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Magnetic Separation of Nanometer Size Iron Catalyst from Fischer-Tropsch Wax

R. R. Oder

EXPORTech Company, Inc., P.O. Box 588, New Kensington, PA 15068-0588, USA
724-337-4415 / FAX 724-337-4470, roder@magneticseparation.com

1. Abstract

This paper presents preliminary results using the Magnetic Micro-Particle Separator, (MM-PS, patent pending) which was conceived for high throughput isothermal and isobaric separation of nanometer (nm) sized iron catalyst particles from Fischer-Tropsch wax at 260 °C. Using magnetic fields up to 2,000 gauss, F-T wax with 0.3- 0.5 wt% solids was produced from 25 wt% solids F-T slurries at product rates up to 230 kg/min/m^2. The upper limit to the filtration rate is unknown at this time. The test flow sheet is given and preliminary results of a scale-up of 50:1 are presented.

2. Background

The novel separation technology (Magnetic Micro-Particle Separator, Patent Pending) evolved from a magnetic method for breaking solids-stabilized emulsions [1] which required implanting a ferromagnetic seed into the internal phase of the emulsion. The internal phase was coalesced in a magnetic field and drawn to collecting magnetic rods or wires where it was withdrawn from the separator under force of fluid flow. This is illustrated in Fig. 1 where the magnetic elements are permanent magnet rods. For the case of water in oil emulsions, iron ligno-sulfonate was used as the ferromagnetic seed. The technology was tested in recovery of organic acids from crud produced in caustic washing of crude oil but not pursued because of technical problems at the time with recovery of the magnetic additive. In the Fischer-Tropsch (F-T) application, however, there is no emulsion and the catalyst particles are magnetic so development of magnetic methods for separation of nanometer (nm) catalyst particles from F-T wax is expected to be straightforward.

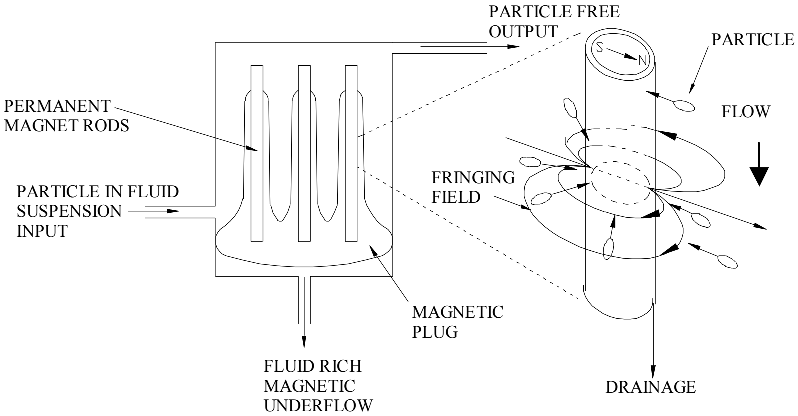

Fig. 1. Magnetostatic Coalescer

The significance of the magnetic technology lies in the fact that the force on magnetic particles, $m\sigma\nabla H$ can be strong, where m is the particle mass, σ is the magnetic moment per unit mass, and ∇H is the gradient of the magnetic field at the location of the particle. For example, the force of attraction of a 10 μm diameter magnetic particle located on the surface of an alnico permanent magnet rod is about 100 times that of gravity, i.e., $\sigma\nabla H/g \approx 100$, where g is the acceleration due to gravity. In the example, $\sigma \approx 50$ emu/g, the alnico magnet is 1/8 inch (3.2 millimeters) in diameter and is magnetized transverse to its length so that $\nabla H \approx 300$ gauss/cm. In the case of mutual magnetic attraction of submicron size magnetized particles, the field gradient at the surfaces of the particles can be orders of magnitude greater than 300 gauss/cm, resulting in rapid coalescence and producing stable agglomerates.

The MM-PS is significantly different from the coalescence technology in that no rods or wires are present inside the separation chamber. The new approach takes advantage of rapid coalescence of the catalyst particles in an applied magnetic field and overcomes problems associated with plugging that can occur when strongly magnetic materials are present inside the separation chamber. Information has been presented elsewhere on characterization of the F-T catalyst/wax slurry and results of separation with the novel technology [2]. The discussion below presents information on scaling of the technology.

3. Apparatus and Fischer-Tropsch Catalyst Particle Slurries

The Magnetic Micro-Particle Separator (MM-PS) used in this work is shown in Fig. 2 as it was being assembled. The structures on the skid-mount are the electromagnet, power supply and chiller. The three tanks to the right in the photo are the feed, product, and underflow vessels.

Fig. 2. Magnetic Micro-Particle Separator Being Assembled

The apparatus, when completed, was tested in separation of nm iron catalyst particles from F-T wax at 260 ^{o}C at rates up to 59 BPD. The specific filtration rate for achieving 0.3-.5 wt.% wax from 20-25 wt.% feed slurry is greater than 230 kg/min/m^2. The upper limit is not known and the process was not optimized because of physical limitations in the apparatus and slurry supply. The process instrumentation and control diagram is shown in Fig. 3.

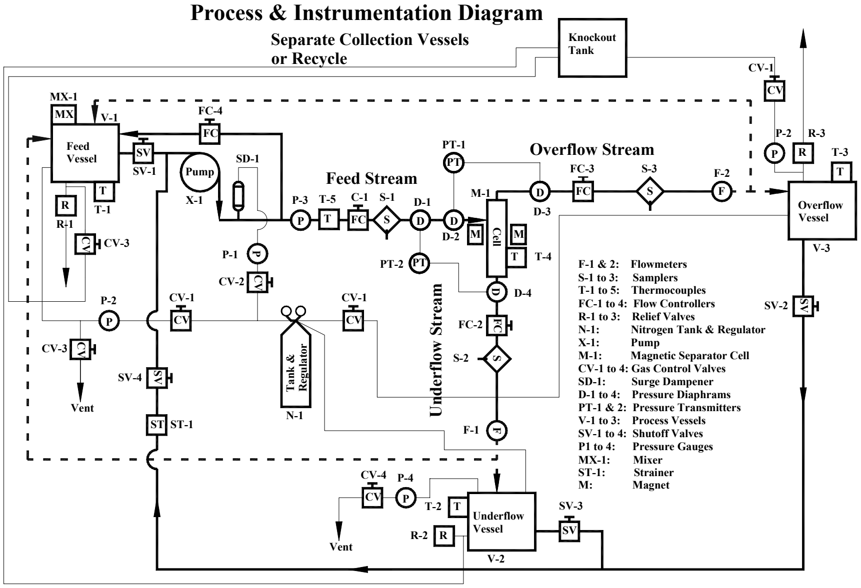

Fig. 3. Process and Instrumentation Diagram

The separation vessels employed in the work are shown in Fig. 4. The vessel lengths are 9, 12 and 16 inches. The volume was scaled up by more than a factor of 52 in the course of the work.

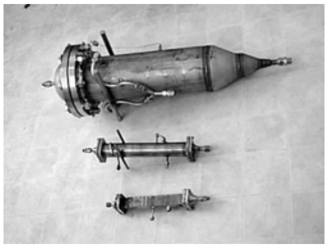

Fig. 4. MM-PS Separators

Catalyst particle images made using computer controlled transmission electron microscopy (TEM) showed that the catalyst particles are actually loose aggregates of very small particles ranging from a few nm to 60 nm in size. From the TEM images, it can be seen that many of the particles were agglomerated or chained together. Fig. 5 shows nm sized particles agglomerated in the MM-PS overflow. The sizes of individual particles range from 20 to 50 nm. Fig. 6 shows larger agglomerates of these particles observed in the MM-PS underflow.

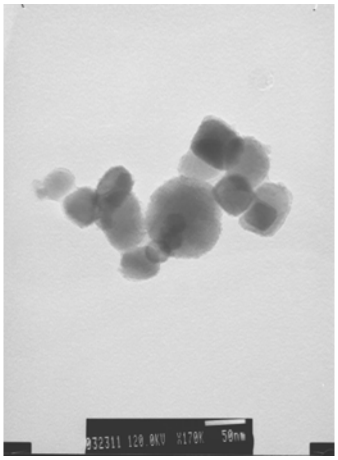

Fig. 5. Overflow Agglomerated Particles

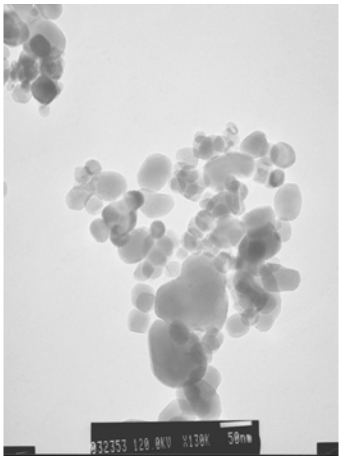

Fig. 6. Underflow Agglomerated Particles

4. Results

Canister Size and Field Effects. The general effects of canister size and magnetic field strength can be seen in Fig. 7, which has been compiled using the results of runs under many different operating conditions. At low overflow rates, generally below 70 kg/min/m^2, changes in the operating variables, i.e., magnetic field strength, canister size, inlet and outlet port dimensions and configurations, inlet flow rate, and recycle ratio (the rate of underflow divided by the rate of overflow), etc., have little effect on overflow ash until an upper level in overflow rate is achieved where further increases in the flow rate make a precipitous increase in overflow ash as can be seen in the figure. The largest overflow rate achieved for the six-inch cell at 2,000 gauss for which the overflow ash was less than 0.5 wt.% was nominally 230 kg/min/m^2. This limit was imposed by the physical limitations in apparatus and supply of catalyst impregnated wax and not by the configuration employed with the six-inch diameter canister.

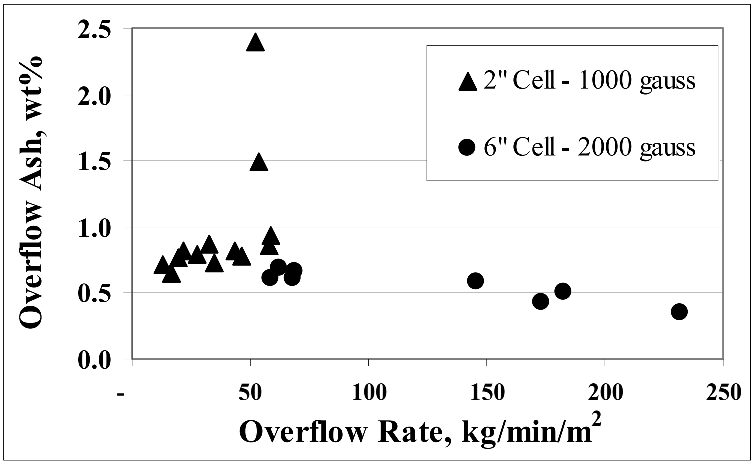

Fig. 7. Overflow Ash vs. Overflow Rate for Different Canister Diameters

*Scale-Up***.** The maximum values of throughput achieved in the work reported here are shown versus canister diameter in the first two rows of Table 1. The range of values shown in the third row are projections based on scaling to 15 gpm product with underflow ten times product. The feed is 165 gpm.

Table 1. Scale-up to 15 gpm

	Ash			Product Rate		
ID inches	**Feed wt%**	**Product wt%**	**Reduction %**	**gpm**	**gpm/Ft^2**	**kg/min/m^2**
1.86	18.48	0.86	95	0.04	2.1	56
6.07	21.45	0.35	98	1.43	7.1	221
13 - 20				15	7 - 16	221-517

Projection of these data yields a filtration rate of 517 kg/min/m^2. Such a projection is probably an understatement, however, because a maximum rate was not achieved for the six-inch canister. Accordingly, scale up uses the measured throughput of 221 $kg/min/m^2$ which yields **very** conservative estimates. The range of 13-20 inches for the canister diameter for processing 15-gpm slurry product is obtained using the projected value and the measured rate of 221 $kg/min/m^2$, respectively.

The measured value of 221 for the specific filtration rate, $kg/min/m^2$, shown in Table 1, is 17 times greater than the Stokes settling rate for particles of density 1.75 g/cc in a fluid of 0.78 g/cc density similar to the operating conditions for the F-T separation, nominally 20 wt.% solids. This rate is estimated to be more than 400 times greater than Davis' estimate for the Sasol commercial unit and 95 times greater than that reported by Davis for one run with the University of Kentucky CAER one liter Fischer-Tropsch unit [3]. Table 2 shows an estimate of the size of MM-PS capable of preparing a low solids 15 gpm product. The estimate is based on using the conservative value of filtration, 221 $kg/min/m^2$. It is interesting to note that the cross-sectional area of the MM-PS could be increased by a factor of 10 and still occupy less than one percent of the volume of the Sasol reactor [3].

Table 2. Separator Footprint

Sasol Slurry Reactor	
Slurry Reactor Volume	288 m^3 (Liquid Fill)
Wax Production Rate	12,960 kg/hr
Magnetic Filter	
Filtration Rate @ 99.6% Catalyst Return	221 $kg/min/m^2$
Filter Cross-Sectional Area	0.98 m^2
Filter Volume	0.2 m^3
Percent of Slurry Reactor Volume	0.08%

5. Conclusions

Magnetic filtration shows potential for primary separation of nanometer iron catalyst from F-T wax and has achieved very high throughputs when compare to more conventional methods.

6. Acknowledgements

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