Section 1

Introduction and Objectives

This report describes the catalyst separation technique study that was conducted by Bechtel in support of Air Products and Chemicals, Inc. (APCI) in the latter's DOE-funded Alternative Fuels and Chemicals from Synthesis Gas project. This is the first report under Task 1.3 of Bechtel's effort in support of APCI.

1.1 INTRODUCTION

Development of a reliable and cost-effective method of wax/catalyst separation is a key step toward a commercially viable slurry reactor process with iron oxide-based catalyst for Fischer-Tropsch (F-T) synthesis of hydrocarbon transportation fuels. Although a variety of suitable catalysts (including, for example, cobalt-based catalysts) are available, iron oxide-based catalysts are preferred for coal-derived, CO-rich syngas because, in addition to catalyzing the F-T reaction, they simultaneously catalyze the reaction shifting CO to H_2, obviating a separate shift process block and associated costs.

Because of the importance of development of this wax/catalyst separation, a study was initiated in February 1991. P. Z. Zhou of Burns and Roe [Ref. 1] reviewed the status of F-T wax/catalyst separation techniques. This led to the selection of a filtration system for the separation. Pilot tests were conducted by Mott Porous Metal Products in 1992 to develop this system. Initial results were good, but problems were encountered in follow-up testing. As a result of the testing, a filter was selected for use on the pilot plant.

In LaPorte, Texas, APCI has been operating a pilot plant for the development of various synthesis gas technologies with DOE and industry support. The APCI F-T program builds on the DOE-sponsored laboratory-scale work by Mobil, reported in the mid-1980s, which used an iron oxide catalyst to produce high-quality F-T liquids in relatively compact reactors. Separation of the catalyst solids from the wax still represents a challenge. In the summer of 1992, testing of the selected filter was begun as part of the pilot plant testing. The filter performed poorly. Separation of the catalyst was primarily by sedimentation. It was recommended that the wax/catalyst separation be developed further.

In work under DOE Contract DE-AC22-91PC90027 (Topical Report, October 1994), Bechtel and Amoco provided a baseline design and the economics for a slurry reactor F-T synthesis plant. This baseline will serve as an economic basis for comparison with alternative processes.

1.2 OBJECTIVES

The objectives of this study are to (1) describe state-of-the-art techniques for the separation of the F-T wax from iron catalysts, (2) discuss the potential for a commercially viable separation method, and (3) present follow-up recommendations.

Section 2

Separation Process Selection

The approach used to develop state-of-the-art techniques for separation has been to (1) survey the literature, (2) contact both separation equipment suppliers and people who have direct wax/catalyst separation experience, (3) review the results to date of this project and others requiring separation, (4) develop a design basis, (5) review previous results, and (6) select a few processes for further evaluation. In Section 3, these selected separation technologies serve as a basis for contacting vendors and developing a comparison between the different processes. Section 4 summarizes the results and recommendations.

2.1 DESIGN BASIS

The design basis was developed using the baseline design for the wax/catalyst separation system evaluated in DOE Project No. DE-AC22-91PC90027, entitled "Baseline Design/Economics for Advanced Fischer-Tropsch Technology."

2.1.1 Baseline Design

Figure 2-1 shows the wax/catalyst separation process diagram used in the baseline design. The wax/catalyst separation is based upon the Kerr-McGee ROSE solvent extraction process. The wax/catalyst mixture from the F-T reactor is first rough-separated by hydrocyclones. This removes the larger catalyst particles and recycles them with some of the wax to the F-T reactor. The remaining mixture enters the Kerr-McGee ROSE solvent extraction process. The clean wax then goes to a cracker to produce additional liquid hydrocarbons. The potential difficulty with the system seems to be that the heavy wax returning to the F-T reactor can react to form undesirable, still heavier wax. The ROSE process is discussed in more detail in Appendix 1.

Costs were developed for this separation process in the baseline design. The capital cost for the solvent extraction part of the process (but not the initial separation) was estimated at $50 million, with the equipment costing $16 million and installation accounting for the remainder. This equipment cost will serve as a guide for evaluating the other separation processes. It is assumed that any other process costing this amount or less would be included in this report.

2.1.2 Background Information

As a prelude to quantitative studies of separation techniques, Bechtel reviewed a number of documents and obtained opinions from experts.

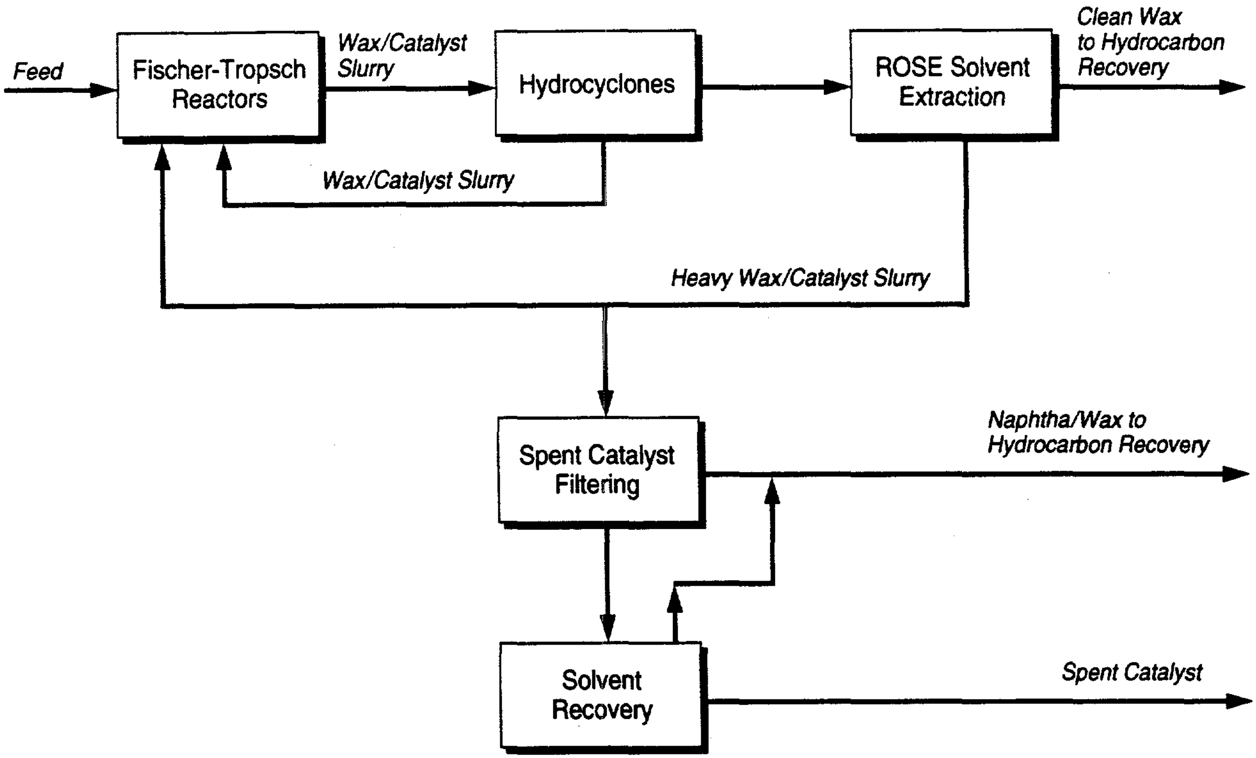

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Figure 2-1 Baseline Design – Wax/Catalyst Separation with Solvent Extraction

Document Review

Documents included the survey of techniques by Zhou [Ref. 1], data on the critical solvent extraction process of Kerr-McGee, and the SASOL application for a U.S. patent filed January 25, 1994 [Ref. 2]. Dick Tisher of DOE and Bert Davis of the University of Kentucky were also consulted. Each of these references is summarized briefly here. Fuller summaries are provided as appendices.

P. Z. Zhou. Status Review of Fischer-Tropsch Slurry Reactor Wax/Catalyst Separation Techniques. The 63-page document gives typical properties of reactor wax and catalyst particles, discusses separation requirements, and reviews separation techniques under nine major headings. The document contains eight tables and 13 figures. Salient facts are the following:

- Density and viscosity are the wax properties that have the greatest effect on the difficulty of separation, and particle size and density are the most important catalyst properties.
- Zhou quotes hydrocracker licensers as desiring a maximum of 2 to 5 ppm of particulates in the hydrocarbon feed.
- Filtration inside the reactor would be the least expensive separation technique, but the risk of plug-up may make it undesirable.
- Vacuum distillation is not feasible because the heaviest wax has too high a boiling point.
- Thermal cracking is infeasible because too much of the wax is converted to coke and gases.
- Sedimentation is often tried, inside and outside the reactor. Too much catalyst seems to be lost with the product wax. Techniques to enhance separation include insertion of surfaces to minimize the distance a particle must fall. No quantitative assessment is provided.
- Filtration, a preferred technique in principle, has been tried at several installations. No quantitative data are presented.
- Centrifuging, including hydrocyclones, is indicated as a good approach to enhance particle fall rate compared to gravity separation. No quantitative data are presented. The author seems to assume that, in contrast with hydrocyclones, centrifuges must run in the batch mode.
- The use of a solvent to assist separation by reducing liquid viscosity is recommended, but no analysis is given.
- High-gradient magnetic separation (HGMS) is described in some detail as a natural scheme for removing iron-based catalysts, but no quantitative analysis is given.

- Chemical methods such as conversion of iron to gaseous iron carbonyl are mentioned, but dismissed as excessively costly and complex.

Kerr-McGee ROSE Process for Critical Solvent Extraction. As mentioned above, the ROSE process was included in a recent Bechtel conceptual baseline design of an F-T plant. However, 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 that must be disposed of. The following 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, about 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 the 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 would 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 viscosity 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 Reactor.* 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 an 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, ensures 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. See Appendix 1 for more details.

SASOL January 25, 1994 Application for a U.S. Patent. The 38-page patent application of B. Jager et al. of SASOL describes a F-T slurry reactor with an internal filtration system. The internal system seems capable of removing only large catalyst particles, suggesting that a second separation system outside the reactor removes fine particles from the product. More details are provided in Appendix 2.

A significant difference from the baseline design is that SASOL produces wax as a product. No information was found on the further separation of the catalyst from the wax after this initial filtration. The resultant wax is exported, and the United States is a large importer of this product.

In its patent application, SASOL points out that its system of separation internal to the reactor has two major advantages over schemes for separating particles external to the reactor:

- The internal system is intrinsically less expensive, since all external systems add extra vessels, piping, mechanical equipment, and process steps, which raises 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. External systems, on the other hand, subject the catalyst to transport through pumps and pipes; rough handling in minicyclones, centrifuges, and magnetic separators; and jostling and

collisions in systems to reslurry the concentrated solids. Because iron-based catalyst is unusually susceptible to size degradation, gentle handling should be at a premium.

The conclusions from the SASOL patent application are:

- 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 than external separation systems in its handling of fragile iron-based catalyst.
- 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, largely because of the particles sizes. SASOL mentions removals to levels of 200 ppm to 2 ppm in the product liquid. If the slurry concentration is at a typical level of 20 wt% solids, an exit solids level of 200 ppm represents removal of all but 1 part in 1,000 and an exit solids level of 2 ppm represents removal of all but 1 part in 100,000. It can be conjectured that SASOL's 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 aforementioned 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 such a good job of primary removal means that residual removal will be easier and presumably less expensive to carry out.

Personnel Contacts

April 18, 1995 Telephone Conference with Dick Tisher. During its life in the F-T reactor, the catalyst solid goes through large changes in crystal volume (oxide to metal to carbide), which could account for the observed rapid attrition of the iron-based catalysts (see Section 2.1.3). Certain characteristics of colloidal systems seem to have been observed in lab and pilot tests which suggest substantial amounts of the catalyst are in the colloidal size range (1 micron and smaller). More details are given in Appendix 3.

April 19, 1995 Telephone Conference with Bert Davis. Iron oxide-based F-T catalyst is made by precipitation from iron nitrate titrated with ammonia. The precipitated oxide forms solid spheres 1 micron in diameter. The precipitate is processed into porous pellets which might be as large as 60 microns in diameter, but these pellets tend to break down and reform as 1-micron spheres. SASOL seems resigned to having to replace catalyst at a significant rate. More details are given in Appendix 4.

2.1.3 Physical Properties

As a first step in developing quantitative indicators of the efficacy of various separation techniques, Bechtel has selected typical values for the most relevant properties of the solid and liquid components. These values and the rationales for their selection are given below.

Catalyst Properties

In wax/catalyst separation, the most important properties of the iron oxide-based catalyst are the particle size spectrum, the particle density, and (if magnetic separation is considered), the magnetic susceptibility.

Particle Size. The baseline design assumed steady-state size spectrum had a weight average diameter of 15 microns with 84 wt% greater than 10 microns. This spectrum lent itself to convenient separation using hydrocyclones followed by Kerr-McGee critical solvent extraction. Catalysts with such a size spectrum or with larger particles are commonly considered. For instance, in a January 25, 1994 application for a U.S. patent for its F-T system with internal filtration, SASOL mentions as an example a spectrum with upper size of 300 microns and 95 percent greater than 22 microns. In the same paragraph beginning at line 23 on page 2 of the draft, SASOL indicates that the types of catalysts envisioned in its system are iron-based catalysts and cobalt-based catalysts [Ref. 1].

Nevertheless, experimental work with iron oxide-based catalysts under DOE sponsorship has dealt almost exclusively with size spectra where the particle diameters are considerably smaller than those in the baseline design. It is not obvious that the wax/catalyst separation methods in the baseline design are applicable to catalyst material of the size spectrum experimentally observed.

Consequently, for some time DOE has been alert to the need for separation methods adequate to the observed size spectrum.

For the particle size spectrum, the spectrum used is the one designated "STATOIL" and shown in Figure 2-2. The spectrum ranged from 100 to 0.08 micron, with a mean diameter of 4 microns and 16 wt% smaller than 1 micron. However, it is well known that one can manufacture the catalyst with a significantly larger mean diameter. For instance, APCI has used fresh catalyst with a mean particle size of 13 microns (and mean wt% at perhaps 20 microns). Thus, it seems reasonable to assume that such a catalyst would have a spent catalyst spectrum like the measured STATOIL spectrum.

If satisfactory wax/catalyst separation cannot be achieved with this iron oxide catalyst, it will be necessary to switch to a cobalt-based catalyst that does not break down easily. Early attempts to remove the iron oxide-based catalyst by filtration have been unsuccessful because of filter blind-off, and, accordingly, the subject of appropriate separation techniques has to be reexamined.

More recent work at Sandia and the University of New Mexico [Ref. 3] indicates even smaller particles could result. Their work was to understand the effects of pretreatment and reaction on the characteristics of iron catalysts. They discovered that "these morphological transformations involve a major breakdown of the single crystal hematite particles into smaller crystallites of carbide." The hematite crystals are about 1 micron long, 0.3 micron wide, and approximately 0.04 to 0.05 micron thick. These break down into carbide crystallites that are 0.02 to 0.03 micron in diameter. They also concluded that "the iron oxide transforms from hematite to magnetite and finally into an iron carbide phase." These transformation affecting both particle size and physical characteristics can obviously influence the final selection of the separation process.

Density. For particle density, a value of 4.53 g/cm^3 is used, inferred from catalyst bulk density as follows: the catalyst has a typical bulk density of approximately 193 lb/ft^3 or 3.1 g/cm^3. Assuming hexagonal close pack in bulk, the density of a single spherical pellet in air is 43 percent higher, or 4.44 g/cm^3. Assuming the solid portion of the sphere has the density 5.12 g/cm^3 proper to ferric oxide (hematite), pore volume constitutes 13.3 percent of the total volume. When the pores are filled with wax of density 0.7 g/cm^3 in the reactor, the total density of the sphere becomes 4.53 g/cm^3.

Magnetic Properties. For particle magnetic susceptibility, a value appropriate to hematite (Fe_2O_3) is used. Hematite is at the upper end of paramagnetic behavior. The choice of hematite is conservative, in that the iron oxide in the catalyst pellets may be partially in the form of magnetite (Fe_3O_4), which has 100 times the susceptibility. The numerical magnitude and dimensions of the assumed susceptibility are presented in the discussion below of magnetic separators.

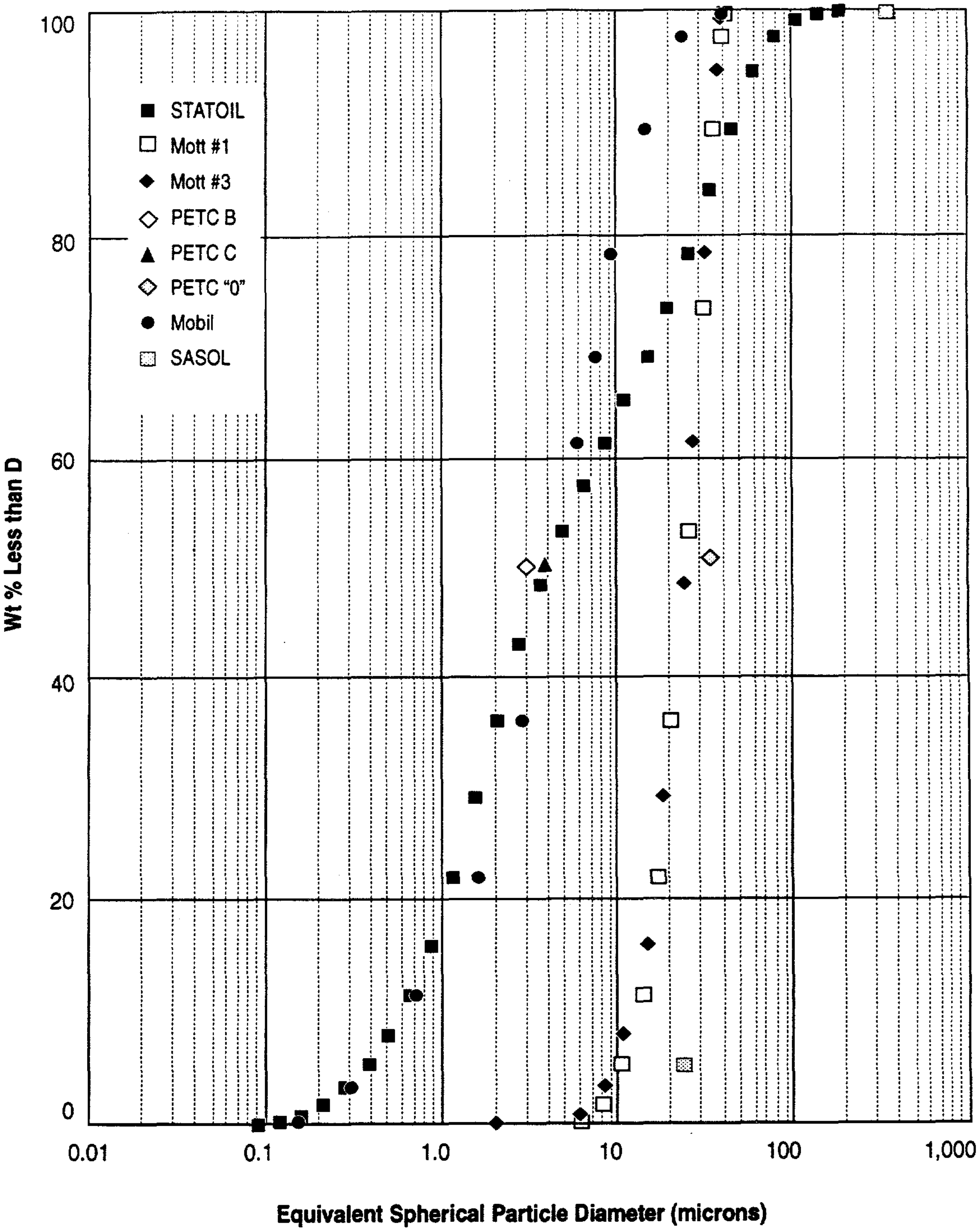

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Figure 2-2 Iron Oxide Catalyst Particle Size Measurements by Various Organizations

Wax Properties

In catalyst/wax separation, the most important properties of the wax are the fluid density, the viscosity, and the very low magnetic susceptibility (wax is diamagnetic).

Density. For wax density, a value of 0.696 g/cm^3 is used. The value is intermediate between the two entries for Mobil wax in Table 4 on page 40 of Zhou, and corresponds to a temperature of 229°C, a reasonably likely separation temperature for a reactor discharging at approximately 250°C.

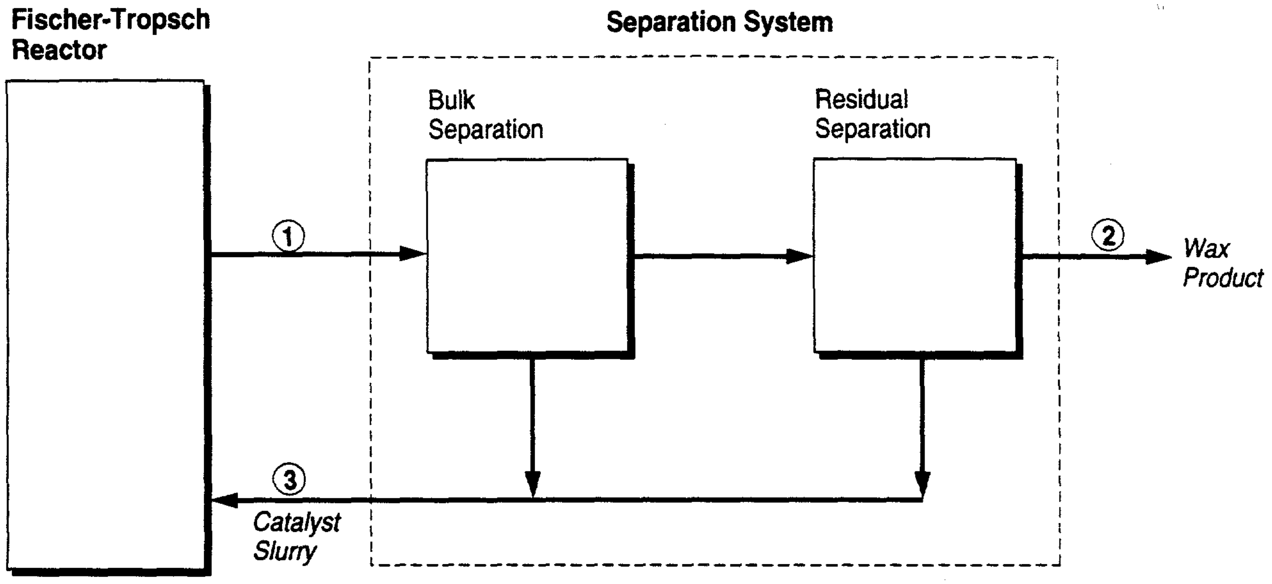
Viscosity. For wax viscosity, a value of 3 cp (3 mPa-s) is used. This value is intermediate between the two entries for Mobil wax in Zhou's Table 4 mentioned above, appropriate for a separation temperature of 229°C. Zhou's two entries fit a formula $\mu = 0.05956 \exp(1{,}966/T)$, where the viscosity μ is in cp and the absolute temperature T is in °K.

2.1.4 Design Basis

The objective of the separation is to produce a solids-free liquid. The removed solids do not need to be liquid-free since the solids are to be recycled to the reactor. The degree of completeness of catalyst removal depends on the required feed quality for wax entering downstream catalytic cracking systems. Catalytic cracker licensors currently wish to limit residual heavy metals (including iron) in typical feedstocks to 2 to 5 ppm, for reasons of avoiding poisoning of the cat cracker catalyst. The Bechtel/Amoco baseline design assumed a clean wax with no catalyst but a catalyst makeup rate equivalent to about 1,600 ppm to account for solids deliberately purged.

Figure 2-3 summarizes the important system design parameters for 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. In this simplified system sketch, a block represents the separation process. Within this block, there could be several steps to accomplish the required separation. The design parameters have been discussed in preceding sections. An exception is the return flow to the F-T reactor. The slurry emerges from the reactor at 488°F and 289 psig, under which conditions the wax is a liquid with a viscosity of 2.5 cp. At 444°F, wax viscosity is 3.0 cp and wax density is 0.696 g/cm^3. The catalyst particles have a calculated density of 4.5 g/cm^3 (this includes wax in the pores). It is assumed that the flow must return at the same pressure as the operating pressure in the reactor. Additionally, it is assumed the return flow cannot exceed 60 wt% solids due to difficulties in pumping and handling.

Residual removal must clean the product liquid wax so that it does not contain more than 2 to 5 ppm iron oxide. This is the assumed purity required for feed liquids to the hydrocracking system.



Quantity	Units	1	2	3	Comments
Pressure	psi	289	< 289	289	
Temperature	°F	488			
Flow	lb/hr	>400,000	300,000		
Wax Density	g/cm^3		0.7		
Wax Viscosity	cp		3		
Concentration	ppm, wt%	< 25%	< 5 ppm	< 60%	1 to 5 ppm in wax product by Zhou
Catalyst Density	g/cm^3	4.5			
Catalyst Size Distribution		Fig. 2-2			

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Figure 2-3 Wax/Catalyst Recovery System

As has been discussed, the particle size distribution is a very significant design parameter. To achieve a wax residual solids level as low as a few ppm, starting with a catalyst concentration of 25 percent, requires a catalyst removal efficiency of 99.999 percent. This implies that essentially all of the catalyst has to be removed from the wax.

Inspection of size spectra in Figure 2-2 shows that to obtain the efficiency for the Mott #1 spectrum, only particles 7 microns and larger would have to be removed, whereas for the STATOIL spectrum, particles as fine as 0.1 micron would have to be removed. Given the required removal efficiency, it is the particle size distribution, specifically how far it extends into the smaller sizes, that determines the degree of difficulty of the separation.

After reviewing the available information on the size of the iron oxide-based catalysts, Bechtel decided to use the STATOIL spectrum shown in Figure 2-2 for the design catalyst size distribution. This was felt to be the best estimate of the distribution in the wax/catalyst mixture discharged from a slurry reactor. The distribution was provided to separation equipment vendors to use in making their equipment selection. The very small sizes in the STATOIL distribution implies that the separation is quite difficult.

The complete design basis is summarized in Appendix 5, which is an attachment to the information request letter sent to separation equipment vendors.

2.2 SEPARATION TECHNIQUES

Regarding practical process design, modern textbooks stress the need to begin with a broad slate of potential techniques, to use rough criteria to screen alternatives down to those most applicable, and then to consider the remaining candidates in increasing depth, each technique singly, or combinations of techniques. For separations, the initial slate is established by focusing on exploiting physical or chemical differences between the components to be separated.

2.2.1 Potential Techniques

A schematic overview of liquid/solids separation schemes has been developed by Woods [Ref. 3], and considers the following eight parameters for purposes of separation:

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

- Wettability
- Vapor pressure

A number of the above techniques can be eliminated from consideration for the F-T system. In the brief discussion below, the techniques are discussed in reverse order of listing.

For the wax/catalyst separation, exploiting vapor pressure (evaporating the wax away) has proved infeasible because the boiling point of the wax is too high.

Exploiting wettability requires introducing a gas phase for flotation, and probably is not feasible for the F-T system because of the added system complexity.

Exploiting the solubility of the solids phase is infeasible for the F-T system.

Solubility of the liquid phase is exploited by the ROSE process, which was considered in the Bechtel/Amoco 1994 baseline F-T plant design. However, it seems infeasible as designed (unless some process modifications are made) for the following reasons:

- The recycled heavy wax will react in the slurry reactor to form even longer wax molecules, which is undesirable.
- The solids carryover with the ROSE process in the baseline design is predicated on a larger size spectrum, and the carryover probably would be unacceptably high with the spectrum being considered in this document.

Exploiting electric charges to produce enhanced separation has not been mentioned to date as a technique to facilitate F-T solids/wax separation. Studies of the potential of this technique could be carried out before electric charge the approach is eliminated.

Exploiting magnetic susceptibility differences between the solids and the wax should be technically feasible, and the susceptibility properties are known for both the solids and the wax. The solids, moreover, contain iron, a highly magnetic element, so that an easy separation may be possible. Accordingly, this process is discussed in more detail in Section 3.

Alteration of wax viscosity by solvent addition is discussed in more detail in Section 3.

Alteration of the solids size spectrum by chemical addition with coagulation is a concept that could be considered for further study. The potential for coagulation to improve ease of separation is discussed in more detail in Section 3.

Exploiting solids/wax density differences by settling and centrifuging is an obviously applicable technique and is discussed in more detail in Section 3.

Exploiting particle size by filtration is a preferred technique and is discussed in more detail in Section 3.

2.2.2 Detailed Considerations

As a result of the above examination, the following processes have been selected for more detailed consideration:

- Filtration
- Sedimentation/centrifuging
- Enhancement by viscosity reduction and size enlarging
- High-gradient magnetic separation

Techniques for further study include methods for coagulation of catalyst solids, exploitation of electric charge, and exploitation of wettability for flocculation.

There is a strong possibility that several techniques will be needed simultaneously in a final suitable separation system. In particular, the final system may need a bulk removal process and a residual removal process in series. Processes to remove the finest of the particles often work best as residual separation systems operating on an already fairly solids-free feed, since residual processes often must be carried out in a batch mode, and the switchover frequency is proportional to the concentration of entering solids. The distinction between bulk and residual processes will probably be the fineness of the particles that can be removed by the bulk removal system.

Consideration of solids loss mechanisms can shed light on the requirements of the separation systems. Design considerations will include:

- Solids leaving in the product liquid will not return to the reactor. By the product purity criterion given in Figure 2-3, the product liquid wax should contain no more than 5 ppm (0.0005 wt%) solids. The liquid from the slurry reactor is typically 20 percent solids and 80 percent wax. This means that the product wax takes 4 times 0.0005 wt%, or 0.002 wt% of the solids in the reactor.
- As shown in Figure 2-2, the separation system must remove all particles with diameters greater than 0.1 micron.
- Because the catalyst is known to become more and more finely divided with time and it is suspected the finer particles are a better catalyst [Ref. 3], it may be desirable to retain the smallest particles and recycle them to the F-T reactor.
- A catalyst removed with a filter aid would be contaminated by the filter aid material. It might not be practical to separate the catalyst from the filter aid.

It should be noted that catalyst makeup must equal the amount of solids lost with the product wax plus the amount lost with the purge stream, thus affecting catalyst economics.

2.3 INITIAL SCREENING OF CANDIDATE SEPARATION PROCESSES

Design conditions were established in Section 2.1.4. Based upon a review of potential separation techniques, several were identified as possible separation methods. Table 2-1 lists these methods and indicates the type of removal (bulk, residual, or both) for which each method is suitable in a separation operation. After screening, several of these separation methods were selected for further investigation. This is described in Section 3.

Table 2-1 Comparison of Separation Methods

Separation Method	Typical Processes	Action	Suitable for:			Comments
			Bulk	Residual	Both	
Particle size	Filtration	More detail	X	X	X	
Density differences	Sedimentation/ centrifuging	More detail	X			
Alternation of properties:						
Wax viscosity	Sedimentation	More detail			X	
Solid size by chemicals	Coagulation	More study		X		
Magnetic differences	HGMS	More detail		X		
Electrical charge		Modify				Include as part of coagulation study
Solubility	K-M ROSE	Modify		X		
Wettability	Flotation	Eliminate		X		
Vapor pressure	Evaporation	Eliminate			X	