28

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DEVELOPMENT OF AN ADVANCED, CONTINUOUS MILD GASIFICATION PROCESS FOR THE PRODUCTION OF CO-PRODUCTS

Quarterly Technical Progress Report
for the Period April - June 1990

by

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1.0 INTRODUCTION

Work performed during the quarter included the refractory cure of the carbonizer, and then shakedown of the carbonizer, water quench system, and char removal system. Construction continued on the tar/oil quench system. Sulfur capture tests carried out at AMAX included iron oxide sorbent scouting studies.

2.0 PDU CONSTRUCTION

Construction consisted mainly of placing peripheral systems and instruction into place.

2.1 Area 100-200 - Coal Preparation-Utilities

A low pressure coal hopper was constructed and placed above the pressurized hopper. This will enable operation for 24 hours before refilling the hopper is necessary. The hopper was added due to the difficulty of changing the feed hopper and char hoppers on every shift.

2.2 Area 300-400 Carbonization-Calcination

New distributor caps were made for the carbonizer because during the curing of the reactor, it was discovered the caps were made from an "L" grade stainless steel. The caps were replaced with Haynes alloy C-276. Also, the top 4 inches of the pneumatic coal feed tube (which is exposed to the highest reactor temperature) was replaced with the alloy due to breakdown of the material during the cure.

2.3 Area 500 - Gas Quench & Liquid Separation

The gas piping from the exit of the oil sieve column to the second water scrubber was modified to enable the temperature of the gas stream to be dropped as low as possible. Also, a demister was added prior to the product gas meter and flare. All remaining heat tracing had been installed. The capacitor probes for measuring liquid level in the second venturi scrubber had been removed due to the coating of tar that built up on the devices. The tar nuclear source was moved to this scrubber. The tar venturi and Water Scrubber 1 will share a nuclear source. Modifications have been made to allow movement of the source and detector in a matter of minutes.

3.0 PDU REACTOR CURE AND SHAKEDOWN TESTS

3.1 Refractory Cure

The refractory in the carbonizer and calciner plenums was cured in separate steps using a natural gas burner. Each cure took approximately 40 hours, and involved raising the gas temperature inside the refractory from ambient to 2200°F (1200°C) in several ramp and hold steps. After the plenums were cured, they were placed on the bottom of the reactors. The two high temperature hoppers were then individually cured along with the char transfer piping.

The carbonizer was cured using the main natural gas burner. The product gases were vented through the rupture disk vent line. After curing, the plenum and distributor plate were removed and the reactor was inspected for defects in the refractory. It was observed that the distributor caps were completely carbonized. Apparently, the caps were made of "L" grade stainless steel. New replacement caps were made. Cracks had also developed around the refractory surrounding the water-cooled pneumatic coal feed line. The refractory was removed and replaced. This soft, high temperature refractory, which is present as a small cone in the center of the plenum, may require replacement over the course of many hours of operation.

After inspection was complete, the carbonizer was reassembled and pressure checked. No leaks were found in the vessel. Several of the knife valves, however, did leak when pressures of about 15 psi were achieved. Since all of the runs are scheduled to be performed at low pressures, these valves are satisfactory.

3.2 Carbonizer Shakedown Tests

Three tests were conducted to shakedown the carbonizer/water quench system. The first test was performed with hot gas, the second with petroleum coke, and the third with Wyodak coal.

The hot gas test included shaking down: the carbonizer, carbonizer pressure purges, main reactor secondary heaters, pneumatic coal feed system, gas transport system and heater, primary and secondary cyclone heaters, overhead gas line heater, the two water scrubber quench systems, flare, and the data acquisition and control system; and determining the stoichiometric operation parameters for the natural gas burner, temperature loss in the freeboard, and the heat loss associated with the primary and secondary cyclone heaters. The test was conducted over two 24-hour periods with the system operating without any major problems.

Temperature at the bottom of the reactor was maintained at approximately 1100 to 1200°F, with the gas exiting the freeboard at 800°F. The temperature dropped to 600°F at the entrance to the first water scrubber. This was lower than the design temperature of 700-900°F, but was due to not having coal in the reactor. Water Scrubber 1 cooled the gas from 600°F to between 60 and 180°F, and maintained the desired setpoint. Water from combustion of the natural gas was removed in this unit operation and pumped down to the temporary storage tank. Water Scrubber 2 cooled the gas down to 60°F. The product gas was then piped to the flare.

The second test involved feeding petroleum coke into the reactor. This test was performed to shakedown the pneumatic coal feed system, the char removal system, and the primary and secondary cyclone particulate removal systems. Small amounts of tar were observed in the wastewater produced from this test, which was conducted over a 12-hour period with no major problems.

The third and last test involved feeding Wyodak coal into the carbonizer. An 8-hour test was conducted with a normal shutdown. The reactor only reached a temperature of 750°F, due to the high heat load generated by endothermic coal devolatilization reactions. The burner was unable to supply more heat because of inappropriate flow instrumentation settings. These settings were corrected after shutdown, and higher temperature runs (1100-1300°F) were conducted at later dates. The heat loss across the freeboard dropped to 50-100°F. The char removal system worked satisfactorily. Due to the low reaction temperatures, the char volatile content only dropped from 32% to 20%.

The run was halted after 8 hours because of cooling capacity loss in the two water quench venturis. The exit temperature from Water Scrubber 2 rose to 180-200°F. This was accompanied by an oily mist spraying from the flare. Upon inspection of the water injection system, it was discovered that the nozzle selected for water injection was prone to plugging. Also, a cylindrical "dirty" tar ring had formed around the vortex finder of the first venturi cyclone.

The capacitance probes used in Water Scrubber 2 became caked with tar and did not give a true water level reading. Due to an insufficient reservoir size (caused by the caking tar) the venturi ran dry and could not cool the product gas.

The backup coolant injection system was installed and a nuclear level gauge was put on the second venturi scrubber. Also, a demister was placed prior to the product gas meter and flare. Another backup coolant system was added so that makeup water could be added to the scrubber system from the control room if necessary. It was also determined during the tests that under certain gas flow rates, recycle coolant flow rates, and product gas temperatures, water became entrained in the product gas and would come out in the product gas lines further downstream in the process. After varying operational parameters, these flow and temperature regimes have been defined sufficiently to enable continuous operation.

All products were stored on site for the duration of the quarter to enable product separations testing during the next quarter.

4.0 CHAR UPGRADING

4.1 Iron Oxide Sorbent for Sulfur Capture

Scouting tests to determine the effect of the iron-to-coal ratio on sulfur capture and char upgrading were resumed during May. Indiana No. 3 coal from the AMAX Chinook Mine washing plant was subjected to batch tests at 1020°F (550°C) with and without iron oxide additions. Coal and iron oxide extrudates (prepared from Ruthner process iron oxide powder) were sized to 14 x 100 mesh prior to testing. Iron additions equivalent to 0, 1, 2, and 4 times the theoretical stoichiometric requirement to convert all coal sulfur to FeS were utilized. Samples were heated to 1020°F (550°C) and held at that temperature for 0.5 hours. Tests were performed in 100-ml quartz crucibles with lids. A nitrogen purge was maintained in the furnace during the tests.

After cooling, agglomerates were crushed to pass 14 mesh and magnetic separations were performed. The results of proximate and sulfur forms analysis of the char/iron oxide agglomerates are shown in Table 1. The Chinook feed coal for these tests contained about 3.8% sulfur, which is somewhat lower than the sulfur contents of other similar feeds used in the past. During this test series, a clean char with a sulfur content of about 1.6% was obtained using an addition of iron oxide to the feed coal and a magnetic separation on the product char. A clean char containing about 1.8% sulfur was obtained in the non-magnetic fraction of char produced without iron oxide addition. These results show only a small incentive to add iron oxide to the feed coal in terms of producing a low-sulfur product char.

However, sulfur retention in the char is increased by iron oxide additions. Based on the product weights and sulfur analyses, about 45% of the sulfur in the feed coal is retained in the char (before magnetic separation) without iron addition. The iron additions increased sulfur retention by the char to the range of about 65-75% of that in the feed coal.

Permanent roll magnetic separation tests were performed on the feed coal and on two chars produced under the same conditions as those shown in Table 1. Chars produced without iron oxide and with twice the stoichiometric ratio of required iron oxide were used. The permanent roll magnetic separator exhibits significantly greater field strength than the hand magnet used for previous scouting tests. Table 2 compares the results obtained using the permanent roll magnetic separator and the hand magnet. Proximate and sulfur analyses are pending.

The effect of removing the magnetic fraction from the 14 x 50 mesh fraction of the feed coal will be determined. A batch laboratory test to produce char at 1020°F (550°C) without iron addition will be performed. A magnetic separation of the char will also be performed.

4.2 Product Char Characterization

A sample of Wyodak coal char, produced in the 4-lb/hr mild gasification unit at UNDEERC, was received by AMAX R&D for evaluation. The proximate analysis of the char is shown in Table 3, which also shows proximate and ultimate analysis data obtained at UNDEERC. Ultimate and sulfur forms analyses are pending. The char is also under evaluation for applicability as a feedstock for the AMAX Char-to-Carbon process.

TABLE 1

EFFECT OF RUTHNER IRON OXIDE ADDITIONS ON INDIANA NO. 3 CHAR PROPERTIES

		Recovery, Wt% (dry basis)		Analysis, % (dry basis)					
Treatment	Fraction	As-Is Char	Based on Feed Coal	Ash	Vol.	S(tot)	S(pyr)	$S(SO_4)$	S(org)
None	Feed Coal	---	---	12.16	37.27	3.82	0.93	1.05	1.85
No Iron Addition; 1020°F (550°C) Char	Non-mags	45.3	31.2	15.45	12.04	1.83	0.09	0.16	1.59
	Magnetics	54.7	---	21.81	11.87	3.08	0.38	0.29	2.41
	Calc Char	100.0	---	18.93	11.95	2.52	0.25	0.23	2.04
1x Stoich. Fe_2O_3 addn; 1020°F (550°C) Char	Non-mags	47.7	38.4	26.79	7.70	1.64	0.02	0.06	1.56
	Magnetics	52.3	---	45.08	16.25	4.75	0.15	0.19	4.42
	Calc Char	100.0	---	36.35	12.17	3.26	0.09	0.13	3.05
2x Stoich. Fe_2O_3 addn; 1020°F (550°C) Char	Non-mags	28.0	25.5	15.05	10.97	1.74	0.04	0.05	1.66
	Magnetics	72.0	---	44.03	17.14	3.58	0.09	0.23	3.26
	Calc Char	100.0	---	35.93	15.41	3.07	0.08	0.18	2.81
4x Stoich. Fe_2O_3 addn; 1020°F (550°C) Char	Non-mags	24.6	26.8	13.30	10.97	1.67	0.05	0.06	1.56
	Magnetics	75.4	---	72.61	29.47	2.49	0.23	0.33	1.93
	Calc Char	100.0	---	58.01	24.92	2.29	0.18	0.26	1.84

Note: 14 x 100 mesh Indiana No. 3 coal used for each test.
Ruthner iron oxide extrudes crushed to 14 x 100 mesh prior to addition.
30-min residence time used in static system (purged w/N_2 first).
Calc Char values are for char including Fe addition.

TABLE 2

COMPARISON OF PERMANENT ROLL MAGNETIC SEPARATION AND HAND MAGNET TEST RESULTS

		Weight Recovery, % (dry basis)	
Treatment	Fraction	Perm Roll	Hand Magnet
None (Feed Coal; 14 x 50M)	Non-mags	85.1	100
	Magnetics	14.9	0
None (Feed Coal; 50 x 10M)	Non-mags	31.7	100
	Magnetics	68.3	0
No Iron Addition; 1020°F (550°C) Char	Non-mags	22.8	45.3
	Magnetics	77.2	54.7
2x Stoich. Fe_2O_3; 1020°F (550°C) Char	Non-mags	3.0	28.0
	Magnetics	97.0	72.0

TABLE 3

PROXIMATE ANALYSIS OF WYODAK COAL MILD GASIFICATION CHAR (WT%)

	AMAX	EERC
Moisture	0.00	0.00
Ash	10.53	10.53
Volatile Matter	12.25	15.58
Fixed Carbon	77.22	73.89
Hydrogen		2.26
Carbon		80.41
Nitrogen		1.20
Sulfur		0.50
Oxgen (by difference)		5.10
Ash		10.53

END

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