

***Magnetic Separation of Catalysts from
Fischer-Tropsch Wax***

*Final Report, Phase II
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ABSTRACT

During Phase I a novel magnetic method employing flow past magnetized rods (patent pending) was shown to separate iron catalyst particles from a simulant of Fischer-Tropsch wax at 390 °F. In Phase II a Magnetic Micro-Particle Separator (MM-PS) – a new concept for continuous magnetic separation of micron-sized particles from viscous flow – was investigated and successfully tested using a proprietary slurry of sub-micron sized iron catalyst agglomerates in Fischer-Tropsch wax at 500 °F at ambient pressure.

The goal of this SBIR Phase II was to prepare an engineering evaluation and cost estimate for a 15 gallon per minute product pilot unit for testing magnetic separation of iron catalysts from Fischer-Tropsch wax.

The following objectives were established to meet this goal:

- Build an experimental test unit capable of feeding slurry containing 15 – 20 wt.% catalyst to the separator at the rate of nominally two gallons per minute (gpm) with continuous production of wax containing less than one wt% catalyst at the rate of nominal 0.2 gpm which is the equivalent of 7 barrels per day (BPD).
- Test High Gradient Magnetic Separation as a polishing operation for the first stage continuous separator.
- Design and cost an apparatus for 15 gpm product operation based on the results of Phase II.

The goal of 7 BPD throughput was exceeded. A slurry with 0.35 wt% catalyst was produced at the rate of 50 BPD in true continuous operation at 500 °F using the MM-PS. The upper limit of catalyst reduction and throughput measured in Phase II was imposed by the limited supply of slurry and by the experimental apparatus which had been designed for a lower throughput – not by the ability to separate catalyst particles from the slurry. High Gradient Magnetic Separation (HGMS) was tested to polish the MM-PS overflow. Catalyst concentrations in the 500 ppm range were obtained; these results were similar to those obtained in Phase I. The level of post-HGMS particle concentration may have been associated with contaminants in the slurry.

A cost estimate for an instrumented 15 gpm MM-PS pilot test unit was prepared based on a very conservative projection of the size unit required for this throughput. Using this cost estimate as a basis and employing a scaling factor of 0.8, capital and operating costs were approximated for 10,000 and 100,000 BPD MM-PS commercial units. The capital costs are 30 and 19 \$/BPD; operating costs, including capital amortized over 20 years, are 0.69 and 0.44 \$/B, respectively. Multi-stage operation or post MM-PS polishing will be required to achieve a catalyst concentration in the 10 ppm range if required for downstream processing.

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EXECUTIVE SUMMARY

Overview

During Phase I a novel magnetic method employing flow past magnetized rods (patent pending) was shown to separate iron catalyst particles from a simulant of Fischer-Tropsch wax at 390 °F. In Phase II a Magnetic Micro-Particle Separator (MM-PS) – a new concept for continuous magnetic separation of micron-sized particles from viscous flow – was investigated and successfully tested using a proprietary slurry of sub-micron sized iron catalyst agglomerates in Fischer-Tropsch wax at 500 °F at ambient pressure.

The goal of this SBIR Phase II was to prepare an engineering evaluation and cost estimate for a 15 gallon per minute (gpm) product pilot unit for testing magnetic separation of iron catalysts from Fischer-Tropsch wax.

The following objectives were established to meet this goal:

- Build an experimental test unit capable of feeding slurry containing 15 – 20 wt% catalyst to the separator at the rate of nominally 2 gpm with continuous production of wax containing less than one (1) wt% catalyst at the rate of nominal 0.2 gpm which is the equivalent of 7 barrels per day (BPD).
- Test High Gradient Magnetic Separation (HGMS) as a polishing operation for the first stage continuous separator.
- Design and cost an apparatus for 15 gpm product operation based on the results of Phase II.

Sixty-five gallons of Fischer-Tropsch wax slurry were procured for use in the test program.

The goal of 7 BPD throughput was exceeded. A slurry with only 0.35 wt% catalyst was produced at the rate of 50 BPD in true continuous operation using the MM-PS. The upper limit of catalyst reduction seen in Phase II was imposed by the limited supply of slurry and by the experimental apparatus which had been designed for a lower throughput – not by the ability to separate catalyst particles from the slurry. High Gradient Magnetic Separation (HGMS) was tested to polish the MM-PS overflow. Catalyst concentrations in the 500 ppm range were obtained; these results were similar to those obtained in Phase I. The level of post-HGMS particle concentration may have been associated with contaminants in the slurry.

A cost estimate to build an instrumented 15 gpm MM-PS pilot test unit was prepared based on a very conservative projection of the size unit required for this throughput. Using this cost estimate as a basis and employing a scaling factor of 0.8, capital and operating costs were approximated for 10,000 and 100,000 BPD MM-PS commercial units. The capital costs are 30 and 19 \$/BPD; operating costs, including capital amortized over 20 years, are 0.69 and 0.44 \$/B,

respectively. Multi-stage operation or post MM-PS polishing will be required to achieve a catalyst concentration in the 10 ppm range if required for downstream processing.

Apparatus

A test bed was designed and built to contain the slurry, mix it, pump it to the separator, monitor it, and receive the product and return streams. A high pressure mixing tank and overflow and underflow tanks were insulated to maintain the wax slurry at 500 °F. ETCi supplied the electromagnet used in the program and designed the separators which were fabricated at a local machine shop. The entire apparatus was installed within an isolated air-swept enclosure which was continuously monitored for explosive gaseous buildup.

The initial test runs employed a two-inch diameter canister which was later replaced by a six-inch diameter canister.

Characterization and Analytical Measurements

Magnetization measurements were performed by Dr. S. Chu at Carnegie Mellon University using a vibrating sample magnetometer. Computer Controlled Scanning Electron Microscopy (CCSEM), Computer Controlled Transmission Electron Microscopy (CCTEM), melting range, and iron content were measured by the RJ Lee Group, Inc. Mössbauer measurements, performed by Dr. H. Hamdeh of Wichita State University, gave qualitative information on the superparamagnetic content of the catalyst particles. Solids concentration of overflow and underflow samples for each experimental run was determined by a modified ASTM ashing procedure, D-482.

Results

The focus of the test work was (1) to identify the parameters affecting catalyst separation during continuous operation and (2) to determine the maximum throughput that could be achieved with the MM-PS with the constraint that the concentration of the catalyst in the separator output not exceed 1 wt%.

A typical run consisted of feeding wax slurry containing 21.45 wt% catalyst into a separation vessel through feed lines. The underflow contained 23.33 wt% ash and the overflow contained 0.35 wt% ash. The ash level in the overflow was 98% less than that in the feed.

The residence time in the apparatus was 11 seconds. For the six-inch canister the total process flow during a two-hour run had a volume equal to 660 times that of the empty separation canister. Thus, a volume equal to 660 times that of the canister was processed without signs of plugging.

Application to Fischer-Tropsch Synthesis

Figure ES-1 is a flow diagram of one method of applying the technology to Fischer-Tropsch synthesis. The figure shows a Fischer-Tropsch reactor with a slurry zone containing a

liquid comprising waxes made in the reactor and magnetic catalyst particles. Synthesis gas comprising hydrogen and carbon monoxide is added at the bottom of the reactor. Vapors are removed from the reactor overhead. Slurry is drawn from the slurry zone into the vapor liquid separator. Vapors are returned to the reactor and the slurry flows through a valve into the MM-PS. The magnetic particles exit the bottom of the separator and may be pumped back into the reactor to recycle the catalyst particles. The exit stream is shown passing through an optional demagnetizing coil.

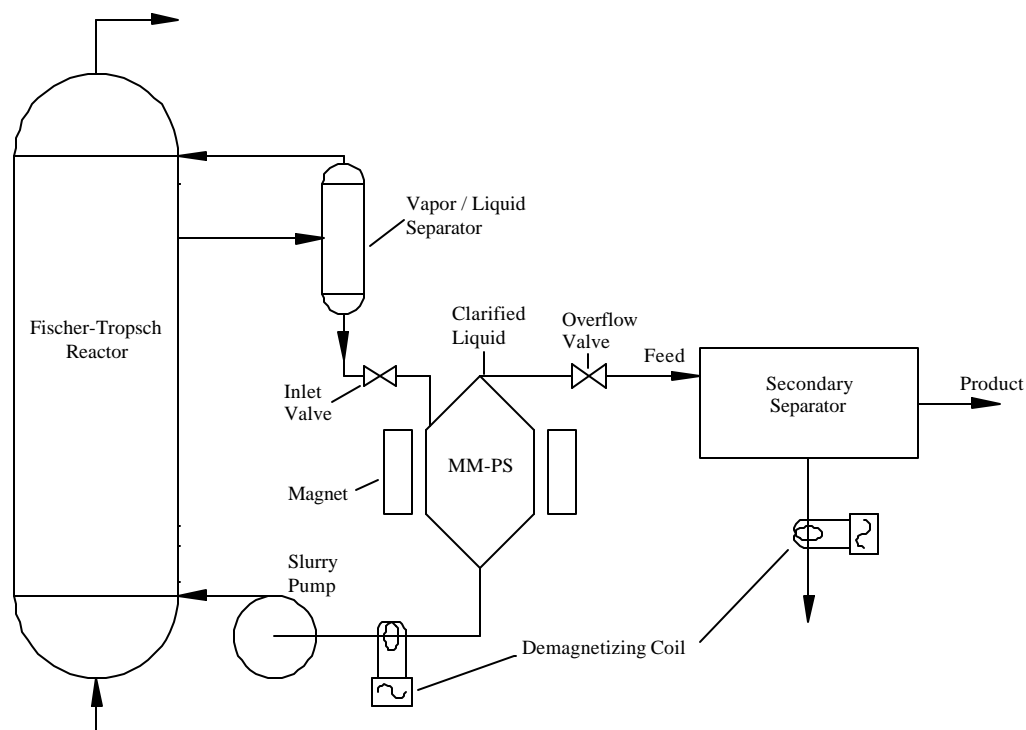


Figure ES-1. Fischer-Tropsch Synthesis.

A clarified liquid is removed at the top of the continuous magnetic separator to a secondary separator for further processing. High Gradient Magnetic Separation (HGMS) may be used as the second stage separator, although other filters may be used as well. It is not as important that this process step be continuous since it is not integrated into the synthesis process.

Conclusions

Micron size agglomerates of sub-micron size catalyst particles with an individual particle size ranging between 2 and 60 nm have been separated from Fischer-Tropsch wax at 260 °C by the MM-PS method at a rate of 50 barrels per day. These particles can be returned to the reactor or discarded as is required in the process application.

INTRODUCTION

Fischer-Tropsch synthesis (F-TS) is a well established technology for production of high quality synfuels.¹ The technology employs catalysts generally based on either iron (Sasol) or cobalt (Shell). Tubular fixed bed reactors originally employed at Sasol in 1955 and currently employed by Shell at its Bintulu, Indonesia plant have developed into slurry bed reactors primarily for coping with the high heat release from the exothermic F-TS reactions.² The catalyst is removed from the slurry bed reactor along with the wax products.

Currently, cobalt catalysts are of great interest because of the potential of cobalt based F-TS for monetizing stranded natural gas. Iron catalysts are attractive, however, because of the highly olefinic nature of their products and because of the activity for the water-gas shift reaction that permits use of low H₂/CO syngas derived from the gasification of coal and other disadvantaged hydrocarbons.³ Unsupported iron catalysts have been developed which are micron sized agglomerates of nanometer magnetite particles. In the F-TS reactor, the magnetite particles react with carbon monoxide to produce iron carbides which are the actual source of catalytic activity. The carbide particles differ in crystal structure from the oxide and as such are prone to break away from the surface of the agglomerate in the reactor due to attrition.⁴ More stable supported iron catalysts have not yet been developed for commercial application. It is apparent that one of the major operational problems associated with the use of the iron catalyst for F-TS using slurry reactor technology is separation of the catalyst from the product wax. Conventional methods of separation have proven to be ineffective.

ExxonMobil has a patented magnetic method⁵ using High Gradient Magnetic Separation technology. This is a batch technology used commercially to separate micron sized discolorant particles from slurries of kaolin clay in a batch operation.⁶ Even though HGMS is capable of separating micron sized particles, it is expected to be limited to separation of low concentration catalyst from F-TS wax because of the nature of the magnetic filter employed. The need in the F-TS application, however, is for continuous separation of nominal 20 wt% catalyst streams making HGMS appear to be a better candidate for solids polishing than for primary separation.

This report describes a novel magnetic method for rapid continuous removal of sub-micron sized iron catalyst particles from F-TS wax. Testing was carried out at 500 °F and ambient pressure. The ability to rapidly separate concentrated sub-micron sized particles from viscous wax is of great importance because of the need to maintain temperature and pressure for efficient return of the catalyst particles to the reactor. Justification for the technology derives from the fact that iron and cobalt are strongly magnetic. Magnetite is ferrimagnetic and carbides formed in F-TS are also magnetic.

¹ Anderson, R. B. *The Fischer-Tropsch Synthesis*, Academic Press, New York (1984).

² B. Jager, R. Espinoza, Advances in low temperature Fischer-Tropsch synthesis, *Catalyst Today* **23** (1955) 17-28.

³ L. Xu, S. Bao, R.J. O'Brien, A. Rajee, and B.H. Davis, Don't rule out iron catalysts for Fischer-Tropsch synthesis, *Chemtech* (January 1998) 47-58.

⁴ Burtron Davis, Poster No. 6, Fischer-Tropsch Synthesis, <http://crtc.caer.uky.edu/fischt.htm>.

⁵ James A. Brennan, Arthur W. Chester, Yung-Feng Chu, Separation of catalyst from slurry bubble column wax and catalyst recycle, US Patent 4,605,678 (August 12, 1986).

⁶ R.R. Oder and C. R. Price, "Brightness beneficiation of kaolin clays by magnetic treatment," *TAPPI* **56** (1973) 75.

Goals and Objectives

The goal of this SBIR Phase II was to prepare an engineering evaluation and cost estimate for a 15 gallon per minute (gpm) pilot unit for testing magnetic separation of iron catalysts from Fischer-Tropsch wax.

The following objectives were established to meet this goal:

- Build an experimental test unit capable of feeding slurry to the separator at the rate of nominally two (2) gpm with continuous production of wax containing less than one (1) wt% catalyst at the rate of nominal 0.2 gpm, the equivalent of 7 barrels per day (BPD).
- Test High Gradient Magnetic Separation (HGMS) as a polishing operation for the first stage continuous separator.
- Design and cost an apparatus for 15 gpm operation based on the results of Phase II.

MAGNETIC MICRO-PARTICLE SEPARATOR

The Magnetic Micro-Particle Separator

During Phase II EXPORTech built and tested an apparatus and a method to separate magnetic particles from non-magnetic fluid in which the particles are entrained. The particles and fluid are passed through a separation chamber located between the poles of a magnet. The particles are swept to an underflow outlet at the bottom of the chamber where a concentrated stream of particles exits the container; a fluid diminished in particles flows out the top of the chamber. Non-magnetic particles follow the slurry flow, the bulk of which exits the bottom of the chamber. The high solids slurry exiting the bottom of the separation chamber through the exit valve could be returned to the slurry source if appropriate. Likewise, the low solids slurry exiting the top of the separation chamber through the overflow valve could be subjected to additional separation employing this or other methods such as cross flow filtration, sedimentation, centrifugation, or high gradient magnetic separation, etc.

The feed, overflow, and underflow rates are controlled. A pump was employed to force the fluid through the separation canister. Valves are employed with the pump to control the rates of high solids underflow and diminished solids overflow respectively. The magnetic fields employed need only be large enough to saturate the magnetism of the particles.

The apparatus has been found to be especially effective in separating micron sized particles and especially sub-micron sized iron catalyst particles from Fischer-Tropsch wax at elevated temperatures. Magnetic fields of 1500-2000 gauss are sufficient for separation of high throughput (i.e., 200-300 kg/min/m²) micron size precipitated iron catalyst particles from Fischer-Tropsch wax at 260 °C. The experimental apparatus used in Phase II is capable of separating 20 ~ 25 wt.% concentration sub-micron sized iron catalyst to produce a Fischer-Tropsch wax concentrate with catalyst concentration in the 0.1 – 0.5 wt.% range on a continuous

basis at throughputs 20 to 50 times greater than can be achieved by sedimentation. When High Gradient Magnetic Separation is employed as a second stage of separation, diamagnetic wax slurries with particle concentrations in the 0.01 to 0.05 wt.% range have been prepared.

The benefit of this technology is that flows containing high concentration of magnetic particles of a very broad size range can be efficiently separated in a true continuous mode of operation. Further, the throughputs achievable with this method are much higher than possible with conventional sedimentation or filtration so that the separation apparatus can be kept small by comparison. This is advantageous where high temperature and high pressure are involved as is the case in commercial separation of magnetic catalysts from Fischer-Tropsch wax.

EXPERIMENTAL

This section gives the results of characterization of the Fischer-Tropsch slurry material, describes the experimental apparatus, and summarizes the experimental procedures employed.

Characterization of Fischer-Tropsch Slurry

Nominally 65 gallons of Fischer-Tropsch (F-T) wax with iron catalyst was used in the test program. The material was supplied in two 55 gallon barrels, one of which had been punctured in transit as shown in Figure 1. The wax was contaminated with parts of electrical wiring insulation and other debris which were hand-picked before loading into the apparatus. An in-line filter was installed ahead of the slurry pump to prevent large pieces of debris from entering the separator.



Figure 1. Damaged Drum

Overflow and underflow samples were characterized for ash content for each experimental run. Magnetization measurements were performed in Phase I on selected samples by Lakeshore Cryotronics, Inc. and in Phase II by Dr. S. Chu at Carnegie Mellon University using a vibrating sample magnetometer. Computer Controlled Scanning Electron Microscopy (CCSEM), Computer Controlled Transmission Electron Microscopy (CCTEM), melting range, and iron content were measured by the RJ Lee Group, Inc. Mössbauer measurements, performed by Dr. H. Hamdeh of Wichita State University, gave qualitative information on the superparamagnetic content of the catalyst particles. Solids concentration was determined by a modified ASTM ashing procedure, D-482.

CCSEM

The elemental concentration of the proprietary slurry employed in Phase II was determined by CCSEM and is shown in Figure 2. The majority of the particles were iron rich. Mössbauer measurements identified the oxidation states of the iron and, along with transmission electron microscopy (TEM) of similar materials, identified how much of the iron was in the nanometer size range.

Melting Point: 83-90°C
16.4 wt% ash
13.5 wt% Fe (by ICP)

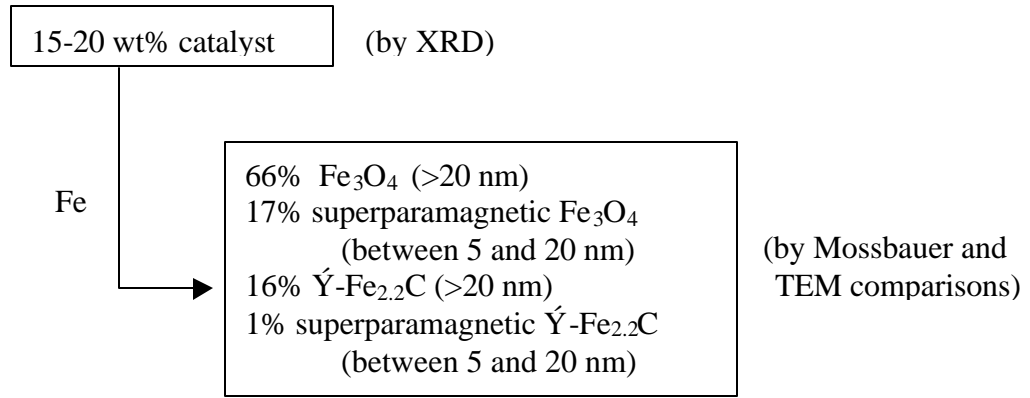


Figure 2. Slurry Analysis of Melting Point, Catalyst Concentration, and Iron Oxidation States

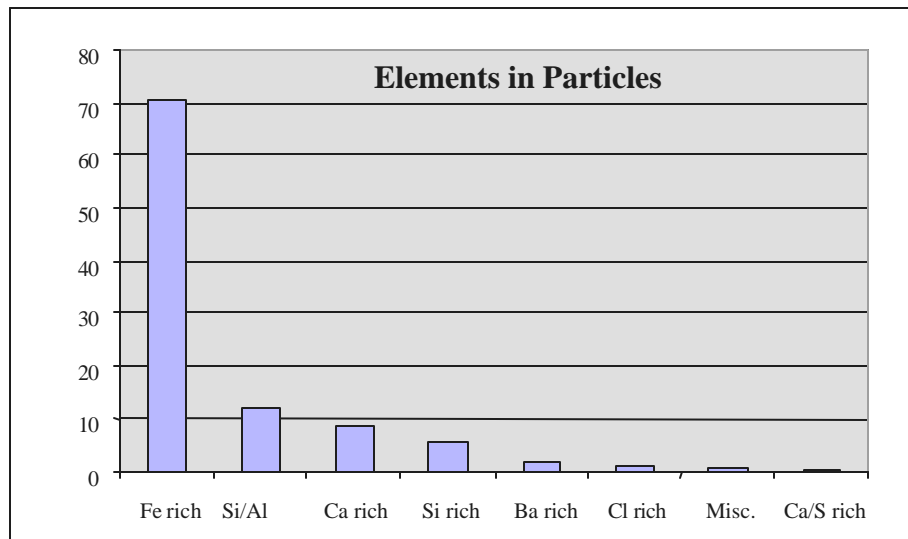


Figure 3. Particles Rich in Certain Elements

Particle Size Distribution

Measurements of particle size distribution (PSD) made in Phase I are shown in Figure 4 to indicate the effects of attrition observed after hundreds of passes through the apparatus.

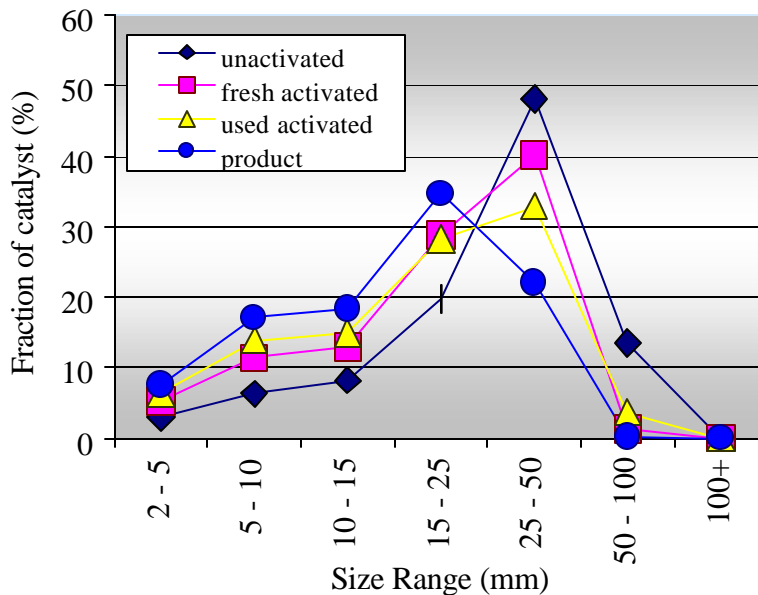


Figure 4. Particle Size Distributions Measured in Phase I

The PSD shifted slightly toward smaller particle sizes upon CO activation with the greatest change for the 50-100 μm range. When the catalyst had been run through the separator for several recycle experiments (a total of hundreds of passes), the PSD shifted slightly again to smaller particle sizes. The PSD for the output from the first stage MM-PS separator is shifted significantly to smaller particle sizes with greater than 75% of the catalyst smaller than 25 μm .

Additional particle size distribution measurements were made using CCTEM by the R.J. Lee Group. These measurements showed that the micron-sized particles are actually loose aggregates of particles ranging from a few nm to 60 nm in size. Figure 5 shows nm sized particles; the sizes of individual particles range from 20 to 50 nm.

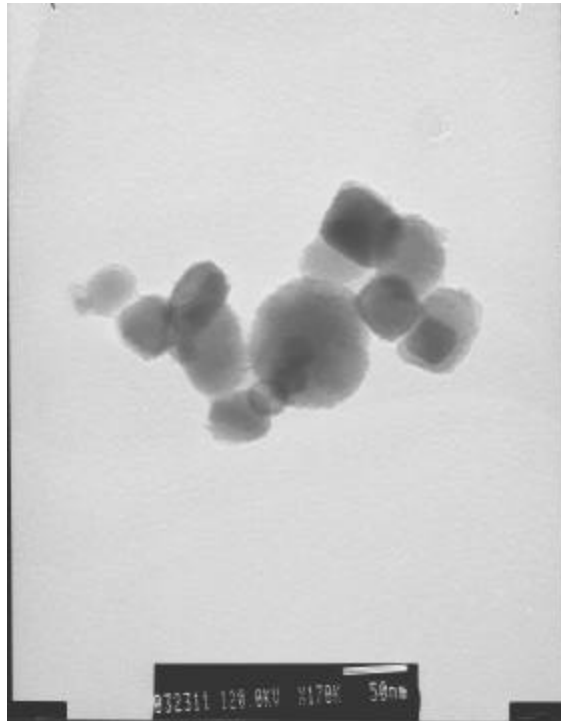


Figure 5. Nano-sized particles

Kinematic Viscosity

Viscosity was measured at different temperatures using Spindle LV-2 at 60 RPM of a Brookfield viscometer. Slurry density measured at 19.7 wt% solids and 310 °C was determined to be 0.945 g/cm³ by mass and volume measurement.

Magnetization Measurements

Magnetization measurements were made by Carnegie Mellon University using a Foner vibrating sample magnetometer (VSM). Problems were experienced with the CMU sample holder, which became strongly paramagnetic upon cycling the temperature between ambient and 300 °C. Further, the test sample was a liquid at temperatures above the melting point so the magnetic particles could move in the liquid. Because of these problems, values such as magnetic moment, etc., determined from these measurements are qualitative at best.

VSM measurements for the MM-PS feed are presented in Figure 6.

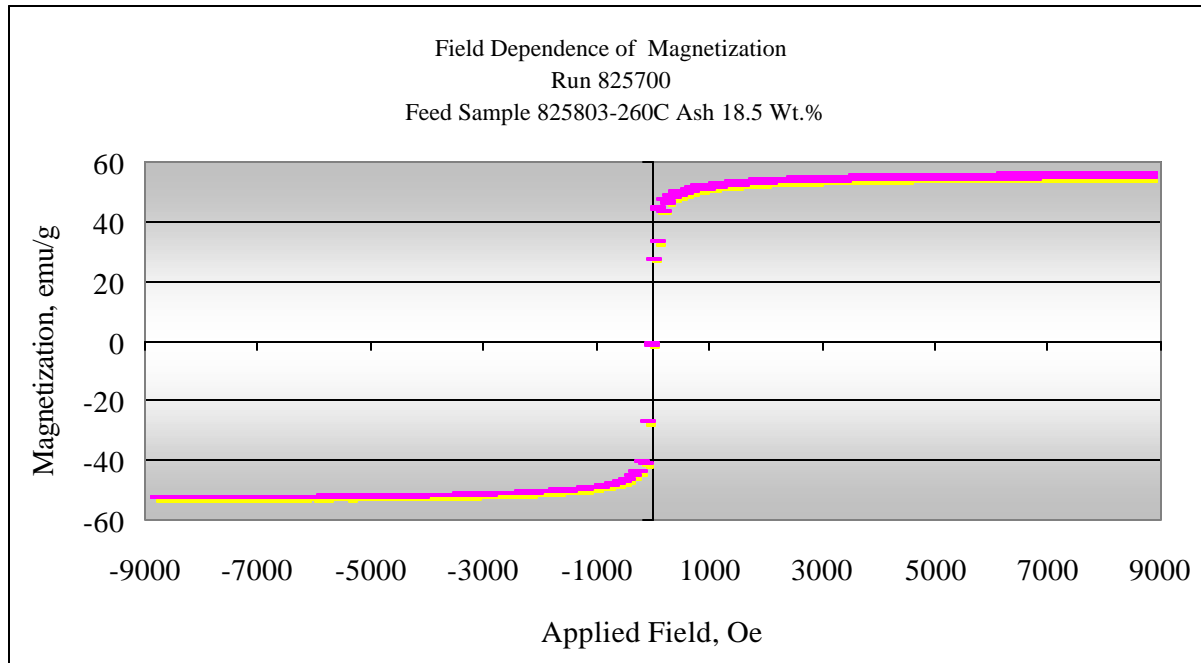


Figure 6. Field Dependence of the Magnetization per gram of the MM-PS Feed.

The feed exhibits strong collective magnetism. There is also a large paramagnetic component superimposed on the magnetic moment of the feed. Qualitatively, from the data it is estimated that the saturation magnetic moment is of the order of 50 emu/g, the remanent magnetization is about 20 emu/g, the coercivity is 30-40 Oe, and the paramagnetic susceptibility is about 7500 micro-emu/g-Oe. The underflow is similar to the feed.

Test Bed Assembly

A test bed was designed and built to contain the slurry, mix it, pump it to the separator, monitor it, and receive the product and return streams. Figure 7 is an illustration of the process flow and monitoring equipment. The product stream is reduced in catalyst concentration relative to the feed and is called the overflow. The return stream has a higher catalyst concentration than the feed and is called the underflow. The overflow rate is significantly less than the underflow rate (typically one tenth). Therefore, while the catalyst concentration in the overflow is reduced by more than 95 % (i.e., from 20 wt% in the feed to 0.7 wt% in the overflow), the concentration in the underflow is increased by less than 10 % (i.e., from 20 wt% in the feed to 21.9 wt% in the underflow).

Process & Instrumentation Diagram

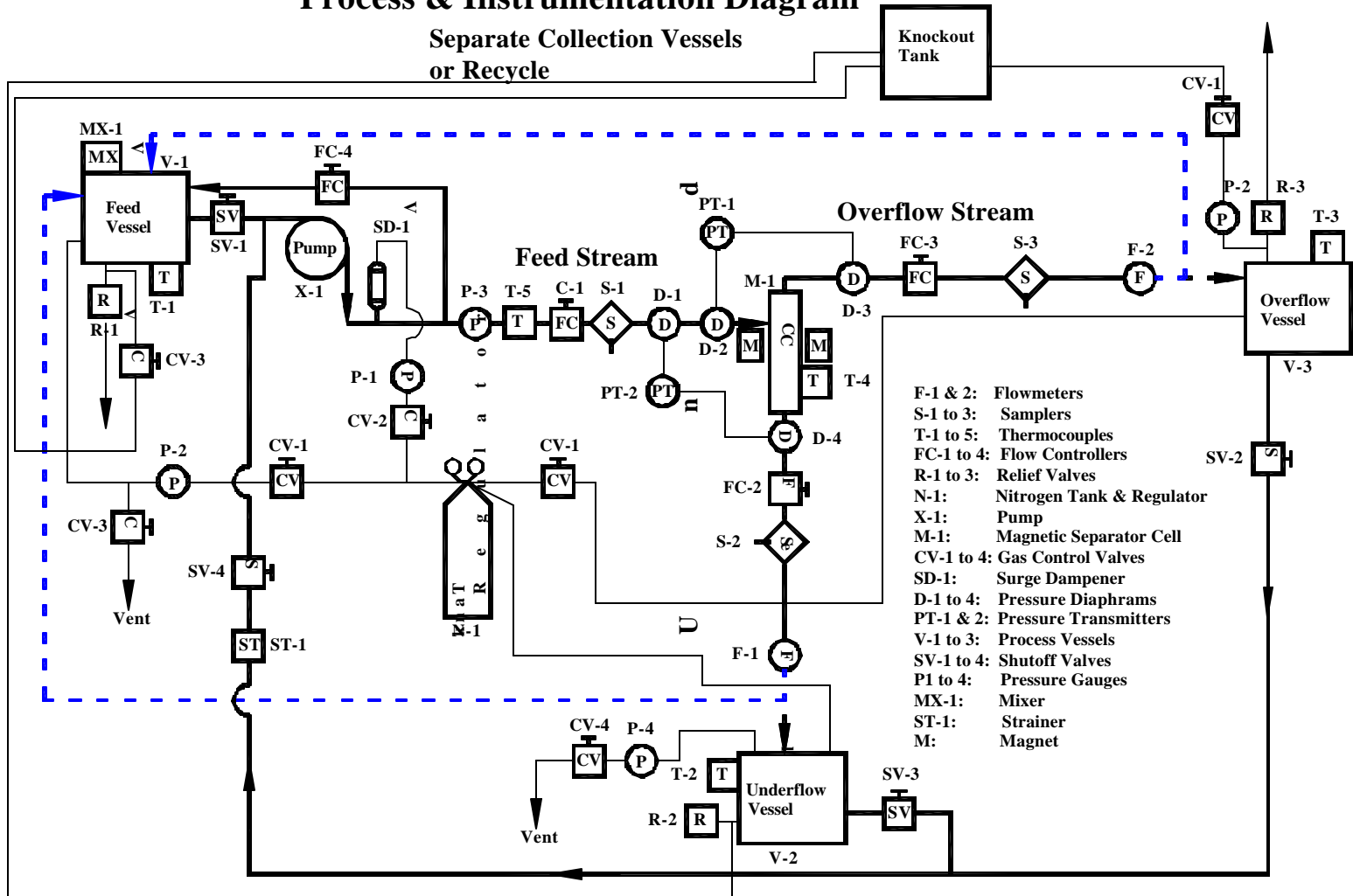


Figure 7. Process & Instrumentation Diagram

The 109 gallon feed tank (Figure 8) was manufactured by the Gaumer Company. It has the necessary inlets and outlets for the slurry. The tank was designed to withstand pressures of 150 psi; a 165 psi pressure relief valve was also provided for safety. Band heaters and insulation were included to heat and maintain the slurry at 500°F. GLD Company also supplied the cable heaters used on the slurry transport lines, the pump, and the overflow and underflow tanks. A constant flow of nitrogen was maintained in the tank to inert the atmosphere within it as long as a slurry was in the tank.

A Lightnin Mixer is shown in Figure 8 mounted at the top of the feed tank. The mixer's speed and blades were chosen to keep the slurry well mixed while minimizing the shear on the particles.

The underflow and overflow storage tanks (shown on the right in Figure 8) were purchased from McMaster-Carr Supply Company. The underflow tank has a volume of 120 gallons, and the overflow tank has a volume of 60 gallons. These tanks were used only when the entire volume of separated slurry was stored separately instead of being returned to the feed tank. They were also used to mix and store the slurry when the feed tank had to be filled or drained. All tanks were insulated and slurry lines heat traced and insulated.

The slurry pump (Figure 9) was manufactured by the Tuthill Corp. Its pumping rates are approximately one gpm to more than 50 gpm at 5 psi. In order to operate below one gpm, a recycle line was "teed" off the separator feed line back to the tank through a control valve. The control valve regulated the flow rate of slurry recycled back to the tank and the remaining flow went to the separator. A surge ballast (not shown) was installed on the pump outlet to reduce the fluctuations in flow.

ETCi designed the separators which were manufactured by Alkab Contract Manufacturing, Inc. The electromagnet (Figure 10) was manufactured by Everson Electric Company and designed and supplied by ETCi. The electromagnet generated fields up to 7700 Gauss.



Figure 8. Feed Tank with Mixer on Top. Overflow and Underflow Tanks Are Shown on the Right covered with Insulation.



Figure 9. Pump



Figure 10. Electromagnet

Figure 11 shows the control panel and power supply. Figure 12 shows feed and overflow lines before thermal insulation was added. The entire apparatus was installed within an isolated air-swept enclosure which was continuously monitored for explosive gaseous buildup.



Figure 11. Control Panel and Power Supply



Figure 12. Feed and Overflow Lines

Experimental Setup

A Tuthill Model HD-70A lobe pump shown in Figure 9 above was used to pump a slurry of Fischer-Tropsch wax containing iron catalyst particles through a continuous magnetic separator. The catalyst particles consisted of iron oxide particles which had been subjected to Fischer-Tropsch synthesis conditions. Portions of the catalyst particles had been converted to iron carbides upon exposure to carbon monoxide and hydrogen in the Fischer-Tropsch reactor. The individual particle size ranged from 2 to greater than 60 nm. The kinematic viscosity of the slurries ranged from less than 1 cS to greater than 600 cS for solids ranging from 0.5 to 35 wt% at nominal 500 °F. A Brookfield viscometer was employed to measure the viscosity. Slurry density measurements were approximated from mass and volume measurements at 500 °F.

Experimental Procedure

The temperatures in the tank, the feed line, and the separator were monitored during each run. Flow rates and differential pressures were also measured. Following each run, the solids concentrations in the feed, overflow, and underflow samples were determined by ashing.

RESULTS OF TESTING

The focus of the test work was (1) to identify the parameters affecting catalyst separation during continuous operation and (2) to determine the maximum throughput that could be achieved with the MM-PS with the constraint that the concentration of the catalyst in the separator output not exceed 1 wt%.

No maximum in throughput was observed with the experimental apparatus. Although the original goal of Phase II was to achieve an output of 0.2 gpm, we operated up to 1.7 gpm without sacrifice of performance. The throughput was limited by the experimental apparatus, i.e., the pump, connecting lines, electromagnet gap, etc., and not by an ability to achieve an overflow concentration of less than 1%. Optimum operation will be determined by a tradeoff involving costs.

Typical Run

A slurry containing 21.45 wt% catalyst was fed into a separation vessel at a low magnetic field. The underflow contained 23.33 wt% ash. The overflow 0.35 wt% ash. The ash level in the overflow was 98% less than that in the feed.

The residence time in the apparatus was 11 seconds. The total process flow during the two-hour run had a volume equal to 660 times that of the empty separation canister. Thus, a volume equal to 660 times that of the canister was processed without signs of plugging.

General Overview

The general effects of canister size and magnetic field strength can be seen in Figure 13 which has been compiled using the results of early runs under many different operating conditions. At low overflow rates, where the overflow ash is generally in the range of 0.3 to 1.0 wt%, changes in the operating variables have little effect on overflow ash until an upper level in overflow rate is achieved where further increases in the flow rate makes a precipitous increase in overflow ash as can be seen in the figure. The leveling off of ash concentrations above the breakpoint of the curve on the high flow rate side, while not fully understood, is believed to be a result of interplay between the many process variables which were tested.

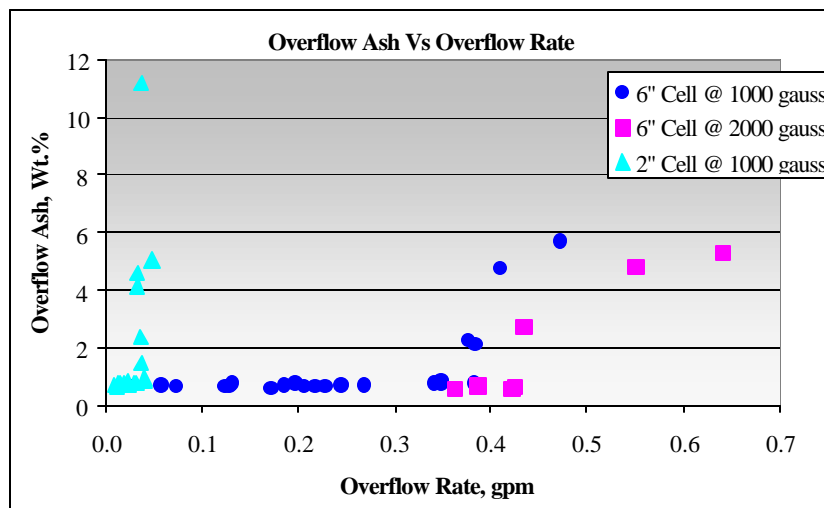


Figure 13. Overflow Ash vs. Overflow Rate for Different Canisters and Levels of Applied Magnetic Field

Magnetic Field Effects

To determine the effects of magnetic field strength, a slurry of nominal 19.1 ± 0.5 wt% ash was fed to the separator at an average rate of 11.3 gpm. The magnetic field strength was varied from 0 up to 2000 gauss. Valves were used to maintain a constant overflow rate and recycle ratio for the measurements. The ash levels in the feed, underflow, and the overflow were measured by flaming the wax sample and then ashing using ASTM procedure D-482. The percentage reduction in ash was calculated as $\% \text{ reduction} = [(\text{ash in feed} - \text{ash in overflow}) / \text{ash in feed}] * 100$.

The applied magnetic field has a major effect on the separation process. For separation of more than 96% by weight of the catalyst, the applied field must be sufficient to bring the magnetic moment of the catalyst particles to 95% of saturation. The relationship between the particle separation, as measured by percent difference between the feed and overflow ash levels, and the magnetization of the slurry of 19.1 ± 0.5 wt.% catalyst is shown in Figure 14.

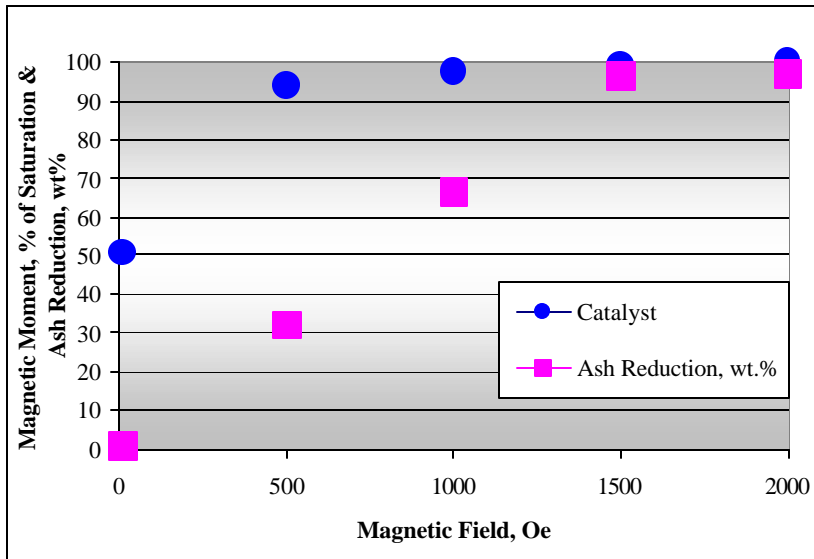


Figure 14. Relationship Between Magnetic Moment and Ash Reduction, Saturation Moment 53 emu/g @ 260°C

Comparison to Stokes Settling

Values of the specific settling rate for Stokes Law can be calculated for comparison with the measured values for the unoptimized MM-PS. Stokes Law values are illustrated in Figure 15 for particles and fluid of densities similar to those for the Fischer-Tropsch wax used in Phase II. testing.

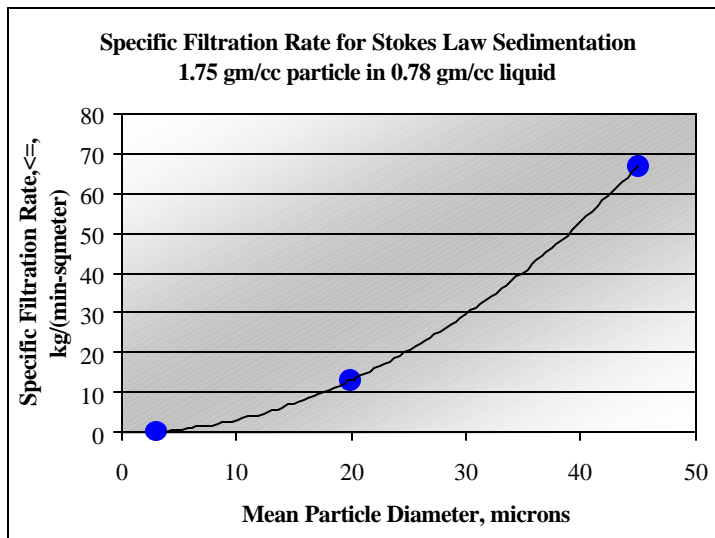


Figure 15. Calculated Values of Stokes Settling Rate, kg/min/m²

Measurements of particle size made in Phase I show that the peak in particle size for the MM-PS is in the 15-25 μm size range. Using 20 microns as the average in this interval, the Stokes settling rate calculates to 13 $\text{kg}/\text{min}/\text{m}^2$. With an average of 232 $\text{kg}/\text{min}/\text{m}^2$ potential, the unoptimized MM-PS employed in Phase II separates at a rate 18 times higher than Stokes settling. The measured value of 227 for the specific filtration rate, $\text{kg}/\text{min}/\text{m}^2$ is 17 times greater than the Stokes settling rate. The rate conservatively estimated by Davis for the Sasol commercial unit is 0.53 $\text{kg}/\text{min}/\text{m}^2$. The measured specific separation rate for the MM-PS is 400 times that estimated by Davis for the Sasol unit and 95 times greater than that reported by Davis for one run with the University of Kentucky CAER one liter Fischer-Tropsch unit!⁷

ENGINEERING EVALUATION

An engineering evaluation for a 15 gallon per minute (gpm) Magnetic Micro-Particle separator designed to be retrofitted to a single Fischer-Tropsch reactor column to produce product at the rate of 15.5 gpm was carried out.

The data used for this evaluation are based on results of testing the two inch diameter and the six inch diameter Magnetic Micro-Particle Separators (MM-PS) employed in Phase II.

The bare 15 gpm MM-PS consists of the separation canister with feed inlets, overflow outlet, underflow outlet, a pressure relief valve, temperature control, and electromagnet. The canister diameter has been scaled to 19.64 inches to provide 15.5 gpm employing the throughput rate, nominally 7.13 gpm/Ft^2 , measured for the six inch diameter canister in Phase II. The magnet design employed in Phase II is used in the cost evaluation although less expensive magnets are visualized for a commercial unit.

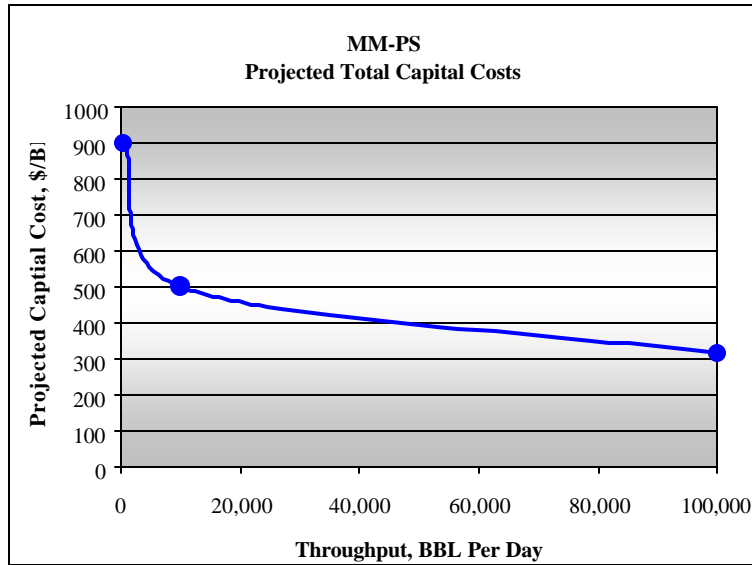
Cost Estimate

Based upon the cost estimates made for the 15 gpm unit, capital and operating costs have been projected for 10,000 and 100,000 BBL per day MM-PS installations using a scaling factor of 0.8. It is assumed that capital is paid out over 20 years. The operating cost includes a capital charge as well as labor, maintenance, and power. This result is shown in Figure 16.

⁷ Burtron H. Davis and Enrique Iglesia, Technology Development for Iron and Cobalt Fischer-Tropsch Catalysts, *Quarterly Report, April 1 – June 30, 1999, US Department of Energy Contract DE-FC26-98FT40308*, p. 28.

Final Report

Throughput BPD	Total Capital Cost \$	Total Capital Cost \$/BPD
531	\$476,252	\$896
10,000	\$4,982,886	\$498
100,000	\$31,439,885	\$314



Throughput BPD	Operating Expense Capital Amortized Over 20 Years	
	\$/Yr	\$/B
531	\$259,309	\$1.34
10,000	\$2,713,073	\$0.74
100,000	\$17,118,331	\$0.47

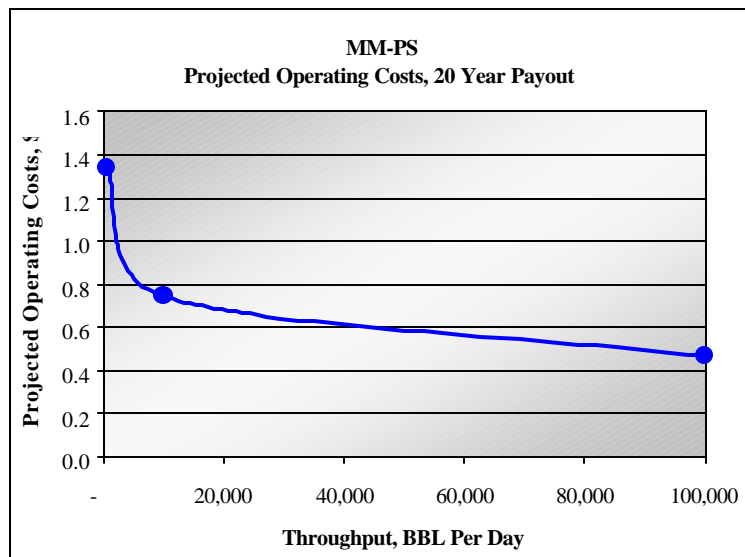


Figure 16. Estimated Capital and Operating Costs for 10,000 BBL and 100,000 BBL Per Day MM-PS Installations

CONCLUSIONS

Agglomerates of sub-micron size catalyst particles with an individual particle size ranging between 2 and 60 nm have been separated from Fischer-Tropsch wax at 260 °C by the MM-PS method at a rate of 50 barrels per day. These particles can be returned to the reactor or discarded as is required in the process application.

An estimate of the cost to build an instrumented 15 gpm MM-PS pilot test unit was prepared based on a very conservative projection of the size unit required for this throughput. Using this cost estimate as a basis and employing a scaling factor of 0.8, capital and operating costs were approximated for 10,000 and 100,000 BPD MM-PS commercial units. The capital costs are 30 and 19 \$/BPD respectively, and operating costs are 0.69 and 0.44 \$/B respectively. Multi-stage operation or post MM-PS polishing will be required to achieve a catalyst concentration in the 10 ppm range if required for downstream processing.

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