# MAGNETIC SEPARATION OF NM IRON CATALYST FROM FISCHER-TROPSCH WAX

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**Abstract**

This paper illustrates the potential for separation of nm magnetic particles from viscous flows using the Magnetic Micro-Particle Separator (MM-PS, patent pending). The MM-PS was designed for high throughput isothermal and isobaric separation of nm iron catalyst particles from Fischer-Tropsch wax at 260 $^{o}$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 75:1 are presented.

**Background**

The novel MM-PS separation technology 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 The internal phase was coalesced in a magnetic field and drawn to collecting magnetic rods or wires where it was withdrawn from the separator under force of fluid flow.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 Figure 1 where the magnetic elements are permanent magnet rods. For the case of water in oil emulsions, iron lignosulfonate 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 nm catalyst particles from F-T wax is expected to be straightforward.

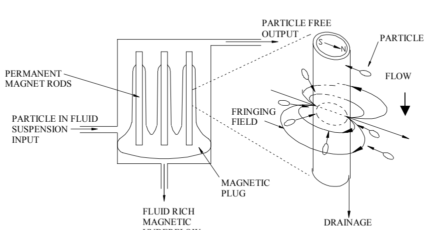


**Figure 1.** Magnetostatic coalescer

The significance of the magnetic technology lies in the fact that magnetic forces are strong, i.e., $\sigma \nabla H/g \approx 100$,where $\sigma \approx 50$ emu/g is the magnetic moment per gram of catalyst agglomerates of nominal 10 μm diameter, $\nabla H$ is the gradient of the magnetic field, which is of the order of 300 gauss/cm for a 1/8 inch diameter alnico permanent magnet rod magnetized transverse to its length, and g is the acceleration due to gravity. The field gradient can be orders of magnitude higher near submicron size magnetized particles resulting in rapid coalescence 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.

**Apparatus**

The Magnetic Micro-Particle Separator (MM-PS) used in this work is shown in Figure 2 as it was being assembled. The structures on the orange 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. The apparatus, when completed, was tested in separation of nm iron catalyst particles from F-T wax at 260 $^{o}$C at the rate of 59 BPD. The specific filtration to achieve producing 0.1-.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 Figure 3.

**Figure 2**. Magnetic Micro-Particle Separator being assembled

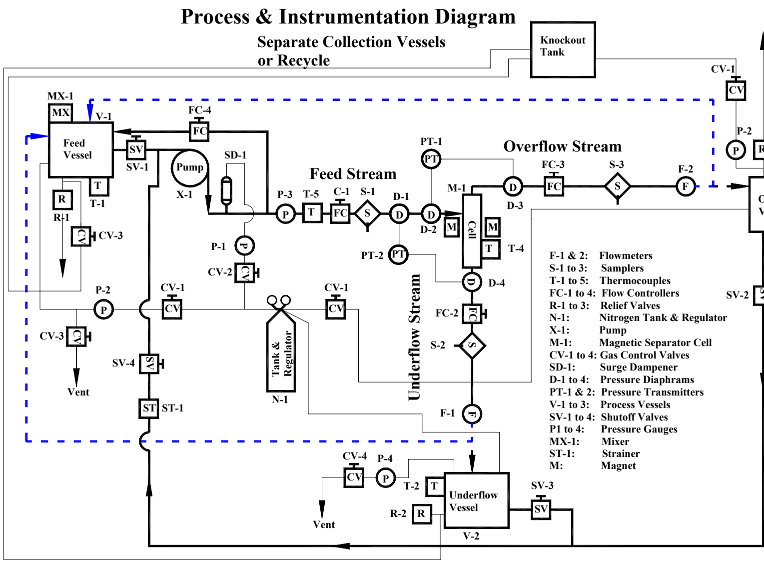


**Figure 3**. Process & instrumentation diagram

The separation vessels employed in the work are shown in Figure 4. The active separation lengths are nominally the same for all three vessels. The cross-section was scaled up by more than a factor of 75 in the course of the work.

**Figure 4**. MM-PS separators

## Results

**Canister Size and Field Effects**. The general effects of canister size and magnetic field strength can be seen in Figure 5 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 0.5 to 1.0 wt%, 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 makes a precipitous increase in overflow ash as can be seen in the figure. The maximum overflow rate ultimately achieved for which the overflow ash was less than 0.5 wt.% was nominally 1.4 gpm or 230 kg/min/m$^2$.

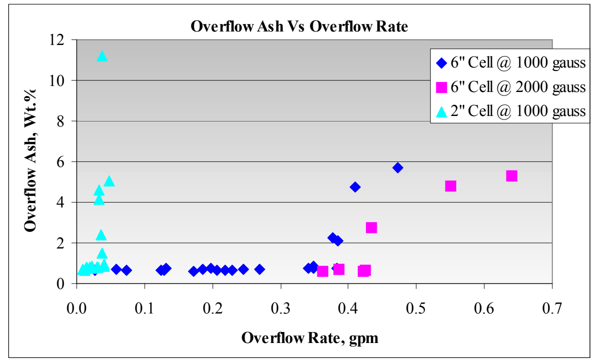


**Figure 5**. Overflow ash vs. overflow rate for different canister diameters and levels of applied magnetic field.

**Scale-Up.** The maximum values of throughput achieved in the work reported here are shown versus canister diameter in Table 1.

Table 1. Measured MM-PS Canister Size and Throughput

| **Canister ID (inches)** | **Test Results** | | |
|---|---|---|---|
| | **gpm** | **gpm/Ft$^2$** | **kg/min/m$^2$** |
| 0 | 0 | 0 | 0 |
| 2 | 0.04 | 2.1 | 67 |
| 4 | 1.43 | 7.13 | 227 |

Projection of these data yields very high filtration rates. Such a projection is not justified, however, because a maximum rate was not achieved for the 6 inch canister. Accordingly, scale up uses the measured throughput of 227 kg/min/m$^2$ which yields very conservative estimates.

The measured value of 227 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]

## Conclusions

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

## References

[1] U.S. Patent 5,868,939 (February 9, 1999).
[2] R. R. Oder, "Magnetic Separation of Iron Catalysts from Fischer-Tropsch Wax," Prepr. Pap. – Am. Chem. Soc., Div. Pet. Chem. 2004.
[3] B. H. Davis and E. Iglesia, Technology Development for Iron and Cobalt Fischer-Tropsch Catalysts, *Quarterly Report, April 1 – June 30, 1999, US DOE Contract DE-FC26-98FT40308*, p. 28.