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COMBINED SHIFT AND METHANATION IN A FLUIDIZED-BED REACTOR

Quarterly Progress Report for January 1—March 31, 1980

By
Robert C. Streeter

April 1980

Work Performed Under Contract No. AC01-79ET14815

Bituminous Coal Research, Inc.
Monroeville, Pennsylvania

MASTER

U. S. DEPARTMENT OF ENERGY

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**COMBINED SHIFT AND METHANATION IN A
FLUIDIZED-BED REACTOR**

**QUARTERLY PROGRESS REPORT
FOR THE PERIOD
1 JANUARY 1980 - 31 MARCH 1980
(BCR Report L-1091)**

by

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APRIL 1980

**WORK PERFORMED UNDER CONTRACT
DE-AC21-79ET14815**

for

**U.S. DEPARTMENT OF ENERGY
DIVISION OF ENERGY TECHNOLOGY**

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BITUMINOUS COAL RESEARCH, INC.
DOE-SPONSORED RESEARCH PROGRAM

COMBINED SHIFT AND METHANATION IN A FLUIDIZED-BED REACTOR

Report No. DOE/ET/14815-8
(BCR Report L-1091)

ABSTRACT

Construction of the new bench-scale reactor system was completed during this report period, and the system was put into operation on March 26.

A bench-scale activity test, using the older life-test apparatus, was conducted to evaluate two samples of a Ni/Mo catalyst with a stoichiometric synthesis gas ($H_2/CO = 3/1$). One of the samples had been regenerated, by heating in air at 450 C, to remove a significant amount of carbon (18 weight percent) that had been deposited in an earlier PEDU test. The activity of the regenerated sample appeared to be equivalent to that of the as-received catalyst.

A second life test (with the older apparatus) was conducted to evaluate the activity and stability of a new catalyst, BCR Lot No. 4438, with a feed gas H_2/CO ratio of 3/1. The catalyst was extremely active, yielding essentially equilibrium conversions at all temperatures in the range of 800 to 1050 F. After being heated to 1100 F, however, the catalyst was deactivated. This was not unexpected, since the catalyst manufacturer had advised that the maximum operating temperature was 932 F. Arrangements have been made to obtain a PEDU-scale sample of this catalyst for further studies in the fluidized-bed reactor.

A 5-day PEDU test was conducted during the week of February 25-29, 1980, using a sample of the BI-GAS pilot plant methanation catalyst. The system was operated with feed H_2/CO ratios of 2/1 (without steam) and 1/1 (with a steam/gas ratio of 0.12) for 36 and 14 hours, respectively. The catalyst remained active throughout the test, even though a post-test analysis showed an accumulation of 2 percent carbon. Useful conversions during the 2/1 and 1/1 periods were 97 and 96 percent, respectively, while product gas methane contents were 92 and 89 percent, respectively, on a CO_2 -free, dry basis. Two reformer upsets and failure of the mechanical seal on the main Dowtherm pump forced premature termination of the test, and it is planned to repeat the $H_2/CO = 1/1$ data period in a future PEDU test.

A purchase order was issued by BCR to Dr. Calvin Bartholomew of Brigham Young University on January 24, 1980, authorizing BYU to proceed with support studies on carbon deposition and regeneration of fluidized methanation catalysts.

I. INTRODUCTION AND PROJECT OBJECTIVE

This report presents progress achieved during the period January through March 1980 on the program "Combined Shift and Methanation in a Fluidized-bed Reactor," being conducted by Bituminous Coal Research, Inc., for the U.S. Department of Energy. The program was initiated under Contract No. DE-AC21-79ET14815 on September 25, 1979.

The continuing objective of this program is to demonstrate the feasibility of employing a gas-phase catalytic fluidized-bed reactor to carry out water-gas shift and methanation in a single pass. Successful attainment of the objective will permit methanation of a nonstoichiometric feed gas in an SNG process.

II. SCOPE OF WORK

Tests will be conducted using both bench-scale equipment and a process and equipment development unit (PEDU). The PEDU reactor is a 6-inch I.D., 15-foot long vessel with a reaction zone of approximately 8 feet. The nominal catalyst charge is about one cubic foot.

The combined bench-scale and PEDU studies will be conducted to develop a fluidized-bed process for use in upgrading a coal-derived synthesis gas. Tests will involve the use of synthesis gases with H_2/CO ratios of 2/1 and 1/1, containing CO_2 and CH_4 , to simulate the gasifier products (sulfur-free basis) of several first- and second-generation coal gasification processes. Some tests will involve the addition of steam to the feed gas in an effort to determine its role in inhibiting carbon deposition, as well as its possible adverse effects on the catalyst (e.g., sintering). Nominal test conditions will be 1000 psig pressure and temperatures in the range of 800 to 1100 F.

Additional studies will be undertaken at Brigham Young University on the effects of gas composition, catalyst composition, and support material on the kinetics of carbon formation under representative reaction conditions, and on the kinetics of carbon removal by oxygen. Information from these studies will afford a comparison of the relative sensitivities of the catalyst to carbon fouling, and may provide sufficient basis for an in situ catalyst regeneration process.

III. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

A. Bench-scale Studies:

1. Life Test of Regenerated Ni/Mo Catalyst: As discussed in the last quarterly progress report, a bench-scale test was conducted using the older life test apparatus to evaluate several samples of catalyst, BCR Lot No. 4265,

which had been employed during PEDU Tests No. 36 and 37 under the earlier contract.* This is a Ni/Mo catalyst on a silica-alumina support. One of the objectives of that bench-scale test was to compare the activity of one sample, originally containing 18 percent carbon but regenerated by heating in air at 450 C (842 F) for 2 hours, with that of the fresh (previously unused) catalyst. However, because of a pressure upset during the test, the results were inconclusive, and it was decided to repeat the test during this report period.

Once again, the older life-test apparatus was used. The catalyst samples were reduced in pure H₂ overnight at 1000 F and 1000 psig, then the temperature was decreased to 800 F and a stoichiometric synthesis gas (20 percent CO, 60 percent H₂, 20 percent CH₄) was introduced. The temperature was then gradually increased through the range 800 to 1100 F, then decreased again to 800 F at the conclusion of the test.

The results of this test are shown in Figure 1 in terms of useful conversion[†] versus reactor temperature. It is apparent that the activity of the regenerated catalyst throughout the test was not significantly different from that of the fresh catalyst. Thus, it can be concluded that the heat treatment to remove carbon did not significantly impair the activity of the spent catalyst. In contrast to the results of the earlier life test, discussed in the last quarterly report, neither of the two catalyst samples achieved useful conversions above 90 percent. However, in the earlier test, the temperature was cycled several times to 1000 F, and the fresh catalyst tended to show higher activity following each cycle. This behavior has been observed in previous activity studies with these Ni/Mo catalyst systems, and is believed to be due to the relatively slow reduction of molybdenum-containing sites, enhanced at the higher temperatures. In fact, it may be noted in Figure 1 that the activities of these two catalysts were tending to increase at the end of the test, following their exposure to 1100 F.

2. Life Test of Catalyst BCR Lot No. 4438: Also during this report period, a new catalyst sample (BCR Lot No. 4438) was evaluated in the older life test apparatus using a stoichiometric feed gas (H₂/CO ratio = 3/1). The sample was obtained from United Catalysts, Inc. (UCI), and its composition has not been disclosed. The catalyst supplier had advised that the maximum operating temperature for this catalyst was 500 C (932 F), so one of the

* Gas Generator Research and Development - BI-GAS Process: Contract No. EF-77-C-01-1207.

$$^{\dagger} \text{Useful conversion } (\alpha) = \frac{(\text{CO} + \text{H}_2)_{\text{in}} - K(\text{CO} + \text{H}_2)_{\text{out}}}{(\text{CO} + \text{H}_2)_{\text{in}}} \times 100 \text{ (percent)}$$

where $K = C_{\text{in}}/C_{\text{out}}$. The terms and rationale for this expression were discussed in the last quarterly progress report.

Figure 1. Life Test Results With Two Samples of Methanation Catalyst
BCR Lot No. 4265 (H₂/CO Ratio = 3/1)

objectives of this test was to observe any changes in catalyst activity at the higher reactor temperatures.

Because of the possible sensitivity of the catalyst to the higher temperatures, the catalyst was reduced overnight at 800 F (instead of 1000 F) in a stream of pure H_2 . Initial activity data were obtained at 800 F for 2 days, then the temperature was increased by 50-degree increments to 1000 F. Through the range 900 to 1000 F, the temperature was held at each level for at least 5 days to observe any changes in catalyst activity with time. Following the 1000 F treatment, the temperature was decreased to the baseline value of 800 F for 3 days. No decline in catalyst activity was evident, so the temperature was increased further to 1050 F (followed by another baseline check at 800 F), and finally to 1100 F. The overall duration of the test was 1,825 hours.

The results of this life test are shown in Figure 2. It is apparent that the UCI catalyst showed extremely high activity, giving essentially equilibrium conversions (indicated by the horizontal dashed lines) through most of the test. The catalyst also appeared to be thermally stable to at least 1050 F (565 C), exceeding the manufacturer's specifications. However, after exposure of the catalyst to 1100 F for 2 days, a definite decline in activity was observed, as shown in Figure 2. Although the catalyst was still quite active on the first day following the 1100 F treatment, conversions eventually decreased from 96 to 60 percent and were still dropping when the test was concluded. Thus, the catalyst was evidently thermally deactivated at 1100 F. This is not considered to be a serious drawback, however, since the fluidized-bed PEDU reactor does not normally operate at temperatures above 1000 F. Higher temperatures have been reached, but only infrequently during thermal excursions of short duration (the emergency N_2 purge system in the PEDU is automatically activated if the temperature exceeds 1060 F).

The UCI catalyst was also subjected to the laboratory accelerated attrition test. In this test, a 50-gram sample of catalyst, previously sieved to 100 x 230 mesh, is fluidized by air at velocities of about twice those achieved during normal operation of the PEDU reactor. The catalyst is withdrawn periodically, and the amount of minus 325-mesh material generated (both entrained and remaining in the bed) is determined.

The results of the attrition test are shown in Figures 3 and 4. Figure 3 shows the cumulative buildup of fines with time, and Figure 4 shows the change in the average particle diameter of the catalyst during the course of the test. Also included are comparative data from an earlier attrition test with catalyst BCR Lot No. 3631, a split of the Harshaw alumina-supported Ni/Cu/Mo methanation catalyst from the Homer City BI-GAS pilot plant. The data indicate that the UCI catalyst exhibits somewhat better attrition behavior than the Homer City catalyst in terms of fines production during operation. On the other hand, Figure 4 shows that the rate of attrition is essentially the same for the two catalysts; the UCI catalyst appears to be superior simply because its initial size distribution is coarser than that of the Homer City catalyst (e.g., 0.4 percent minus 325-mesh material in No. 4438 vs. 43.7 percent in No. 3631, both on an as-received basis).

USEFUL CONVERSION (α), PERCENT

BCR 8070G2

Figure 2. Life Test Results for Methanation Catalyst
 BCR Lot No. 4438 (H₂/CO Ratio = 3/1)

CUMULATIVE MINUS 325 MESH MATERIAL
FROM 50-GRAM SAMPLE, GRAMS

BCR 807003

Figure 3. Laboratory Attrition Test Data for Catalysts
BCR Lots No. 4438 and 3631

**Figure 4. Change in Average Particle Diameter With Time
During Laboratory Attrition Test of Catalysts
BCR Lots No. 4438 and 3631**

On the basis of the encouraging results from both the life test and attrition test, the supplier of catalyst BCR Lot No. 4438 has been requested to arrange for the preparation of a 1.5 cubic-foot sample for further evaluation in the fluidized-bed PEDU reactor. Preparation and delivery of this sample will require approximately 3 months.

3. New Bench-scale Reactor System: Construction of the new bench-scale reactor system was completed during this report period, and a photograph of the completed apparatus is shown as Figure 5. The system has been located in the PEDU control room area to take advantage of the available low pressure nitrogen supply for the pneumatic control valves.

Shakedown tests of the system were conducted during the early part of March. One of the three flow control valves was found to be defective due to scoring of the seat by a metal burr on the valve stem. The manufacturer has agreed to replace the valve trim at no charge to BCR.

The first test with the new reactor system was initiated on March 26. This test will evaluate the performance of the UCI catalyst and the Harshaw Ni/Cu/Mo catalyst at 800 F and 1000 psig, using a steam-free feed gas with a H_2/CO ratio of 2/1. Results of this and subsequent bench-scale tests of other catalysts at $H_2/CO = 2/1$ will be presented in the next quarterly report.

B. PEDU Studies

1. PEDU Test No. SM-1: PEDU Test No. SM-1* was conducted over the 5-day period of February 25 to 29, 1980. The purpose of this test was to evaluate a fresh batch of the Ni/Cu/Mo BI-GAS pilot plant methanation catalyst with extended operation at feed H_2/CO ratios of 2/1 (without steam addition) and 1/1 (with steam addition).

a. Catalyst Preparation and Activation: The catalyst (70 pounds) was received from the Homer City plant on February 19 and designated as BCR Lot No. 4533. The sample was sieved at 16 mesh to remove extraneous debris, then a charge of 66 pounds (1.16 cu ft at minimum fluidizing velocity) was introduced to the reactor. This is slightly more than the 62.5 pounds (1.1 cu ft) normally employed, because it was intended to allow for the withdrawal of catalyst samples during the course of the test.

During startup on February 25, the reactor was purged initially with N_2 , followed by CH_4 ; then H_2 and CO from the steam reformer were gradually blended into the CH_4 until a stoichiometric synthesis gas composition was obtained (60 percent H_2 , 20 percent CO, 20 percent CH_4 ; $H_2/CO = 3/1$). The addition of H_2 and CO to the feed gas was initiated at 3:00 p.m. on the first day of the test with bed temperatures at about 470 F. Within one hour, the desired feed gas composition had been achieved, and bed temperatures were close to 600 F, which has been observed previously to be the "light-off" temperature for this catalyst.

*SM denotes combined Shift/Methanation.

**Figure 5. New Bench-Scale Methanator Reactor System
(March 1980)**

For the next three hours, bed temperatures rose smoothly and leveled out at about 950 F. The first data period was started at 7:00 p.m.

b. Summary of PEDU Test No. SM-1 Results: Data on test conditions, feed and product compositions, material balances, and conversions are summarized in Table 1 for the three steady-state data periods of PEDU Test No. SM-1. In all three periods, all of the feed gas was introduced through the bottom inlet (distributor zone) of the reactor, with the result that significant bed temperature differentials (>100 F) were measured (i.e., most of the reaction was occurring in the bottom of the catalyst bed). Consequently, the average bed temperature has been arbitrarily redefined as the average between the middle bed and distributor zone temperatures only.

Period 1 was a short baseline activity check with a stoichiometric feed gas ($H_2/CO = 3/1$). The results shown in Table 1 are typical, based on previous PEDU test experience with this catalyst, confirming that the catalyst had reached steady-state activity.

Period 2 was the first combined shift/methanation data period at a H_2/CO ratio of 2/1, without steam addition. The reactor temperature was kept as high as possible to minimize carbon formation. Evidence that the shift reaction was occurring is shown by the much lower H_2 content of the product gas and the reduced production of by-product water compared with Period 1. This period was continued for 36 hours, during which time there was no perceptible decrease in catalyst activity. On a CO_2 -free, dry basis, the product gas composition from Period 2 was 4.1 percent H_2 , 2.1 percent CO , 92.4 percent CH_4 , and 1.4 percent C_2H_6 .

It is also significant that this data period represents the first time that as much as 30 percent CO_2 was fed to the reactor. The CO_2 content of the feed gas has never been higher than 20 percent in any previous PEDU test, and there was concern that attempts to increase it further would lead to difficulties in maintaining high reactor temperatures because of the comparatively high heat capacity of CO_2 . However, no such problems were encountered.

Period 3 was started at 6:00 p.m. on the third day of the test. The feed H_2/CO ratio was decreased to 1/1, and steam was added at the maximum achievable rate of about 16 pounds/hour corresponding to a steam/dry gas ratio of 0.12. The system ran smoothly for 7.5 hours, until an upset in the reformer occurred, causing an interruption of 6.5 hours in the data period. The period was resumed at 7:00 a.m. on the fourth day of the test, and steady-state conditions were maintained for another 6.5 hours, at which time a second reformer upset occurred. During each of these upsets, reactor temperatures dropped to nearly 500 F. To avoid possible carbon deposition under these conditions, the feed gas composition was readjusted to that employed in Period 1 ($H_2/CO = 3/1$), and steam was removed from the feed until the reactor temperatures recovered.

TABLE 1. PROCESS MATERIAL BALANCE DATA FOR
METHANATION PEDU TEST NO. SM-1

DATA PERIOD	1	2	3
PERIOD DURATION, hours	2	36	14
TEMPERATURES, F			
Feed	673	778	728
Distributor	958	979	936
Middle Bed	935	907	889
Top Bed	863	812	815
Bed Average	946*	943*	912*
PRESSURE, psia	1003	1008	1008
COMPOSITION DATA, mole percent			
Feed			
Hydrogen	59.1	40.2	27.6
Carbon Monoxide	20.0	20.0	27.4
Carbon Dioxide	0.7	29.9	19.4
Methane	20.2	9.6	14.2
Ethane	0.0	0.3	0.7
Steam	--	--	10.7
Product (dry basis)			
Hydrogen	21.2	1.8	2.8
Carbon Monoxide	0.3	0.9	1.1
Carbon Dioxide	6.0	56.7	52.3
Methane	72.4	40.0	42.6
Ethane	0.1	0.6	1.2
HYDROGEN/CARBON MONOXIDE RATIO IN FEED	2.95	2.01	1.01
MASS FLOW RATES, lb/hr			
Gas In	92.6	175.3	163.1
Steam In	--	--	16.3
Total Out	92.8	174.2	179.0
Gas Out	68.3	159.7	163.6
Water Out	24.5	14.5	15.4
USEFUL CONVERSION (α), percent	86.0	97.3	95.5
FEED, scfh (dry basis)			
Distributor	3383	3135	2866
Middle Zone	0	0	0
Total	3383	3135	2866
SPACE VELOCITY, hr ⁻¹	2916	2703	2471
PRODUCT, scfh (dry basis)	1748	1901	2027

* Average bed temperature based on distributor and middle bed temperatures only (see text).

Just about the time the system had recovered from the second reformer upset, the main Dowtherm pump (supplying the finned-tube cooling coils) developed a leak through the mechanical seal, forcing premature termination of the test. During the 14 hours that the system was in stable operation, however, there was no evidence of catalyst deactivation.

The data in Table 1 again show the increased useful conversion (relative to Period 1) during Period 3. On a CO₂-free, dry basis, the product gas composition from Period 3 was 5.9 percent H₂, 2.3 percent CO, 89.3 percent CH₄, and 2.5 percent C₂H₆.

c. Changes in Catalyst Properties: As noted earlier, it was intended to withdraw catalyst samples periodically during the PEDU test to monitor any carbon buildup. However, although the ball valves for the catalyst sampling system had been rebuilt and appeared to be working properly before the test, one of the valves began leaking early in the test, precluding the taking of catalyst samples.

The carbon content of the bulk spent catalyst at the conclusion of the test was 2.0 percent; thus, some carbon deposition evidently took place, and it is believed likely that this was aggravated by the temperature upsets that occurred near the end of the test. However, as discussed previously, no decline in catalyst activity was evident throughout the test. In previous PEDU tests with this catalyst it has been observed that its activity is not affected until the carbon content reaches about 3 percent.

The particle size distributions of the as-charged and bulk spent catalyst samples from PEDU Test No. SM-1 are shown in Table 2. The changes in particle size distribution during the test are considered normal; data from previous PEDU tests have shown that the fines content (minus 325-mesh material) usually increases to a limiting value of 55-60 percent during the course of the test.

d. General Conclusions Concerning PEDU Test No. SM-1: The data from PEDU Test No. SM-1 confirm that combined shift and methanation in a fluidized-bed reactor is feasible with the BI-GAS pilot plant methanation catalyst. The catalyst operated successfully at H₂/CO ratios of 2/1 (without steam) and 1/1 (with minimal steam addition), yielding product gases containing 92 and 89 percent CH₄ (CO₂-free, dry basis), respectively, on a single pass through the reactor, without recycle, and at space velocities from 2500 to 2700 hr⁻¹.

Unfortunately, the data period at H₂/CO = 1/1 was cut short because of several unexpected system upsets. It is planned to repeat this data period over a longer time interval in a future PEDU test.

C. BYU Studies: A purchase order was issued by BCR to Dr. Calvin Bartholomew of Brigham Young University on January 24, 1980, authorizing BYU to proceed with support studies on carbon deposition and regeneration of fluidized methanation catalysts.

TABLE 2. SIZE DISTRIBUTION OF AS-CHARGED AND BULK SPENT
SAMPLES OF CATALYST BCR LOT NO. 4533 BEFORE AND AFTER PEDU TEST NO. SM-1

Mesh Size (U.S. Series)	Cumulative Weight, percent*		Mesh Size Fraction	Amount in Designated Mesh Size Fraction, percent	
	No. 4533 As-charged	No. 4533 Bulk Spent		As-charged	Bulk Spent
+ 100	1.6	0.3			
+ 120	2.9	0.7	> 120	2.9	0.7
+ 140	3.6	1.3			
+ 170	8.4	3.6	120 x 170	5.5	2.9
+ 200	12.3	6.0			
+ 230	31.9	20.6	170 x 230	23.5	17.0
+ 270	35.1	24.4			
+ 325	50.9	40.3	230 x 325	19.0	19.7
- 325	100.0	100.0	< 325	49.1	59.7
Mean Particle Diameter, microns	33.1	29.5			

*Determined by dry sieve analysis.

D. Visitors During January, February, and March 1980

January 29, 1980

Chang Bi-Jiang
Bao Han-Chen
Huang Ke-Quan
Yang Gui-Lin
Institute of Coal Chemistry
Chinese Academy of Sciences
P. O. B. 165
Taiyuan, Shanxi, China

E. Trips and Meetings During January, February, and March 1980

None

IV. WORK PLAN AND SCHEDULE

Tests with the new bench-scale reactor system will be continued.

PEDU tests will be continued to evaluate the shift/methanation behavior of other catalysts as they become available. The next PEDU test is scheduled for the week of April 14-18, 1980.

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