The National Carbon Capture Center
at the Power Systems Development Facility

Test Run Summary Report

Test Run R12
November 20, 2013 – December 13, 2013

DOE Cooperative Agreement
DE-NT0000749

Prepared by:

Southern Company Services, Inc.
Power Systems Development Facility
P.O. Box 1069, Wilsonville, AL 35186
Phone: 205-670-5840
Fax: 205-670-5843
http://www.NationalCarbonCaptureCenter.com

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List of Abbreviations

CCAD	Continuous Coarse Ash Depressurization
DEPG	Dimethyl Ether of Polyethylene Glycol
DOE	Department of Energy
HSX	Hybrid Siloxane
MTR	Membrane Technology & Research
NCCC	National Carbon Capture Center
NETL	National Energy Technology Laboratory
OSU	Ohio State University
PDAC	Pressure Decoupled Advanced Coal
PDMS	Polydimethyl Siloxane
PRB	Powder River Basin
R01 through R12	Test Runs 1 through 12
SCL	Syngas Chemical Looping
WPI	Worcester Polytechnic Institute

1.0 EXECUTIVE SUMMARY

The National Carbon Capture Center (NCCC) at the Power Systems Development Facility supports the Department of Energy (DOE) goal of promoting the United States' energy security through reliable, clean, and affordable energy produced from coal. As part of its mission to develop technologies for clean coal power generation, the NCCC conducts testing of advanced gasification and CO_2 capture processes.

Gasification run R12 commenced from November 20 through December 13, for 516 hours of on-coal operation. The run was stable and demonstrated high carbon conversion while using Powder River Basin (PRB) coal as the feedstock. Test objectives related to gasification technologies included further development and evaluation of coal feeders, gasifier automatic controls, hot gas filter components, and water-gas shift and COS hydrolysis catalysts. In addition, initial commissioning of equipment in the Ohio State University (OSU) Syngas Chemical Looping (SCL) process began during the run. Test objectives involving pre-combustion CO_2 capture technologies consisted of operation of gas separation membranes from Worcester Polytechnic Institute (WPI) and Membrane Technology & Research (MTR), and a CO_2 solvent from the DOE's National Energy Technology Laboratory (NETL).

2.0 GASIFICATION TECHNOLOGIES

2.1 Coal Preparation and Feed

Table 1 lists the as-received and as-fed properties of the PRB coal used in R12.

Table 1. R12 PRB Coal Properties

Coal Property	
As-Received Carbon, wt %	53.88
As-Received Hydrogen, wt %	3.93
As-Received Nitrogen, wt %	0.77
As-Received Sulfur, wt %	0.33
As-Received Ash, wt %	8.78
As-Received Oxygen, wt % (by difference)	16.33
As-Received Volatiles, wt %	45
As-Received Fixed Carbon, wt %	30.2
As-Received Heating Value, Btu/lb	10,900
As-Received Moisture Concentration, wt%	19.97
As-Fed Moisture Concentration, wt%	18.35
Moisture Content Reduction, %	6.23
As-Fed Mass Median Diameter, micron	278
As-Fed Oversize (>1,180 micron) Content, wt%	36

Both the original coal feeder and the developmental Pressure Decoupled Advanced Coal (PDAC) feeder operated consistently in R12 without any forced downtime. The original feeder operated with flow rates ranging from 1,000 to 2,500 lb/hr. The PDAC feeder operated at flow rates ranging from 1,000 to 5,000 lb/hr, and the PDAC trim logic controller maintained a consistent feed rate by varying conveying nitrogen.

2.2 Transport Gasifier

R12 consisted of 44 steady state periods. Figure 1 plots the average steady state operating parameters for the run. Steady state carbon conversion ranged from 98.4 to 99.9 percent, averaging 98.8 percent. During part of the run, from about hours 180 to 280, a tube leak in the primary gas cooler precluded operation that was sufficiently consistent for meeting the criteria for steady state data. Throughout the run, the gasifier automated temperature control maintained the gasifier outlet temperature to within 5°F of setpoint.

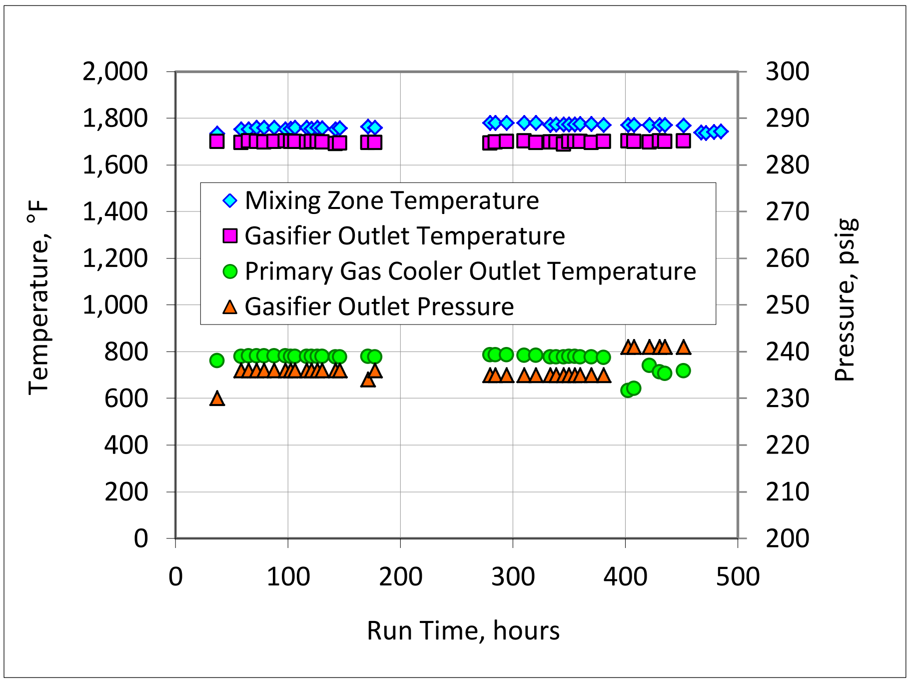

Figure 1. Steady State Operating Conditions

2.3 Particulate Control Device

The particulate control device operated with excellent filtration performance during the run, with particulate outlet loading consistently below the sampling detection limit of 0.1 ppmw. Long-term material evaluation continued for the hot gas filter elements and failsafes. Some of the older Iron Aluminide elements were removed from service due to high pressure drop observed with post-run, cold-flow testing.

2.4 Water-Gas Shift and COS Hydrolysis Catalysts

Testing was conducted with the previously tested water-gas shift catalyst and a new COS hydrolysis catalyst with a honeycomb configuration from the same developer. Parametric testing for the hydrolysis catalyst included varying pressure, temperature, and steam addition, and gas analysis included syngas concentrations of COS, hydrogen cyanide, and H_2O at the reactor inlet and outlet. The two catalysts were tested around 440 hours each during the run.

2.5 Ohio State University Syngas Chemical Looping

During the run, multiple tasks were performed for the OSU SCL pilot project. Commissioning work completed involved instrumentation and the programmable logic controller. The NCCC supported various activities in the commissioning phase, performing a pre-startup safety inspection with OSU and identifying final construction items. NCCC engineers also assisted with utility checkouts on the unit, and in coordination with OSU, identified utility process flow rates and checked out safety trips and interlocks for each system.

3.0 PRE-COMBUSTION CO_2 CAPTURE TECHNOLOGIES

3.1 Worcester Polytechnic Institute Membrane

WPI tested a palladium-alloy hydrogen membrane tube in R12. The system was heated to the operating temperature of 840^{o}F in a nitrogen atmosphere. Once the membrane reached operating temperature, bottle hydrogen was introduced into the feed stream to achieve 26 vol% hydrogen concentration. Membrane performance data collected while operating with the hydrogen/nitrogen atmosphere served as baseline data. On November 26, syngas was introduced into the membrane module while hydrogen enrichment continued. This resulted in hydrogen concentration of 38 vol% in the feed.

Initially, the membrane produced high purity hydrogen as in previous tests. However, the membrane began to show signs of leaks beginning on November 28 as evidenced by a reduction of hydrogen purity in the permeate stream. Testing of the first membrane was terminated on December 5 after 336 hours of testing, including operation with hydrogen/nitrogen and with hydrogen/syngas. WPI shipped a second membrane tube which was installed on December 6, and testing resumed on the same day initially with hydrogen/nitrogen and later with hydrogen/syngas. During this period of operation, no leaks occurred, and the membrane showed stable performance with greater hydrogen product purity than that from first membrane. Testing concluded on December 13 when the R12 run ended. The second membrane operated for a total of 154 hours, including operation with hydrogen/nitrogen and with hydrogen/syngas. Both membranes were returned to WPI.

3.2 Membrane Technology & Research Membrane System

MTR has been testing its polymeric hydrogen membrane from runs R03 through R10 using either a 1-lb/hr stamp cell or a 10-lb/hr-scale spiral-wound module, both operating at 300^{o}F.

Positive results from this testing have led to MTR's decision to scale up membrane testing to 50 lb/hr using a commercially representative manufacturing process producing a four-inch standard module.

To test this scaled-up hydrogen membrane, MTR initiated modifications to the existing 50 lb/hr CO_2 membrane skid at NCCC for higher temperature operation. The modifications included the additions of new syngas heater, heat tracing to keep the module and inlet, residual, and permeate streams at operating temperature, and new instrumentation. All these modifications were completed in early November. The hydrogen skid temperature was initially set at 200°F, to be gradually increased to 300°F over the course of testing.

Testing began on November 21 as shifted syngas was introduced to the hydrogen membrane skid. A few hours into operation, data indicated a blockage on a welded check valve located on the permeate stream. This caused high permeate pressure and led to reduced membrane hydrogen separation performance. The system was shut down, and the check valve was replaced with NCCC's assistance. Following the valve replacement, testing resumed, and the permeate pressure was reduced from 16 to 10 psig. Two identical hydrogen membrane modules were tested for a combined 417 hours during the run, at temperatures ranging from 200 to 300°F. The membranes enriched hydrogen concentration from 10 to 14 percent in the feed gas to 22 to 34 percent in permeate stream, depending on the temperature. Although the performance was stable, the hydrogen separation was lower than expected based on results from the previous stamp cell testing. Modifications will be made prior to further testing for improved temperature control, which is expected to improve membrane performance. Both hydrogen membrane modules were returned to MTR for post-run analysis.

3.3 CO_2 Solvent Batch Reactor Testing

Testing of the hybrid siloxane (HSX) physical solvent supplied by NETL started in the bench-scale batch reactor using bottled gases. The capacity of the solvent was determined at 70°F with 35 percent CO_2 in nitrogen and 165 and 315 psia (CO_2 partial pressure of 50 and 100 psi). The data are plotted in Figure 2 along with data at 70°F for dimethyl ether of polyethylene glycol (DEPG) and polydimethyl siloxane (PDMS) collected in previous test runs. For HSX, the two high points at about 100 psi were measured after the regenerated solvent sat overnight, while the lower one was run immediately after regeneration. This may indicate a slow release of CO_2 similar to that found when testing the University of Alabama solvent.

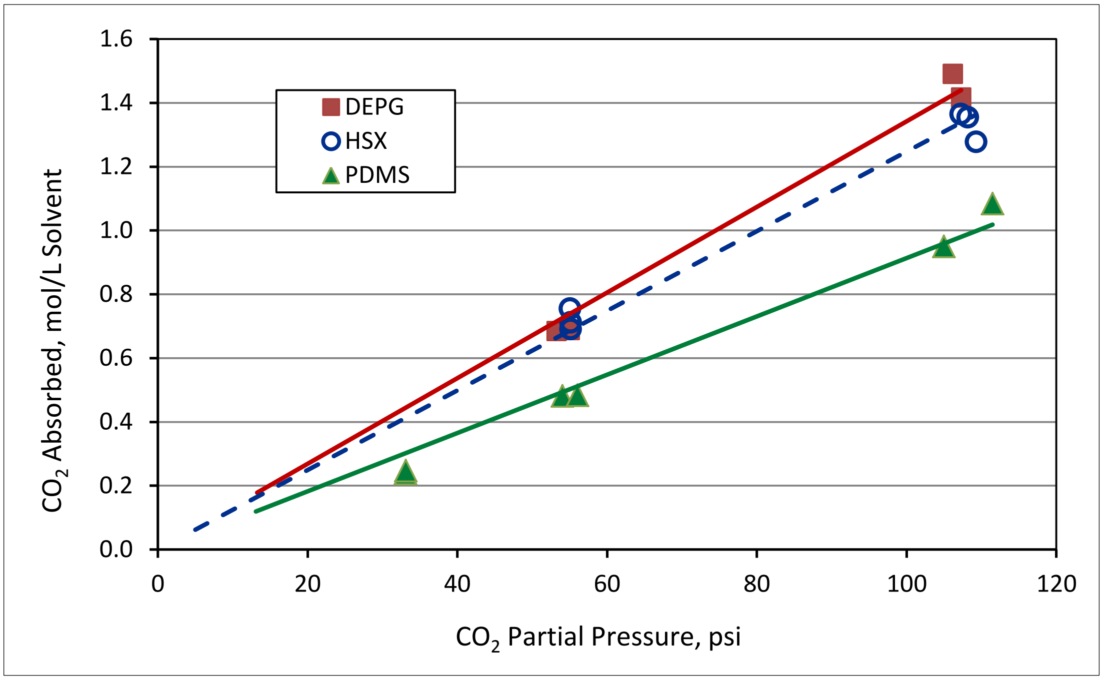

Figure 2. Absorption of CO_2 for Hybrid Siloxane, DEPG, and PDMS Solvents

Liquid collected in the exit piping indicated that measurable amounts of solvent were carried over during absorption. This has not been the case for other solvents tested. To desorb the solvent, the pressure was reduced with the stirrer operating. The solvent was then poured into a container to determine solvent losses during the absorption/regeneration process. When poured into the container, about one inch of foam was generated on the surface of the solvent, indicating that additional CO_2 was being released. Foaming has not been observed with other solvents. Additional tests were planned to determine CO_2 capacity at 100°F, as well as H_2S absorption capacity.