

## **Task 4. Wax/Catalyst Separation**

The objective of this task is to develop techniques for the separation of catalysts from FT reactor slurries.

### **A. Slurry Bubble Column Reactor (SBCR) Activities**

#### **ABSTRACT**

In this report we describe results and operating experiences for two SBCR pilot plant runs completed during this reporting period. For the first time since overhauling the SBCR plant, a high alpha iron-based F-T catalyst run was completed. The objective of the run was to test the efficiency of the new catalyst/wax filtration system for removing heavier F-T products. Conversion and catalyst activity decline of the high alpha catalyst in the SBCR were compared to that of CSTR experiments. The activity decline measured in the SBCR system was shown to be similar to that of the CSTR experiments.

A catalyst attrition study was conducted using a spray-dried iron catalyst. Attrition of the catalyst particles occurred in two stages: 1) a rapid breakdown during catalyst loading and activation and 2) after two days time on stream an exponential decay period. SEM images of the particles indicate that breakdown of the particles was more physical than chemical in nature. During the end of the attrition test, the effect of syngas composition was investigated by changing the H<sub>2</sub>/CO ratio from 0.7 to 2.0. After changing the H<sub>2</sub>/CO ratio to 2.0, the trend of losing reactor slurry level (as shown from a previous SBCR run) was verified. The cause of the slurry level decrease was found to be due to a decreased gas hold-up with the reactor vessel.

#### **INTRODUCTION**

A Slurry Bubble Column Reactor (SBCR) is a gas-liquid-solid reactor in which the finely divided solid catalyst is suspended in the liquid by the rising gas bubbles. SBCR offers many

advantages over fixed-bed type reactors such as: 1) improved heat transfer and mass transfer; 2) an isothermal temperature profile is maintained; and 3) relatively low capital and operating cost. Fischer-Tropsch Synthesis (FTS) takes place in a SBCR where the synthesis gas is converted on catalysts suspended as fine particles in a liquid. The synthesis gas flows in a bubble phase through the catalyst/wax suspension. The bubbles in the catalyst slurry are produced by a gas distributor in the bottom of the reactor. The volatile products are removed with unconverted gases, and the liquid products are separated from the suspension.

Heavy wax products must be separated from catalyst particles before being removed from the reactor system. Achieving an efficient wax product separation from the catalyst is one of the most challenging technical problems associated with slurry-phase F-T. The breakdown of the catalyst particles and the production of heavy wax using high alpha catalysts can further exasperate the filtration problem (1). Thus, designing a physically robust catalyst without compromising activity is an important factor for a stable and economical F-T process. However, small-scale catalyst attrition tests may not be adequate in simulating the environment within a bubble column reactor. In previous studies (2,3) an air-jet attrition tester as outlined in the ASTM-D-5757-95 test method was used to compare the relative breakdown resistance of catalysts. This method is useful in comparing the relative strength between catalyst candidates; however, it does not yield information as to the rate of attrition during synthesis. Attrition data in CSTR systems are compromised by the extreme impact and shear forces from the impeller blade on catalyst particles.

As discussed in the previous reports (4,5), the CAER 5.08 cm diameter SBCR plant was overhauled and redesigned to incorporate automatic slurry level control and wax filtration systems. The wax filtration system was designed to accept a variety of filter elements. These

additions enhance the stability of the reactor for long-term tests useful for both catalyst deactivation and attrition studies under SBCR synthesis conditions.

In the following discussion, we will detail the results and operational experiences of a two experimental SBCR runs using an precipitated/spray-dried iron catalysts. Objectives of the runs were to: 1) Determine the effectiveness of the catalyst/wax filtration system using a high-alpha catalyst with heavy reactor wax F-T products; 2) Compare the SBCR performance of a high-alpha catalyst with that of the smaller scale continuous stirred tank reactor (CSTR) system; and 3) Quantify the attrition performance of the catalyst during activation and synthesis in the SBCR environment.

## **EXPERIMENTAL**

The SBCR apparatus, shown in Figure 1 (see a detailed description in the last quarterly report, ref. 5), was originally designed as a direct coal liquefaction reactor. In the current configuration, the bubble column has a 5.08 cm diameter and a 2-m height with an effective reactor volume of 3.7 liters. The synthesis gas was passed continuously through the reactor and distributed by a sparger near the bottom of the reactor vessel. The product gases and slurry exit the top of the reactor and pass through an overhead receiver vessel where the slurry was disengaged from the gas-phase. Vapor products and unreacted syngas exit the overhead vessel, enter a warm trap (100 °C), and a cold trap (3°C). A dry flow meter down stream of the cold trap was used to measure the exit gas flow rate.

A dip tube was added to the reactor vessel so that the F-T catalyst slurry could be recycled internally via a natural convection loop. The unreacted syngas, F-T products, and slurry exited into a side port near the top of the reactor vessel and entered a riser tube. The driving force for the recirculation flow was essentially the difference in density between the fluid column

in the riser (slurry and gas) and that of the dip-tube (slurry only). The dip tube provided a downward flow path for the slurry without interfering with the upward flow of the turbulent syngas slurry mixture. Thus, to some degree, back mixing of the slurry phase and wall effects in the narrow reactor tube were minimized.

Catalyst Activation and SBCR Startup Procedure. In preparation for catalyst activation, the SBCR was filled with 2.8 liters (~75% of the reactor volume) of a slurry consisting of 20 wt.% iron catalyst and Shell C<sub>30</sub> oil. An additional 1.3 liters of the C<sub>30</sub> oil was isolated in the overhead separation vessel. The reactor was pressurized with flowing CO gas at 175 psig (12 atm) while the slurry temperature was increased to the desired activation temperature at a 50 °C/hour rate. Once the reactor temperature stabilized, the exit gas was periodically monitored for CO<sub>2</sub> to observe the progress of activation. During the activation period, the down-comer leg from the overhead vessel to the reactor was valved-off so that the catalyst remained isolated inside the reactor. Likewise, the C<sub>30</sub> oil in the overhead vessel did not mix with reactor catalyst during activation.

After the catalyst had been activated (~24 hours), hydrogen gas flow was phased in with the CO feed gas. Once the desired gas space velocity had been attained, the down-comer valve used to isolate the C<sub>30</sub> oil in the overhead vessel was opened to allow circulation between the reactor, riser and down-comer legs. Once the C<sub>30</sub> oil became mixed with the activated catalyst slurry and the reactor temperatures were stabilized, CO, H<sub>2</sub>, and syngas conversions were calculated at least once a day to monitor the reactor performance.

Wax Filtration and Catalyst Particle Sampling Procedure. For the current study, a filter tube from Mott Metallurgical Corporation was installed in the liquid down comer pipe below the overhead separation vessel. The filter unit is a flow-through device having a sintered metal (2-5

µm average pore size) tube in a shell. Filtered wax was extracted radially through the tube while slurry flows downward in the axial direction. The shear force of the axial slurry flow prevented excessive caking of the catalyst around the filter media. Filtered wax was metered into a storage tank through a letdown valve operated by the overhead liquid level controller. Pressure drop across the filter media was varied manually by varying the wax storage tank pressure. The filter assembly was configured such that the filter media could be replaced on-line, without aborting or interrupting the reactor run.

The level or volume of the slurry within the receiver was continuously monitored by measuring the differential pressure across the height of the vessel. Argon was purged through each of the pressure legs to keep the lines free of slurry. Slurry volume within the receiver was controlled to be no more than 1.3 liters by removing wax from the reactor system via the level control valve. The unfiltered slurry flowed back to the reactor via a natural convection loop through a dip-tube exiting near the bottom of a reactor.

Samples of the unfiltered slurry were taken from the bottom of the overhead receiver tank on a daily basis. Wax products were Soxhlet extracted according to the method of McCartney et al. (6). The particle size distributions of the extracted catalyst particles were quantified using a light scattering technique by a Cilas 1064 liquid/particle analyzer.

## **DISCUSSION OF RESULTS**

### **High Alpha Catalyst Performance**

A high alpha iron-based, catalyst was prepared from a 75-kg batch of a precursor spray-dried catalyst having a composition of 100 Fe/4.4 Si/1.0 K. Approximately 750 kg of the high alpha catalyst was prepared from the precursor by adding Cu and K promoters. The final

composition of the high-alpha catalyst was 100 Fe/5.1 Si/2.0 Cu/ 5 K (determined by in-house analysis).

Comparisons between CSTR and SBCR runs. The run/activation conditions for the enhanced SBCR system along with the comparison CSTR run are listed in Table 1. A nominal space velocity of 5.0 slph/g was used for both reactor systems. The target activation temperature for the SBCR reactor test was intended to be 270°C; however, due to operator error, the catalyst was activated at 230°C. Consequently, we repeated the activation procedure for the comparison CSTR tests in order to judge the catalyst performance on an equal basis.

The gas conversions versus time-on-stream for the SBCR and CSTR systems are displayed in Figure 2. Hydrogen, CO, and syngas conversions in the SBCR reached a maximum after 50 hours time-on-stream (TOS). After this catalyst initiation period, the syngas conversion started to steadily decline to about 14% after 288 hours TOS. Maximum conversions in the CSTR were attained after only 24 hours of TOS. Overall, the syngas conversions in the CSTR were consistently less than that of the SBCR. This observation is consistent with the previously reported SBCR results (4,5) attributed to the plug-flow nature of the SBCR reactor system. Corresponding carbon dioxide and methane selectivities are shown in Figure 3. Carbon dioxide selectivity varied between 31 and 38% and 21 to 26% for the SBCR and CSTR systems, respectively. The methane selectivity for the SBCR was consistently below 2% while that of the CSTR experiment varied between 3 and 4%.

Catalyst Deactivation Rate Comparisons. Catalyst deactivation rates were compared between the different reactor configurations using the activity function defined as:

$$a(t) = \frac{r_{FT}(t')}{r_{FT}(t'=0)} \approx \frac{X_{CO}(t')}{X_{CO}(t'=0)}$$

Where  $t'$  is the time after attaining the maximum total reaction rate or conversion. The relative activity functions were calculated from the maximum conversion and plotted in Figure 4 versus the relative  $t'$  timescale. In this fashion, each of the deactivation rates could be compared on an equal basis, independent of the conversion levels.

The deactivation rates of the high-alpha catalyst calculated for the SBCR and CSTR cases followed a linear zero order fashion with decay constants of 0.055 and 0.0558 day<sup>-1</sup>, respectively. The relatively rapid deactivation was likely due to the low activation temperature. As previously reported (4), the same catalyst (without promoters) had activity decay constants in the range of 0.013 and 0.014 day<sup>-1</sup> using a 270 °C activation temperature with 100% CO.

F-T Product Distribution. The averaged alpha plot distributions for the SBCR and CSTR high alpha experiments are displayed Figure 5. The durations of the runs were not sufficient to purge the system of the C<sub>30</sub> startup oil, as evidenced by the protuberance in the alpha plot near n=28-32 in both the SBCR and CSTR plots. Similar single alpha values of 0.86 and 0.87 were calculated for the SBCR and CSTR respectively. The weight percent distribution of light gas, gas, gasoline, diesel, and wax for both the CSTR and SBCR systems are listed in Table 2. Although the single alpha values were similar for the two reactor systems, the SBCR produced 60.9% C<sub>12</sub>+ versus 44.5% in the CSTR.

Corresponding plots of the alkene ratios for both reactor systems are shown in Figure 6. Beyond carbon number 10, the SBCR products were considerably more olefinic than the CSTR products. This is another indicator of the plug-flow nature of the SBCR. In addition, the higher conversion achieved in the SBCR would also tend to promote the formation of heavier alkenes.

### **Catalyst Attrition Study**

One objective of this study was to monitor the attrition performance of a spray-dried iron catalyst under synthesis conditions. A CAER-prepared catalyst having an elemental composition ratio of 100 Fe/4.4 Si/2.0 Cu/1.44 K and a mean diameter of 22  $\mu\text{m}$  was chosen for the study.

The activation and synthesis conditions for the attrition run are displayed in Table 3.

Conversion and  $\text{CO}_2/\text{CH}_4$  selectivity versus time-on-stream are plotted in Figures 7 and 8, respectively. Syngas conversion remained quite stable more than 136 hours on stream despite the fact that catalyst was removed from the reactor when slurry samples were taken. During this time period, the  $\text{H}_2/\text{CO}$  ratio was fixed at 0.7. After the attrition test, the  $\text{H}_2/\text{CO}$  ratio was increased to 2.0 in order to view the response of slurry level and gas hold-up (see the discussion in the following section).

A SEM image of the raw catalyst (before activation) is displayed in Figure 9. Generally, the particles had the appearance typical of a spray-dried powder: uniform spherical particles with a mean diameter of 22  $\mu\text{m}$ . Sampling of the reactor slurry was started immediately following the catalyst activation period and continued every 24 hours thereafter for five days during the synthesis operation. In order to remove the F-T product wax from the catalyst for subsequent analyses, slurry samples were soxhlet extracted in warm ortho-xylene for 24 hours followed by a 24 hour drying period in a 200°C oven.

A SEM image of the day-five sample (TOS 136 hours) is shown in Figure 10. Some spherical elements of the catalyst were visible and appeared to be of the same scale as the particles shown in Figure 9. However, smaller fragments of the catalyst particles were becoming more predominant in the day-five sample. This qualitative trend in decreasing particle size was verified by a particle size distribution analysis of the extracted slurry samples shown in Figure

11. The cumulative particle size distribution curves consistently move toward the left (to smaller particle diameters) for each day of time on stream. In terms of mean diameter, the catalyst particles seem to have rapidly attrited from 22 mm down to about 14 mm during activation, as shown in Figure 12. This observation may be misleading since the location of the slurry collection port (located at the bottom of the overhead receiver) may bias the samples toward smaller particles. Large diameter particles may accumulate in the reactor while finer particles are entrained in the riser tube into the overhead receiver. After 90-hours on stream, the mean diameter decreased in an exponential decay fashion down to 8 mm.

### **Effect of Syngas Composition on SBCR Performance**

In previous pilot plant runs, we encountered a problem of maintaining slurry level in the overhead receiver after increasing the  $H_2/CO$  ratio from 0.7 to 2.0. This resulted in a precipitous drop in gas conversion and significantly lowered the production rate of heavy waxes to point where the slurry level in the overhead separation vessel was apparently depleted after only 24 hours. Simultaneously, the selectivity was shifted toward lighter products such as methane. However, based on an overall mass balance of the system, this was not enough to account for the rapid drop in slurry volume (nearly 1.5 liters of slurry). Another explanation is that the gas hold-up within the reactor vessel had decreased after changing the syngas composition. Since the level had dropped so quickly below the measurable range, we were unable to conduct gas-hold measurements to verify this hypothesis.

At the conclusion of the attrition test, the syngas  $H_2/CO$  ratio was increased to 2.0 in order to revisit the slurry level problem in previous pilot plant runs. A plot of the average slurry level during the syngas transition period is shown in Figure 13. The slurry level dropped from 6.5" w.c. to 3.5 " w.c. (a slurry decrease of 1 liter in the overhead receiver) within 18 hours after

changing the syngas composition. If F-T products within the reactor were being depleted, the mass balance should have been greater than 100%, indicating a non-steady-state condition where the mass rate of products and unreacted syngas exiting the reactor would exceed the input mass rate of CO and H<sub>2</sub>. However, during this sampling period, the mass balance was only 93%, therefore, the drop in slurry level was not due to the reactor products being vaporized or consumed. Indeed, the low mass balance would indicate an accumulation within the reactor.

Gas hold-up measurements were not determined online. Instead, a procedure for estimating gas hold-up was developed (4) using the overhead vessel slurry level. The syngas flow was temporarily shutdown and the resulting drop in liquid level was recorded. This volume of slurry corresponding to the liquid level decrease was equivalent to the volume of gas that resided in the reactor during synthesis conditions. Previous gas hold-up measurements for the 0.7 H<sub>2</sub>/CO syngas ratio were in the range of 18-20%. However, the gas hold-up measurements 18 hours after changing the H<sub>2</sub>/CO ratio to 2.0 were only in the range of 7-8%. This decrease could be due to changes in both the gas and liquid phases. The gas phase density was considerable smaller due to the increased H<sub>2</sub> concentration in the syngas. Lighter gas bubbles would have a higher rise velocity through the reactor slurry, thereby lowering the gas hold-up to some degree. Chemical changes in the reactor wax could influence the slurry surface tension and viscosity which could also affect the average bubble rise velocity (6-8). Further studies are needed to elucidate cause of this phenomenon.

## **CONCLUSIONS**

Measured high alpha catalyst deactivation rates in the SBCR system were comparable to that of CSTR experiments under similar conditions. As observed in previous pilot plant tests, the SBCR exhibited properties of a plug-flow reactor as evidenced by its higher gas conversion and

the more olefinic properties of the F-T products as compared to that of the CSTR. Overall alpha distributions between the two reactor systems were quite similar.

Attrition tests in the SBCR system indicated that the most of the catalyst breakdown occurred between the time period of loading and activation; however, further research will be required to investigate the possibility of particle size classification/segregation at the sampling points. After activation, the catalyst particle mean diameter decreased in an exponential decay fashion.

A decreased gas hold-up within the SBCR was found to be the main cause of losing slurry level after changing the H<sub>2</sub>/CO ratio from 0.7 to 2.0. Lighter gas bubbles and changes in the reactor wax over time could influence average bubble rise velocity.

## NOMENCLATURE

a(t)	Catalyst activity function
k <sub>d</sub>	First order deactivation rate constant, day <sup>-1</sup>
r <sub>F-T</sub>	Rate of Fischer-Tropsch synthesis, mole s <sup>-1</sup> Fe·g <sup>-1</sup>
SV	Gas space velocity, Sl h <sup>-1</sup> Fe·g <sup>-1</sup>
t'	Time after maximum CO conversion, hours
TOS	Time-on-stream, hours
U <sub>g</sub>	Superficial gas velocity based on inlet reactor conditions, cm s <sup>-1</sup>
X <sub>CO</sub>	CO conversion
ε <sub>g</sub>	Gas hold up fraction in the reactor vessel, l l <sup>-1</sup>

## REFERENCES

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<b>Table 1</b>		
<b>Operating Conditions: SBCR and CSTR Comparison Experiments</b>		
	SBCR High Alpha	CSTR High Alpha
Catalyst	100Fe/5.1Si/2.0 Cu/5K	100Fe/5.1Si/2.0 Cu/5K
Initial Cat. loaded wt%	20	20
<b>Catalyst. Activation:</b>		
Gases	CO	CO
Gas space velocity (SL/hr-g Fe)	1.0	1.0
Temperature (°C)	230	230
Pressure (atm.)	12	12
<b>Synthesis Conditions:</b>		
H <sub>2</sub> /CO	0.7	0.7
Gas space velocity (SL/hr-g Fe)	5.0	4.95
Temperature (°C)	270	270
Pressure (MPa)	1.21	1.21
Gas superficial velocity (cm/sec)	3	Stirred speed 750 RPM

<b>Table 2</b>			
<b>F-T Product Report</b>			
<b>CSTR/SBCR Comparison: High Alpha Catalyst</b>			
<b>Groups</b>	<b>Range</b>	<b>CSTR wt% (2 samples)</b>	<b>SBCR wt% (4 samples)</b>
<i>Light Gas</i>	C <sub>1</sub>	4.8 ± 1.8	2.2 ± 0.8
<i>Gas</i>	C <sub>1</sub> to C <sub>4</sub>	19.7 ± 4.1	13.1 ± 4.4
<i>Gasoline</i>	C <sub>5</sub> to C <sub>11</sub>	31.0 ± 8.1	23.8 ± 8.6
<i>Diesel</i>	C <sub>12</sub> to C <sub>18</sub>	17.2 ± 2.6	27.2 ± 4.7
<i>Wax</i>	C <sub>19</sub> plus	27.3 ± 11.3	33.7 ± 14.1
	C <sub>12</sub> plus	44.5 ± 13.9	60.9 ± 12.3
	C <sub>5</sub> plus	75.5 ± 5.9	80.85 ± 5.2

<b>Table 3</b>	
<b>Operating Conditions: SBCR Attrition Experiment</b>	
	SBCR
Catalyst	100Fe/4.4 Si/2.0 Cu/1.44 K
Initial Cat. loaded wt%	20
<b>Catalyst Activation:</b>	
Gases	CO
Gas space velocity (SL/hr-g Fe)	1.0
Temperature (°C)	270
Pressure (atm.)	12
<b>Synthesis Conditions:</b>	
H <sub>2</sub> /CO	0.7
Gas space velocity (SL/hr-g Fe)	5.0
Temperature (°C)	270
Pressure (MPa)	1.21
Gas superficial velocity (cm/sec)	3

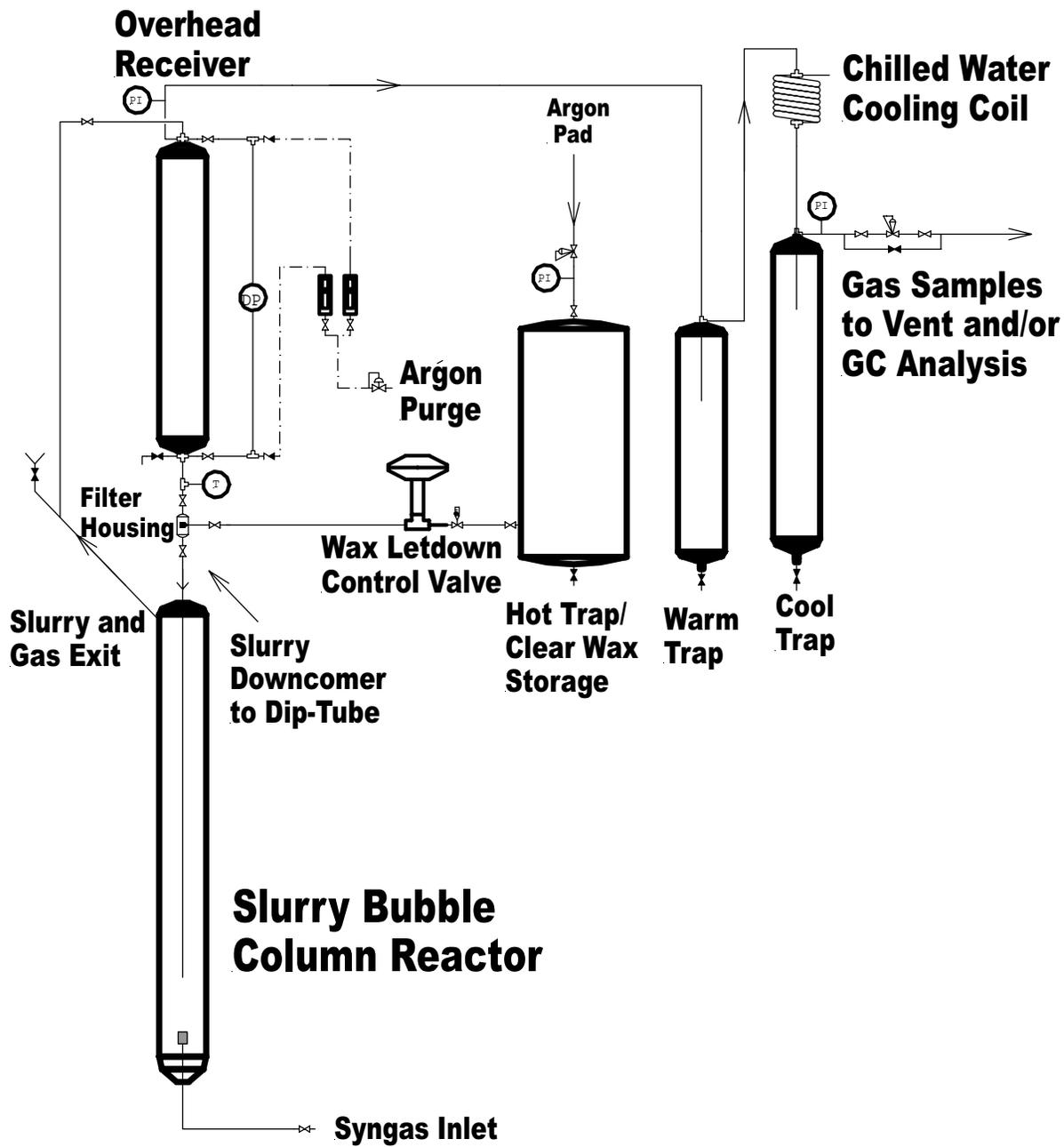
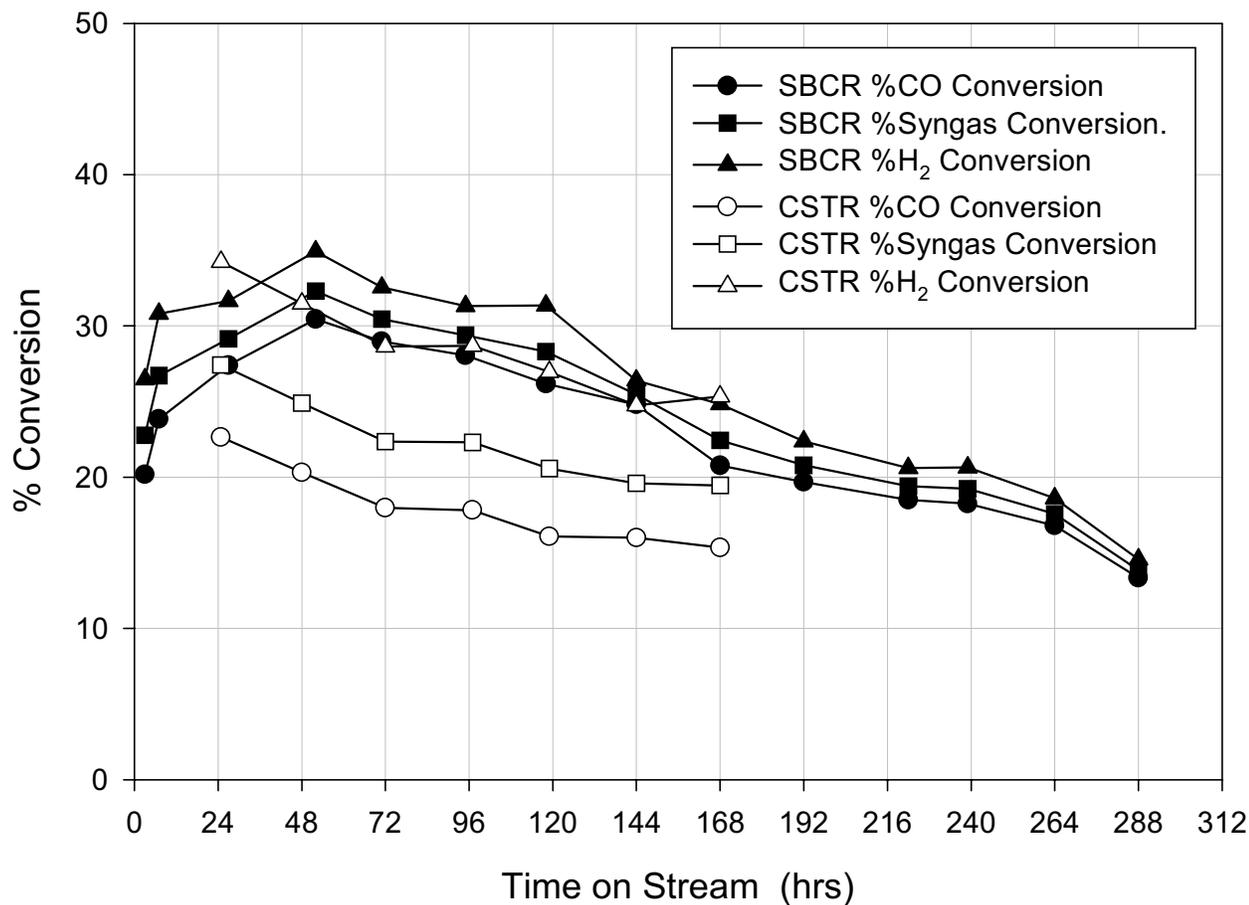


Figure 1. Experimental SBCR system.

## Conversion Comparison of SBCR and CSTR Reactors

SBCR Run JKN-002; CSTR Run BAO-064



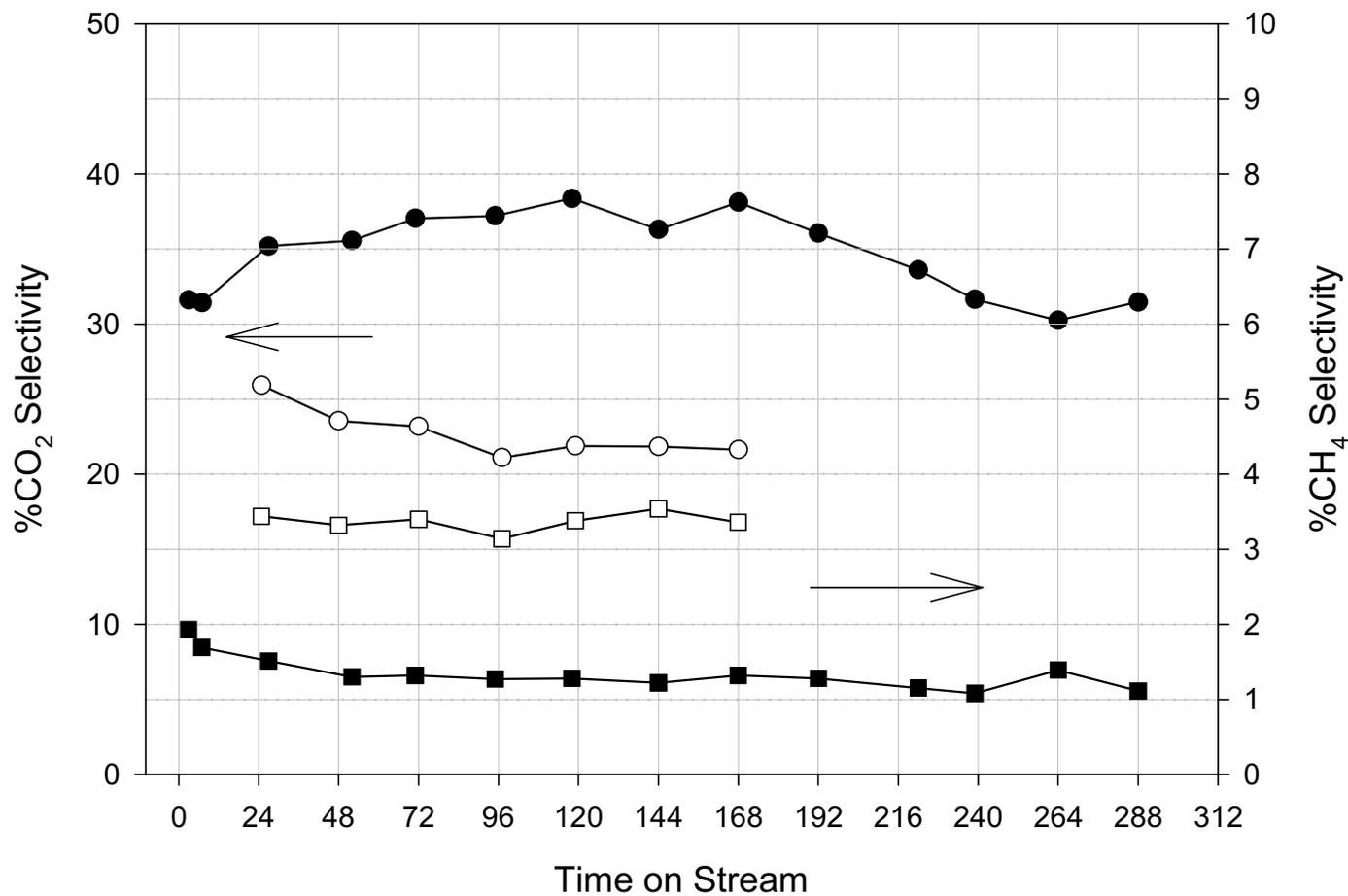
Starting Conditions:

T = 230 °C; P = 175 psig; H<sub>2</sub>/CO Ratio = 0.7; SV = 5.0 slph/g Fe

Catalyst: RLS 100 Fe/5.1 Si/2.0 Cu/5K

Figure 2. Conversion comparison: SBCR and CSTR.

5 cm-SBCR Run JKN-002  
1/22/01



Starting Conditions:

T = 230 °C; P = 175 psig; H<sub>2</sub>/CO Ratio = 0.7; SV = 5.0 slph/g Fe

Catalyst: RLS 100 Fe/5.1 Si/2.0 Cu/5K

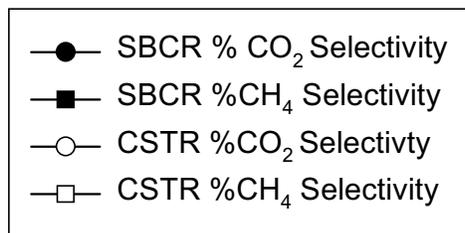


Figure 3. Selectivity comparison: SBCR and CSTR.

Activity Decline Comparison  
 SBCR JKN-001 and CSTR BAO-064  
 High alpha Catalyst: 100Fe/5.1Si/2.0Cu/5K  
 Activated at 230 °C with CO

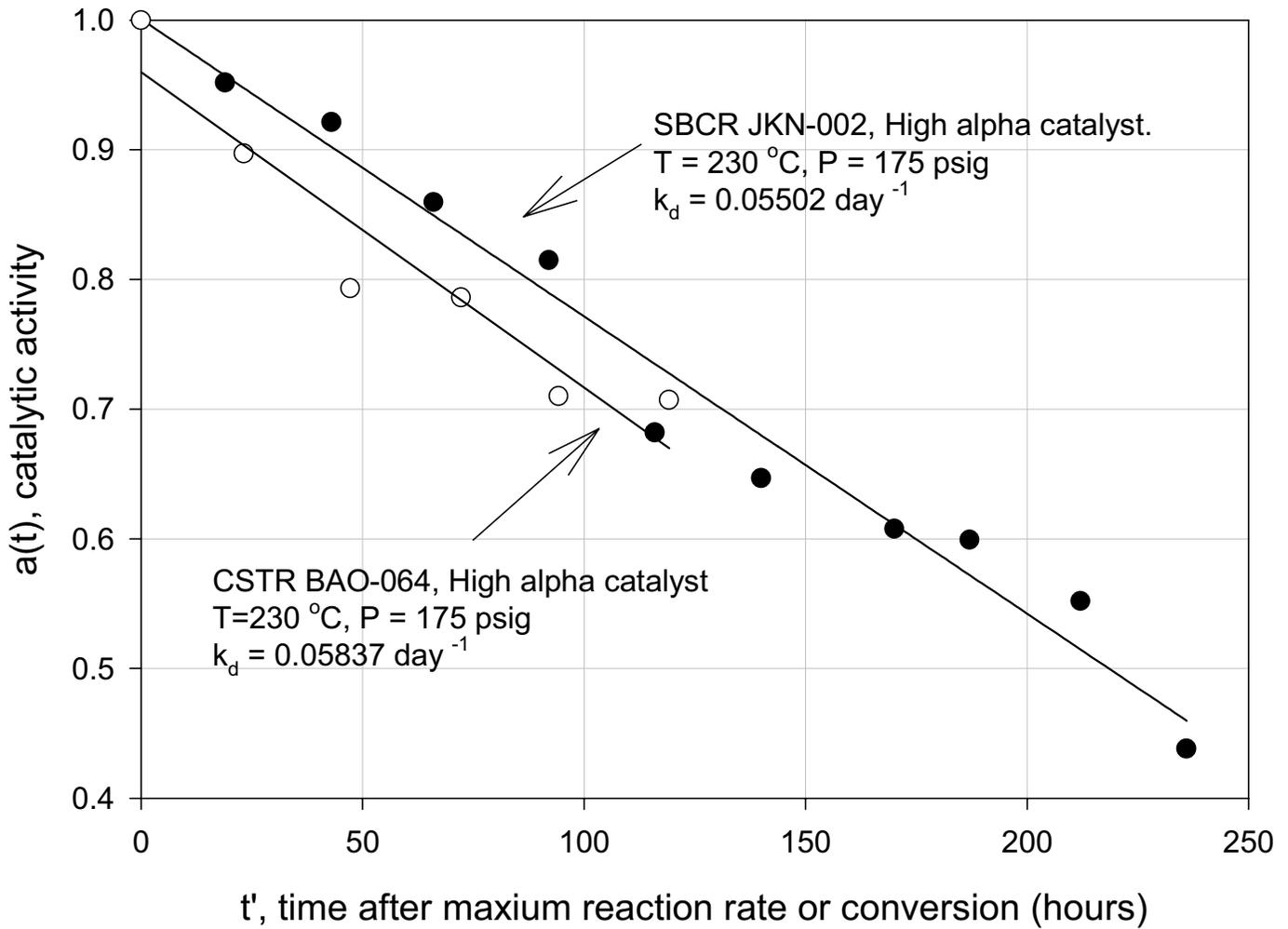


Figure 4. Catalyst deactivation: SBCR and CSTR.

# Alpha Plot Comparison Between Reactor Types

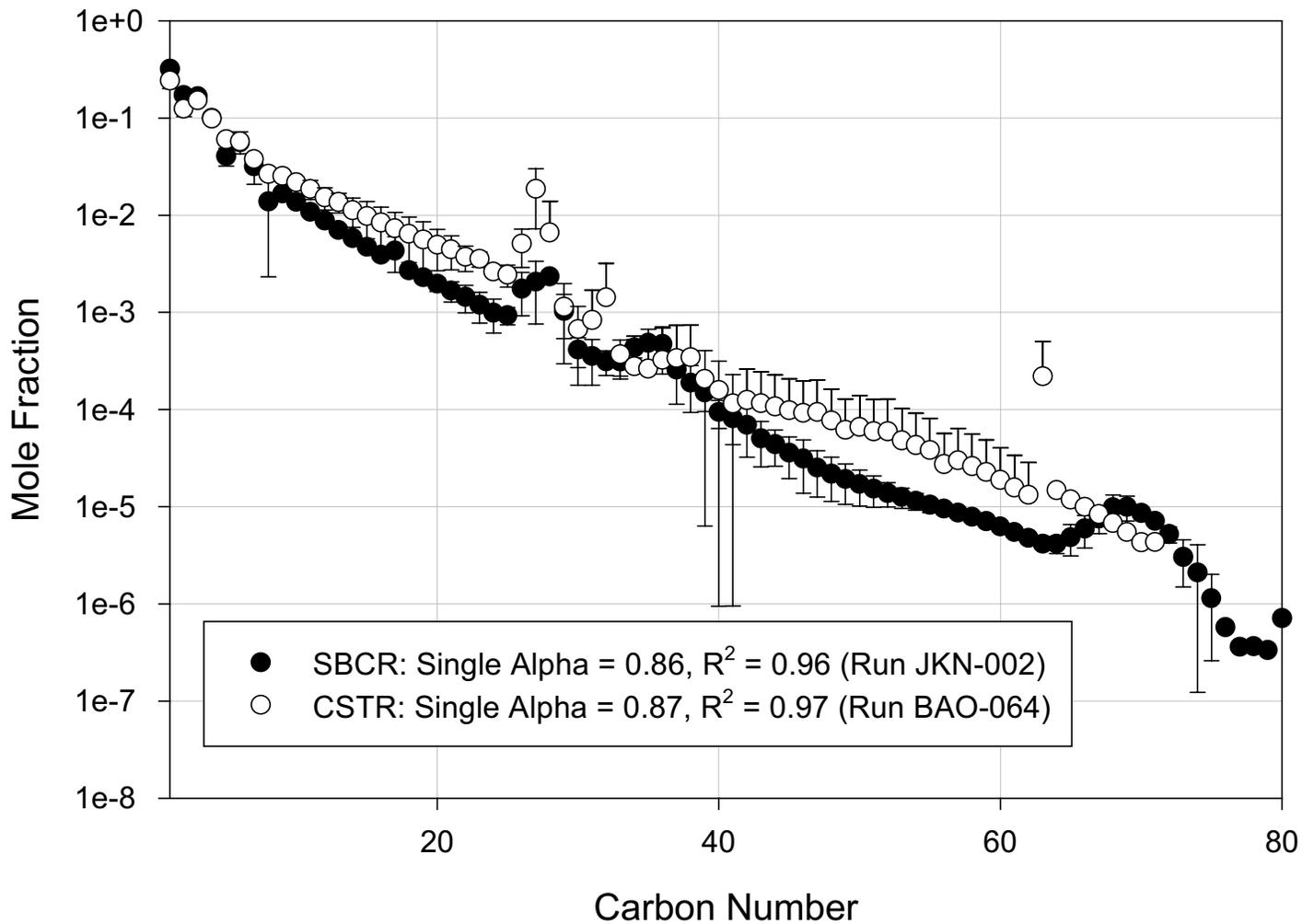


Figure 5. Alpha plot comparison between SBCR and CSTR.

# Alkene Ratio Comparison for SBCR & CSTR

SBCR Run JKN-002 and CSTR Run BAO-064

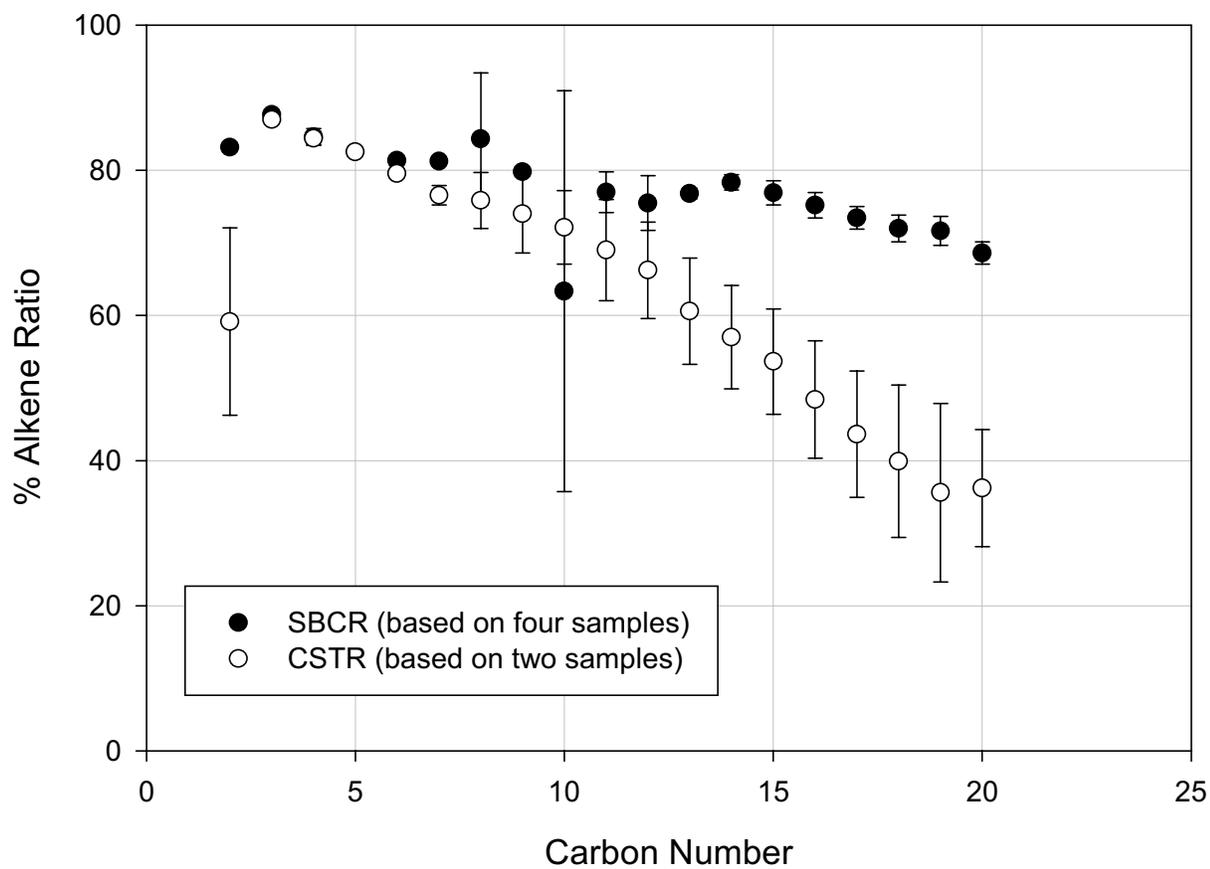
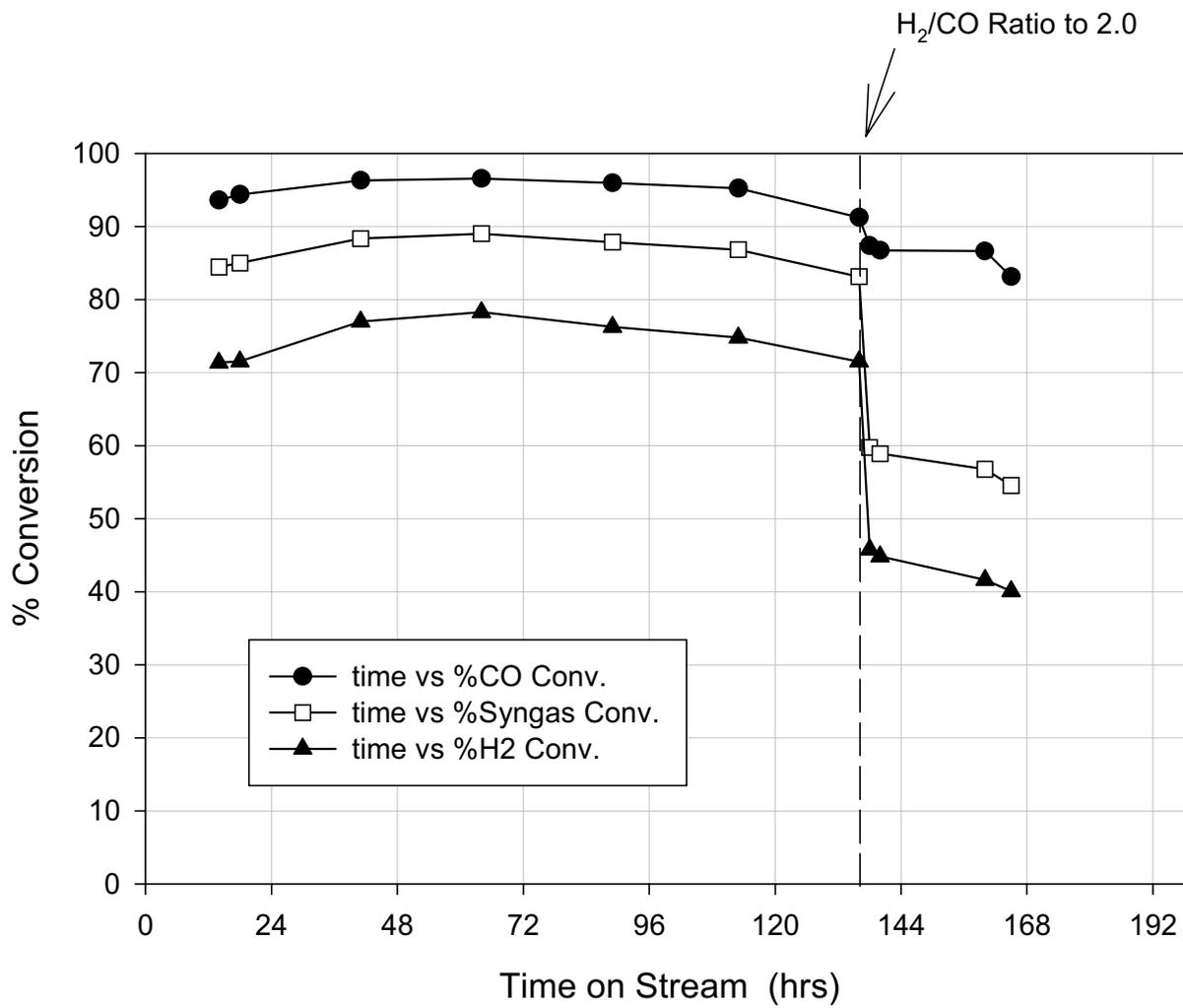


Figure 6. Alkene ratios: SBCR and CSTR comparison.

5 cm-SBCR Run JKN-003  
3/1/01



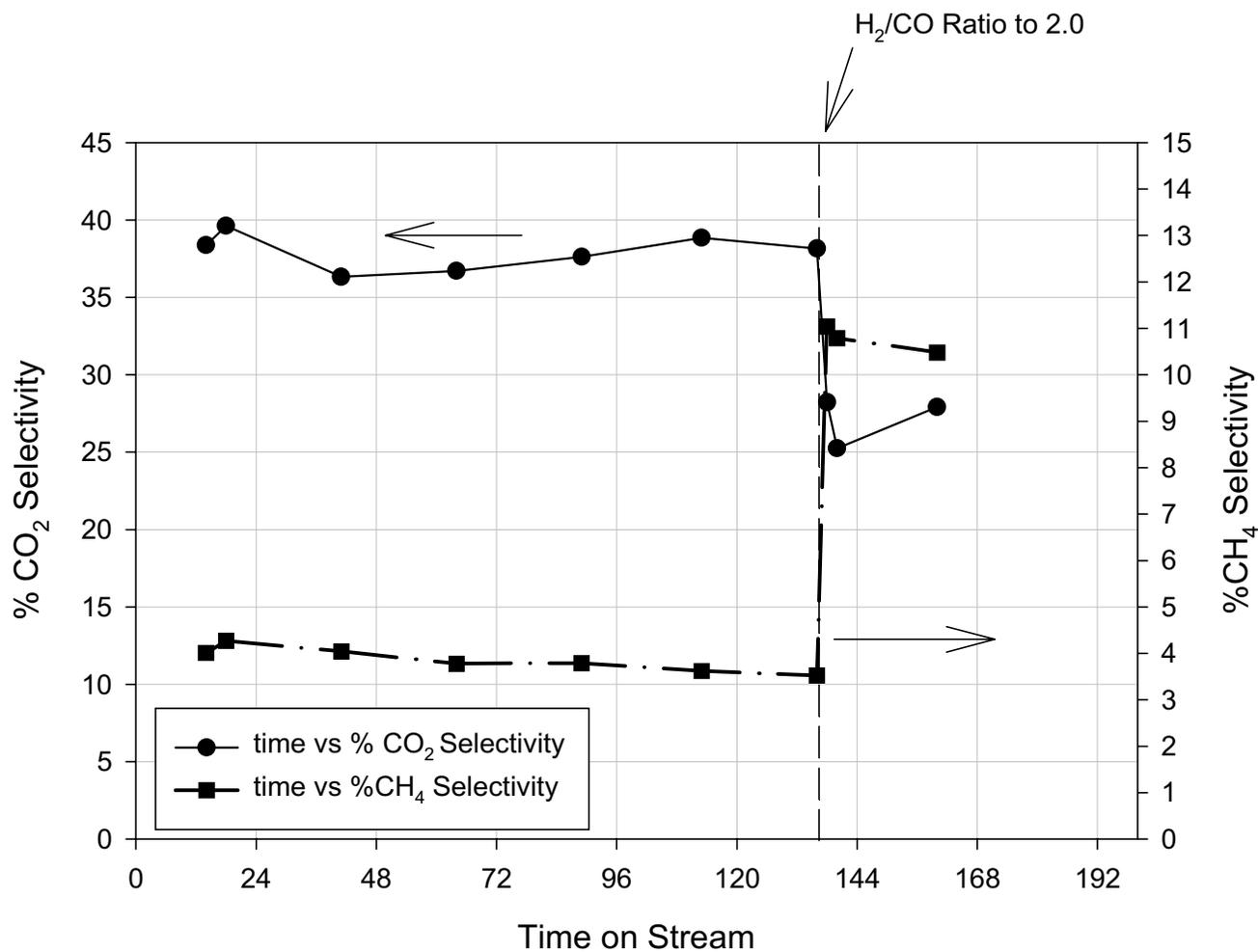
Starting Conditions:

T = 270 °C; P = 175 psig; H<sub>2</sub>/CO Ratio = 0.7; SV = 5.0 slph/g Fe

Catalyst: 100 Fe/4.4 Si/2.0 Cu/1.44K

Figure 7. Conversion versus TOS, Run JKN-003 (attrition run).

5 cm-SBCR Run JKN-003  
3/1/01



Starting Conditions:

T = 270 °C; P = 175 psig; H<sub>2</sub>/CO Ratio = 0.7; SV = 5.0 slph/g Fe

Catalyst: 100 Fe/4.4 Si/2.0 Cu/1.44K

Figure 8. CO<sub>2</sub> and CH<sub>4</sub> selectivity, Run JKN-003 (attrition run).

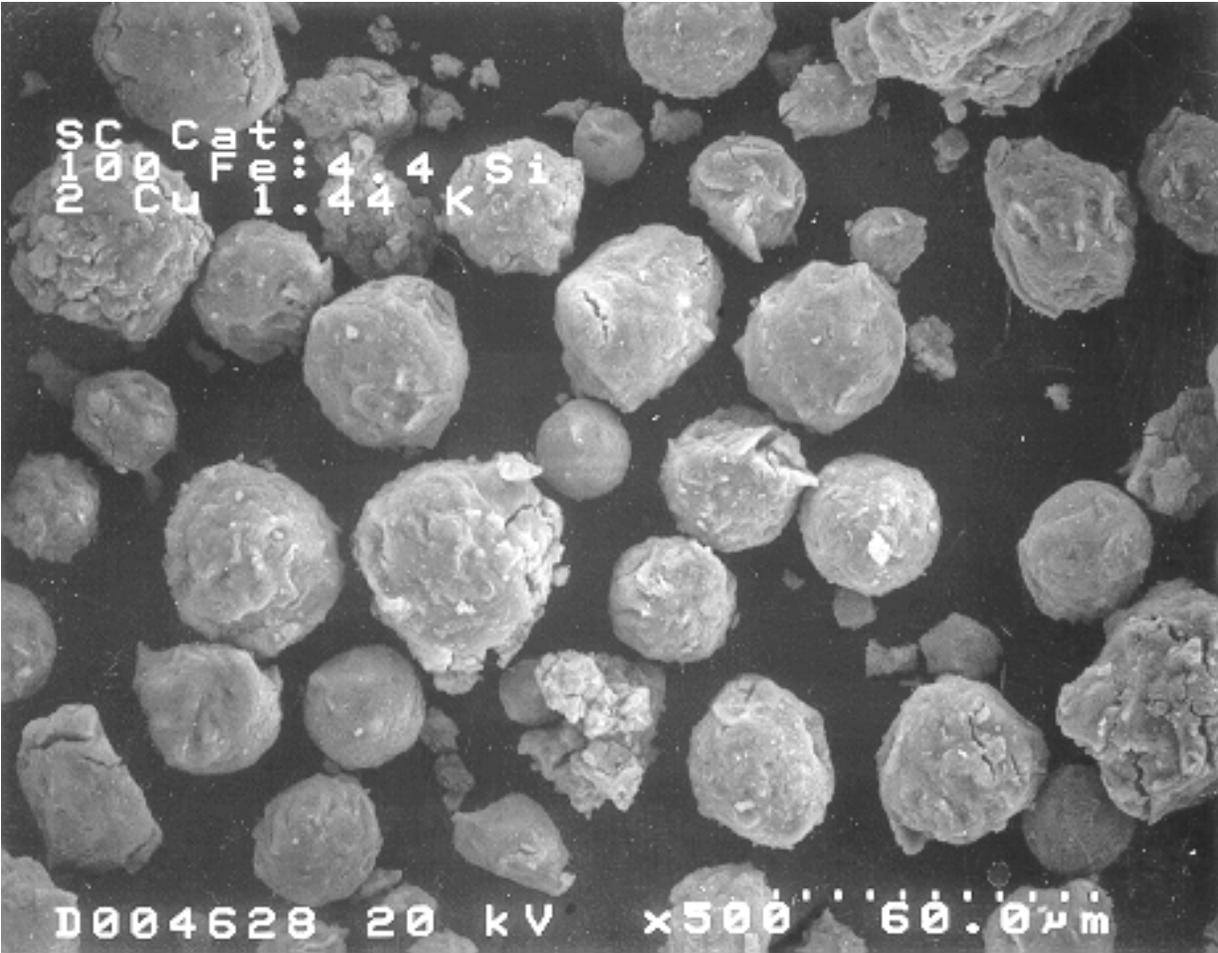


Figure 9. Catalyst SEM image (before activation).

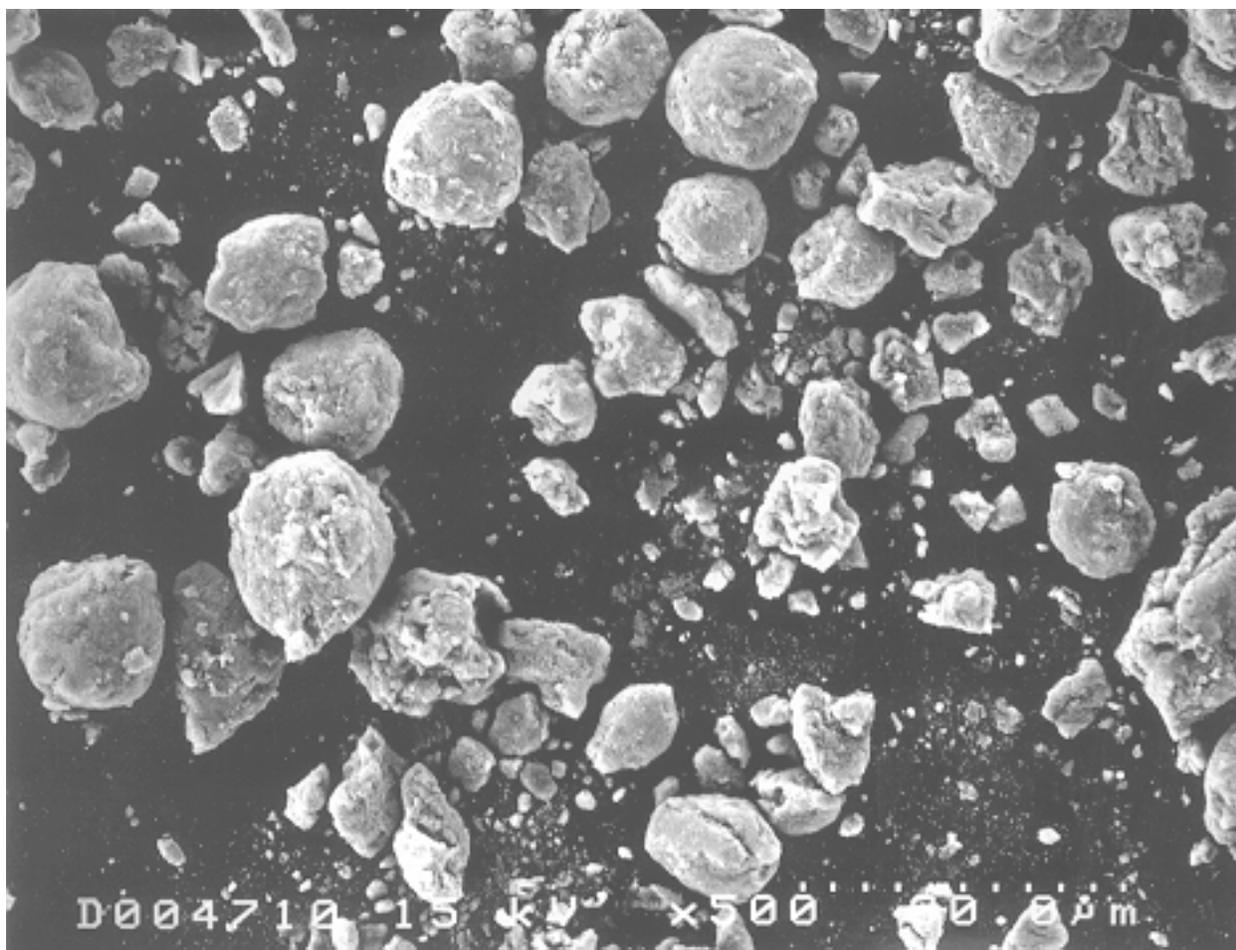


Figure 10. Catalyst reactor samle after five days on stream.

# SBCR Particle Size Distribution: Run JKN-003

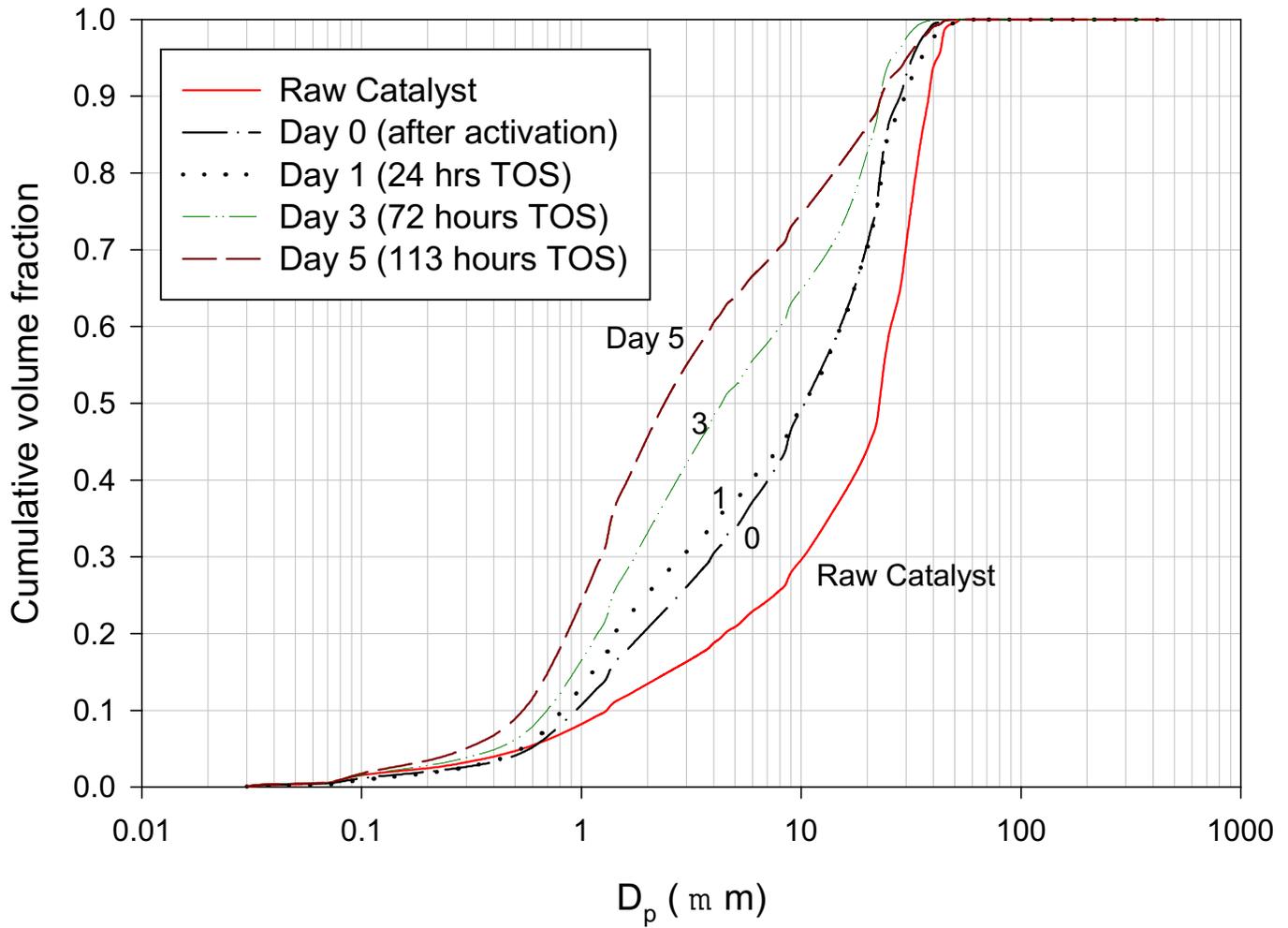
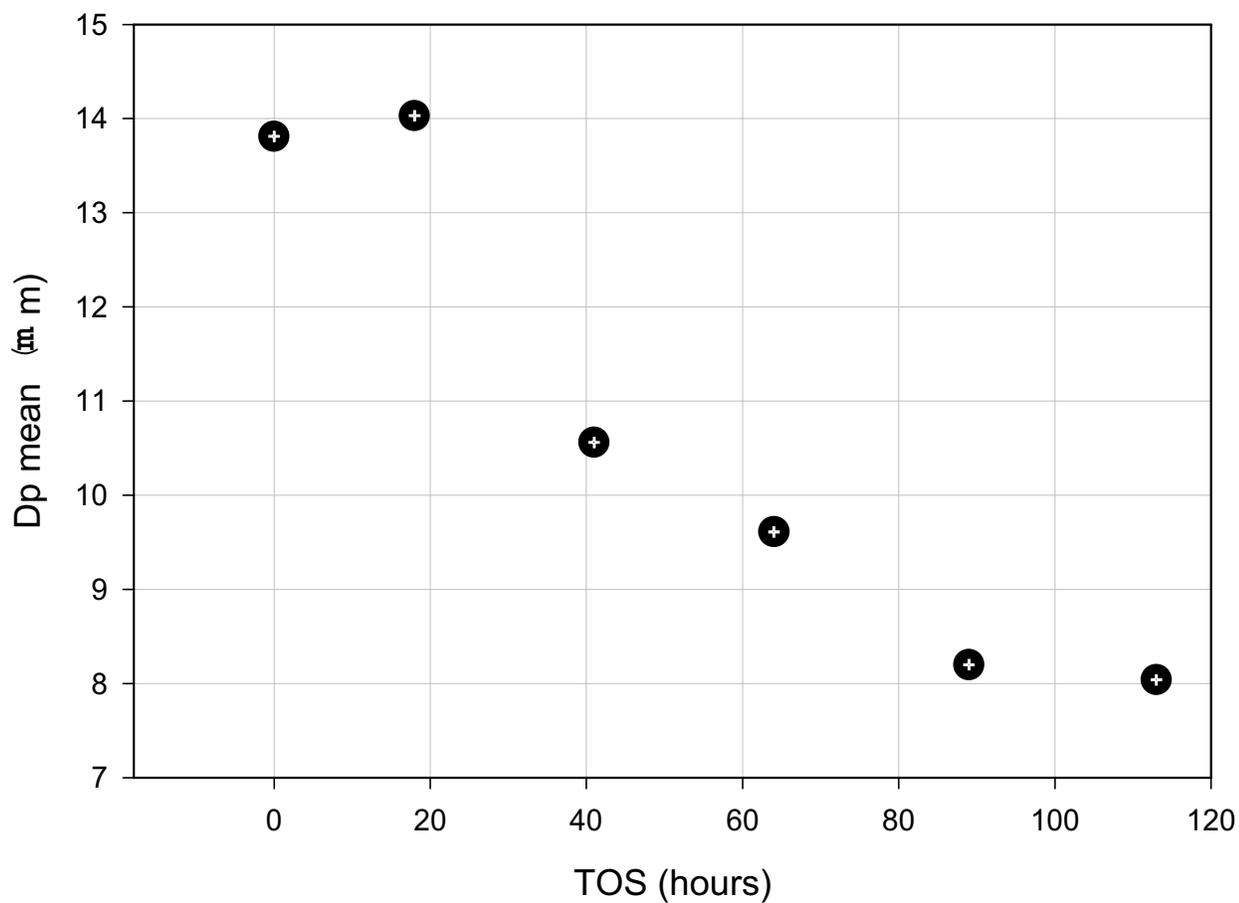


Figure 11. Catalyst size distribution for various days.

## Catalyst Mean Particle Size vs. Time on Stream



SBCR Run JKN-003

Spray Dried Catalyst: 100 Fe:4.4 Si:2.0 Cu:1.44K

Starting Dp mean: 22.05 μm before activation

SV= 5.0 slph/g Fe, T=270 °C, P = 175 psi

Figure 12. Catalyst mean particle size versus TOS.

### Reactor Slurry Level for SBCR Run JKN-003 (after transitioning to 2.0 H<sub>2</sub>/CO ratio)

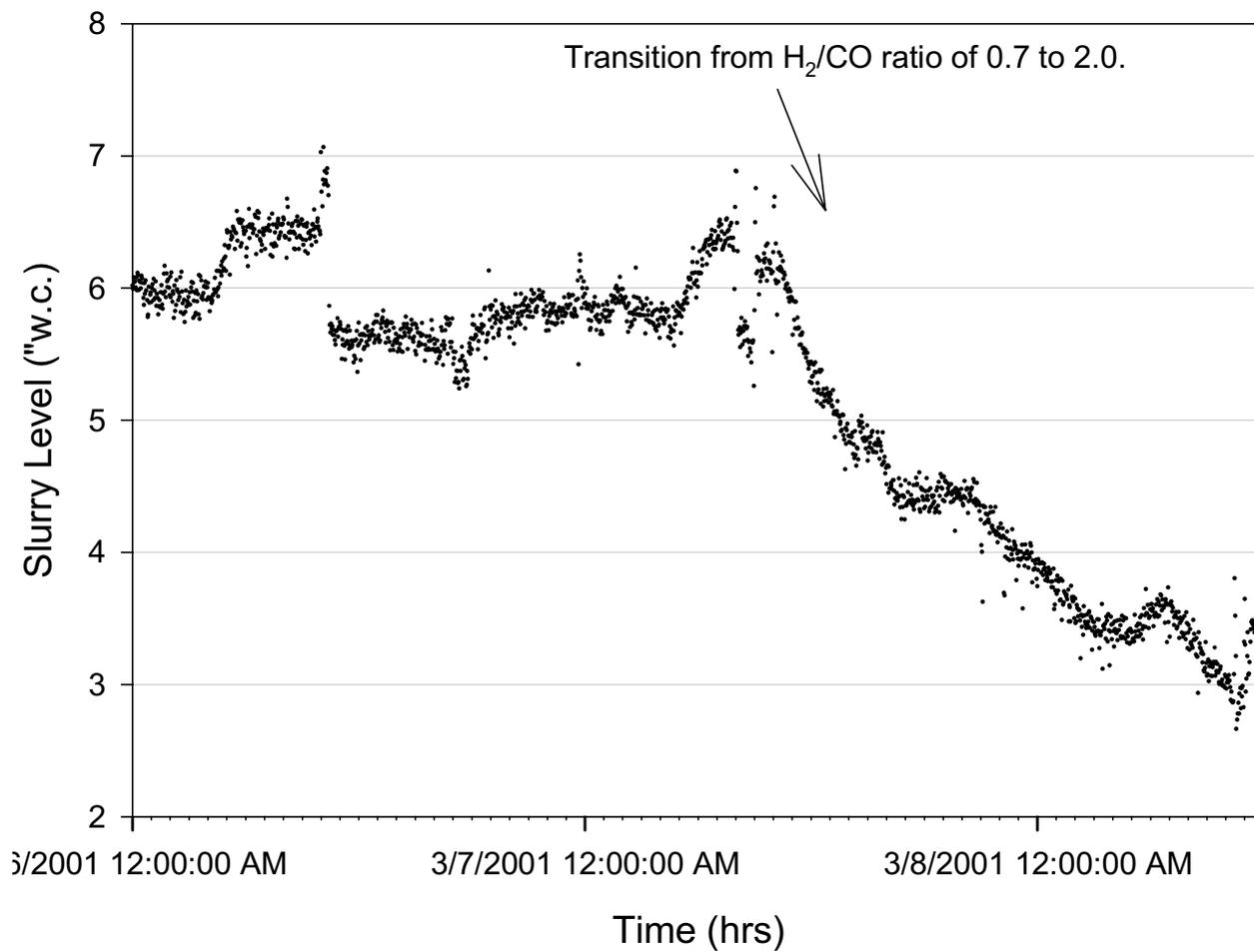


Figure 13. Reactor slurry level signal trace during syngas transition.