FE-2247-14
Distribution Category UC-90d

GASIFICATION OF RESIDUAL MATERIALS FROM COAL LIQUEFACTION

Type II Preliminary Pilot Plant Evaluation of SRC-II Vacuum Flash Drum Bottoms

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Date Published - May, 1978

PREPARED FOR THE UNITED STATES
DEPARTMENT OF ENERGY

Under Contract No. EX-76-C-01-2247

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ABSTRACT

About 3 3/4 tons of SRC-II Vacuum Flash Drum Bottoms from the liquefaction of Kentucky coal were successfully gasified in a pilot plant at Texaco's Montebello Research Laboratory under DOE contract EX-76-C-01-2247.

A 98 percent conversion of the carbon in the feed to syngas was achieved yielding 30.4 SCF of dry syngas per pound of residue charged. The dry syngas contained over 93 volume percent carbon monoxide and hydrogen.

The short, 7.3 hour, pilot plant run confirmed the operability of the Texaco Coal Gasification Process with this feedstock and the data obtained confirm our earlier predictions of performance efficiency.

INTRODUCTION

Objective

The objective of a Type II Preliminary Pilot Plant Evaluation is to confirm the operability of the Texaco Coal Gasification Process on candidate feedstocks selected by DOE from various residual materials from DOE-sponsored coal liquefaction projects. These short pilot plant evaluations will permit refining the estimates of preferred processing conditions, product gas yield and composition and will identify unexpected operating problems.

Up to 20 barrels (a total of 8000 pounds) of each candidate feedstock will be charged to the Texaco pilot plant gasifier at a rate between 600 and 1000 pounds per hour, depending on the feedstock properties, and with proper ratio of oxygen and steam determined from Texaco correlations.

Background

Almost all coal liquefaction processes, which are being developed to reduce our dependence on foreign oil, require hydrogen or synthesis gas (a mixture of hydrogen and carbon monoxide) to solubilize the coal. In order to obtain a favorable product yield in such a coal liquefaction plant it is desirable to produce the needed hydrogen or synthesis gas primarily from the non-liquefied fraction of the coal. This material, together with the inorganic ash and some fraction of the converted coal, may be recovered in various forms depending on the particular process. Many of these streams will make excellent feedstocks for gasification using the Texaco Coal Gasification Process to produce synthesis gas or hydrogen.

Texaco developed the non-catalytic partial oxidation process in the late 1940's to convert natural gas to synthesis gas which was then reacted with steam to form additional hydrogen. Further developments enabled the use of light oils, residual oils, and asphalts as feedstocks. Recently, Texaco has carried out work that has demonstrated the feasibility of gasifying coal-water slurries.

The Texaco Synthesis Gas Generation Process has been licensed for use in more than 70 plants in over 20 countries throughout the world using a variety of liquid feedstocks.

The process which has been modified to handle high ash feedstocks such as coal and coal liquefaction residues has been designated the Texaco Coal Gasification Process.

Exploratory pilot plant runs conducted in the summer of 1975 demonstrated the feasibility of gasifying pumpable coal derived residues which contained as much as 28 percent ash. Early in 1976, under contract with the Electric Power Research Institute, Inc. (EPRI), approximately 40 tons each of two high ash coal liquefaction residues were successfully gasified. The residues were obtained from the H-Coal liquefaction of Wyodak and Illinois No. 6 coals and contained

11 percent and 20 percent ash respectively.

To determine the suitability of various residual materials from DOE-sponsored coal liquefaction projects as feedstocks to the Texaco Coal Gasification Process, DOE currently is sponsoring a series of tests to be conducted at Texaco's Montebello Research Laboratory.

Scope

This report covers work performed at Texaco's Montebello Research Laboratory under contract Ex-76-C-01-2247 with the United States Department of Energy (DOE) during the week of March 12, 1978.

About 7500 pounds of SRC-II Vacuum Flash Drum Bottoms from the SRC pilot plant at Ft. Lewis, Washington were successfully gasified in a single 7.3 hour continuous run.

The residue was obtained from the liquefaction of Kentucky coal.

PILOT PLANT PROCESS FLOW

The Texaco Synthesis Gas Generation Process is a non-catalytic partial oxidation process that is based on certain reactions between oxygen and hydrocarbons that take place at high temperatures to produce a synthesis gas composed primarily of hydrogen and carbon monoxide. The high temperature reactions occur when the hydrocarbon and a deficiency of oxygen are introduced under pressure into a refractory lined vessel.

In order to control both the temperature and the conversion of hydrocarbon to gas, steam or liquid water is often added to the reactor. Gasification efficiency is enhanced to the degree that the steam-carbon reaction can be made to take place.

Figure 1 is a process flow diagram of the pilot plant.

Approximately 4 tons of solid residue were charged to the 1300 gallon melt tank and melted at 550°F under a nitrogen blanket. The melted residue was transferred to a 1000-gallon day tank in several batches during the run. The day tank is mounted on a scale to allow monitoring the charge rate. From the bottom of the day tank, the molten residue was circulated continuously through a line strainer past the suction of the high pressure residue charge pump and back to the top of the day tank. This was done to insure that a positive pressure was maintained at all times at the suction of the high pressure

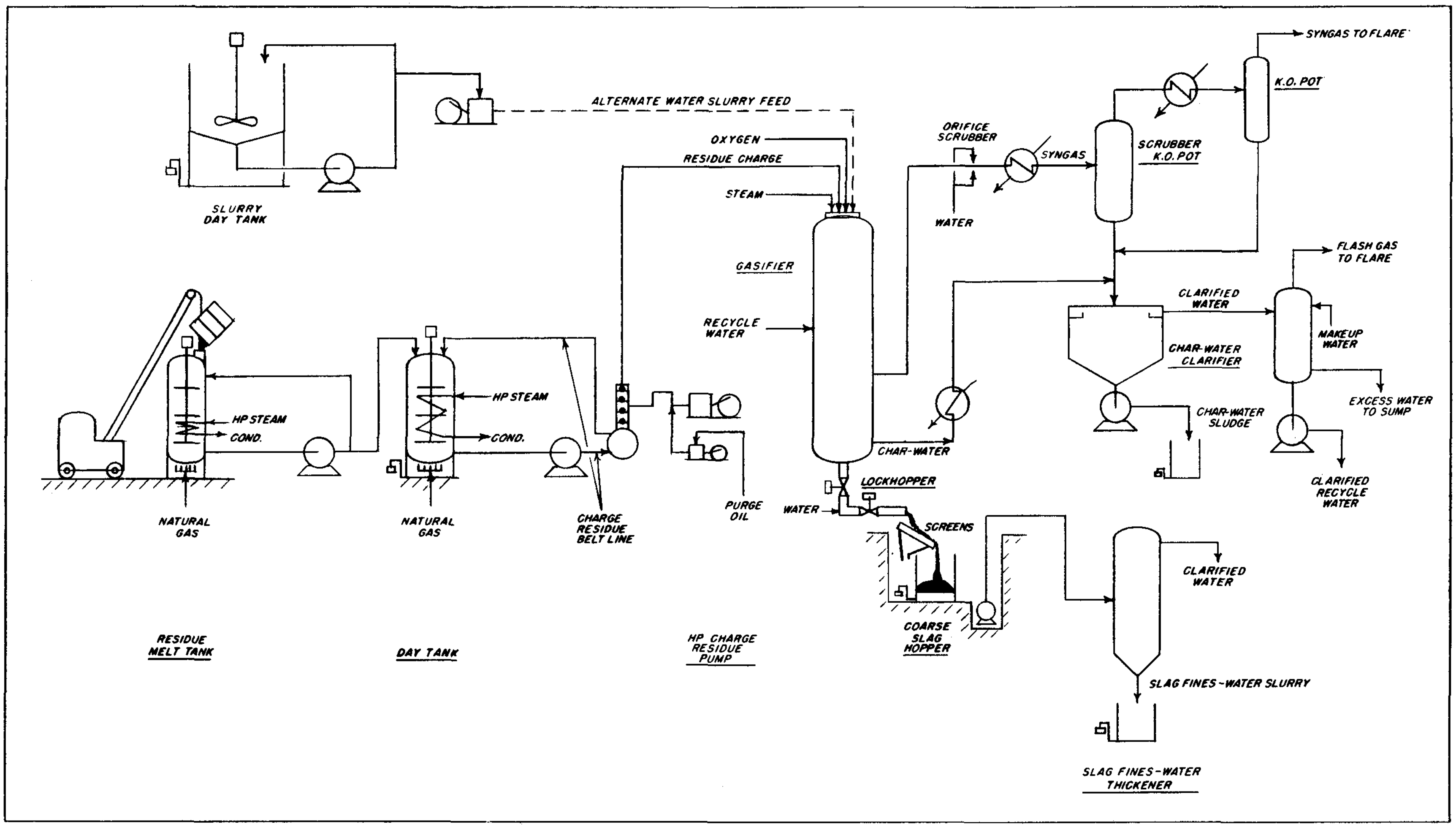

Figure 1 Pilot Plant Process Flow

residue charge pump. The day tank was also blanketed with nitrogen during the run.

Gasifier Feed System

Molten residue, oxygen and steam were fed through a proprietary Texaco burner into the top of the pilot plant gasifier.

The molten residue was pumped to the burner under pressure at a constant rate using a positive displacement plunger pump fitted with external ball check valves. The piping between each check valve assembly and the corresponding pump cylinder was filled with a clean purge solvent. An auxiliary pump was utilized to maintain a small purge rate (2% of residue feed rate) of fresh solvent into each connecting pipe to prevent molten residue from diffusing back to the charge pump cylinders. In a commercial plant the use of a solvent purge most likely would not be required.

Pilot Plant Gasifier

The pilot plant gasifier is a 5 ft. diameter by 20 ft. long vessel which is divided internally into two sections. It is designed to operate at a maximum pressure of 24 atmospheres.

The top section is lined with a special refractory material specifically designed to withstand the severe operating environment expected. In this section, the partial oxidation reactions take place.

The lower section is a quench vessel. A reservoir of water was maintained in the bottom of this vessel at all times. Syngas leaving the top section of the gasifier passed through a water cooled diptube into the water in the quench vessel. Slag, and most of the char, carried with the syngas remained in the water. The saturated syngas was removed from the gas space above the water, cooled to condense and remove most of its water content, metered and then flared. Water was continuously injected into the quench vessel to replace water lost by evaporation and solids removal.

Slag Removal System

Molten slag, which formed during the gasification of the residue, was carried into the quench chamber with the syngas. Upon contacting the reservoir of water in the quench chamber, the molten slag solidified. Most of the slag formed sand-like grains. The remainder of the slag formed teardrop-shaped glassy pieces of up to one inch in length. Slag was removed from the bottom of the quench chamber during each run with a lockhopper system.

The slag and water removed through the lockhopper system were ducted to a vibrating dewatering screen where the slag was separated into coarse and fine fractions. The coarse fraction was collected and weighed as a solid with less than 10 percent moisture. The fine fraction was pumped as a slurry into a settler where it was allowed to concentrate for later weighing, sampling, and disposal.

Char Recovery and Water Recycle System

During the gasification process, a small amount of char is formed which must be removed and recycled when practical.

Most of this material is removed from the syngas in the water reservoir at the bottom of the quench chamber. The char is less dense than slag and tends to remain dispersed in the water.

A continuous side draw-off of char water slurry was maintained during each run. This slurry was cooled and sent to a settling vessel where it was allowed to thicken for later removal, weighing, sampling, and disposal.

The clarified water off the top of the settling vessel was recycled to the process.

Final traces of char were removed in a scrubbing nozzle in which the syngas was contacted with additional water. The dilute char-water was collected in a scrubber knockout pot from which it was continuously withdrawn and combined with the quench water-char slurry in the settling vessel.

DISCUSSION AND RESULTS OF PILOT PLANT EVALUATION RUN

Raw Material Properties

The SRC-II Vacuum Flash Drum Bottoms is a brittle solid at ambient temperature. At 400°F it is a viscous fluid. It was shipped in open top 55-gallon drums in the form of random sized flakes of about ¼ to ½ inch thickness. The residue was charged to the melt tank directly from the drums.

The average ultimate analysis of the residue is shown on Figure 2. It contained almost 28 (wt) percent ash and 63.7 (wt) percent carbon. The ash was in the form of micron sized particles which were uniformly suspended in the carbonaceous material. No tendency for the ash to settle was observed in the molten residue.

Material Balance

Figure 2 summarizes the material balance and analytical data obtained during the run. The residue (+2% purge solvent) was fed to the gasifier at a rate of 1048 pounds per hour. The run lasted 7.3 hours. The entire melt tank charge of 3 3/4 tons of residue was gasified. A 98 percent conversion of carbon in the residue to syngas was achieved at the listed oxygen flow rate, producing 30.4 standard cubic feet of dry syngas per pound of residue gasified. The dry syngas contained 93.5 (vol) percent hydrogen plus carbon monoxide.

Figure 2

Comparison of Predicted vs Actual Performance
For Gasification of SRC-II Vacuum Flash Drum Bottoms

	Predicted Data From DOE Report Fe-2247-9R Figure 3	Actual Data
Charge to Gasifier		
Residue (+2% Purge Solvent), Pounds per Hour	1000	1048
Pure Oxygen, Pounds per Hour	770	764
Steam, Pounds per Hour	300	297
Total Input	2070	2109
Output From Gasifier		
Dry Product Syngas, Pounds per Hour	1694	1694
Char, Pounds per Hour	44	65
Coarse Slag, Pounds per Hour	217	137
Fine Slag, Pounds per Hour	-	109
Forced Water, Pounds per Hour	115	104
Total Output	2070	2109
Analytical		
Process Fuel Analysis, Weight Percent		
C	64.9	63.7
H	3.7	3.6
N	1.2	1.2
S	3.0	2.9
O (By Diff)	1.7	0.8
Ash	25.5	27.8
Higher Heating Value, BTU per Pound	11,300	11,250
Product Syngas Composition, Mole Percent, Dry		
H_2	33.4	34.0
CO	57.7	59.5
CO_2	7.2	5.3

Figure 2 (Cont'd)

Comparison of Predicted vs. Actual Performance
For Gasification of SRC-II Vacuum Flash Drum Bottoms

	Predicted Data From DOE Report Fe-2247-9R Figure 3	Actual Data
N_2	0.54	0.50
H_2S	1.04	0.44
COS	0.08	0.05
CH_4	0.00	0.11
A	0.03	0.07
Carbon on Coarse Slag, Weight Percent	0	1.2
Carbon on Fine Slag, Weight Percent	-	3.7
Carbon on Char, Weight Percent	14	12.9
Percent Carbon Conversion	99.0	98.1
Gasifier Pressure-PSIG	350	350
Dry Product Gas, Standard Cubic Feet per Hour	31,300	31,880
Hydrogen Plus Carbon Monoxide, Standard Cubic Feet per Hour	28,500	29,800
Run Length, Hours	-	7.3

The values shown on Figure 2 have been slightly adjusted by computer to yield 100 percent recoveries of the elements C, H and O and of the ash. Any missing ash was assumed to be coarse slag. The raw data obtained during the run are listed in Appendix A.

Also shown on Figure 2 is the material balance predicted on the basis of a previous Type 1 Laboratory Evaluation of a sample of SRC-II vacuum flash drum bottoms (DOE Report Fe-2247-9R, August 1977). The agreement between actual and predicted performance is excellent.

About 87 percent of the input ash was actually accounted for at the conclusion of the run. The remaining 13 percent was lost either due to sampling errors or hold up on the walls of the gasifier. Assuming all of the missing ash was in the form of coarse slag, the distribution of solids recovered is 44 (wt) percent coarse slag, 35 (wt) percent fine slag and 21 (wt) percent char. An emission spectrographic analysis of the coarse slag revealed that the major components contained Fe and Si with a minor component containing Al. The fluid temperature of the slag in a reducing atmosphere is 2318°F.

The char produced contained one percent less carbon than predicted. About 50 (wt) percent more char than predicted was produced due to the slightly lower ratio of oxygen to residue used in the actual run.

Based on the Type 1 Laboratory Evaluation previously completed, pumping and handling problems were anticipated. The sample viscosity was so high that the use of a diluent to reduce its viscosity was suggested for the pilot plant run. The viscosity of the residue charge used for this run was lower than expected. No diluent was used, and no pumping or handling problems were experienced in the run. The molten residue was pumped at a temperature of 510°F.

The charge pump purge solvent used was a heavy distillate from the SRC pilot plant in Ft. Lewis, Washington. Physical and chemical properties are summarized on Figure 3. It was fed to the charge pump at a rate of about two (wt) percent of the residue feed rate; it was included in the gasifier charge in the material balance.

At the conclusion of the run no evidence of significant refractory attack or burner tip erosion was apparent.

Figure 3

Heavy Distillate Purge Solvent Properties

Gravity, °API	0.6
Wt.%	
C	89.64
H	7.22
N	1.27
S	0.43
Ash	0.31
O(by diff.)	1.13
IBP	580°F
Flash pt (open cup)	321°F

ESTIMATE OF OPERATION FOR TYPE III EXTENDED PILOT PLANT TEST

Based on the data obtained in this short pilot plant run, a revised estimate of operation was prepared for gasifying 1000 pounds per hour of the SRC-II Vacuum Flash Drum Bottoms (plus 2% purge solvent should a Type III Extended Pilot Plant Test be desired. During a Type III Test the steam and oxygen rates would be varied to better define the optimum, however Figure 4 is our best estimate of optimum operation at this time. These conditions could be used as a center-point in a balanced experimental design. The distribution of coarse and fine slag shown on the estimate is somewhat arbitrary as this ratio would be expected to vary from run to run. A 99 percent conversion of carbon to syngas is predicted which will yield 30.8 SCF of dry syngas per pound of carbonaceous feed.

Figure 4

Texaco Coal Gasification Process

Estimate of Operation for Type III Extended Pilot Plant Test

For: DOE Contract EX-76-C-01-2247

Location: Montebello Research Laboratory Pilot Plant

Charge Stock: SRC-II Vacuum Flash Drum Bottoms

Ultimate Product: Hydrogen

Charge to Generator

Hydrocarbon (Fresh, Dry), Pounds per Hour	1000
Ultimate Analysis, Weight Percent, Moisture Free	
Carbon	63.7
Hydrogen	3.6
Nitrogen	1.2
Sulfur	2.9
Oxygen	0.8
Ash	27.8
Higher Heating Value, BTU per Pound	11,250
Pure Oxygen, Pounds per Hour	747.
Steam, Pounds per Hour	300.

Product Composition, Mole Percent, Wet

Carbon Monoxide	54.1
Hydrogen	31.3
Carbon Dioxide	5.8
Water	7.1
Methane	0.01
Argon	0.03
Nitrogen	0.49
Hydrogen Sulfide	0.96
Carbonyl Sulfide	0.07

Figure 4 (Cont'd)

Dry Product Gas, Standard Cubic Feet per Hour	30800
Hydrogen Plus Carbon Monoxide, Standard Cubic Feet per Hour	28400
Coarse Slag, Pounds per Hour	139
Carbon Content, Weight Percent	<0.5
Fine Slag, Pounds per Hour	112
Carbon Content, Weight Percent	2
Soot Discharge, Pounds per Hour	33
Carbon Content, Weight Percent	13
Unconverted Carbon, Percent of Carbon in Feed	1.
Generator Pressure, psig	350.

CONCLUSIONS AND RECOMMENDATIONS

SRC-II Vacuum Flash Drum Bottoms, containing almost 28 percent ash from the liquefaction of Kentucky Coal, is a suitable feedstock for the Texaco Coal Gasification Process. Three and three quarter tons of this residue were successfully gasified at 350 psig in a single 7.3 hour pilot plant run.

The residue was fed to the Texaco gasifier undiluted as a molten fluid at 510 deg. F.

A 98 percent conversion of the carbon to syngas was achieved.

The SRC-II Vacuum Flash Drum Bottoms is a suitable material for a Type III Extended Pilot Plant Evaluation and this should be considered by DOE.

APPENDIX A

Raw Data From Gasification of SRC-II Vacuum Flash Drum Bottoms

Input		
* Fuel Rate, lb/hr	1018.	
Steam Rate, lb/hr	297.	
Oxygen Rate, lb/hr	766.	(9080 SCFH)
Total Measured Input	2081.	
Output		
Dry Product Gas, lb/hr	1687.	(32092 SCFH)
Char, lb/hr	65.	
Coarse Slag, lb/hr	96.	
Fine Slag, lb/hr	109.	
Total Measured Output	1957.	
Forced Water	99.	
Total Output	2056.	
Analytical		
Process Fuel Analysis, Wt. Pct.		
C	63.1	
H	3.6	
N	1.2	
S	2.9	
Ash	28.3	
O (by diff)	0.9	
Product Gas Analysis, Vol%, Dry		
H_2	34.5	
CO	59.0	
CO_2	5.3	
N_2	0.51	

APPENDIX A (Cont'd)

H_2S	0.44
COS	0.05
CH_4	0.11
A	
Carbon on Coarse Slag, Wt.%	1.2
Carbon on Fine Slag, Wt.%	3.7
Carbon on Char, Wt.%	12.9
Carbon Conversion, Wt. Pct.	102.0
Ash Recovery, Wt. Pct.	87.1
Gasifier Pressure, PSIG	350.
Run Length, Hrs.	7.3

*Residue plus 2% purge solvent

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