Chemical-Looping Process in a Coal-to-Liquids Configuration

DOE/NETL-2008/1307

Independent Assessment
of the Potential of Chemical-Looping in the Context of a
Fischer-Tropsch Plant

December 2007

Disclaimer

The Ohio State University Chemical-Looping Process in A Coal-to-Liquids Configuration

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Independent Assessment
Of the Potential of the OSU Chemical Looping Concept

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Acknowledgements

Executive Summary

At the request of the Department of Energy (DOE) National Energy Laboratory (NETL), Noblis performed an independent technical assessment of the potential of chemical looping in the context of a Fischer-Tropsch coal-to-liquids (CTL) plant. Several different concepts of chemical looping are being developed. In this analysis the concept under development by Ohio State University (OSU) was assessed to confirm that the thermochemical operations were in heat balance at temperatures compatible with an operable system, and to develop simulations of an entire coal to Fischer-Tropsch (F-T) liquids process, including the proposed looping scheme. Noblis was also asked to compare the technical performance results of a CTL plant with chemical looping with a conventional coal-to-liquids (CTL) system.

The Ohio State University (OSU) is developing a chemical looping scheme that could find application for treating tail gas from a coal based Fischer-Tropsch (F-T) Coal-to-Liquids (CTL) process. This chemical looping concept uses iron oxide (Fe2O3) to react with the unreacted synthesis gas (H_2 and CO) and light hydrocarbons in the effluent tail gas from an F-T reactor. This reaction that takes place in a Fuel Reactor produces CO_2, H_2O and reduced iron. The reduced iron is then reacted with steam to produce hydrogen that can be recycled to the F-T reactor to adjust the input hydrogen to carbon monoxide ratio.

The steam/iron reaction produces Fe3O4 and this is pneumatically transferred with air to the fuel reactor, during which it is further oxidized back to Fe_2O_3. The hot solids thus transferred are the source of heat for the subsequent reduction of Fe_2O_3 to Fe in the fuel reactor.

This chemical looping concept has several potential technical advantages. The hydrogen produced, when added to the gasifier output, can produce a suitable H_2/CO ratio without the need for the shift reactor, the CO_2 produced in the fuel reactor from the reduction of the iron oxide with the CO and light hydrocarbons can be captured at pressure thus reducing CO_2 compression power requirements, and the waste heat recovered from the processes is of a very high quality, facilitating efficient steam generation equipment.

Noblis performed Aspen Plus and in-house spreadsheet simulations and confirmed that the thermochemical operations were in heat balance at temperatures compatible with an operable system. Then simulations of the entire integrated coal to F-T liquids process, including the proposed looping scheme, were performed and the technical results from this analysis were then compared to a conventional CTL system. Both systems used cobalt F-T catalysts.

The chemical looping CTL configuration analyzed in this report processes a mixture of F-T tail gas and clean synthesis gas. OSU reports that the small amount of CO_2 in the gasifier exit could be handled by the fuel reactor with no significant effect so it is unnecessary to remove CO_2 from the synthesis gas. The two stage Selexol gas cleaning system that is used in the conventional CTL scheme can now be replaced with a cheaper MDEA sulfur removal system. To address concerns over sulfur poisoning of the cobalt catalyst, an additional guard bed was inserted before the F-T reactor.

This overall system is in balance and theoretically very efficient. The high temperatures involved are challenging for materials properties but probably feasible. Handling and transferring large quantities of hot solids between reactors could well be an issue from an engineering viewpoint.

If hydrogen is used to meet fuel gas requirements, then the looping system permits 100% of the carbon in the coal to be retained in the liquid fuels or sequestered. In the conventional CTL case, about 10% of total carbon not retained in the fuel is emitted to the atmosphere. As a result of the lower carbon emissions and greater yield, the total carbon emissions per barrel produced and used is about 19% less for the proposed chemical looping system compared to the conventional CTL configuration. These results of the comparative technical analysis between the chemical looping scheme and the conventional CTL scheme indicate that the looping case increased liquid production from the same coal feed by about 10% (12,076 vs. 11,002 barrels/day).

OSU personnel report that the chemical reactions involved in the looping system go to completion in their tests. To date Noblis has insufficient data to simulate how the system would perform if some equilibrium level of unwanted reactants remained at each stage. This is an important area that should be investigated further because if there is a significant amount of CO and H_2 in the fuel reactor effluent then this would have to be sequestered with the CO_2 with a resulting loss of efficiency.

In addition to this concern, the following areas would need further study in order to better understand this novel process: 1) more detailed system economics; 2) more expansive and detailed simulations to discover the impact of incomplete reactions at each stage and 3) other system integration schemes as well as the case when iron catalyst is used in the F-T process.

Introduction

At the request of the Department of Energy (DOE) National Energy Laboratory (NETL), Noblis performed an independent technical assessment of the potential of one of the Ohio State University's (OSU) chemical looping concepts. Noblis was asked to confirm that the thermochemical operations were in heat balance at temperatures compatible with an operable system, and to develop simulations of an entire coal to Fischer-Tropsch (F-T) liquids process, including the proposed looping scheme. Noblis was also asked to compare the technical performance results of this analysis with a conventional coal-to-liquids (CTL) system.

Background

Liquid fuels such as gasoline, kerosene, and diesel are important fuels to power a modern society. The United States consumes over 380 million gallons of gasoline alone every day (EIA, 2007). At present, liquid fuels are mainly produced from crude oil through the oil refining process. However, the soaring oil price, lack of oil reserve in U.S., and insecure foreign oil supply has made it imperative for the United States to look into alternative feedstocks for liquid fuel production.

Besides crude oil, coal and natural gas can also used for bulk production of liquid fuel through the well known Fischer-Tropsch (F-T) synthesis of coal or natural gas derived syngas. Among the three major fossil fuels, coal is a more favorable choice for the United States due to its abundant resources and relatively stable price.

In the state-of-the-art indirect coal to liquid (CTL) process, coal is first gasified into syngas, which is a mixture of CO and H_2. The resulting syngas is then purified and conditioned. The conditioned syngas is further synthesized into a mixture of hydrocarbon fuels through the F-T synthesis. The resulting hydrocarbon mixture can then be refined to produce liquid fuels such as naphtha, kerosene, and diesel. The elaborate procedures involved in the state-of-the-art CTL process make it capital intensive as well as energy consuming when compared to gas-to-liquid (GTL) and oil refining processes.

Professor L.-S. Fan's research group at the Ohio State University has developed three novel chemical-looping schemes: the coal direct chemical looping process, the syngas chemical looping process, and the calcium looping process (Thomas et al, 2005; Gupta et al, 2005; Iyer et al, 2006 a,b; Fan et al, 2007;). The configuration considered in this report utilizes chemical looping to enhance the yield of the state-of-the-art CTL process by converting the tail gas from the F-T reactor into hydrogen, an important feedstock for the liquid fuel synthesis. The chemical looping CTL process examined in this report is characterized by the ease in CO_2 capture, thereby significantly reducing CO_2 emissions from the CTL process.

The OSU Chemical Looping Concept:

Professor L.-S. Fan's research group at The Ohio State University (OSU) is developing the chemical looping scheme illustrated in Figure 1 for treating tail gas from a coal based F-T synthesis reactor system. The concept uses iron oxide (Fe_2O_3) to react with the unreacted synthesis gas (H_2, CO) and light hydrocarbons in the effluent tail gas from an F-T reactor. This reaction takes place in a so-called **Fuel Reactor** to produce CO_2, H_2O and reduced iron. The reduced iron is then reacted with steam in the **Hydrogen Reactor** to produce hydrogen for recycle to the F-T reactor. The following simplified equations illustrate the concept.

Fuel Reactor:

The following equations are representative of the reactions occurring in the fuel reactor of the chemical looping scheme:

$$3\ CO + Fe_2O_3 \leftrightarrows 3\ CO_2 + 2\ Fe \qquad\qquad \Delta H = -17{,}405\ Btu/\#mole$$

$$3\ H_2 + Fe_2O_3 \leftrightarrows 3\ H_2O + 2\ Fe \qquad\qquad \Delta H = 27{,}290\ Btu/\#mole$$

$$C_2H_4 + 2\ Fe_2O_3 \leftrightarrows 4\ Fe + 2\ CO_2 + 2\ H_2O \qquad\qquad \Delta H = 83{,}920\ Btu/\#mole$$
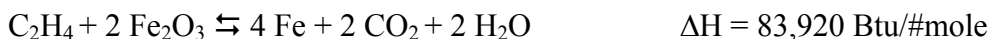
Hydrogen Reactor:

The following equations are representative of the reactions occurring in the hydrogen reactor of the chemical looping scheme:

$$3\ Fe + 4\ H_2O \leftrightarrows Fe_3O_4 + 4\ H_2 \qquad\qquad \Delta H = -42{,}340\ Btu/\#mole$$

The Fe_3O_4 exit from the steam/iron reaction is pneumatically transferred with air to the fuel reactor, during which it is further oxidized to Fe_2O_3.

$$4\ Fe_3O_4 + O_2 \leftrightarrows 6\ Fe_2O_3 \qquad\qquad \Delta H = -207{,}800\ Btu/\#mole$$

This reaction is exothermic and part of the heat generated will heat up the solids. The hot solids thus transferred are the source of heat for the subsequent reduction of Fe_2O_3 to Fe in the fuel reactor.

Several potential technical advantages are offered by the looping system

1. The hydrogen produced, when added to the gasifier output, can produce a suitable H_2/CO ratio without the need for the shift reactor.
2. All of the CO_2 produced in the fuel reactor from the reduction of the iron oxide with the CO and light hydrocarbons can be captured at pressure at the exit of the fuel

reactor. This reduces CO_2 compression power requirements. The water produced can be removed by condensation.

3. The waste heat available from the processes is of a very high quality, facilitating efficient steam generation equipment.
4. There are potential capital cost savings depending on the actual cost of the looping system. This will be discussed later.
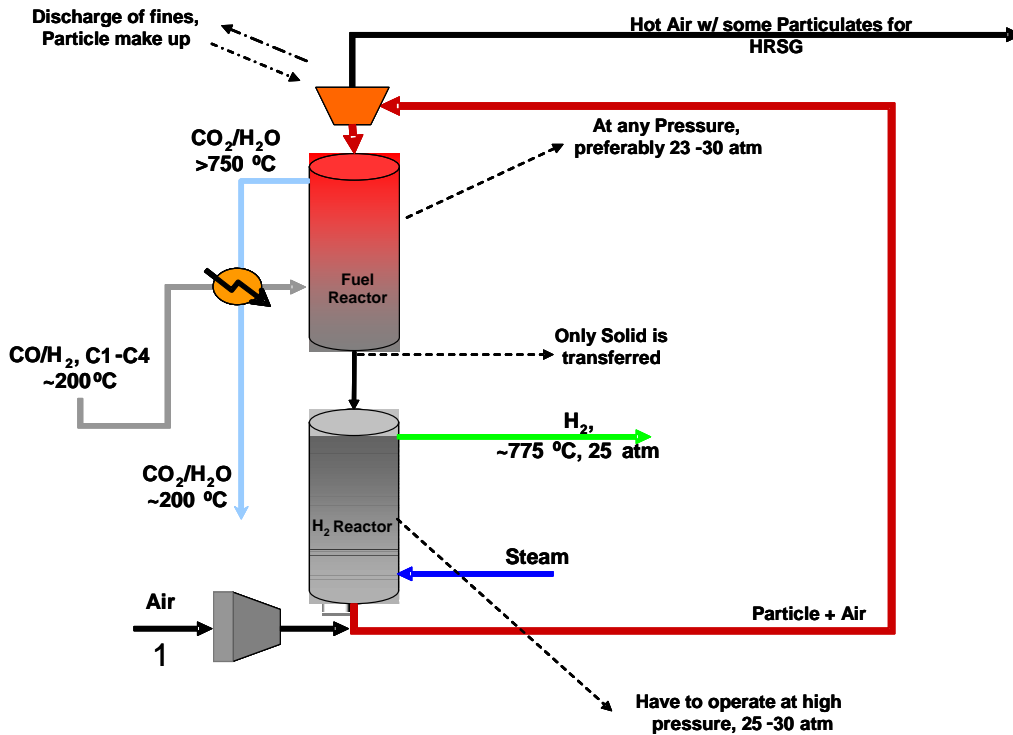
Figure 1: Chemical Looping For Hydrogen Production

Analysis Methodology

Extensive lab scale and bench scale demonstration experiments on the chemical-looping process have been performed at The Ohio State University. Key experimental data for the simulation has been obtained from OSU from publications (Fan et al., 2007, Gupta et al, 2007) as well as from personal communications. According to the demonstrations carried out in both fixed bed and moving bed demonstration units, higher than 99.8% CO, H_2, methane, acetylene, propane, and their mixtures are converted into CO_2 and H_2O by the iron oxide composite particles developed at OSU.

During the experiments, part or all of the iron oxide composite particles are reduced to metallic iron. The reduced iron composite particles are also shown to be able to produce hydrogen with a minimum purity of 99.7% (dry basis) while being oxidized back to Fe_3O_4.

Using the chemical looping performance data provided by OSU, Noblis simulated the complete CTL process from coal to liquid products by developing a conceptual design that integrated the chemical-looping process with the overall CTL process.

Noblis first performed both Aspen Plus and in-house spreadsheet simulations to confirm that the thermochemical operations were in heat balance at temperatures compatible with an operable system. When this proved to be so, simulations of the entire integrated coal to F-T liquids process, including the proposed looping scheme, were performed and the technical results from this analysis were then compared to a conventional CTL system.

It should be noted that the looping scheme investigated in this report is based on an F-T reactor using a Cobalt based catalyst rather than iron based catalyst which is frequently used in a conventional design when using a coal feedstock. With cobalt catalyst the H_2/CO ratio entering the F-T reactor must be about 2.08/1 because, unlike iron-based catalysts, cobalt does not possess any water gas shift capability. Further studies would be necessary to evaluate the performance of this Chemical Looping scheme when iron based catalyst is used.

Analysis

Technical Performance

Early on in the analysis, it was found by iteration that the F-T reactor must operate at very low (~50%) conversion in order for the tailgas to produce enough hydrogen to balance the H_2/CO ratio of the entire gasifier output. It was discovered both by Noblis and OSU that the system could be improved if some clean syngas as well as F-T tailgas, was fed to the fuel reactor.

This modification actually simplifies the looping process by reducing the amount of hydrogen required to balance the H_2/CO ratio of the F-T feed, and permits the F-T reactor to be operated at a more typical conversion per pass of ~75%. It was also found in the simulation that the small amount of CO_2 in the gasifier exit could be handled by the fuel reactor with no significant effect. Because it was now unnecessary to remove CO_2 from the synthesis gas, the Selexol gas cleaning system that is used in the conventional CTL scheme can now be replaced with a cheaper MDEA sulfur removal system. This configuration was used as the Noblis Case 1 for the looping system. To address concerns over sulfur poisoning of the cobalt catalyst, an additional guard bed was inserted before the F-T reactor. The overall configuration is shown in Figure 2. Detailed flows are given in Table 1 (both at the end of the report).

Many different waste heat management configurations were considered for the looping case. In the case shown, H_2 and excess steam are used to heat the feed to the fuel reactor. The very hot (1,100+ ºC) depleted air from the solid transfer/oxidation system is filtered and fed to an expander for power generation. The hot H_2O/CO_2 from the fuel reactor is used to raise and superheat steam in a HRSG. The overall system is in balance and theoretically very efficient. The high temperatures involved are challenging for materials properties but feasible.

If hydrogen is used to meet fuel gas requirements, as we propose, then the looping system permits 100% of the carbon in the coal to be retained in the liquid fuels or sequestered. In the conventional CTL case, about 10% of total carbon not retained in the fuel is emitted to the atmosphere. As a result of the lower carbon emissions and greater yield, the total carbon emissions per barrel produced and used is about 19% less for the proposed chemical looping system compared to the conventional CTL configuration.

Economic Performance

The Noblis effort did not include a detailed economic analysis. The effort was limited to determining capital and operating costs relative to a CTL plant using conventional technology. The majority of the chemical looping plant, including syngas production using a GE type entrained flow gasifier, slurry-phase F-T synthesis and upgrading, would be similar to a conventional plant.

Economic analysis of the chemical looping components has been performed at OSU and was shared with Noblis through personal communications. Personnel at OSU have estimated the delivered cost of the chemical looping system, which comprises a fuel reactor, a hydrogen production reactor, and a particle conveying system, to be about $40 million in 2000 dollars at current scale. The following are the methodologies adopted by OSU for the cost estimation.

The OSU cost estimation approach used a power factor method with an exponent of 0.6 (Peters et al., 2003).

The cost of the reference unit was obtained from the economic data on a high-pressure directly heated fluidized bed biomass gasifier, which operates under similar pressure but higher temperature (830 ºC compared to 750 ºC) than the chemical-looping units (Kraig and Mann, 1996). The installed equipment cost of the gasifier (along with the lock hopper system), which has a volume of 180 m^3, is reported to be $20.97 million in 1990 dollars. Since the installation cost is set to be 15% of the delivered equipment cost in the report, the delivered equipment cost is calculated to be $18.23 million in 1990 dollars, or $21.53 million in 2000 dollars according to Marshall and Swift Index. Moreover, unlike the biomass gasifier, the fuel reactor and the hydrogen reactor of the chemical looping system has no internals and lock hopper systems. Therefore, a factor of 0.75 is applied to the reference unit cost. The resulting reference unit cost is $16.15 million for a volume of 180 m^3.

The sizing of the chemical looping units is estimated based on the capacity of the units and the required gas and solid residence times obtained from the demonstration results obtained from

bench scale reactor operation at OSU. The size of the fuel reactor is estimated to be 216 m^3 and the hydrogen reactor 180 m^3. Based on the power factor method, the estimated delivered cost for the fuel reactor and the hydrogen reactor is $17.99 million and $16.12 million respectively, both in 2000 dollars. The particle conveying system, where particles are transported at a residence time of an order of magnitude less than those in the fuel reactor or hydrogen production reactor, is estimated to be $4.8 million. Thus, the total delivered equipment cost for the whole chemical looping system is at $38.91 million in 2000 dollars.

The chemical looping configuration eliminates the water gas shift reactor, because hydrogen is cycled back to the F-T reactors, and permits the two stage Selexol in the conventional system to be replaced with a cheaper single stage MDEA system, because bulk carbon dioxide removal is not needed. The savings associated with these changes would be about equal to about $40 million. This is almost identical to the cost of the chemical looping system as estimated by OSU. This and other required changes between the chemical looping configuration and the conventional CTL configuration are summarized below in Table 2.

Table 2: Capital Cost Impact of Proposed Looping System

Item	Incremental Cost (MM$)
Add Looping System (OSU Estimate)	40
Eliminate Shift	-15
Eliminate Two Stage Selexol	-65
Add single stage MDEA and polisher	40
Increase F-T Reactor Size	5
Eliminate F-T Recycle	-8
Reduce CO_2 Compression Cost	-8
Improved Heat Exchanger Metallurgy	10
Net Change	**-1**

Within the error of this cost analysis, we conclude that the capital cost of the OSU looping configuration is approximately equal to a conventional CTL system with the same coal feed.

Comparison

Technical performance results were compared with a conventional CTL configuration with cobalt catalyst shown in Figure 3 and Table 3 (located at the end of the report).

These results indicate that the looping case increased liquid production from the same coal feed by about 10% compared to a conventional F-T CTL system: (12,008 BPD vs. 11,002 BPD). The overall process efficiency is increased from 46 percent (HHV basis) in the conventional case to 49 percent in the chemical looping case. Since capital and operating costs are similar, the increase in output would result in an approximate 10% reduction in the selling price required to

yield a desired return on investment. The effect on profitability, in terms of return on investment at a fixed selling price, would be highly dependent on the financial assumptions.

Conclusions & Recommendations

This analysis confirmed that chemical looping systems have the potential to improve CTL economics. The system proposed by OSU is technically sound and it is recommended that further development and scale up of the experimental effort be performed.

OSU personnel reported that the chemical reactions involved in the looping system go to completion in their demonstrations. To date Noblis has not investigated how the system would perform if some equilibrium level of unwanted reactants remained at each stage. This is an important area that should be investigated further. If there is a significant amount of CO and H_2 in the fuel reactor effluent then this would have to be sequestered with the CO_2 with a resulting loss of efficiency.

The following areas would need further study in order to better understand this novel process: 1) more detailed system economics; 2) more expansive and detailed simulations to discover the impact of incomplete reactions at each stage and 3) other system integration schemes as well as a case when iron catalyst is used in place of cobalt

Overall, the chemical looping systems such as that proposed by OSU have the potential to significantly (~10%) increase the yield of the conventional cobalt based F-T process and allow more efficient heat recovery and much lower (~19%) carbon emissions.

Concerns include the impact of high temperature and large volumes of solids transfer on materials and on the engineering design necessary to ensure smooth and reliable performance.

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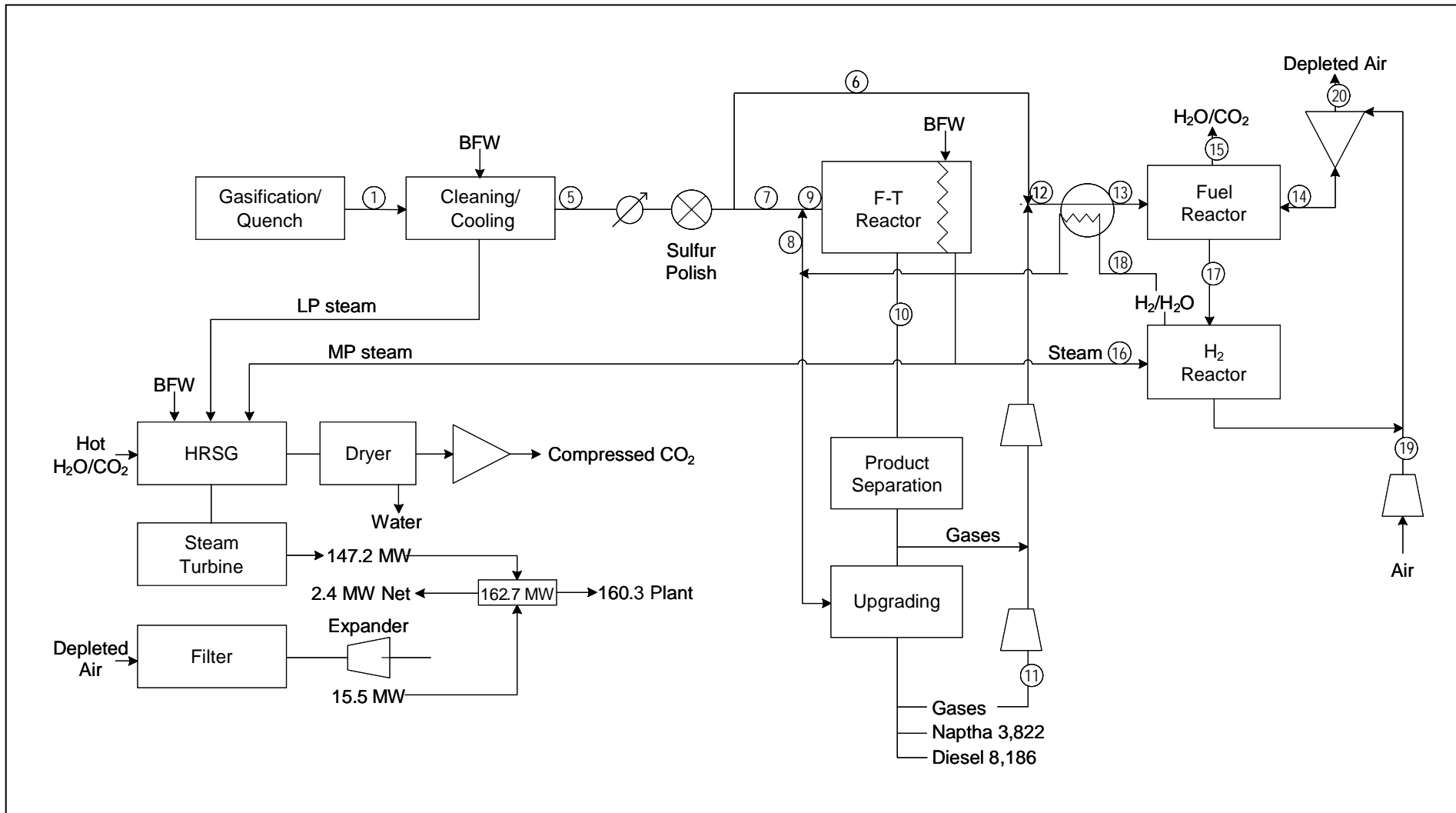
Figure 2: CTL Plant with Chemical Looping

Table 1: Stream Tables for CTL Plant with Chemical Looping

Stream No.	1	2	3	4	5	6	7	8	9
	Quenched Output	COS Hydrolysis	MDEA Feed	Sour Gas	Clean Gas	Fuel Reactor Split	Fresh F-T Feed	Recycle H2	F-T Feed
Species flows									
CH4 (mol/hr)	1	1	1	0	1	0	1		1
H2O (mol/hr)	64,443	64,443	88	0	88	25	64	1,044	1,108
H2 (mol/hr)	12,024	12,024	12,024	6	12,018	3,336	8,682	19,814	28,496
CO (mol/hr)	19,122	19,122	19,122	19	19,102	5,303	13,800		13,800
CO2 (mol/hr)	5,011	5,011	5,011	0	5,011	1,391	3,620		3,620
N2 (mol/hr)	942	942	942	0	942	261	680		680
H2S (mol/hr)	414	414	414	414					
NH3 (mol/hr)									
Total (mol/hr)	101,957	101,957	37,602	439	37,162	10,316	26,847	20,858	47,705
Temperature (F)	418	418	400	104	151	151	500	300	416
Pressure (atm)	32.3			31			25	25	25

Stream No.	10
	F-T Output
Species flows	
C1-C2 (mol/hr)	263
H2O (mol/hr)	11,390
H2 (mol/hr)	7,074
CO (mol/hr)	3,503
CO2 (mol/hr)	3,628
N2 (mol/hr)	680
C3-C4 (mol/hr)	174
C5-C6 (mol/hr)	40
C7-C11 (mol/hr)	82
C12-C18 (mol/hr)	86
C19+ (mol/hr)	222
Oxgnts (mol/hr)	0
Total (mol/hr)	27,142
Temperature (F)	455
Pressure (atm)	23.75

Table 1 Continued: Stream Tables for CTL Plant with Chemical Looping

| Stream No.	11	12	13	14	15	16	17	18	19	20
	Upgrader Offgas	Fuel React Inp Compr	Heated	Recycle Solids	Fuel Reactor Output	Steam Input	Fe/Steam Input	Fe/Steam Output	Compressed Air	Depleted Air
Species flows										
CH4 (mol/hr)	3.1	175	175							
C2H4 (mol/hr)		0	0							
C2H6 (mol/hr)	1.7	93	93							
C3H6 (mol/hr)		0	0							
C3H8 (mol/hr)	33.4	122	122							
C4H8 (mol/hr)		0	0							
iC4H10 (mol/hr)		1	1							
nC4H10 (mol/hr)	60.8	145	145							
H2O (mol/hr)		229	229		12,486	21,922		1,044		
H2 (mol/hr)		10,410	10,410					20,878		
CO (mol/hr)		8,805	8,805							
CO2 (mol/hr)		5,019	5,019		15,010					
N2 (mol/hr)		680	680		680				5,522	5,522
O2 (mol/hr)									1,456	151
Fe2O3 (mol/hr)				7,829						
SiC (mol/hr)				9,348	9,348		9,348	9,348		
Fe (mol/hr)					15,658		15,658			
Fe3O4 (mol/hr)								5,219		
Total (mol/hr)	99	25,679	25,679	17,177	53,182	21,922	25,006	36,489	6,978	5,673
Temperature (F)	60	148	991	1,921	1,382	450	1,382	1,491	1114	1,921
Pressure (atm)		30	30	30	28.5	28.8	27.08	26	33	30
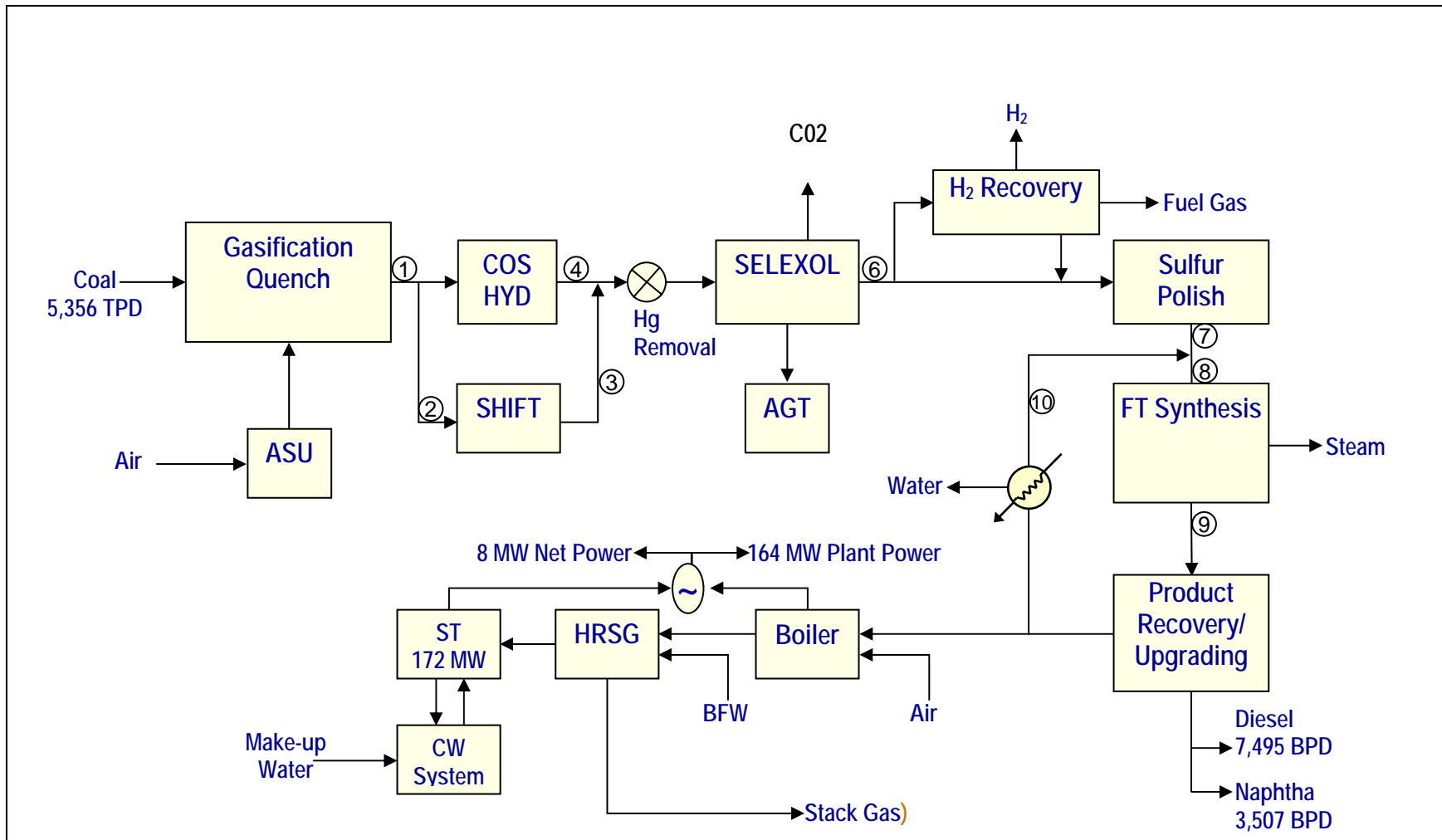
Figure 3: CTL Plant 10,000 BPD, Bituminous Coal, with Simple Recycle (Cobalt Catalyst)

Table 3: Stream Tables for CTL Plant with Simple Recycle

Stream No.	1	2	3	4	5		6	H2	7
	Quenched Output	Shift Input	Shift Output	COS Hydrolysis	Selexol Feed	Sour Gas	Clean Gas	Recovery	Fresh F-T Feed
Species flows									
CH_4 (mol/hr)	1	0	0	0	1	0	1		1
H_2O (mol/hr)	63,218	37,931	28,721	25,287	123	0	123		123
H_2 (mol/hr)	11,948	7,169	16,379	4,779	21,158	11	21,147	826	20,321
CO (mol/hr)	18,941	11,365	2,155	7,576	9,732	10	9,722		9,722
CO_2 (mol/hr)	4,908	2,945	12,154	1,963	14,117	13,835	282		282
N_2 (mol/hr)	334	200	200	133	334	0	334		334
H_2S (mol/hr)	410	246	246	164	410	410			
NH_3 (mol/hr)									
Total (mol/hr)	99,760	59,856	59,855	39,902	45,875	14,266	31,609	826	30,783
Temperature (F)	411	411	400	411	115	115	115	400	411
Pressure (atm)	30	30		29.1				27.6	30

Stream No.	8	9	10	11	12	13
		F-T	Upgrader	85%	Boiler	Water &
	F-T Input	Output	Feed	Recycle	Fuel	Oxygenates
Species flows						
C1-C2 (mol/hr)	1,417	1,667		1,417	250	
H_2O (mol/hr)	123	9,936				9,936
H_2 (mol/hr)	24,440	4,846	661	4,119	727	
CO (mol/hr)	11,861	2,517		2,139	378	
CO_2 (mol/hr)	483	236		201	35	
N_2 (mol/hr)	2,224	2,224		1,890	334	
C3-C4 (mol/hr)	952	1,120		952	168	
C5-C6 (mol/hr)		38	38			
C7-C11 (mol/hr)		77	77			
C12-C18 (mol/hr)		80	80			
C19+ (mol/hr)		204	204			
Oxgnts (mol/hr)		25				25
Total (mol/hr)	41,500	22,970	1,060	10,718	1,892	9,961
Temperature (F)	500	455	100	100		
Pressure (atm)	28.3	26.17	26.17	26.17		