal) due to a shortage of the kaolin. At Hour 396, the kaolin rate was increased to about 370 lb/hr (0.1 pounds kaolin per pound of coal). The steam rate was varied between 500 and 1,100 lb/hr during PRB operation and was varied between 1,200 and 1,600 lb/hr while operating with lignite. The steam rate was slightly higher with the lignite tests to prevent any local hot spots that might cause sodium related agglomerations.

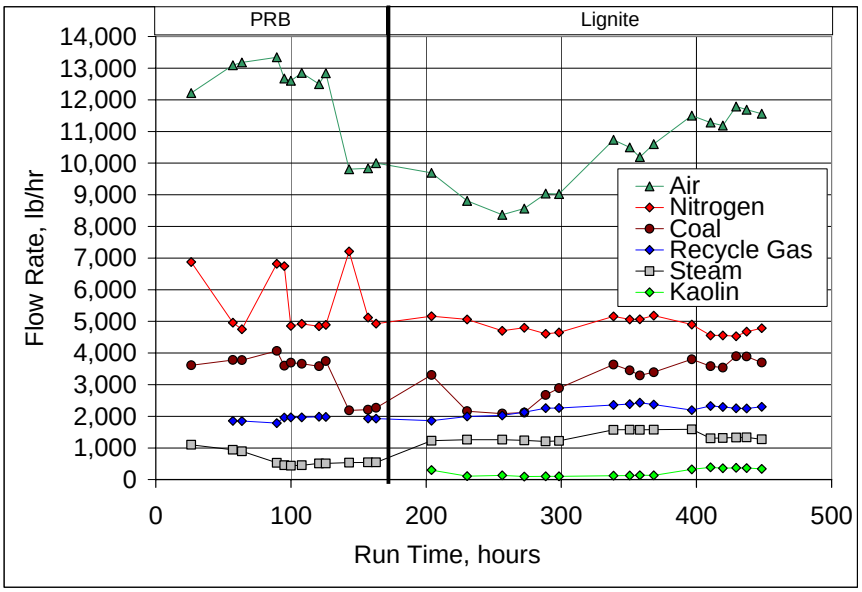


Figure 10. Gasifier Feed Flow Rates.

Figure 11 shows the standpipe levels (measured as differential pressures) and the riser differential pressures. The standpipe level was between 150 and 215 inH_2O for PRB testing and was controlled by the operation of the continuous coarse ash depressurization (CCAD) system. For the first two steady state periods during lignite operation, the standpipe level was maintained at about 187 inH_2O. When the kaolin and coal feed rates were decreased at Hour 230, the standpipe level started to decrease, and the CCAD system operation was suspended. The standpipe height decreased to 84 inH_2O by Hour 298 and then begin increasing around Hour 300 as the coal feed rate was increased. When the kaolin rate was increased to about 370 lb/hr around Hour 396, the standpipe level increased quickly up to 150 inH_2O. As expected, the riser differential pressure tracked the standpipe level.

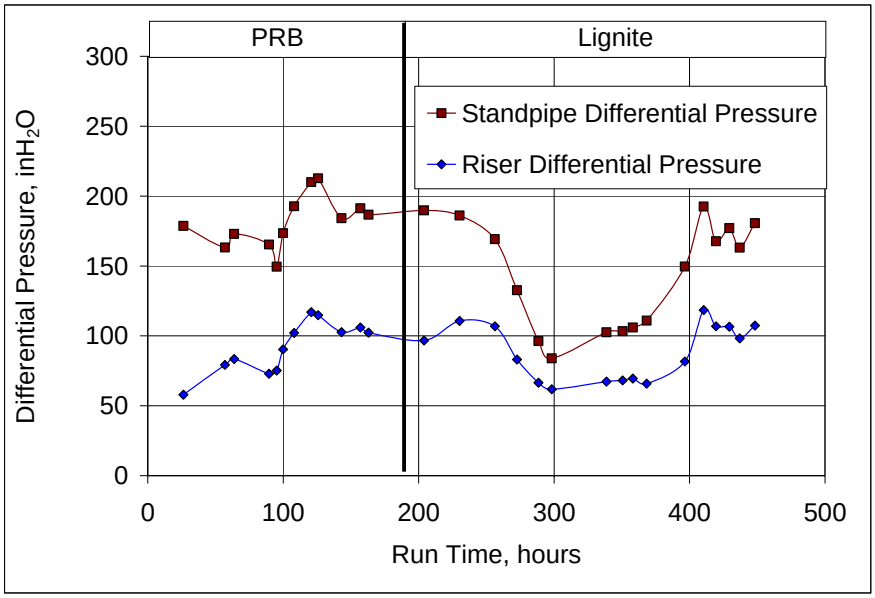


Figure 11. Gasifier Standpipe and Riser Differential Pressures.

Figure 12 shows the primary gas cooler plugging ratio over the course of the test campaign. The plugging ratio is the percentage of lost cross sectional area in the cooler tubes due to plugging. It is calculated based on the tube side pressure drop normalized to a constant temperature, pressure, and gas flow and compared to the cooler pressure drop at the same normalized process conditions with no restrictions. During PRB operation, the plugging ratio was consistently below ten indicating no plugging; however, during lignite operation, the plugging ratio increased dramatically in two steps. There was a gradual increase from Hour 224 to 300 and from Hour 425 until the unit was shutdown.

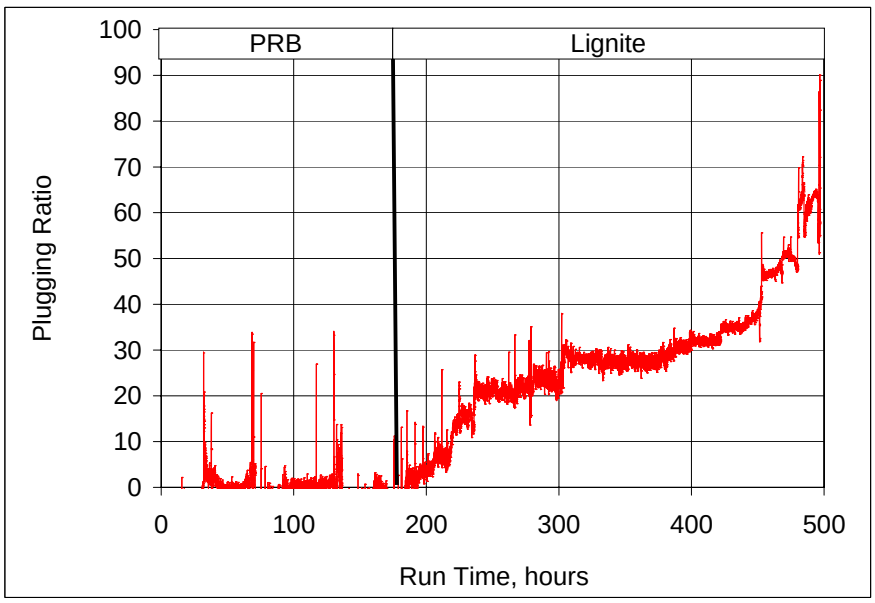


Figure 12. Plugging Ratio.

2.3.2 Gasifier Performance, Solids Analysis

Figure 13 gives the TC23 SMD particle sizes for circulating solids sampled from the standpipe and the seal leg. The PRB standpipe and seal leg particle sizes were between 100 and 150 microns. The PRB coarse solids had two periods of decreasing particle sizes. The first was from the startup sand particle size of 150 microns which then decreased down to 100 microns at Hour 82. The second one started at Hour 90 when the CCAD system was turned off to increase the standpipe level. This allowed larger particles to accumulate in the gasifier, thus increasing the standpipe solids particle size to 140 microns. The standpipe particle size again decreased down to 110 microns at Hour 164.

The standpipe and seal leg particle sizes were constant around 100 to 150 microns during the first fifty hours of lignite feed. After Hour 230, when the lignite and kaolin feed rate were decreased resulting in a decrease in standpipe level and the suspension of CCAD operations, the particle sizes began increasing to about Hour 300. This increase in particle size in the samples was also observed during PRB operations when the CCAD system operation was suspended around Hour 90. From Hour 300 to 400 the coarse solids particles sizes varied between 150 and 300 microns. At Hour 396 the kaolin feed rate was increased to 370 lb/hr and CCAD was started at Hour 402. After Hour 400 the coarse solids particle size decreased down to about 125 microns. The particle size during the last hours of lignite operation was similar to the sizes at the beginning of lignite feed.

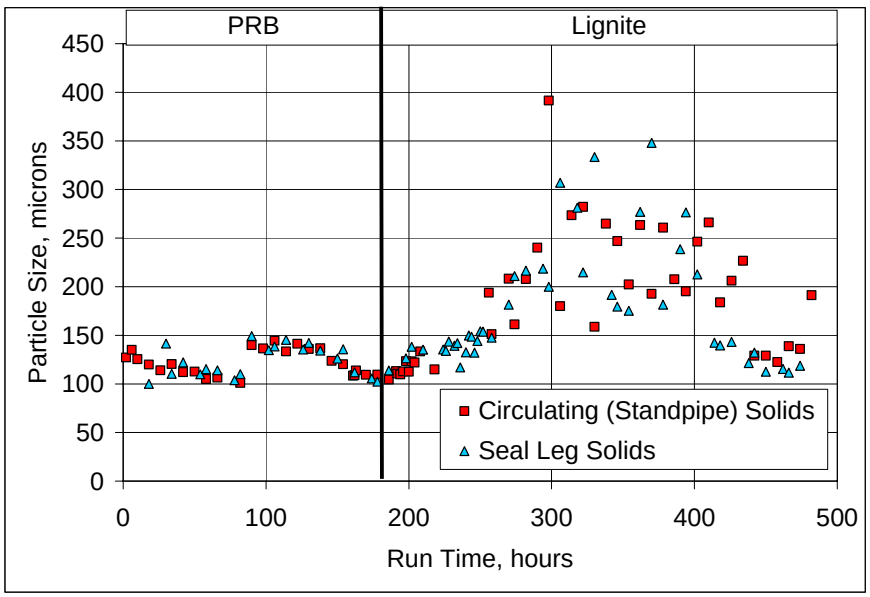


Figure 13. Gasifier Solids Particle Sizes.

Figure 14 shows the solids rates for the gasification ash removed from the PCD by the continuous fine ash depressurization (CFAD) system and the coarse gasification ash removed from the gasifier standpipe by the CCAD system. The PCD solids rates were determined from the PCD inlet solids concentration, and the CCAD rates were determined by a system ash

balance. The CFAD system discharged fines from the PCD at rates up to 540 lb/hr. The maximum CCAD system withdrawal rate of hot ash from the gasifier was about 580 lb/hr. The CCAD system effectively cooled the ash from around 1,700 to 200°F. Between 11 and 69 percent of the solids were removed by the CCAD system. The solids removed by the CCAD system were between 11 and 29 percent of the total gasifier solids for PRB and were between 36 and 69 percent of the total gasifier solids for lignite operation. CCAD withdrawal rates are preliminary and based of estimated fuel ash contents and the LOI of the fines removed by the PCD. The final values will be based on final fuel and PCD fines ash concentrations.

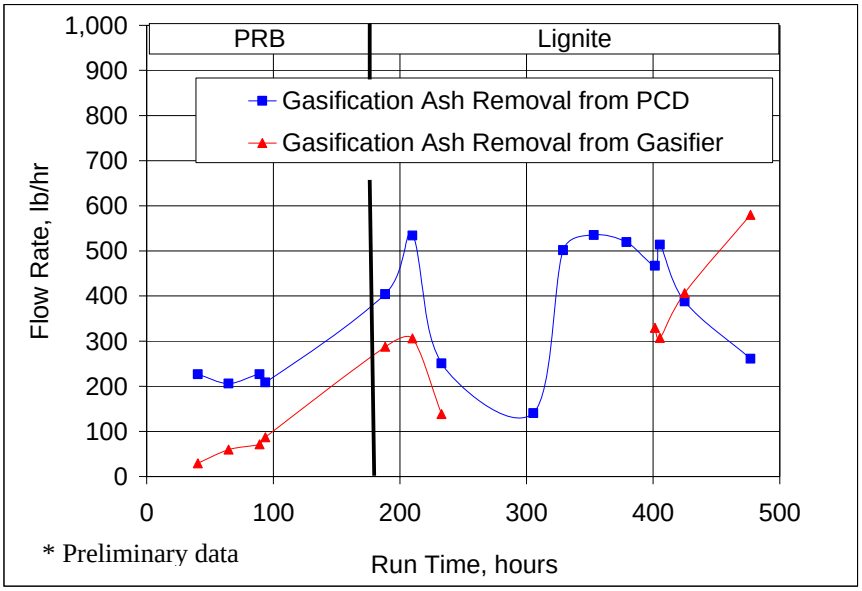


Figure 14. Gasification Ash Removal from PCD and Gasifier.

CFAD and CCAD operated well, accumulating a combined 800 operating hours. Figure 15 shows the particle sizes for the gasification ash removed by CFAD and the PCD inlet sample. The particle sizes varied from 6 to 20 microns. The range of particle sizes processed during TC23 for CFAD and CCAD are shown in Figure 16 with CCAD effectively processing a wide range of particle sizes

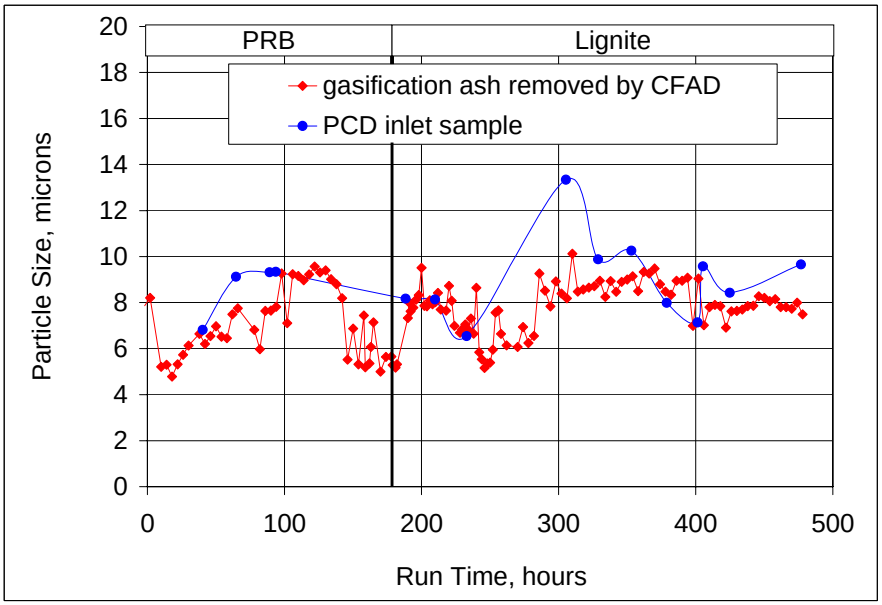


Figure 15. Gasification Ash Removed by CFAD and PCD Inlet Sample.

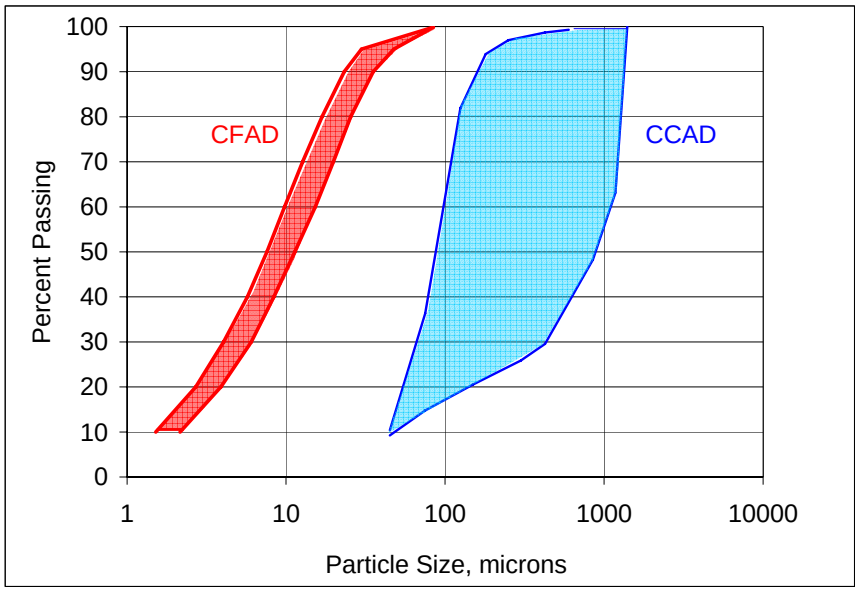


Figure 16. Particle Size Distribution Range Processed in CCAD.

Figure 17 gives the LOI for the gasifier solids. The standpipe and seal leg solids LOI values were typically less than 1 percent.

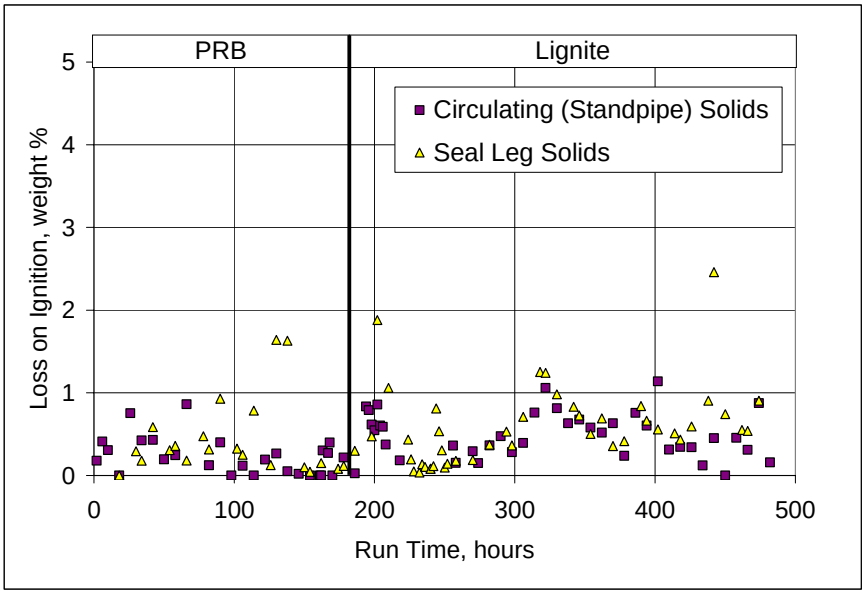


Figure 17. Gasifier Solids LOI.

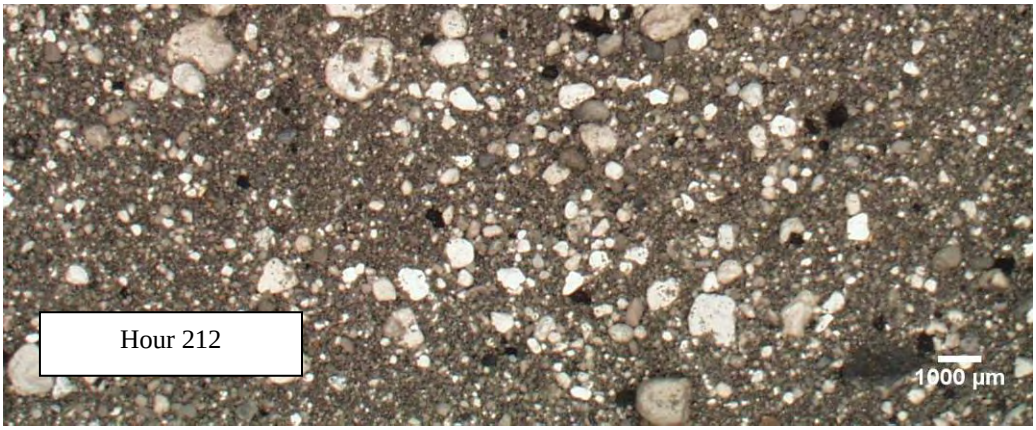
After the transition from PRB coal to high sodium lignite with kaolin addition, the gasifier solids were closely monitored to detect any signs of agglomeration. In addition to monitoring the particle sizes, the gasifier solids were visually inspected using an optical microscope. Figure 18 shows the gasifier solids as sampled from the standpipe at various times. Observations by the visual inspection of the samples corresponded well with data provided by the measured particle sizes.

Hour 100 in Figure 18 gives the baseline gasifier solids on PRB coal. The Hour 196 sample shows the solids about 16 hours after the transition to lignite and kaolin. Some fine ash remained and a few raw kaolin particles were seen. Samples taken at Hour 204 and 212 have a similar appearance as Hour 196, however the Hour 236 samples is noticeably different. There were less fine ash appearing and larger ash and kaolin particles were more visible. This sample was taken about 6 hours after the lignite and kaolin feed rates were reduced and CCAD operation was suspended. The lignite feed rate was increased at Hour 273 and the sample taken at Hour 276 shows mostly large kaolin particles. Samples taken at Hour 300, 324, and 372 contained mostly larger particles as well. These samples had a high percentage larger than 1000 microns. When the kaolin feed rate was increased at Hour 396, the increase in kaolin-to-sodium ratio combined with the removal of solids from the gasifier through CCAD altered the gasifier solids composition and size. The samples at Hour 460 and Hour 476 showed an accumulation of finer ash material and fewer larger particles.

Hour 100
1000 µm

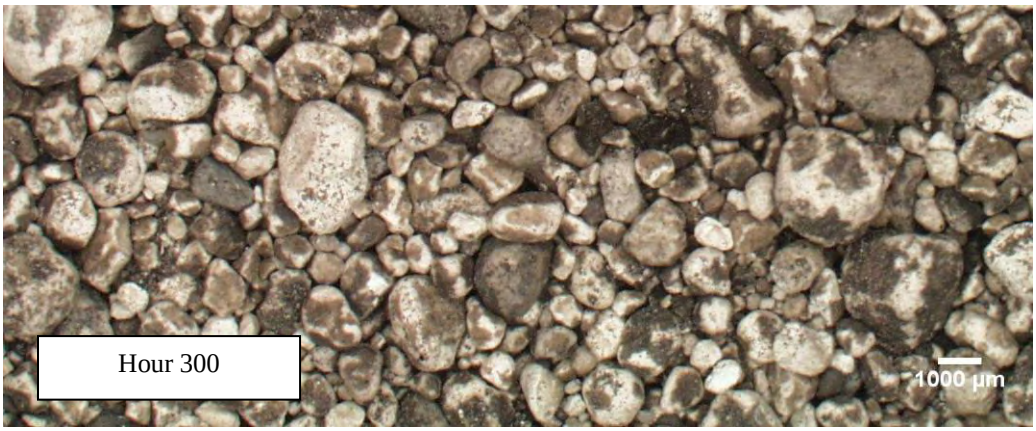
Lignite Feed Started at Hour 180
Hour 196
1000 µm

Hour 204
1000 µm


Hour 212
1000 µm

Lignite and kaolin feed rate reduced and CCAD stopped at Hour 230
Hour 236
1000 µm

Lignite feed rate increased at Hour 273
Hour 276
1000 µm


Hour 300
1000 µm

Hour 324
1000 µm

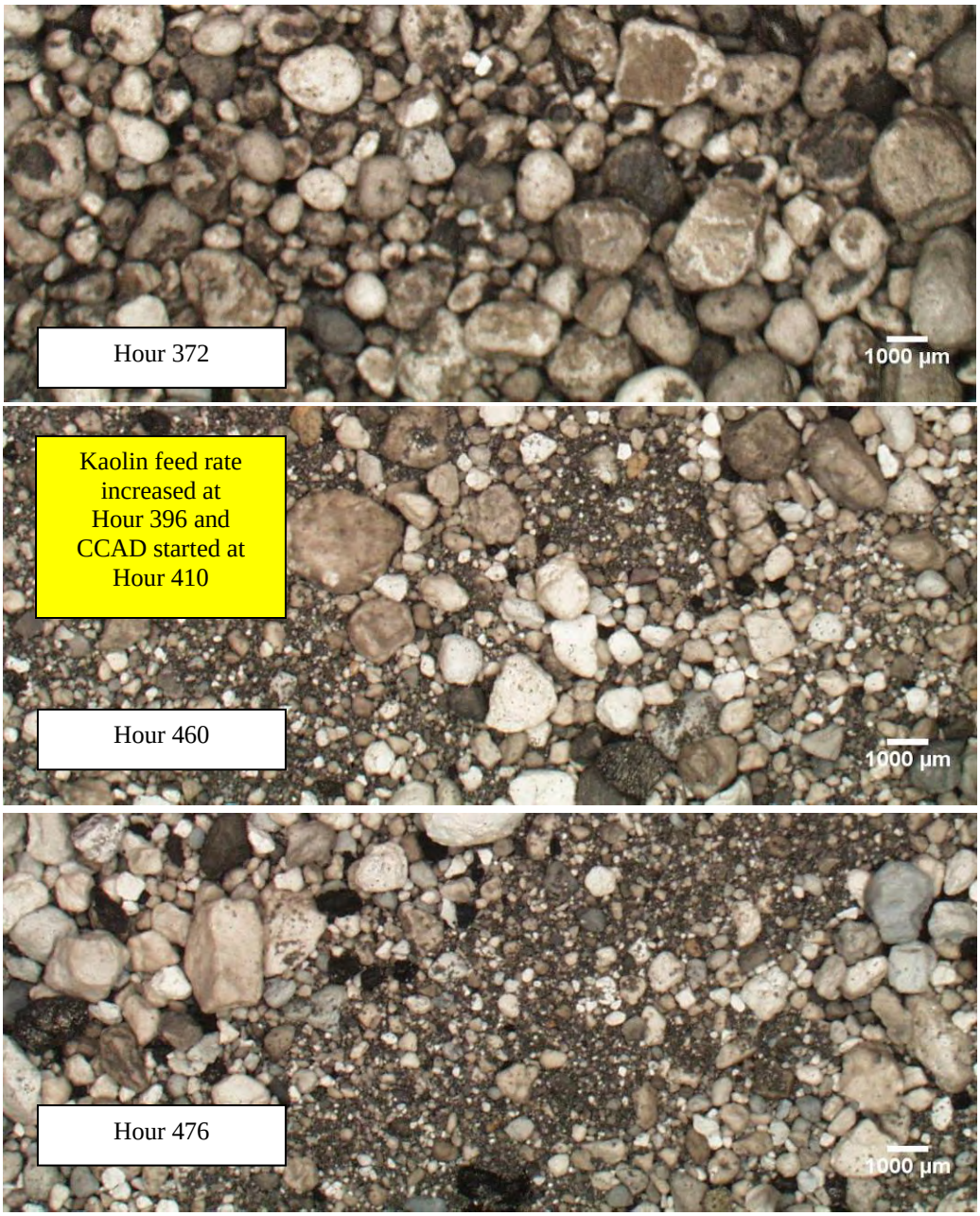
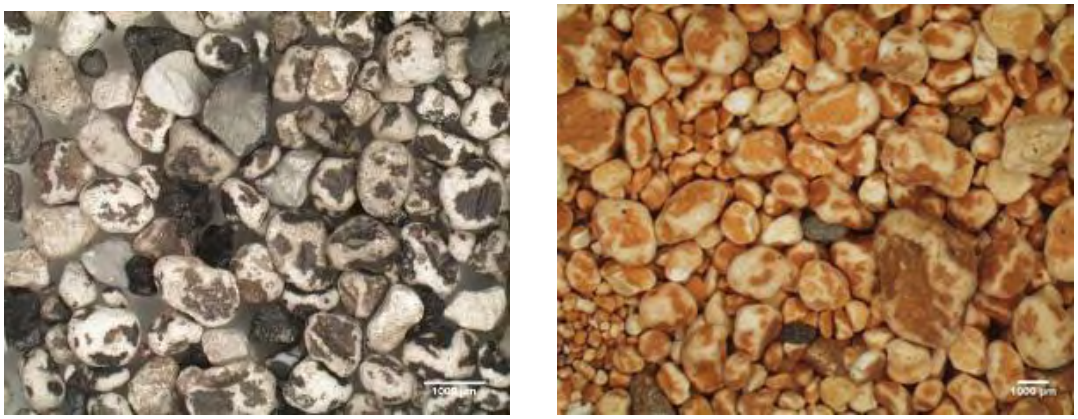


Figure 18. Gasifier Solids.

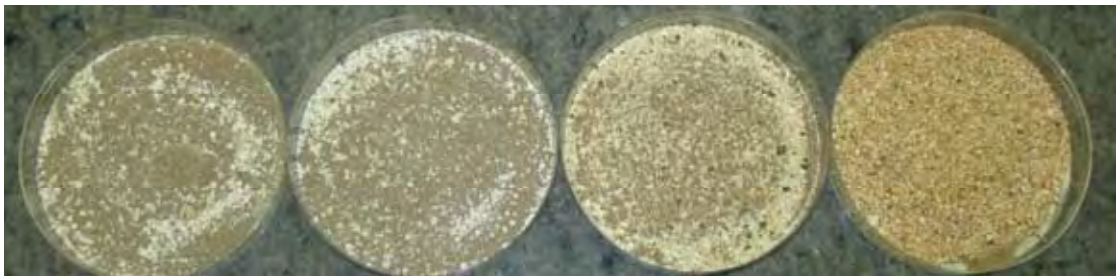
The gasifier solids were baked in a laboratory muffle furnace to determine if exposure to higher temperatures would cause sintering and agglomeration before increasing the operating temperature after the initial stabilization of operating conditions with lignite. No evidence of sintering or agglomeration was found in any of the samples tested. However, the large kaolin particles that were retained in the standpipe gradually became coated with a thin layer of very fine ash. When the samples were heated at 1,800°F for five hours, the gray coating of ash was converted to a reddish-brown ash as shown in the micrographs in Figure 19.



Before Baking After Baking

Figure 19. Standpipe Sample Before and After Baking.

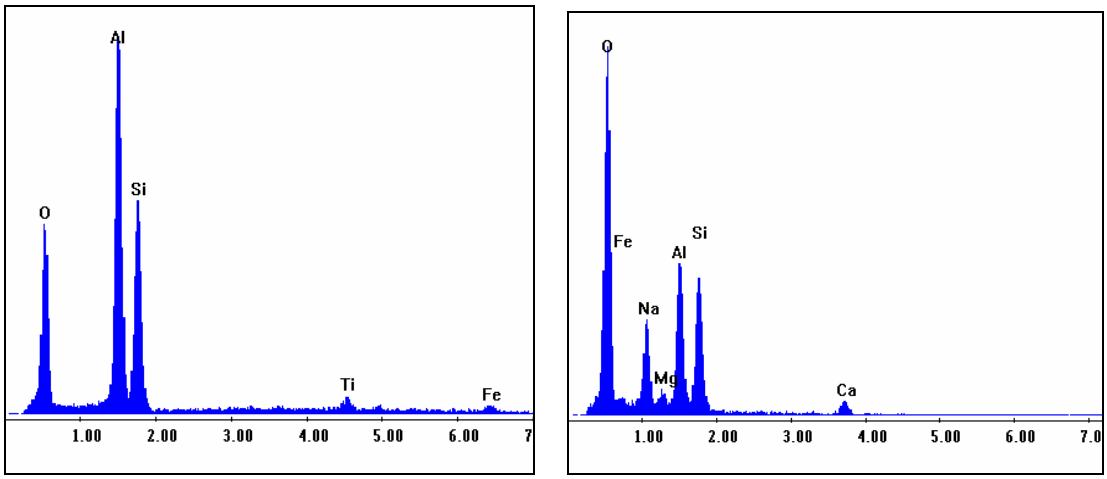
Based on the coloration of the standpipe samples, the amount of ash coating on the surface of the kaolin seemed to increase during the run as shown in Figure 20, but there was no evidence that this had any adverse effect on the performance of the kaolin.



Hour 178 Hour 186 Hour 202 Hour 298

Figure 20. Progressive Change in the Coloration of Baked Standpipe Samples.

The kaolin particles in the TC23 standpipe samples were analyzed by EDX analysis under the scanning electron microscope (SEM) to determine if the kaolin had captured sodium. The same type of analysis was performed on the raw kaolin for comparison, and the results are shown in Figure 21.



Raw Kaolin Kaolin Particle in Standpipe Sample

Figure 21. Energy-Dispersive X-Ray Analysis of Raw Kaolin and Kaolin Particle in Standpipe Sample.

The comparison of the EDX spectra clearly shows that the kaolin particle in the standpipe sample from Hour 350, has captured sodium. This analysis was obtained from an area of the kaolin particle surface that was not coated with ash. In cases where spectra were obtained from ash-coated surfaces, the spectra showed much higher peaks for calcium, iron, and other elements associated with the ash. In those cases, it was difficult to determine how much of the sodium was associated with the ash as opposed to the kaolin surface. However, in cases where there was no ash coating on the kaolin, there was clear evidence of sodium capture. Even in the particles that had captured significant sodium, there was no evidence of sintering between particles. Therefore, it appears that these larger kaolin particles can capture sodium without becoming sticky and agglomerating. This observation is consistent with the findings of Kosminski et al (1) who reported that kaolin reacts with sodium under gasification conditions to form solid nepheline ($Na_2O \cdot Al_2O_3 \cdot 2SiO_2$) without the formation of any liquid silicates.

2.3.3 Gasifier Performance, Gas Analysis

Figure 22 gives the preliminary carbon conversion values (based on the fines LOI) and the raw dry syngas lower heating values (LHVs) for the TC23 steady state periods. For PRB, the carbon conversion was between 95.5 and 98.7 percent and averaged 97.2 percent. With lignite operation, carbon conversion was between 91.3 and 97.6 percent and averaged 94.4 percent. The dry LHV was between 42 and 77 Btu/SCF for PRB and between 34 and 64 Btu/SCF for lignite.

Figure 23 gives the moisture content of the syngas as measured by the Fourier transform infrared spectroscopy (FTIR) analyzer and calculated based on the PCD outlet in situ sampling. There was good agreement with the analyzer and the in situ samples.

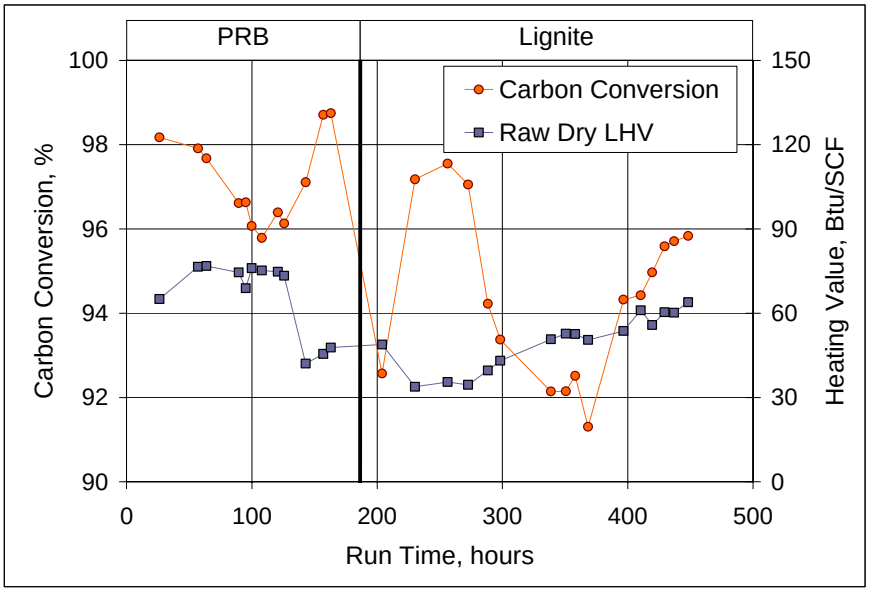


Figure 22. Carbon Conversion and Raw Dry Syngas Lower Heating Value.

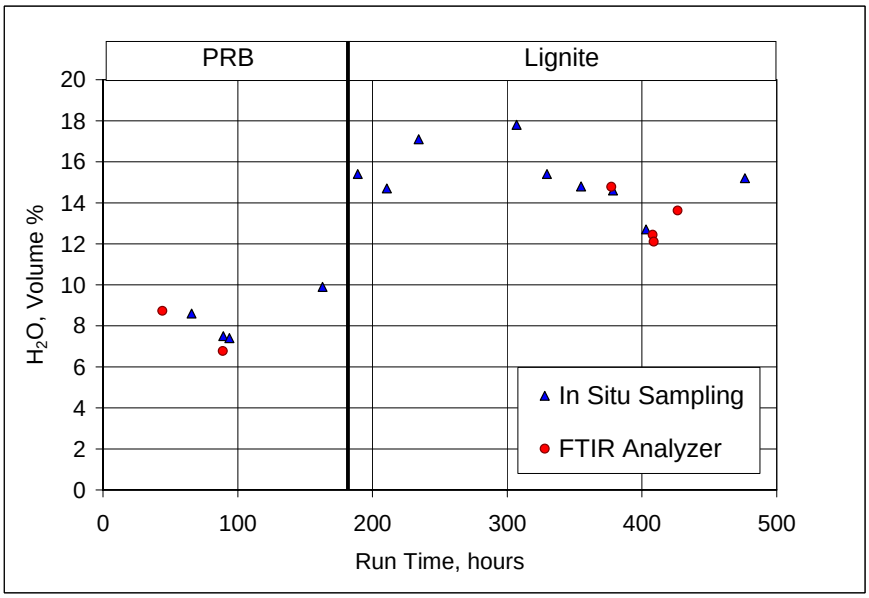


Figure 23. Moisture Content in Syngas.

Figure 24 gives the syngas hydrogen (H_2), carbon monoxide (CO), carbon dioxide (CO_2), and methane (CH_4) concentrations on a wet basis (volume percent). The gas compositions were measured by a gas chromatograph (GC) on a moisture free basis and converted to wet gas concentrations using the H_2O concentrations. Note that for PRB the CO was higher than the H_2, while for the lignite the H_2 was higher than the CO. This is the result of the higher lignite feed moisture content and the higher steam rates used during the lignite testing. The additional moisture in the syngas shifted CO to H_2 via the water gas shift reaction.

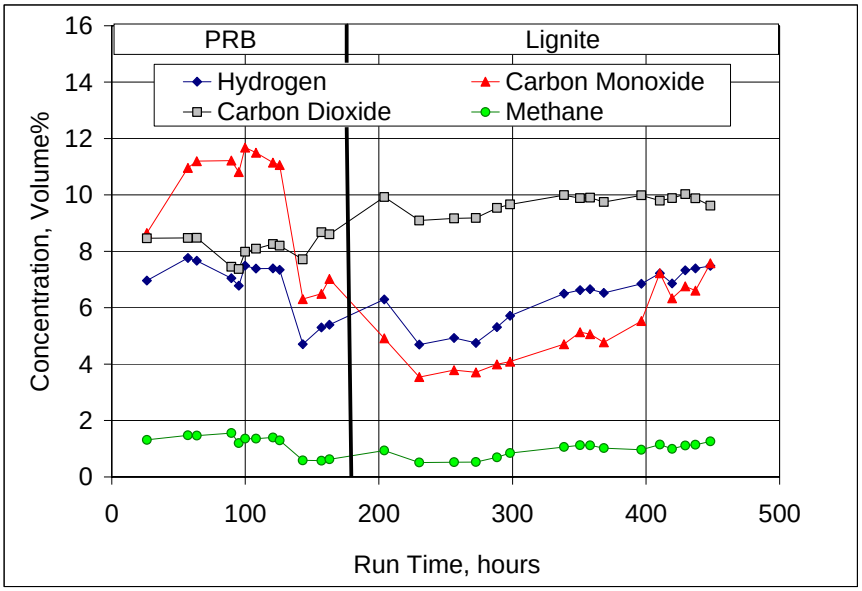


Figure 24. Syngas Hydrogen, Carbon Monoxide, Carbon Dioxide and Methane Concentrations.

Figure 25 gives the syngas sulfur concentrations as measured by the syngas hydrogen sulfide (H_2S) analyzer. As expected, the higher sulfur content lignite produced a syngas that had a higher H_2S concentration, than the lower sulfur content PRB.

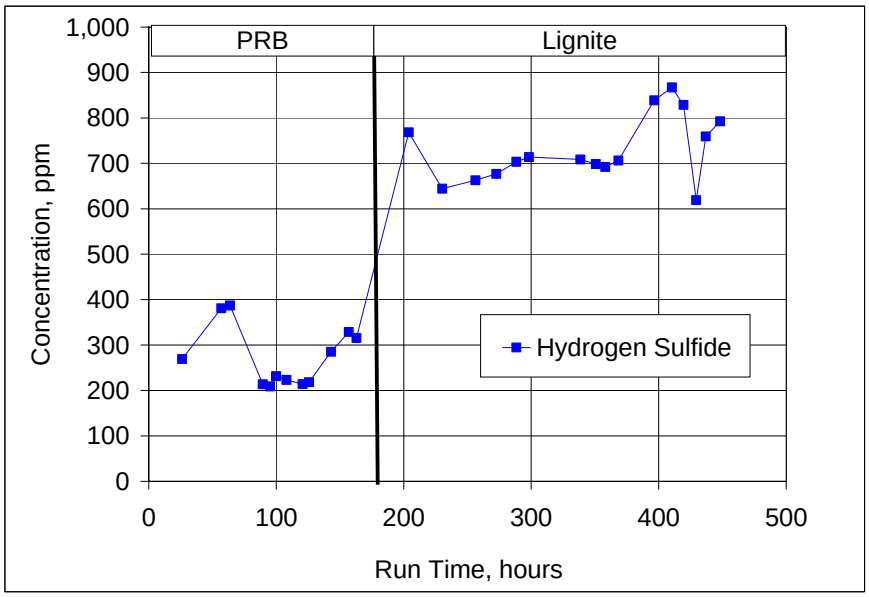


Figure 25. Syngas Hydrogen Sulfide Concentrations.

The syngas ammonia, benzene, naphthalene, and phenanthrene concentrations are listed in Appendix 2 and the syngas H_2S, syngas carbonyl sulfide (COS), and percent syngas sulfur converted to COS are listed in Appendix 3.

2.3.4 Gasifier Performance, Parametric Testing

A number of tests were performed to establish boundary conditions of Transport Gasifier operations with high sodium lignite. The parametric testing completed included gasifier temperature, gasifier pressure/riser velocity, steam to coal ratio, and coal feed rate effects on gasifier performance. In addition, the effect of standpipe level on gasifier circulation rate was examined.

Partial analyses of these parametric tests are presented below. Further analysis is ongoing, and results will be included in the final TC23 topical report. To obtain meaningful analyses, data were analyzed using selected steady state periods which held other variables nearly constant to focus on the variable of interest.

Figure 26 gives the effect of gasifier temperature on carbon conversion at a gasifier pressure of 185 psig, air-to-coal mass ratios between 3.2 and 3.3, and coal feed rates ranging from 3,400 to 3,600 lb/hr. As expected, the data shows a linear correlation between carbon conversion and temperature.

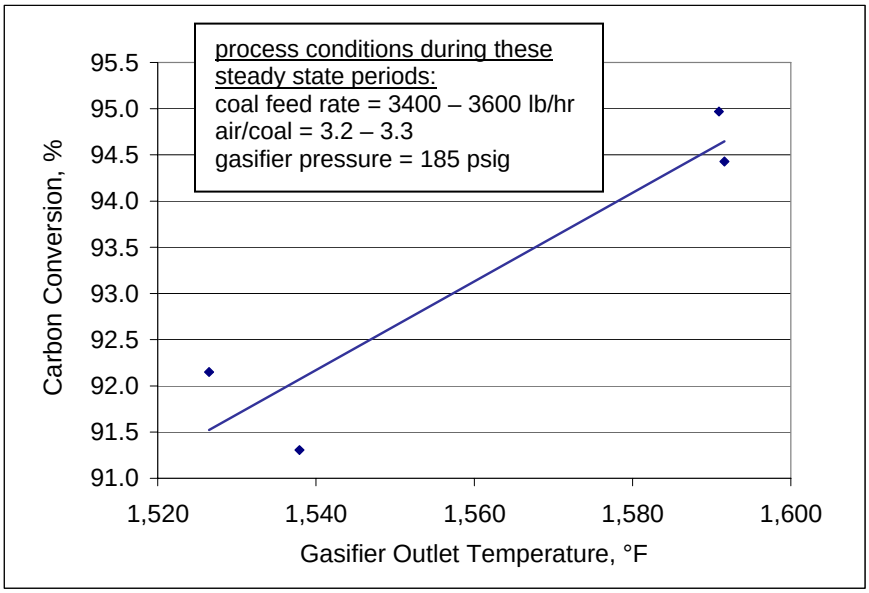


Figure 26. Carbon Conversion as a Function of Gasifier Temperature.

Gasifier pressure (which directly controls riser velocity) was varied to quantify its effects on gasifier performance. Figure 27 illustratively shows the effect of pressure on the syngas methane content, represented by a relative value, the methane factor. The methane factor is calculated based on the actual methane content but uses an equation to normalize the data so that inert gas dilution effects are eliminated. An additional factor is used to place the data on a zero to one scale. During the steady state periods from which the data was extracted, the air-to-coal mass ratios were maintained at about 2.9 to 3.2, the gasifier mixing zone temperature ranged from about 1,600 to 1,630^{o}F, and carbon conversions were between 91.3 and 92.6 percent. The

limited data suggest a linear relationship correlating to an increase in methane factor with increasing pressure.

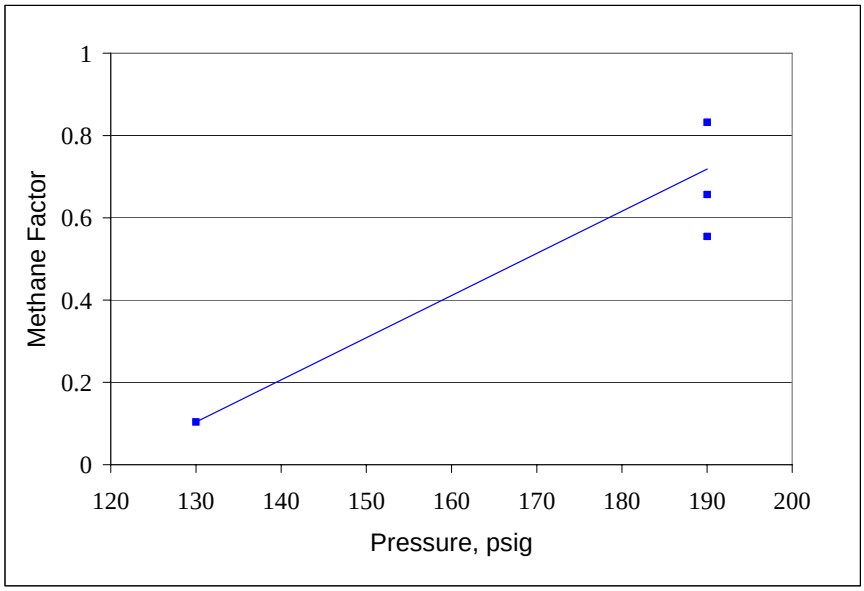


Figure 27. Methane Factor as a Function of Gasifier Pressure.

Figure 28 plots the syngas hydrogen-to-carbon monoxide ratio and the steam-to-coal ratio. The curves follow the expected trend of increasing hydrogen-to-carbon monoxide ratio with increasing steam-to-coal ratio due to the water gas shift reaction. The lignite has higher hydrogen-to-carbon monoxide ratio at similar steam-to-coal ratios.

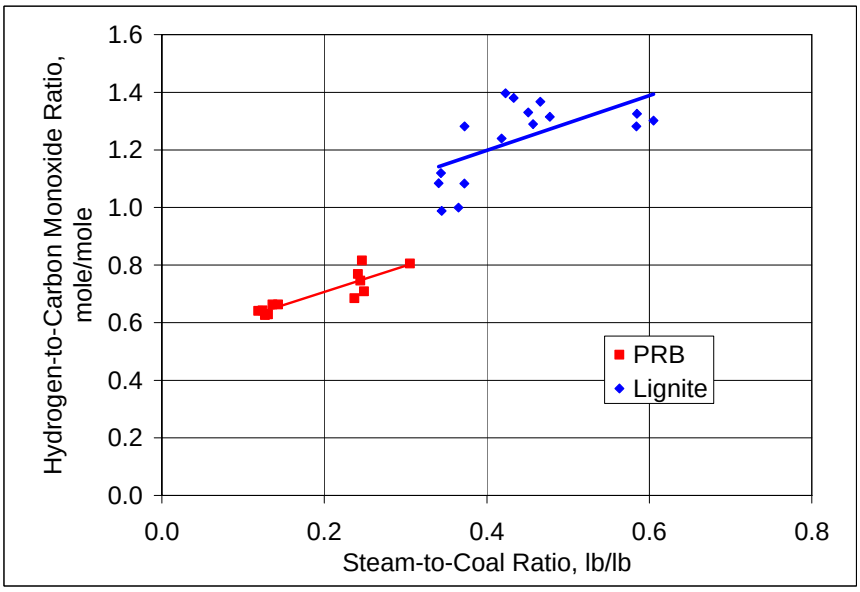


Figure 28. Syngas Hydrogen-to-Carbon Monoxide Ratio versus the Steam-to-Coal Ratio.

Figure 29 shows the raw dry syngas heating value as a function of coal feed rate. This plot included the steady state data taken when the air-to-coal mass ratio was between 2.9 and 4.1. As shown in the figure, the syngas heating value increases as the coal feed rate increases. The increase is due to a lessened effect of nitrogen and a lower percentage of heat loss relevant to the heat input.

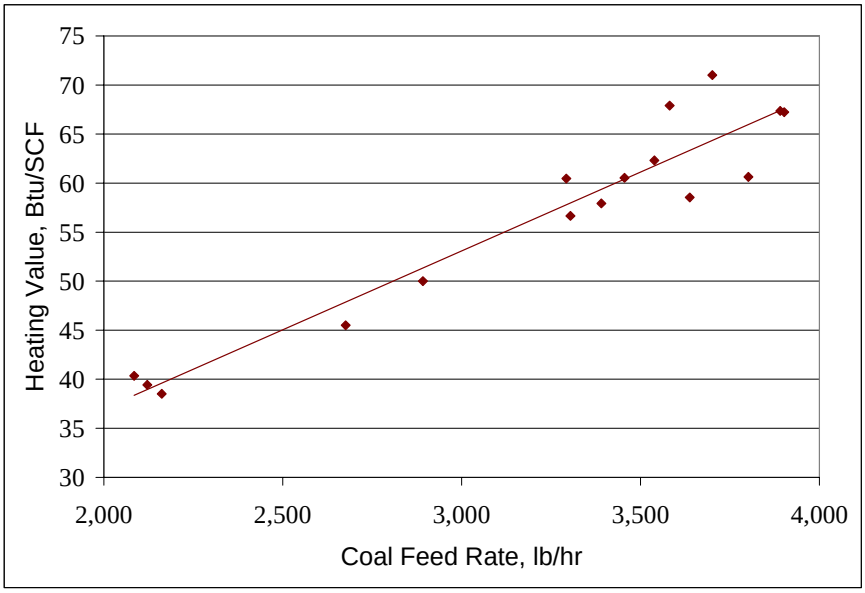


Figure 29. Dry Syngas Heating Value as a Function of Coal Feed Rate.

Figure 30 shows the relative solids circulation rate as a function of gasifier standpipe level. As expected, the data indicate a strong linear relationship.

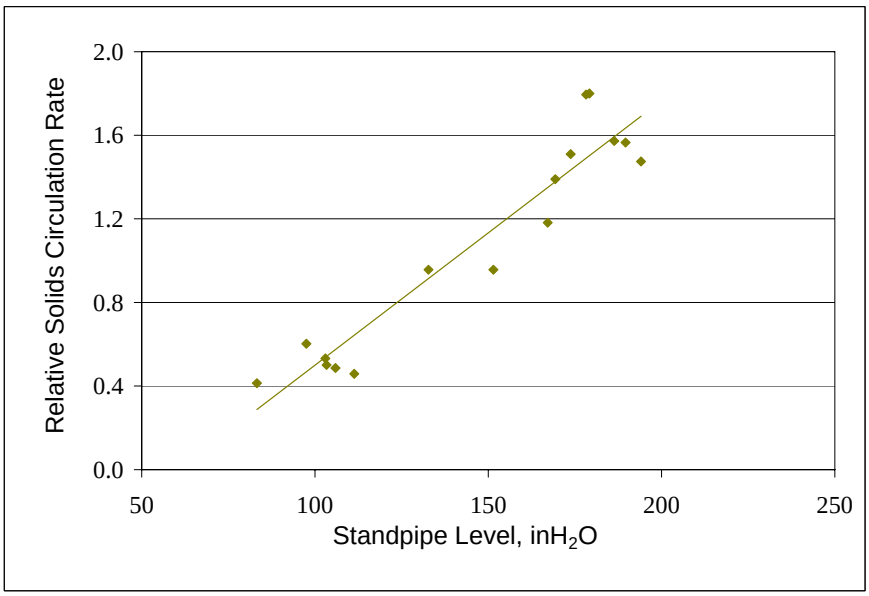
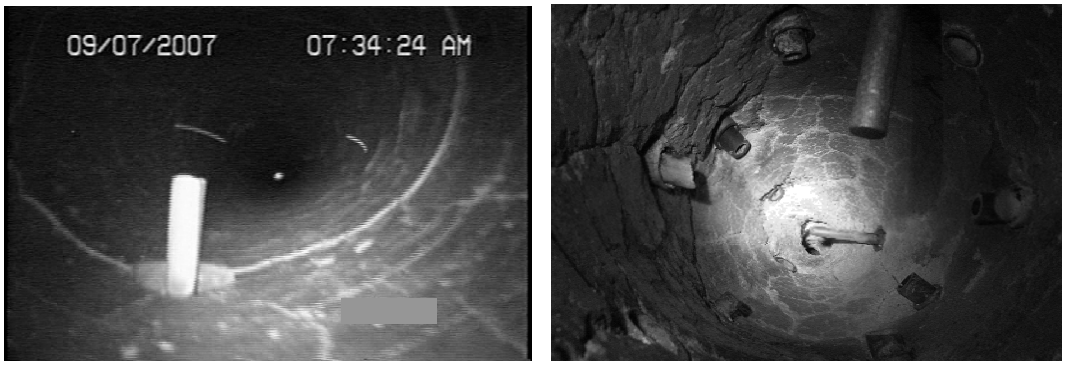


Figure 30. Effect of Standpipe Level on Circulation Rate.

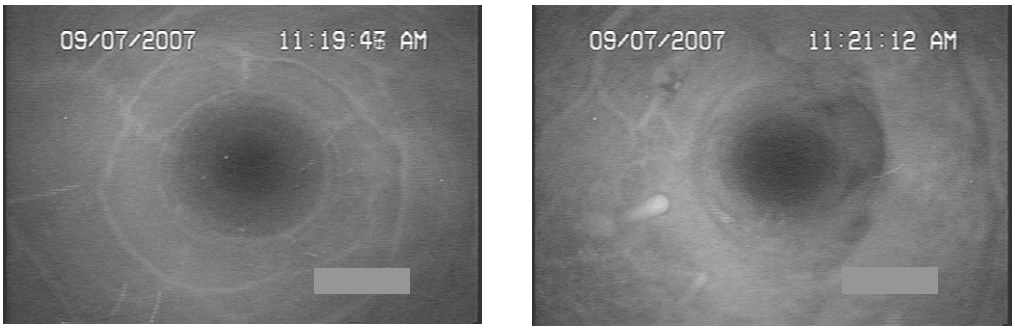
2.3.5 Gasifier Inspections

Detailed inspections of the gasifier and related equipment were performed following the conclusion of TC23. Based on previous operations with high sodium lignite, there was a concern that solids would agglomerate in the gasifier. However, inspections confirmed that no agglomerations had formed in the gasifier during this test run. Figure 31 shows borescope photographs of the riser and lower mixing zone taken during the inspections. In general, the gasifier refractory was found in good condition, although a portion of refractory was missing in the first stage solids separation inlet. Also, some cracks were noted in this solids separation device. The refractory in the lower standpipe, which has been in service for over 19,000 hours of solids circulation and experienced over 100 temperature cycles remains in excellent condition as shown in Figure 32.



Riser Lower Mixing Zone

Figure 31. Riser and Lower Mixing Zone.



Middle Lower Standpipe Lower Standpipe

Figure 32. Lower Standpipe Inspection Photographs.

During the gasifier modifications in 2006, different types of refractory were installed in various sections of the gasifier based on the operating conditions in each area to test different types of refractory. Actchem 85 VC was installed in the first stage solids separation device to evaluate its performance at these conditions. The Actchem 85 VC properties include a high erosion resistance and high coefficient of thermal expansion, which make it very resistant to wear but susceptible to cracking. The Actchem 85 VC refractory life is heavily dependent on the number

of thermal cycles and is rated for about 30 thermal cycles (about 10 year commercial life). Since installation in the first stage solids collection device, the Actchem 85 VC refractory has experienced approximately 15 thermal cycles. Based on the thermal cycles experienced and current refractory condition, the refractory is about halfway through its useful life. Figure 33 shows examples of the cracking in this refractory.

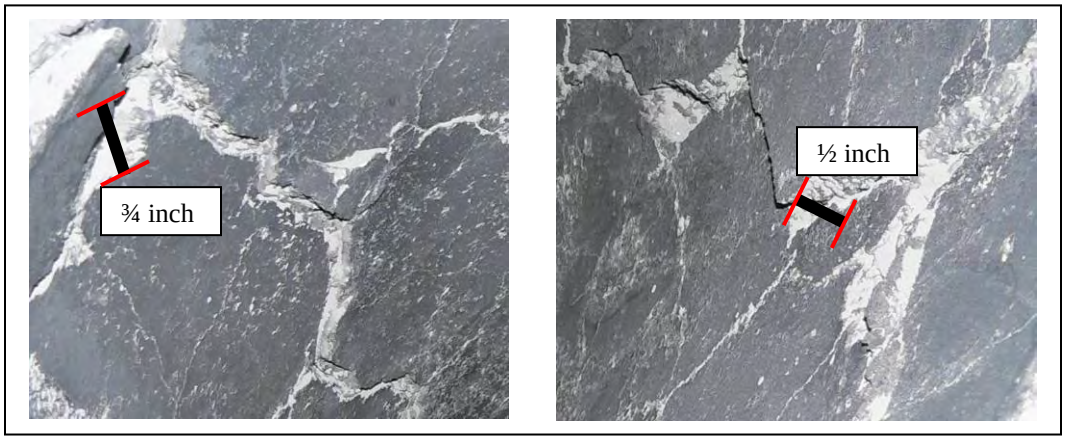


Figure 33. Cracking Observed in Actchem 85 VC Refractory.

Inspections of the primary gas cooler, shown in Figure 34, revealed a majority of the tubes (about 60 percent) were plugged with material by the end of the run consistent with the increases in the plugging ratio as shown in Figure 12. The material was soft and easily removed. Some soft agglomerated material was also found in the process piping downstream of the cooler. Since the solids exiting the gasifier are small particles, about 10 to 12 microns, it is possible that the smaller particles containing the sodium based silicates agglomerated in the gas cooler as the temperature decreased. This is consistent with the laboratory tests performed at the PSDF prior to the test campaign that showed some consolidation when testing the kaolin and kaolinite fine material. Samples of the material plugging the tubes were taken for additional analysis to confirm the deposition mechanism.

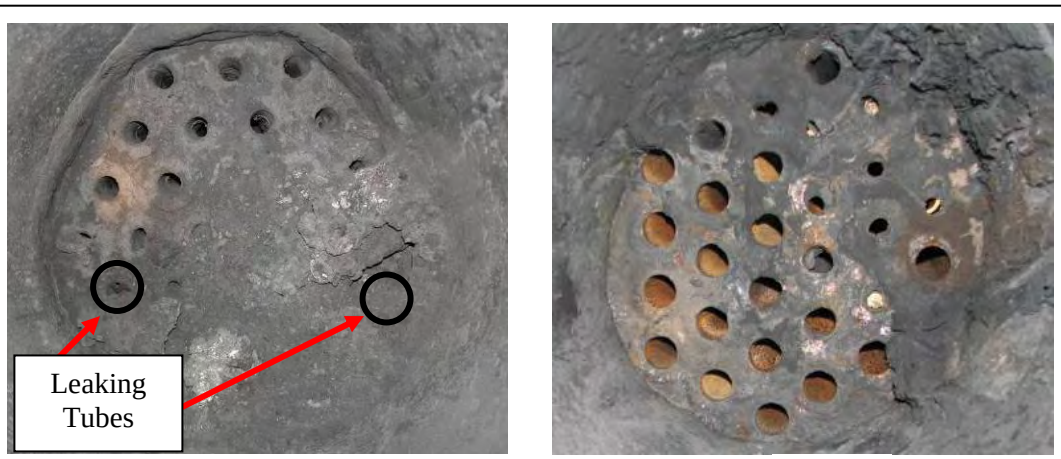


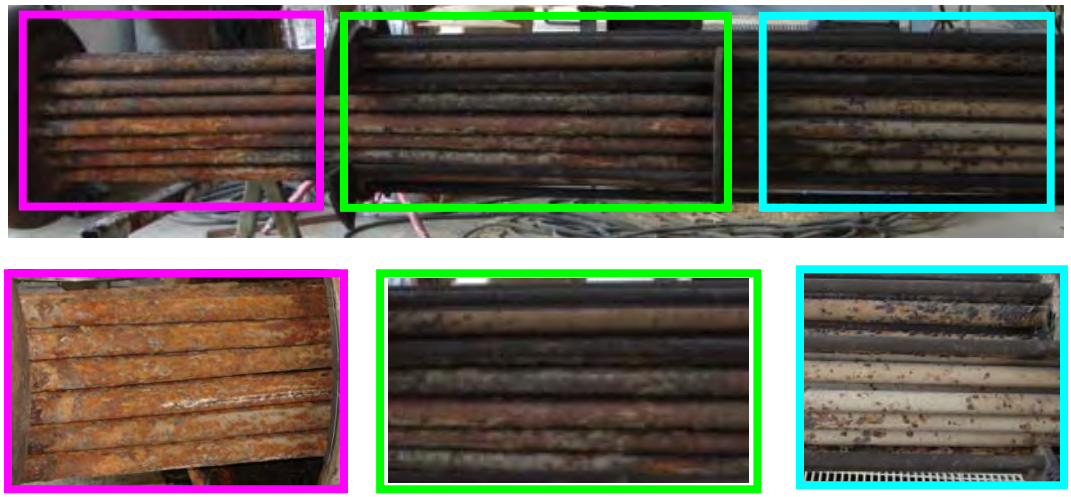
Figure 34. Inlet and Outlet of Primary Gas Cooler.

The tubes that remained opened had some signs of wear. Hydrostatic testing of the cooler showed two leaking tubes. The two leaking tubes located in the outer ring of the array (as noted in Figure 34) had cracked in the upper section of the cooler near the gas inlet.

Further inspections revealed that the water side of the tubes in the upper section of the cooler had suffered significant corrosion. Surface pitting possibly caused from oxygen attack was noted and believed to be caused by downtime corrosion and water chemistry issues resulting from a balance of plant heat exchanger leak that allowed untreated water to enter the steam system. Additional analysis revealed high levels of sulfur on the tube outer diameter which was also linked to the water chemistry issues. The lower section of the tubes where the water entered was in very good condition. Since extensive tube repair was likely, it was decided to replace all of the heat exchanger tubes.

Figure 35 shows the gas cooler tube bundle after it was removed from the process. The left side of the photograph is the syngas inlet and saturated steam exit. Excessive corrosion had occurred in this area as evident in the photograph. The middle section and the water inlet and syngas outlet shown on the right side of the figure were in acceptable condition.

The failed tubes were analyzed to determine to the root cause of the failure. Based on information received to date, the tube wall thickness had decreased during the last several test campaigns likely due to corrosion on the water side. Wall thickness measurements are underway to evaluate if erosion on the gas side also contributed to the failures. As the tubes became plugged with material during the later stages of TC23, the temperature differential between the blocked tubes and tubesheet increased as the blocked tubes cooled. The higher thermal stress in combination with a thinned tube wall likely caused the tubes to crack.



Syngas inlet: steam exit | Middle section | Syngas exit: water inlet

Figure 35. Primary Gas Cooler.

Inspection of the secondary syngas cooler showed that tar condensation had not occurred. As part of the preventive maintenance program the cooler was hydrostatically tested and three tubes were found to be leaking. The leaking tubes were repaired. An evaluation of the water chemistry is ongoing to determine if improvements to the water chemistry is needed to prevent corrosion in the future.

2.3.6 Tracer Tests

A tracer gas test was performed during PRB operation in TC23 to evaluate gas underflow from the first solids separation device to the standpipe, gas and solids mixing in the riser, riser velocity, and vapor evolution profiles. A tracer gas, Argon-41, was injected into the gasifier at two locations, the mixing zone and riser, at separate times under various operating conditions. The riser velocity was varied from 20 to 28 ft/s and the standpipe level measured as differential pressure was varied from 140 to 180 inH_2O. Analysis of the data is underway.

The tracer gas equipment setup, injection, and raw data acquisition was performed by a third-party contractor, Tracerco. Tracerco has extensive experience in tracer gas testing in the oil refining and exploration industries. Injection nozzles were installed for Tracerco to connect a tracer injection apparatus to the system. Non-intrusive detectors were strapped to the perimeter of the gasifier to measure tracer concentrations in the syngas. Two injections were made initially for calibration of the detection equipment. The next six injections and subsequent measurements lasted for approximately 3 hours. The tracer gas, isotope Argon-41, is commonly used in tracer gas testing because it has a short half-life of 1.8 hours, is chemically inert, and can be measured reliably with non-intrusive detectors. Radiation levels were monitored during injection of the tracer gas, and no detectable levels were observed outside of the gasifier.

2.4 Sensor Development

2.4.1 Ceramic-Tipped Pressure Differential Measurements

One area of focus of the sensor development program has been low flow, ceramic-tipped pressure differential measurement ports. Conceptually, this measurement method lowers instrument purge flow requirements and improves sensitivity. Three sets of these low flow ports were evaluated in previous test runs. Two port designs were evaluated: a high differential pressure design and very low differential pressure design. The low differential pressure design matched traditional measurements well, but the high differential pressure design often plugged due to nitrogen purge flow control issues. Installation of low nitrogen flow meters was proposed but further testing was suspended due to the low impact on commercial operations and cost.

2.4.2 Thermowell Materials

Two types of thermowell materials, metal (HR-160) and ceramic, were tested during TC23 for continued evaluation of thermowell durability and longevity. There were no failed gasifier temperature measurements; however, post-run inspections revealed that three thermowells in the mixing zone just above the coal feed nozzle had extensive metal loss. The tip degradation was severe with wear to the inner sheath. These same three elements had shown some wear during

TC21 when exposed to high velocities due to deposit formation in the mixing zone. Figure 36 shows a typical example of these worn thermocouples, and a TC21 post-run inspection photograph is shown for comparison. Metallurgical analysis of the worn HR-160 elements revealed that sulfur species may have attacked the nickel contained in the HR-160. The SEM results from the original material compared to the exposed HR-160 thermowells will be included in the final report. The increased gasifier solids particle size and hardness from kaolin addition may have accelerated the thermowell tip degradation in TC23. Additional analysis will be conducted to determine the main source of degradation.

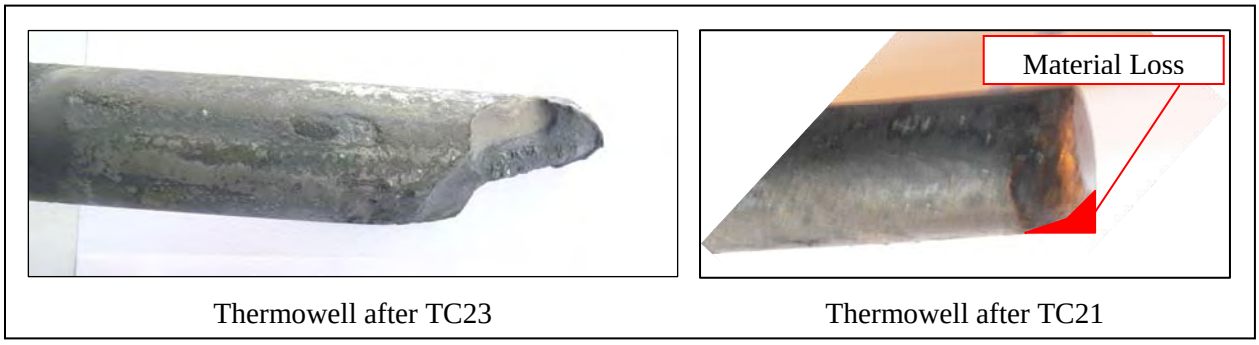


Figure 36. Eroded HR-160 Thermowell Element Removed after TC21 and TC23.

All other HR-160 elements removed from the mixing zone and riser had only minimal wear. A typical example of these elements is shown in Figure 37. These elements have accumulated over 2,200 hours of operation.

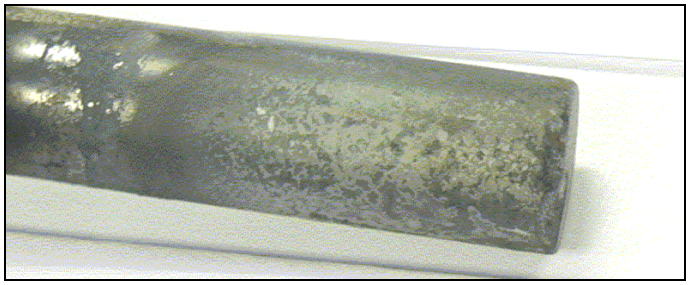


Figure 37. HR-160 Thermowell Element with Minimal Wear Removed after TC23.

Eight ceramic thermowells were tested in TC23. The ceramic thermowells have operated over 2,200 hours and show only minimal wear on the thermowell tips; however, failures incurred during removal of the thermowells continues to be an issue. Figure 38 shows a ceramic thermowell that was broken during removal; however, the tip shown on the right did not show any significant wear.

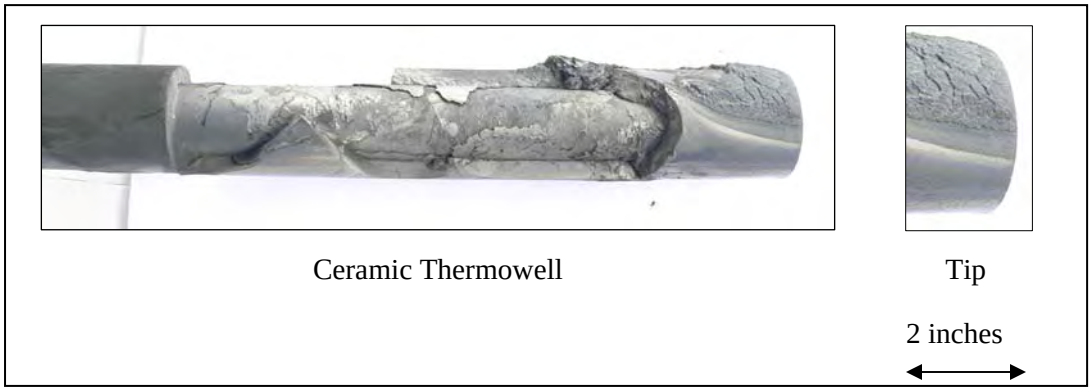


Figure 38. Broken Sheath on Ceramic Thermowell and Thermowell Tip.

Testing of various thermowell materials will continue, but future testing will focus on the HR-160 thermowells since they generally perform acceptably, require little maintenance, and are significantly less expensive than ceramic thermowells. A new material for thermowell application will be tested in future runs as well. This material is a high strength metal alloy suitable for coal gasification application developed by Oak Ridge National Laboratory. Rods of this material, without thermocouples, will be installed in high erosion areas of the gasifier.

2.5 Particulate Collection Device (PCD)

The PCD installation for TC23 included 36 previously exposed iron aluminide (FEAL) elements, 33 previously exposed HR-160 elements, and 3 new Porvair Fecralloy sintered metal fiber elements. Of the installed elements, several were reference elements, i.e. elements which have been subjected to detailed inspection: six Pall FEAL sintered metal powder elements with exposure times ranging from 1,801 to 9,478 hours and two Pall HR-160 sintered metal fiber elements with exposure times of 2,217 hours and 2,526 hours. The monitoring of these reference elements will be continued.

Additionally, the filter element installation included one section of a Pall FEAL element that had been coated with a corrosion resistant material, six Pall FEAL test coupons that were coated with various corrosion resistant materials, and 60 sintered metal powder corrosion test coupons of various materials provided by Mott Corporation. The test coupons were installed on the clean side of PCD tube sheet for exposure to filtered syngas.

Failsafes tested included 34 FEAL fuses, 33 PSDF-designed devices, and 5 internal failsafes made of reversed-media Dynalloy material. One of these Dynalloy failsafes was installed in the failsafe tester for online exposure to unfiltered syngas.

2.5.1 PCD Performance

As in previous test campaigns, the particulate loadings measured at the PCD outlet during TC23 were slightly elevated initially but dropped below the limit of detection after the first six days on coal. The material collected on the outlet sampling filters was a very fine particulate that

appeared to be similar to samples that were previously identified as iron sulfide. The material did not appear to be gasification ash that had penetrated through the PCD. Energy-dispersive X-ray analysis will be performed on the material to determine whether it is the iron sulfide seen in previous tests.

Particle loadings measured at the PCD inlet during TC23 suggested that the solids carryover with the high sodium lignite was about twice the carryover rate measured with the PRB coal used during the initial portion of the run. The higher mass loadings generated with the lignite were partly caused by the higher ash content of the lignite and partly due to the addition of the kaolin to prevent agglomeration. Despite the higher mass loadings with the lignite, the PCD pressure drop increased by only about 20 percent because the transient drag of the lignite ash was about 60 percent lower than the drag of the PRB ash.

Table 2 shows results of the PCD inlet and outlet in situ sampling, which includes particle loading and characteristics.

Table 2. PCD Inlet and Outlet In Situ Sampling Results.

	PCD Inlet						PCD Outlet		
Test Date	Run No.	Particle Loading, ppmw	Particle Loading, lb/hr	Sample L.O.I.,* %	Sample MMD, microns	PCD Drag, **	Run No.	H_2O Vapor, vol %	Particle Loading, ppmw
PRB Combustion									
8/7/07	--	--	--	--	--	--	1	8.3	0.46
PRB Gasification									
8/09/07	1	8820	227	37.1	9.2	95	2	--	0.13
8/10/07	2	8270	206	28.1	14.0	60	3	8.6	1.86(1)
8/11/07	3	8300	211	47.7	12.8	112	4	7.5	0.16
"	4	7940	192	46.1	12.4	90	5	7.4	0.29(1)
8/14/07	--	--	--	--	--	--	6	9.9	0.29(1)
PRB Average		8300	209	39.8	12.1	89		8.4	
Lignite Gasification									
8/15/07	5	18600	404	28.8	12.0	57	7	15.4	0.10
8/16/07	6	23700	532	22.8	12.6	50	8	14.7	<0.10
8/17/07	7	12200	251	10.5	10.5	22	9	17.1	<0.10
8/20/07	8	6400	141	21.8	18.9	102	10	17.8	<0.10
8/21/07	9	19600	500	35.9	13.5	46	11	15.4	<0.10
8/22/07	10	21700	536	34.1	14.7	43	12	14.8	<0.10
8/23/07	11	21700	518	32.3	11.0	40	13	14.6	0.13(2)
8/24/07	12	18900	467	19.2	10.3	56	14	12.7	<0.10
"	13	21400	513	27.4	13.9	56	--	--	--
8/25/07	14	15000	389	32.7	11.5	80	--	--	--
8/27/07	15	9900	261	31.1	12.7	37	15	15.2	<0.10(2)
Lignite Average		17200	410	27.0	12.9	54		15.3	

Notes: 1. Some particles are not believed to be char. No PCD leak is indicated.
2. Failsafe injection test.
*Loss on Ignition
** Units of inwc/(ft/min)/(lb/ft2) - normalized to 70°F and 29.92 inHg.

Figure 39 shows the PCD normalized baseline pressure drop (i.e., the pressure drop following a backpulse cycle, normalized for a constant temperature and face velocity) for the test campaigns performed since the gasifier modifications were completed in 2006. Progression of corrosion of the filter elements may have influenced the pressure drop.

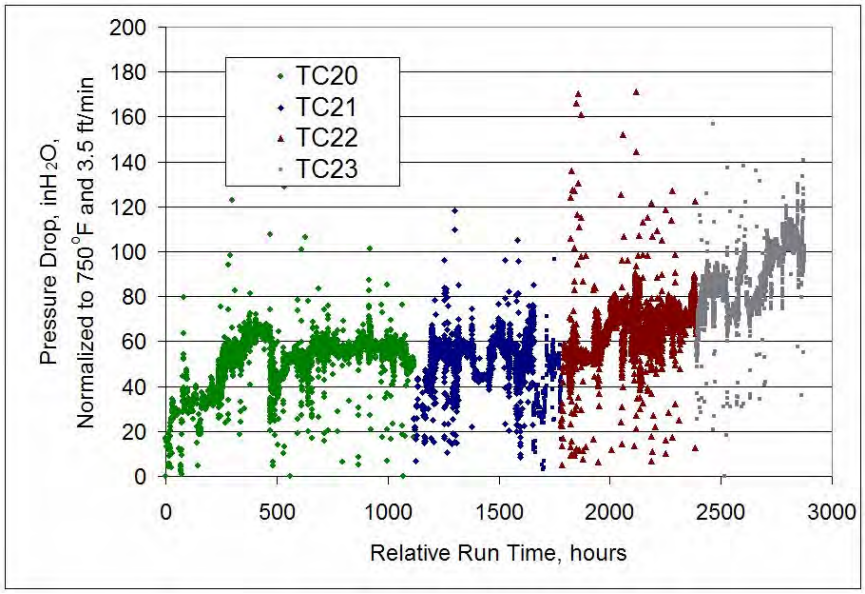


Figure 39. Normalized PCD Baseline Pressure Drop.

2.5.2 PCD Inspections

At the conclusion of TC23, the PCD backpulsing was discontinued one backpulse cycle prior to the termination of coal feed so that the dust cakes formed during lignite operation could be examined. During inspection, both the transient and residual dust cakes were largely intact on the filter elements.

On the top plenum, lumpy dust cakes were noted on the older iron aluminide elements. The cake cross sections had several different layers with a very thin, lighter colored layer on the surface. The lighter colored outer layer may have been material that was carried over after shutdown. On the bottom plenum, the dust cakes on the HR-160 elements were all very smooth. Separate samples of the transient and residual cakes were collected from both the top and bottom plenums. These samples were submitted for analysis of chemical and physical properties.

The thickness measurements made on the top and bottom portion of two HR-160 elements and two iron aluminide elements are summarized in Table 3. On average, the dust cakes were a little thicker on the HR-160 elements, which would be expected from the lower flow resistance of the HR-160 fiber media. The dust cakes were thicker on the bottom than on the top portions of the elements, although this phenomena has not been consistent in previous inspections.

Table 3. Filter Element Transient Dust Cake Thickness Measurements.

Element Location	Element Type	PSDF ID Number	Exposure Hours	Dust Cake Thickness, in	
				Top	Bottom
B8	HR-160	1724	3007	0.1178	0.1379
B14	HR-160	1760	2698	0.1327	0.1453
T8	FEAL	2034	2801	0.0959	0.1037
T14	FEAL	2040	2282	0.1044	0.1307

During inspection, two of the FEAL fuse failsafes were found to be failed. As observed in previous inspections, the fuses broke at the weld between the filter media and the flange, indicating weakness in the weld. One fuse had apparently already fallen into the filter element during operation, while the other snapped off when touched. Microscopic examination showed that very little fresh metal surface was exposed, indicating that the material had begun failing some time ago.

The Fecralloy coupons showed considerable corrosion after 2,200+ hours, but the newer Fecralloy elements (481 hours) did not. Neither the coated FEAL coupons nor the Mott coupons have observable corrosion yet with 481 hours of exposure.

All of the TC23 filter elements were removed from the PCD, and a series of tests were performed on them. Flow resistance tests were performed with the filters in the dirty state (air blown only). These results indicate that the dirty flow resistance of the Pall FEAL filters continues to increase with syngas exposure time. The filters were then pressure washed with water and the clean flow resistance measurements repeated. The clean flow resistance for FEAL filters also continues to increase with age, although not as fast as the dirty resistance. Visual inspection indicates that the corrosion of the FEAL is continuing to seriously affect additional filters as they reach about 5,000 hours of exposure.

The oldest Pall HR-160 filters have reached 3,007 hours of syngas exposure and still do not show obvious corrosion. The dirty flow resistance of these filters appears to also increase with age, but not to the extent of the FEAL filters. After pressure washing, the pressure drop of the HR-160 filters was too low to measure accurately.

Inspection and measurements of flow resistance of all filter elements indicates that corrosion of the FEAL elements is continuing as they age and that flow resistance is increasing with the corrosion. A photograph of the FEAL filter elements that were installed in TC23 is shown in Figure 40. The filters were pressure washed and are arranged in order of exposure age, with the exposure age indicated.

Figure 40. Iron Aluminide Filter Elements in Order of Increasing Exposure Age.

2.6 Advanced Syngas Cleanup

TC23 marked the initial testing of several new syngas cleanup technologies. These included testing of water-gas shift reactions with a catalytic filter element and a fixed bed reactor, direct oxidation of H_2S in a fixed bed reactor, and CO_2 capture in a batch reactor. The initial phase of testing was conducted in offline mode with bottled gases. Observations from the offline and online tests are preliminary and qualitative in nature.

2.6.1 Water-Gas Shift Reaction Testing

For water-gas shift reaction testing, a superheated steam line was installed along with appropriate instrumentation and controls. The catalytic element and the fixed bed were arranged in series, with the outlet stream routed to the CO_2 capture reactor. The approximate composition of the Sud-Chemie T-2822 shift catalyst used for the water-gas shift testing is shown in Table 4.

Table 4. Composition of T-2822 Shift Catalyst.

Component	Weight Percent
Aluminum Oxide	50—70
Magnesium Oxide	15—35
Molybdenum (III) Oxide	5—15
Cobalt Oxide	1—10
Calcium Oxide	3—7

Catalytic Filter Element. For water-gas shift testing, the T-2822 water-gas shift catalyst was pulverized and sieved into a particle size range of 180 to 500 microns for packaging in an iron aluminide filter element. This testing was conducted during the initial portion of TC23 when the gasifier was operating on PRB coal. Testing with the catalytic filter element showed a need for increased catalyst material due to difficulties controlling the low gas flow rate. Pressure typically ranged from 150 to 200 psig, and temperatures varied from 500 to 600ºF, with a short period of testing done at temperatures up to 800ºF. The syngas flow ranged from 6 to 30 lb/hr, and the steam flow was varied from 0 to 6 lb/hr.

Fixed Bed Reactor. Fixed bed reactor testing of water-gas shift was also conducted during gasifier operation with PRB coal and employed the T-2822 catalyst in pellet form. Operating pressures ranged from 110 to 190 psig with temperatures of 550 to 800°F. Syngas flow through the fixed bed reactor ranged from 15 to 30 lb/hr while steam flow rates varied between 0 and 18 lb/hr. Approximately 22 hours of testing with the fixed bed water-gas shift reactor was completed, and this preliminary testing gave results of 40 to 88 percent shift conversion, with higher rates achieved at higher temperatures.

2.6.2 Direct Oxidation of H_2S

Direct oxidation of H_2S was performed offline with bottled gases (nitrogen, hydrogen sulfide, and sulfur dioxide) in preparation for online testing with syngas. The primary objective of this test was to investigate bulk sulfur removal efficiency by converting H_2S at low concentrations to elemental sulfur using sulfur dioxide (SO_2).

The oxidation catalyst used was BASF DD-431 activated alumina in 6 mm diameter spherical form. Offline testing was conducted at 200 psig pressure and gas flow rates of 11.5 to 12 lb/hr. The first test was performed at a reactor bed temperature of about 475ºF and achieved conversions up to about 88 percent. During the second test, the reactor bed temperature was maintained at 417ºF, and the H_2S conversion was slightly higher, at about 93 percent.

TC23 testing of H_2S oxidation employed the same DD-431 catalyst used in offline testing and took place using syngas during the high sodium lignite portion of the test campaign. One objective of the test was to ensure that high H_2S conversions achieved during offline testing could be achieved with actual syngas containing low H_2S concentrations. Another test objective was to observe how a stoichiometric amount of SO_2 selectively reacts with H_2S in the presence of CO and H_2. Levels of COS were monitored to ensure that additional amounts of COS were not formed.

The online testing took place for about 40 hours. Operating conditions included pressures from 110 to 200 psig, temperatures of 260 to 430°F, and a syngas flow rate of 30 lb/hr. Bottle gas containing 1 percent SO_2 and 99 percent N_2 was fed at rates from 0 to 3 lb/hr. During the testing, the reactor inlet concentration of H_2S generally ranged from 0.07 to 0.10 percent on a dry basis.

The first part of the direct oxidation online test was performed at temperatures up to 550ºF. It was observed during this test that H_2S concentration at the outlet of the reactor increased with the increase of SO_2 flow, indicating that SO_2 was reacting with H_2 present in the syngas, forming additional H_2S. No oxidation reaction of H_2S with SO_2 to form elemental sulfur was observed.

The second part of the direct oxidation online test was demonstrated at a lower temperature of about 280ºF. It was noticed in this test that the H_2S concentration at the outlet of the reactor was unchanged, and ammonia present in the syngas was significantly reduced from the reactor inlet to the outlet. Direct oxidation of H_2S was observed only when excess SO_2 flow was used.

2.6.3 CO_2 Capture

For CO_2 capture testing, a syngas line was installed with instrumentation and controls to measure and regulate syngas flow to the CO_2 reactor. Also, a heat exchanger was installed to cool the syngas before it enters the CO_2 reactor.

Carbon dioxide capture from syngas was performed for a few hours with syngas from the fixed bed, water gas shift reactor. The initial commissioning test with syngas was short due to problems associated with the condensate removal system in the inlet syngas line. The batch reactor operated at approximately 110ºF. The limited data showed a decrease in CO_2 concentration.

2.6.4 Inspections

The pulverized Sud-Chemie water gas shift catalyst was removed from the catalytic filter element, and the pelleted shift catalyst was drained from the fixed bed reactor. Both the pulverized catalyst and the pelleted catalyst had changed color from pale green to black. Samples of the used catalysts were sent to Sud-Chemie for analysis.

The BASF DD431 catalyst was also removed from the direct H_2S oxidation fixed bed reactor after about 40 hours of testing. The catalyst shape used was 6 mm diameter spheres and the color had changed from white to grayish black. The used catalyst was sent to BASF for analysis. According to BASF, the sulfur content had increased to about 12 percent, higher than the 3 to 4 percent increase typically seen with oxidation catalysts.

2.7 Recycle Syngas System

The recycle gas compressor is a vertically mounted centrifugal compressor which operates at high temperature (nominally 500 to 600ºF). The compressor was manufactured by Sundyne Corporation, and was designed to have a throughput of nominally 2,000 to 3,000 lb/hr. The

recycle syngas compressor supplied syngas for gasifier aeration for 372 hours during TC23. Figure 41 shows a photograph of the system. The system ran well and experienced no major problems. There was one compressor related trip caused by a faulty vibration probe. The probe was removed from service and will be replaced during the outage. The compressor was restarted within 4 hours of the trip and operated well through the remainder of the test run. Steady state operating conditions at the recycle gas compressor outlet as well as the recycle syngas flow rate to the gasifier are shown in Figure 42. When recycled syngas was used to replace nitrogen aeration, the raw syngas lower heating value increased about 10 percent during both PRB and lignite operations, comparable to previous testing results during PRB operations.

Figure 41. Recycle Syngas Compressor.

The control trip logic for the recycle syngas compressor was modified prior to TC23 to increase the availability of the recycle syngas system. Previously, all gasifier and coal feeder trips would also trip the recycle gas compressor. Generally, coal feeder trips have a short duration, less than 5 minutes, and therefore do not impact operations of the recycle compressor. After completing a thorough design hazard review, the logic was changed to eliminate compressor trips due to coal feeder and certain gasifier trips.

As a result of these modifications, the compressor availability was more than doubled, increasing from about 40 percent in recent test campaigns to over 80 percent in TC23. The higher availability increased test data available for analysis by shortening the time required to re-establish steady state conditions following coal feeder and certain gasifier trips. In addition nitrogen costs were reduced by over $20,000. Based on operations in TC23, a trip condition related to the main air compressor discharge pressure was identified as another trip condition that should be eliminated to further improve availability.

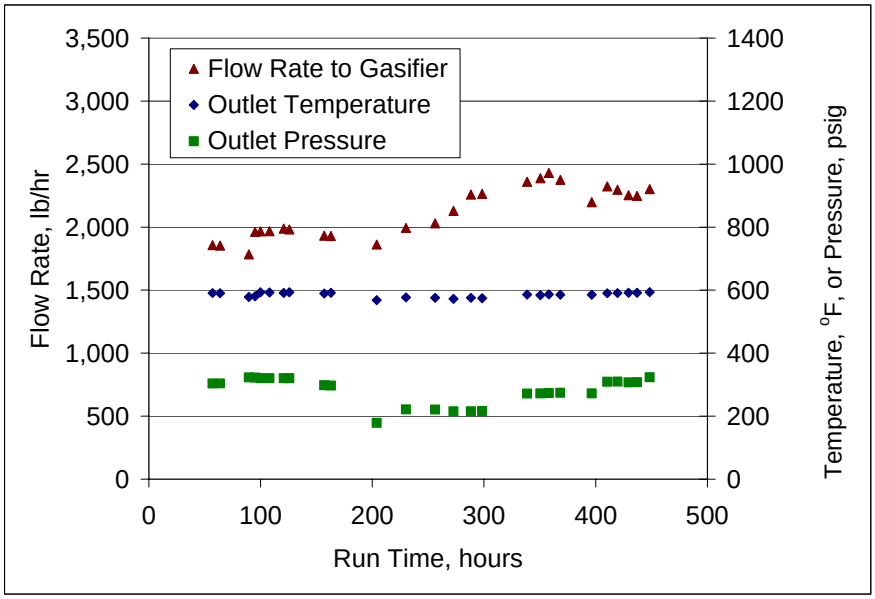


Figure 42. Recycle Syngas Compressor Operating Conditions.

2.8 Air Compliance Testing

Annual air compliance testing was performed during subbituminous coal gasification on August 9, 2007. The testing was conducted in accordance with U.S. Environmental Protection Agency rules and regulations and met the necessary requirements contained in the permit to operate issued by the Alabama Department of Environmental Management. The measurements included particulate matter, sulfur dioxide, nitrogen oxides, carbon monoxide, and volatile organic compounds. Compliance was achieved for all species.

2.9 Slurry Feed System

A slurry feed system was installed to add sand flour to the gasifier to address the agglomeration issues related to the high sodium content in the North Dakota lignite. The slurry feed system consists of a solids feed bin and feed screw, a mix screw, and a progressive cavity pump. The solids feed bin has a hood and vacuum system above the bin to contain the sand flour while being loaded, and the feed system was enclosed in an independent structure. Figure 43 shows the slurry feeder system.

The slurry feed system was commissioned in TC23. Initially the feeder was tested with water and a sand flour and water slurry in an offline mode which allowed for feeding to a receiver vessel that was operated from atmospheric pressure up to 200 psig. The slurry concentrations ranged from 45 to 55 weight percent at flow rates of 100 to 150 lb/hr. Feed screw stalling problems were encountered after transitioning from water to the slurry mix. A larger air motor was added, and the dry box between the feed and mix screws were redesigned to improve feed system operations.

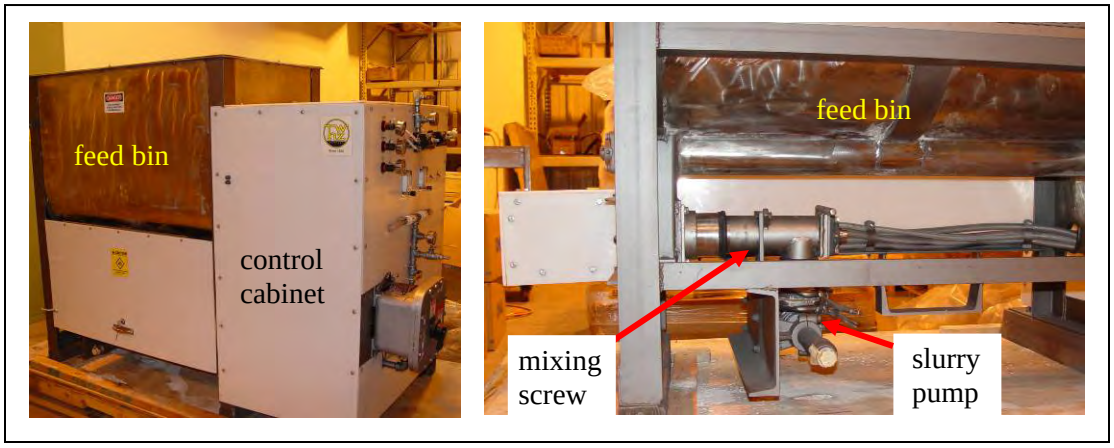


Figure 43. Slurry Feeder System.

After completion of offline testing, the feeder was lined up to the gasifier through the new dispersing nozzles. The feeder was started on water and the safety interlocks and control logic were successfully tested. However, the feeder could not maintain a discharge pressure higher than gasifier pressure. Initial investigation revealed that the rubber stator and stainless steel rotor of the pump need to be replaced. As a result, testing was suspended.

3.0 CONCLUSIONS

Test campaign TC23 resulted in successful gasification operation with PRB and high sodium lignite, with sufficiently high carbon conversions and syngas heating values adequate for gas turbine operation. The use of the kaolin additive successfully prevented agglomeration problems in the gasifier. However, fine kaolin particles may have contributed to deposition in the primary gas cooler. Preliminary results of parametric testing showed effects of temperature, pressure, coal feed rates, steam-to-coal ratios and standpipe level on gasifier operations. Additional testing was continued with sensor development, PCD operations, advanced syngas cleanup, and supporting equipment. Final results of TC23 testing will be presented in the Gasification Test Campaign TC23 Topical Report.

Bibliography

(1) Kosminski, A., D.P. Ross, J.B. Agnew. "Reactions between sodium and kaolin during gasification of low-rank coal" *Fuel Processing Technology*, vol. 87, pp. 1051-1062 (2006).

APPENDIX 1

Steady State Operating Periods and Conditions

Operating Period	End Time	Duration, hours	Run Hour	Gasifier Outlet Temperature, °F	Gasifier Outlet Pressure, psig	Coal Feed Rate, lb/hr	Air Rate, lb/hr	Steam Rate, lb/hr	Recycle Syngas Rate, lbhr	Kaolin Feed Rate, lb/hr	Nitrogen Rate, lb/hr	Syngas Rate, lb/hr	Standpipe Solids Removal Rate, lb/hr	PCD Solids Rate, lb/hr
TC23-1	8/8/07 20:15	4.0	26	1,667	190	3,611	12,215	1,103	0	0	6,878	24,393	18	227
TC23-2	8/10/07 3:00	4.0	57	1,683	200	3,779	13,094	941	1,857	0	4,959	25,983	61	210
TC23-3	8/10/07 11:00	6.5	64	1,688	200	3,776	13,184	895	1,853	0	4,749	25,805	53	223
TC23-4	8/11/07 11:00	3.0	89	1,680	176	4,066	13,345	531	1,784	0	6,819	27,733	99	227
TC23-5	8/11/07 17:15	4.3	95	1,701	208	3,597	12,673	456	1,962	0	6,744	26,646	86	205
TC23-6	8/11/07 22:15	4.7	100	1,688	208	3,695	12,605	438	1,967	0	4,856	24,739	82	227
TC23-7	8/12/07 9:00	10.0	108	1,695	208	3,662	12,851	454	1,968	0	4,923	25,099	72	242
TC23-8	8/12/07 18:30	3.5	121	1,687	208	3,582	12,496	513	1,988	0	4,846	24,680	86	206
TC23-9	8/12/07 23:45	4.0	126	1,700	208	3,746	12,842	511	1,981	0	4,889	25,123	80	228
TC23-10	8/13/07 17:15	4.5	143	1,690	190	2,188	9,809	534	0	0	7,210	21,264	45	126
TC23-11	8/14/07 7:00	4.0	157	1,709	190	2,212	9,838	545	1,932	0	5,120	21,194	14	127
TC23-12	8/14/07 13:30	5.0	163	1,715	190	2,270	10,002	548	1,930	0	4,930	21,133	6	131
TC23-13	8/16/07 6:15	4.5	204	1,532	130	3,304	9,695	1,231	1,862	300	5,163	22,643	285	429
TC23-14	8/17/07 7:30	2.5	230	1,559	150	2,162	8,810	1,264	1,994	112	5,062	22,067	58	281
TC23-15	8/18/07 11:45	7.0	256	1,551	150	2,085	8,367	1,261	2,030	134	4,704	20,990	84	271
TC23-16	8/19/07 3:15	5.3	273	1,547	150	2,121	8,563	1,239	2,130	97	4,798	21,616	0	288
TC23-17	8/19/07 18:30	4.3	288	1,534	156	2,676	9,038	1,206	2,259	106	4,608	22,607	0	361
TC23-18	8/20/07 4:15	4.0	298	1,513	156	2,892	9,023	1,223	2,264	104	4,650	22,680	0	389
TC23-19	8/21/07 21:30	5.8	339	1,531	190	3,638	10,735	1,575	2,361	127	5,156	25,947	0	489
TC23-20	8/22/07 11:15	9.2	351	1,526	190	3,455	10,499	1,578	2,389	128	5,061	25,437	0	465
TC23-21	8/22/07 16:45	5.3	358	1,521	190	3,293	10,192	1,572	2,430	139	5,069	24,942	0	445
TC23-22	8/23/07 3:00	5.3	368	1,538	190	3,391	10,604	1,579	2,375	134	5,182	25,554	0	457
TC23-23	8/24/07 6:30	4.0	396	1,582	190	3,802	11,503	1,590	2,198	325	4,898	26,114	0	536
TC23-24	8/24/07 22:30	8.3	410	1,592	210	3,581	11,281	1,308	2,324	386	4,557	25,031	350	482
TC23-25	8/25/07 6:15	5.5	419	1,591	210	3,539	11,187	1,318	2,296	361	4,555	25,019	343	426
TC23-26	8/25/07 15:30	4.3	429	1,595	210	3,902	11,792	1,330	2,254	371	4,531	25,826	425	378
TC23-27	8/25/07 23:15	4.5	437	1,582	210	3,890	11,691	1,337	2,247	362	4,676	25,813	432	359
TC23-28	8/26/07 10:15	4.0	448	1,584	220	3,700	11,558	1,274	2,302	341	4,783	25,516	419	332

Test Periods TC23-1 to TC23-12 occurred with PRB while TC23-13 to TC23-28 were performed with high sodium lignite.

Note that the standpipe solids removal rates are preliminary and will be finalized when the fuel and fine ash samples analyses is received.

APPENDIX 2

Syngas Ammonia, Benzene, Naphthalene, and Phenanthrene Concentrations

Average Time	Run Time, hours	Coal Type	Ammonia, ppm	Benzene, ppm	Naphthalene, ppm	Phenanthrene, ppm
8/9/2007 12:06	44	PRB	1,691	523	137	209
8/11/2007 8:53	89	PRB	1,800	687	167	260
8/23/2007 9:17	377	lignite	1,579	567	153	*
8/24/2007 15:57	408	lignite	1,576	496	97	117
8/24/2007 16:39	409	lignite	1,571	533	99	119
8/25/2007 10:25	426	lignite	1,642	528	120	150

*** No data available.**

APPENDIX 3

Syngas H_2S, Syngas COS, and Syngas Sulfur Converted toCOS

Average Time	Run Time, hours	H_2S, ppm	COS, ppm	Syngas Sulfur as COS, percent
8/20/2007 13:00	309	889	17	1.9
8/20/2007 14:05	310	770	21	2.7
8/20/2007 14:55	311	729	26	3.5
8/20/2007 15:35	312	760	21	2.7
8/21/2007 8:45	329	715	41	5.5
8/21/2007 9:30	330	662	50	7.0
8/21/2007 9:55	330	675	52	7.2
8/21/2007 10:20	330	733	57	7.2

All data was taken during high sodium lignite operations.