

Section 4

Summary and Recommendations

4.1 SUMMARY

The two most important design parameters for selection of the separation process are required product purity (< 2 to 5 ppm) and catalyst size distribution. The size distribution is shown in Figure 2-2 and is much smaller than in the earlier Mott laboratory testing. The fine particle size makes this a difficult separation process.

In the baseline design report, the Kerr-McGee ROSE process was evaluated. This is a two-step process with a hydrocyclone to separate larger particles before the Kerr-McGee ROSE separation. This design can lead to a buildup of higher-molecular-weight waxes in the reactor.

SASOL has a patent application for wax/catalyst separation which is based upon filtration within the reactor. There are some major differences from the baseline design. These include a much larger catalyst size and wax that is sold as a by-product and not used further to produce a liquid fuel (i.e., the wax may have less stringent product purity limits).

There are many methods to separate the wax and the catalyst. These methods are based on:

- Particle size
- Density differences
- Alteration of properties
- Magnetic differences
- Electrical charge
- Solubility
- Wettability
- Vapor pressure

The product purity should be 2 to 5 ppm of catalyst in the wax. This means that all particles smaller than 0.1 micron be removed if the wax is used in a hydrocracker.

Filtration with microfiltration membranes offers the possibility of removing the particles to the level required. This may be accomplished in one process step.

4.2 RECOMMENDATIONS

The particle size spectrum of Figure 2-2 indicates a significantly more difficult wax/catalyst separation than the particle size spectrum assumed in the baseline design. Not enough is known about the particle size spectra of the catalyst, its constituents, and the desired particle size to maintain in the reactor. This is critical for selecting a separation process. Accordingly, more

measurements of the constituents and size distribution are needed, and a specification must be developed for use in the pilot testing.

Membrane systems are recommended for further evaluation of separation of the wax from the catalyst. This will include a review of the more recent Mott testing. However, there should be a review of the test procedures as recommended in Section 3.2.3 to determine if the testing simulates the actual plant operation.

Asymmetric inorganic membranes should also be considered. This will definitely require additional evaluation of potential membrane systems, with laboratory testing on various membrane systems to determine if the testing criteria recommended in Section 3.2.3 can be met.

Other approaches should be followed if the membrane system does not appear to be able to achieve the testing objectives.

Separation of the catalyst from the wax should become easier as the particle size of the catalyst increases. Catalyst manufacturers and researchers should be encouraged to develop more robust catalysts that resist attrition during handling and use.

Section 5

References

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Appendix 1

The Kerr-McGee ROSE Critical Solvent Extraction Process for F-T Wax/Catalyst Separation

Kerr-McGee's ROSE critical solvent extraction is a candidate process for removal of catalyst from F-T wax/catalyst slurries. Since the technology is proprietary, process details and conditions are not included in the discussion below.

Pilot feasibility tests for the F-T wax/catalyst separation application were carried out by Kerr-McGee beginning in June 1993 and reported to PETC on September 13, 1993. The feedstock tested was provided in drums of congealed slurry containing approximately 4 wt% catalyst. The catalyst particle size spectrum was not mentioned by Kerr-McGee.

The ROSE process uses a solvent liquid near its critical temperature and pressure to preferentially extract a solids-free light wax fraction from the wax/catalyst slurry.

The extraction is a liquid/liquid separation step. The separation mechanism is presumably the difference in density between the critical solvent/light wax phase and the heavy wax/catalyst phase. After leaving the extractor, the light and heavy phases are separately stripped of solvent and sent to their respective destinations. Recovered solvent is recondensed and recycled to the extractor.

In other applications, Kerr-McGee has counted on enhanced settling of solids as a result of agglomeration of the fines by action of certain components of the heavy fraction. The extent to which the F-T wax lent itself to this action in the tests is not known, but Kerr-McGee implied such a mechanism in its interpretation of the test results.

By experimenting with several solvents and a variety of operating conditions, Kerr-McGee discovered a threshold yield of light wax. At yields above the threshold, the light wax contained increasing amounts of carryover solids, up to a yield in which approximately half the entering solids were carried over. At yields below the threshold, the light wax contained less than 0.1 wt% carryover solids. Instruments used during the tests could not resolve solids levels below 0.1 wt%. Later, however, the solids in the light wax yields below threshold were shown to be at the level of a few parts per million, meeting the purity requirements for feedstocks to hydrocracking.

Because acceptable light wax purity was achieved, Kerr-McGee asserts that the tests were successful and that the ROSE process is feasible for wax/catalyst separation for slurry F-T technology using finely divided iron oxide catalyst.

The current configuration of the ROSE process is disappointing, for two reasons. The first is that the threshold light wax yield is only slightly more than half, and consequently the heavy wax/catalyst residue slurry constitutes a major process stream which must be disposed of. Two methods of disposing of the residue slurry have been considered:

- *Residue Slurry Recycled to the Gasifier.* This solution would significantly increase the size of both the gasification and F-T plant sections, and lead to an unacceptably high rate of catalyst makeup (5 percent makeup, over 15 times as high as the baseline design rate of 0.3 percent makeup).
- *Residue Slurry Recycled to the F-T Reactor.* This solution would lead to a desirable reuse of catalyst. It has one major disadvantage: the heavy wax recycling with the catalyst is likely to undergo further synthesis and growth in chain length in the reactor, resulting in an undesirable increase in molecular weight of reactor wax.

The second reason the current configuration is disappointing is that it appears potentially vulnerable to a change of the catalyst characteristics in the feed. A decrease in the particle size will change the removal efficiency of the hydrocyclone and the settling characteristics of the catalyst. The change in the catalyst characteristics will have the following effects:

- The 4 wt% catalyst in the feed to the ROSE unit assumes that upstream hydrocyclones can remove more than 82 percent of the 22.5 wt% catalyst in the slurry leaving the F-T reactor. With the catalyst size distribution assumed in this study, Stokes' law analysis suggests that hydrocyclones will fall far short of 82 percent removal and might even approach the level of 48 percent removal discussed by Bechtel with Kerr-McGee in their correspondence in March 1994.
- Because the ROSE process counts on agglomeration to settle out the finest particles, failure of the agglomeration mechanism or an overload of finer particles could lead to fine particle breakthrough unless a lower yield is accepted.

Throughout its program of study of the application of the ROSE process to the F-T slurry application, Kerr-McGee has seen its objective as extracting a solids-free light wax from the slurry. If, instead, the objective is seen as removing the catalyst solids from both the light wax and the heavy wax, a major improvement in the process might be possible. In particular, the following two changes are proposed:

- *Use Centrifuges to Remove the Solids.* In this case, the Kerr-McGee solvent could be seen as a viscosity-reducer. The literature shows that critical solvents have viscosities on the order of 0.03 cp, a hundredth of the 3 cp level of the F-T wax. Stokes' law analysis suggests that in a centrifuge a 0.1-micron particle in a 0.03 cp liquid will settle as fast as a 1-micron particle in a 3 cp liquid.
- *Use Kerosene (or Some Other Light Hydrocarbon Fraction) to Convey the Catalyst Back to the F-T Reactors.* Kerosene is available in the plant as a coproduct of hydrocracking. Hydrocarbons in kerosene are sufficiently low in molecular weight that they can undergo chain growth in the F-T reactor without excessive buildup of high-molecular-weight wax. Solids at 25 wt% constitute only 5 vol% of a F-T slurry.

If it should prove possible to pump a kerosene/catalyst mixture containing 30 vol% of solids, this mixture would contain 73 wt% solids and only 27 wt% liquid. Thus, such a mixture would be recycling to the reactor only 0.36 pound of liquid for every pound of recycle solids. Slurries containing 50 vol% solids are pumpable by diaphragm pumps in the coal and minerals industries. Finally, the low melting point of kerosene eliminates any concerns about liquid freeze-up in the recycle equipment and lines.

In summary, the modified process proposed above uses ROSE solvent technology to reduce liquid viscosity, assures deep solids removal by use of centrifuges, frees all the F-T wax for hydrocracking, and uses a light hydrocarbon to recycle a slurry at high solids volume fraction.

Appendix 2

SASOL's 25 January 1994 Application for a U.S. Patent for a Fischer-Tropsch Synthesis Reactor, Including Internal Filter

On January 25, 1994, SASOL Chemical Industries (Proprietary) Limited of South Africa filed an application for a U.S. patent entitled "Process for Producing Liquid and, Optionally, Gaseous Products from Gaseous Reactants," with "Flodderbed Reaktor" as a short title. The invention is credited to Berend Jager and five others.

SYNOPSIS OF APPLICATION

The application seeks patent protection for an invention that consists of a process and associated installation configurations that have been piloted.

The process is for producing liquid and gaseous products from a reacting feed gas introduced in the bottom of a slurry bed of solids suspended in a liquid (typically product liquid), allowing the gas to react as it flows upward, and removing liquid product from the reactor using a particular internal filtration process. The solids can be a catalyst for the reaction, the gas a synthesis gas of carbon monoxide and hydrogen used in Fischer-Tropsch synthesis. The slurry bed can be in a reaction vessel. The vessel cylindrical section might be typically 24 meters high with the slurry bed occupying 14 to 18 meters. The reactor can maintain the slurry at temperatures in the range 160°C to 280°C and pressures 18 to 50 bar as needed for the desired Fischer-Tropsch synthesis products. The spent syngas and gaseous products are removed through the top of the vessel.

The filtration process takes place in a filtration zone in the slurry bed, usually toward the top, where there are no mechanical mixing devices, and where the particles are suspended by fast rising gas with a superficial velocity between 5 and 70 cm/sec.

Filtration is a four-step process. In the first step, liquid product is removed by passing it through a filter medium located in the filtration zone against a medium/cake pressure drop up to 8 bar and an average filtration rate exceeding 500 L/hr/m² of medium surface. In the second step, the flow is stopped. In the third step, the filter is backflushed with two or more rapid-onset pulses of liquid, or liquid and gas, so as to dislodge and remove filter cake, where the pulse pressure drops can be up to 10 bar, and the flow rate up to 10,000 L/hr/m² of medium surface. In the fourth step, no flow passes through the filter in either direction for a wait period of 15 to 60 minutes while the cake falls away and is dispersed.

Filtration is accomplished by several filter media arranged in banks. Each medium is an elongated cylindrical element enclosing a filtrate collection zone, with a filtrate outlet at one end, normally the bottom. The medium is mounted in the vessel, usually vertically. The filtrate flows out of the vessel through primary conduits or lines. These lead through secondary and tertiary lines into a rundown tank. Backflush is accomplished through reverse flow through the conduit system. Valves and restrictive orifices provide control of the filtration and backflush fluid flow. The rundown tank can double as the reservoir of backflush fluid, or a separate backflush vessel can be

provided with special backflush conduits. During filtration, driving pressure difference is effected by reducing the pressure in the rundown tank. For backflush, the driving pressure difference is effected by pressurizing the backflush tank with pressurized gas.

SASOL's application discusses in detail a particular design of filter medium consisting of a 12 cm dia. spiral of stainless steel wire embedded in or attached to a cylindrical frame of circumferentially spaced longitudinal support slats extending between top and bottom support plates. The wire is 1.2-millimeter-wide wedge wire with 0.03-millimeter (30-micron) gaps or slots between adjacent loops. The base of the wedge is on the outside as a surface against which the cake can form. Because of the trapezoidal cross section of the wires, the gap between wires diverges as the filtrate flows inward, eliminating the possibility of permanent clogging by small particles passing through with the filtrate.

SASOL claims that the system has operated continuously for several months with repeated flush cycles without permanent clogging or mechanical failure of filters, proving the feasibility of internal filtration.

THE SOLIDS IN SASOL SLURRIES FILTERED

In its discussion, SASOL indicates that its invention applies to any suitable Fischer-Tropsch catalyst, such as cobalt-based catalysts, and also iron-based catalysts produced by precipitation and spray drying.

SASOL asserts that its system can accommodate a slurry containing up to 40 wt% solids (or 9 vol% solids, according to a Bechtel calculation).

SASOL indicates a preferred size spectrum with 300 microns as a top size and less than 5 wt% of the solids smaller than 22 microns, although according to SASOL the system can tolerate as much as 40 wt% smaller than 22 microns.

SASOL found that higher filtration rates are achieved when the content of particles smaller than 5 microns is minimized, but says that it still gets satisfactory filtration rates when towards the end of a run as much as 25 wt% of the particles are smaller than 5 microns.

The liquid product that passes through the filter typically contains between 2 and 200 ppm of solids.

The above assertions of the application suggest that SASOL's filter medium, with gap sizes ranging from 20 to 40 microns, can successfully filter out particles much smaller than the gap size, presumably by forming a cake with tortuous internal channels between the larger particles that are incapable of passing through the gap.

SASOL'S CLAIM OF SUPERIORITY OVER EXTERNAL SOLIDS REMOVAL SYSTEMS

At the end of the section describing the invention, just before the claims, SASOL points out that, compared to schemes for separation of the particles external to the reactor, its system of separation internal to the reactor has two major advantages:

- The internal system is intrinsically less expensive, since all external systems add extra vessels, piping, mechanical equipment, and process steps, which raise both the capital and the operating and maintenance costs.
- The internal system is intrinsically more gentle with the catalyst, since the catalyst always remains inside the reactor suspended in the product liquid, whereas external systems subject the catalyst to transport through pumps and pipes, rough handling in miniclones, centrifuges, and magnetic separators, and jostling and collisions in systems to re-slurry the concentrated solids. Because iron-based catalyst is unusually susceptible to size degradation, gentle handling should be at a premium.

CONCLUSIONS

SASOL appears to have developed a system for filtration internal to the reactor which can perform satisfactorily with iron-based catalysts in relatively finely divided form.

SASOL's wedge-wire filter medium with a divergent gap seems to have overcome the usual objection that filters are intrinsically vulnerable to permanent clogging.

SASOL's internal filtration technology is intrinsically more gentle in its handling of fragile iron-based catalyst than external separation systems.

SASOL's internal filtration technology is in principle less expensive than external separation systems.

The degree of cleanup achieved by the SASOL system is impressive. They mention removals to levels of 200 ppm to 2 ppm in the product liquid. Supposing the slurry concentration is at a typical level of 20 wt% solids, an exit solids level of 200 ppm represents removal of all but one part in a thousand; a level of 2 ppm represents removal of all but one part in a hundred thousand. It can be conjectured that their 2 ppm level represents performance at the beginning of a run when the catalyst particles are fresh and large, and that the 200 ppm level corresponds to the mentioned end-of-run distribution with 25 wt% of the solids less than 5 microns.

It is likely that residual solids levels would be higher than 200 ppm if the SASOL filtration technology were used in the DOE program. DOE program catalyst solids are more finely divided than the SASOL solids quoted in the patent application. Fresh catalyst in the DOE program can be as fine as 50 wt% less than 4 microns, and catalyst after extended operation will be finer still.

Residual solids levels of 200+ ppm are not acceptable for product liquid feed to hydrocracking. Hydrocracking requires that inert solids in the feed be no more than 200 ppm to avoid fouling the catalyst. Heavy metal solids, including iron, must be limited to 2 to 5 ppm to avoid poisoning the catalyst. Accordingly, if one purpose of the synthesis process is to produce a wax feedstock for hydrocracking, the SASOL technology must be followed by a residual removal system, to bring the feed solids down to the required level. However, the fact that the SASOL filtration system does a such a good job of primary removal means that residual removal will be easier and presumably less expensive to carry out. In particular, magnetic residual separation would be easier and less expensive.

Appendix 3

Bechtel Interoffice Memorandum

To: G. N. Choi Date: April 18, 1995
Subject: Bechtel Job No. 22925-100 From: A. I. McCone
Telecon with Dick Tischer on Iron
F-T Catalysts and Separation from Wax Loc./Ext 50-15-C30/8-7045

Copies to: J. T. Newman, S. S. Tam, C. M. Lowe, S. Kramer

In a phone conversation today, Dick Tischer of DOE made the following remarks about the wax/catalyst separation issue:

1. If the industry cannot produce a sufficiently attrition-resistant iron catalyst, it will be necessary to switch to a cobalt catalyst which can be placed on a strong support.
2. Iron-based catalysts undergo large volume changes in the F-T reactor, with the chemical form ranging from hexagonal Fe_2O_3 (hematite), to metallic iron, to iron carbide, and back again to cubic Fe_3O_4 (magnetite). He discussed studies of the various chemical forms. In batch reactors where the conversion is 40 to 50 percent, iron carbide is the stable form. But when the gas contains appreciable amounts of CO_2 and H_2O , the bulk phase goes completely to magnetite. Dick quoted Bert Davis as seeing iron carbide as a surface form.
3. The morphological and volume changes (especially those between the oxide and the carbide forms) will inevitably lead to steady attrition of the iron-based catalysts:
 - a. The active iron-based catalyst is iron oxide, compounded with 5 to 7 percent copper and 1 percent K_2O . The catalyst in Air Products Run One contained some kaolinite as binder, and it exhibited better attrition resistance than the catalyst in Run Two. The Run Two catalyst contained 5 to 10 percent silica added to control sintering in catalyst pretreatment, but the silica did not contribute to greater attrition resistance.
 - b. The Run Two catalyst was so weak that attrition occurred as the result of handling associated with Sandia tests to determine the particle size spectrum.
4. There are clues that under some process conditions, the slurries have exhibited high viscosity colloidal behavior, indicating a significant fraction of solids with diameters 1 to 0.1 micrometer and less:
 - a. In one run at LaPorte, after a certain period of normal operation, the slurry performance dropped significantly and large bubbles were noted.
 - b. Performance seems okay with 5 to 10 percent solids in the slurry, but conversion dropped off at 20 percent solids.
5. Dick considers that heavy wax recycle is the fatal flaw of the Kerr-McGee process as tested.

6. Dick mentioned SASOL and its patent as indication that iron-based catalysts can be satisfactorily separated from wax. His impression is that SASOL may be ready to license its technology.

Appendix 4

Bechtel Interoffice Memorandum

To: G. N. Choi Date: April 19, 1995
Subject: Bechtel Job No. 22925-100 From: A. I. McCone
Telecon with Bert Davis on Iron
F-T Catalysts and Separation from Wax Loc./Ext 50-I 5-C30/8-7045

Copies to: J. T. Newman, S. S. Tam, C. M. Lowe, S. Kramer

In a phone conversation today, Bert Davis of the University of Kentucky made the following remarks about the wax/catalyst separation issue:

1. Iron-based catalysts are preferred to cobalt-based catalysts to avoid the need for a separate shift process step required with cobalt-based. Also, the iron-based catalysts achieve greater activity. However, the cobalt-based catalyst, which is supported with 70 percent alumina, is more attrition resistant than iron-based, and thus the pellets remain large and pose less difficulties in separation. If iron-based separation costs are too high, economics could favor cobalt-based.
2. Iron-based catalysts are usually made by precipitation from ferric nitrate titrated with ammonia. The precipitated oxide forms solid spheres one micrometers in diameter. The precipitate is processed into porous pellets which might be 60 micrometers in diameter. But the pellets are fragile and the material tends to come apart into the one micrometer spheres, which in Bert's opinion do not degrade further into smaller spheres.
3. SASOL's January 25, 1994 U.S. patent application seems to imply that the reactor's internal filter is only to screen out and retain large pellets. Fines leave with product liquid, and are presumably recovered from the wax externally by solvent extraction (probably using xylene) and mechanical separation (perhaps by magnetic gradient separation). The patent application states that the fines might range up to 40 weight percent of the catalyst material. This suggests that SASOL must supply makeup catalyst at a significant rate.
4. During a recent visit to SASOL's plant along with others sponsored by DOE, he noted that off-limits to visitors was a large building that appeared suitable for catalyst manufacture. He notes that SASOL has a market for its spent catalyst at the nearby steel mill. SASOL's wax is sold as wax, which in the local market is four times higher in price than transportation fuel. The wax he saw which he was there was very white, fines-free wax.
5. At the recent "C1" conference at Anaheim, SASOL's Mr. Inga spoke of achieving 98 percent wax in their product slate. At an earlier conference, SASOL's Mr. Gertzmann (sp?) commented that they could never think of commercial operation with more than 35 weight percent solids. Bert thinks that means they can achieve loadings up to 35 percent.

6. We did not discuss whether recovered fines could be reprocessed into larger pellets.
7. His own best catalyst reaches 90 to 100 percent CO conversion, and activity loss is only 1 percent per week of CO conversion capability. He says that when CO conversion exceeds 67 percent, this best catalyst over-shifts, converting too much CO to hydrogen and CO₂. He recommends a draw-off for water removal and then recycle. He'd love to see the economics with and without this.
8. Activation of the catalyst at the start of operation requires converting surface iron oxide to iron carbide. He believes that also formed is a deposit of elemental carbon, which structurally keeps pores open. He also said that activation is best carried out at 1 atm pressure, meaning that it should be done external to the slurry reactor.

Appendix 5

System for Separation of Finely Divided Iron Oxide Catalyst from Liquid Wax in Fischer-Tropsch Synthesis of Transportation Fuels

In support of Air Products Chemicals, Inc., on a DOE-funded study, Bechtel is examining methods for removal of finely divided iron oxide/iron carbide catalyst particles from liquid wax produced in Fischer-Tropsch (F-T) synthesis. The catalyst separation task is part of an ongoing development of F-T technology so that transportation fuels can be made from coal and natural gas as well as from petroleum. Any selected technology for separation would be tested in the DOE pilot plant in LaPorte, Texas.

Suppliers are invited to provide descriptions, scope, utilities, and budgetary quotes on separation systems for a commercial plant. Suppliers are also asked to recommend and price a test program in their own facilities with simulated slurry.

In a 50,000 bpd commercial plant, the separation system must process 400,000 lb/hr (30,000 bpd, 900 gpm) of wax/catalyst slurry containing as much as 25 wt% solids.

The catalyst is present in finely divided form with particles ranging in size from 180 microns down to less than 0.1 micron, and typically with 50 wt% less than 4 microns, 20 wt% less than 1 micron, and 4 percent less than 0.3 micron. The size spectrum is shown on page A5-3.

As much as possible of the separated catalyst is to be recycled to the F-T synthesis reactor. Suppliers should indicate the quantity of liquid wax recycled with the catalyst.

Although it is preferable to carry out the separation in a single stage, the solids removal may in fact require two stages: a first bulk removal stage and a second residual removal state. A proposer should specify whether his equipment is intended for bulk removal or residual removal or both.

It is hoped that bulk removal can remove 96 percent or more of the solids. Since it may be necessary to purge the solids from residual removal, it is desirable to have the greatest possible removal in the bulk stage.

Residual removal must clean the product liquid wax so that it does not contain more than 1 to 5 ppm iron oxide, the purity required for feed liquids to hydrocracking systems.

The slurry emerges from the reactors at 488°F and 289 psig, under which conditions the wax is a liquid with a viscosity of 2.5 cp. At 444°F, the wax has a viscosity of 3.0 cp and a density of 0.696 g/cc. At 350°F and 70 psig, the wax viscosity is 4.75 cp. The catalyst particles have a calculated density as high as 4.5 g/cm³ (this includes wax in the pores).

Element weight percents in typical starting catalysts are 55 Fe, 4 Cu, 5.3 Si, and 3.7 K; the difference is oxygen. During the reaction step, iron carbide is formed. Typical percentages of iron forms are as follows: 25 ferric oxide, 10 magnetite, 25 chi-carbide, and 40 E-carbide. Magnetite content could be as high as 18 and as low as 6 percent.

For the separation, the pressure may be reduced to a convenient level. However, in pressure reduction there will be some flashing of light hydrocarbons and an attendant temperature drop. Suppliers should indicate the pressure they propose.

An alternative embodiment involves separation after addition of a thinning solvent. In this option, the flow volume would be doubled and the viscosity would be 22 percent of commercial plant viscosities cited above. Suppliers should also quote this option.

SIZE DISTRIBUTION OF IRON OXIDE CATALYST PARTICLES

<u>Wt%<D</u>	<u>D, microns</u>
100.0	180
99.8	136
99.3	103
97.7	77.7
94.5	58.7
89.6	44.4
84.0	33.5
78.5	25.3
73.6	19.2
69.2	14.5
65.2	10.9
61.5	8.27
57.7	6.25
53.5	4.72
48.7	3.57
43.1	2.70
36.2	2.04
29.2	1.54
22.1	1.16
16.0	0.88
11.6	0.66
8.0	0.50
5.4	0.38
3.4	0.28
1.8	0.21
0.8	0.16
0.2	0.12
0.001	0.09

