IX.CENTRIFUGATION

While the long settling time for catalyst/wax separation needs to be shortened for commercial operation, centrifugation (using hydroclones or centrifuges) may be another choice.

1. Hydroclones

Mobil workers [1] adopted a hydroclone system in combination with filters for removal of the catalyst in their conceptual process design. The wax produced in the slurry F-T reactors is removed continuously using level controllers and pumped to the hydroclone system for removing the bulk of the catalyst fines. A minimum of 99.9% of the catalyst in the slurry is required to be recovered and recycled back to the reactor. The clear wax overhead from the hydroclone is sent to the wax cleanup section, where the wax containing <0.1 wt% catalyst is filtered through a vertical precoated leaf filter to remove traces of catalyst. To minimize the amount of equipment as well as valves and piping, the slurry F-T reactors are grouped in clusters of five that share a single hydroclone system, which consists of one slurry pump per reactor plus an enclosed system of a number of (approximately 300, 10 mm ID cyclonetts) parallel manifolded hydroclone separators (from Dorr-Oliver, Inc., Stamford, CT).

Hydroclones offer a simple and inexpensive means for solid/liquid separation. They have been employed in the petroleum refining industry in a somewhat similar situation in which cracking catalyst fines are separated from the fractionator bottom slurries. Unlike other types of solids-removal systems, hydroclones can be demonstrated without building a large size unit because commercial hydroclones are supplied in banks of parallel units. Since the same size units as employed in the pilot plant are to be used in a commercial plant, there is no problem associated with scaleup.

It is believed that hydroclones do not produce an overflow with very low solids content. Hydroclones may be used to recover the bulk of the catalyst in the slurry stream, and the overflow needs to be further clarified through other means, such as filtration, to meet the solids content requirement for wax processing. To cost-effectively produce solids-free waxes and completely recover catalyst particles, a combination of two techniques (i.e., hydroclones plus filters) appears to be necessary. However, the hydroclone system has not been demonstrated for F-T slurry separation, although improved performance over the gravity settler is expected. An investigation of the hydroclone efficiency, reliability, and economics for slurry F-T catalyst/wax separation is necessary.

2. Centrifuges

The conversion of the synthesis gas decreases gradually during operation due to deactivation of the catalyst. The acceptable level of activity is assumed to last for a period of approximately 60 to 70 days (equivalent to 815-950 gHC/gFe production,

although the highest production established by Mobil was 350 gHC/gFe before catalyst settling), and, at the end of this period, the catalyst in the reactor needs to be replaced. In Mobil's conceptual process design, the hot slurry from the slurry reactor is transferred to a centrifuge in order to remove most of the catalyst in suspension. The clean wax is then transferred to a surge vessel for later filtration in preparation for upgrading in the same way as the hydroclone overflow is treated.

In the German semi-industrial demonstration plant, when the reactor was emptied, a pump conveyed the catalyst suspension into a centrifuge where the catalyst was freed from the liquid medium. The reactor wax can be used again, and the deactivated catalyst can be regenerated [12].

It seems to be a common practice that centrifuges are used for spent catalyst recovery, while hydroclones are considered for continuous operation of liquid product separation. In both cases, the clarified wax is further filtered to remove traces of solid particles.

X. SOLVENT-ASSISTED CATALYST/WAX SEPARATION

With respect to physical and mechanical separation methods such as sedimentation, filtration, and centrifugation, the problems in common are the limited throughput and the separation efficiency. Reducing the liquid density and especially viscosity, and increasing the solid particle size are two ways to substantially accelerate the separation and improve the efficiency.

In case of gravity sedimentation, assuming spherical particles and Stoke's law region in absence of wall and concentration effects, the terminal settling velocity is directly proportional to the density difference between particles and liquid and the square of the particle diameter. It is also inversely proportional to the liquid viscosity. When the density difference is fixed, changing the particle diameter and the liquid viscosity are two major means to accelerate the settling velocity.

When solid particles are settling through a liquid in a centrifugal force field, the settling velocity is very nearly given by the Stoke's-law relation. The above analysis applies in the same way. One way of reducing the liquid viscosity is using a solvent. Agglomerants or coagulants can increase the particle size, and therefore the settling velocity. So far we are not aware of any tests having been performed in this area, such as:

A. Settling with dilution of the reactor slurry by light liquid fractions such as F-T naphtha or hydrocracked light naphtha from F-T waxes.

B. Employing suitable agglomerants or coagulants that can properly enhance sedimentation in a catalyst/wax separation step but would not create settling problems after the catalyst is recycled back into the reactor.

This also signals that the production of heavier and heavier waxes is disadvantageous to catalyst/wax separation. In commercial production, recycling <320°C fraction distilled from reactor wax, as practiced in the Rheinpreussen Demonstration Plant_(12), rather than recycling reactor wax itself may be preferrable. This will still keep the gas yield low, while the average molecular weight and consequently the viscosity of the reactor wax will be lower. This reactor wax of lower-molecular weight and viscosity, when recycled back to the reactor, may also reduce the congelling point of the wax slurry, and as a consequence lengthen the reaction on-stream time.

It is therefore suggested accordingly that these studies be conducted.

XI. MAGNETIC SEPARATION

Magnetic interactions can achieve separations that are difficult, impractical, or even impossible to achieve by other techniques. In general, magnetic techniques are restricted to separations of particles from suspension. Applications of magnetic separation in chemistry were reviewed by Hirschbein et al [31]. The major commercial applications at present are the impurity removal from kaolin and the waste water treatment for steel mill processing.

Molecules and materials may be divided into two groups: those with unpaired electrons and those whose electrons are spin-paired. Members of the first group are attracted to regions of high magnetic field (magnetic susceptibility positive), and members of the second group are repelled from high field regions (negative). The magnetization M is a measure of the alignment of the individual unpaired electrons of the material, which is placed in an applied magnetic field.

Materials are also classified into four groups according to the strength of their interactions with applied magnetic fields:

Ferromagnets. In these materials (e.g., metallic iron, nickel, and cobalt) each molecule has several unpaired electronic spins. These molecules have large magnetizations and hence very high magnetic susceptibilities.

Ferrimagnets. In these materials (e.g., magnetite FeO.Fe₂O₃, CrO₃) many of the atoms have strongly interacting unpaired spins. These materials are still attracted very strongly by magnetic fields.

Paramagnets. In materials such as NiSO₄, many other transition metal complexes, NO, O₂, and organic free radicals, the electronic spins of each molecule interact weakly or not at all with the spins of other molecules. These materials thus possess a low magnetic susceptibility. Paramagnetic particles are attracted to a magnetic field much more weakly than ferro- or ferrimagnetic particles, but still strongly enough to be-used in magnetic separations.

Diamagnets. These materials (such as SiO₂, NaCl, graphite, alumina, and most organic compounds) have no unpaired spins and are weakly repelled by magnetic fields.

The critical parameters in magnetic separation are the magnetic susceptibilities of the materials to be separated, the magnetic field strength in the regions where partitioning occurs, the rate of change of the strength of the magnetic field between these regions, and the volume of the particles. The magnetic field gradient becomes increasingly important as the volume of the particles to be separated decreases. The main point in magnetic separation is to find practical methods for generating high magnetic field gradients.

Two types of magnetic separation techniques have been suggested: magnetic filtration

and flotation. In magnetic filtration, also known as magnetic collection, particles suspended in a medium are attracted to regions of high magnetic field associated with a magnetic collector. In magnetic flotation, particles suspended in a para- or ferrimagnetic medium float above regions of high magnetic field. The simplest design of a high-gradient magnetic filter consists of magnetic stainless steel wool packed in a tube, which is placed between the poles of a magnet. There may be more complex designs of magnetic filters. Instead of irregularly packed steel wool, regular arrays of steel pins or rods may be used. The axis of the magnet may lie along the tube rather than perpendicular to it. There may be provisions for moving the filter bed out of the high field region or for turning off or reversing the magnetic field to release the material trapped on the filter [31].

Regarding the working mechanism of a magnetic filter [32-34], the external magnetic field interacts strongly with the electrons in the ferromagnetic filament (steel wool or pins, etc.) and causes their magnetic moments to line up along the field axis. The filament of concern essentially becomes a small magnet and generates its own local magnetic field. The external magnetic field plus the local field produce the total magnetic field around the filament (Figure The important feature is that the total magnetic field is nonuniform close to the filament, which is more intense at the sides and less intense at the top and bottom than the field far from the filament, resulting in a magnetic field gradient around the filament. For particles with high magnetic susceptibilities, the magnetic force greatly outweighs the competing hydrodynamic drag and gravitation. These particles are then attracted strongly by and trapped in regions of high field gradient. The size of the region containing the gradient is of the same magnitude as the filament that generates it. The diameter of the matrix filaments is thus determined by the diameter of the particles to be collected. High gradient magnetic separation is applicable to small (<1 micron) and soft particles, and also to liquid droplets. For highly magnetic small particles, it is the most rapid filtration method known. Because the filter often has 95% void volume, the pressure drop across it can be small. The limitations are as follows: the magnet structure is expensive and filter capacity is relatively low [31].

High Gradient Magnetic Separation (HGMS) was first used as a large-scale industrial application for the removal of colored impurities from kaolin clay in 1969 [35-37]. Cyclic HGMS operating in alternate feeding and flushing modes with filter diameters up to 2 m and capacities up to 20 tons/hr are available commercially [31].

The F-T catalysts are ferromagnetic in nature, and magnetic separation can be used to separate the catalyst particles from the wax. Uses of magnetic separation were suggested for the removal of used catalyst-derived impurities from products such as F-T catalysts [31]. Simple methods of generating very high magnetic field gradients have been developed. One such simple design comprises magnetic stainless steel wool pushed into a tube and the tube placed between the poles of a magnet. Magnetic particles, such as in an aqueous suspension, would be retained by the steel wool and the water carrier would pass through unhindered. The high magnetic gradient is created by the intersection of the external magnetic field of the magnet and the local magnetic field produced by the magnetic filter element, e.g., steel wool,

steel rods, filaments, etc.

Conventional HGMSs use an iron-clad solenoid with water-cooled copper conductors to generate the magnetic field. The toroidal solenoid coil surrounds a canister packed with 400 series stainless steel wool. A typical canister may have a diameter of 84 in. and a length of 20 in. with a packing porosity of 93-94%, so that the frictional pressure drop is negligible. A typical flow rate might be 300-500 gal/min (e.g., 28 wt% clay-water slurry to remove trace amounts of paramagnetic impurity particles finer than 2 microns) depending on the desired product quality, with a retention time ranging from 50-90 sec.

The HGMS technique for separating F-T slurry wax/catalyst is described in a South Africa patent [38], as shown in Figure 12. A slurry bubble-column F-T reactor 10 contains a column of iron catalyst dispersed within a hydrocarbon carrier to form a slurry. The syngas is directed to reactor 10 via line 14 and is discharged into the reactor at the bottom thereof. Gaseous and lower molecular weight hydrocarbon liquids formed are withdrawn from the top and high molecular weight wax product is withdrawn from slurry 12 via line 18, which entrains a substantial amount of catalyst fines. The removal of the catalyst fines from the wax product is achieved by a HGMS scheme. The figure illustrates the use of two parallel high gradient magnetic separators, but other separation schemes such as a single separator or a plurality of separators in series will also produce the desired results. Each individual magnetic separator comprises a means 24, which can be a hollow tube in its simplest construction, holding a filter element 26. The latter can comprise steel wool, thin filaments, or thick filaments such as bars or the like, which are capable of being magnetized in the presence of a magnetic field. The high magnetic field gradients are generated by the thin filaments or by small features such as ridges or other surface irregularities on the larger elements. Tube 24 is positioned such that filter element 26 is positioned and magnetized by the magnetic field formed between the opposite poles of magnet 28 upon the application of an electrical current to the latter.

Wax is divided and passed to the respective high gradient magnetic separators 20 and 22, and is generally heated for flow through tube 26. As the wax flows through tube 24 and passes filter element 26, the entrained catalyst fines will be drawn to magnetized filter element 26 and trapped or held thereon while the purified wax flows out through confining means 24. When it is desirable to recycle the separated catalyst particles to reactor 10, the electric current is switched off and the purified wax is flushed back through filter element 26. The mixture of wax and catalyst is returned to slurry reactor 10 via recycle lines 36 and 38. Preferrably, a solvent is added to the purified wax via line 40, which can be a hydrocracked product from the purified wax. The solvent and wax sent back to the slurry will reduce the congelling point of the wax slurry and thereby lengthen the reaction on stream time. The purified wax, before being upgraded, may be passed in contact with an acidic solid such as alumina or an aluminosilicate zeolite, whereupon soluble potassium compounds in the wax are ion-exchanged and basic nitrogen compounds, if present, are adsorbed. This subsequently purified wax product can then be directed to further processing. Table 8 [38] carries examples that illustrate the ability of HGMS to remove iron catalyst fines from the wax product obtained in a F-T slurry reactor.

In a laboratory scale study, Mobil tested a High Gradient Magnetic Separator (HGMS) for cleaning F-T slurry reactor wax. The solids content was reduced from 0.13 wt% to less than 0.015 wt%. No attemp was made to investigate the effect of various parameters or to optimize the process [39].

The Mobil wax with 133 ppm of iron, obtained by magnetic separation of the catalyst particles from reactor wax, was subject to hydrocracking at UOP. Although no measurable catalyst deactivation was observed over the run period, iron accumulation in the reactor section was evidenced and at least 50% of the iron in the feed processes remained in the hydrocracking reactor. The quartz preheater section was stuck together in solid chunks. No iron was found in the vacuum fractionator overhead product, but small amounts were observed in the fractionator bottoms [4].

The magnetic separation technique is employed at Sasol for its slurry F-T reactor system as one of its catalyst/wax separation options and also as a polishing technology [2].

The HGMS happens to be the first entry for the superconducting technology into more conventional commercial applications, while, until recently, superconducting magnetic systems have been limited to specified areas such as particle accelerators, on Tokomak magnets for fusion research, and in nuclear magnetic imaging medical diagnostic devices. The first superconducting magnetic separator has been in operation for kaolin purification since 1986 in Wrens, Georgia, as shown in Figure 13 [40], and the third one was supposed to be ready for start-up in early 1990. The system ideally required a uniform 2 T (20,000 G) field inside of the solenoid and a zero field outside of the coil. The superconducting material is niobium-titanium in 1,008 turns. It is in the form of a cable that is cryostable and has a copper-to-superconductor ratio of 39:1 [40]. A typical flow cycle might be 26 min on line, 1 min of deenergizing, 2 min of cleaning, and 1 min of reenergizing [41].

The capital costs for conventional and superconducting HGMS systems with a field strength of 2T were reported to be \$1.6 and 2.0 MM, respectively [41]. The superconducting HGMS uses 60 kW compared to 300 kW for a conventional magnet, and the incremental annual savings (at \$0.07/kWh power cost) would be about \$0.13 MM in favor of the superconducting system. A field strength of 2T is the limit for conventional iron-clad electromagnets, while the superconducting HGMS operates more economically and can potentially be designed for 5T or more. This would make large-scale HGMS a promising technique in the oil-refining, ore-processing, and coal-cleaning industries [40,41].

XI. CHEMICAL METHODS

Several attempts were made to convert F-T iron catalyst in wax slurries into iron carbonyl by reacting iron with carbon monoxide. The experiments were done in both shaking and continuous autoclaves at pressures ranging between 165-495 psig and temperatures ranging between 480-570°F. The iron concentrations of the Mobil wax did not decrease in any of the experiments [4].

Very little has so far been done on chemical methods for F-T iron catalysts recovery or separation. Generally, chemical methods are expensive, and waste disposal problems may arise. Unless very cost-efficient chemical methods are found, it is preferrable to engage efforts on the development of less expensive physical and mechanical devices for F-T slurry catalyst/wax separation.

XII. COMMENTS

1. General Comments

A continuous and cost-effective catalyst/wax separation technique is required for the commercialization of slurry bubble column F-T reactors. The high wax viscosity and the fine catalyst particles make this solid/liquid separation a difficult task. Various solid/liquid separation techniques have been tested for F-T catalyst/wax slurries with different degrees of success, they are the following: internal filtration, settling, pressure filtration, centrifugation, vacuum distillation plus thermal cracking, high gradient magnetic separation, and chemical conversion.

Catalyst/wax separation inside the reactor is the simplest way, but internal filters have not been satisfactory and are not suitable for commercial-scale operation. HGMS is promising for internal separation, and further study is needed. Chemical methods of slurry F-T catalyst/wax separation were studied very little, and have not been successful. They are very likely expensive and associated with waste disposal problems.

Gravity sedimentation, centrifugation, pressure filtration, and HGMS are technically feasible, either singly or in combination, but their productivity and efficiency need to be improved and their economic viability must be justified. Solvent application to enhance and accelerate settling and centrifugation is worth investigation. Above all, a basic understanding of the settling characteristics of F-T catalyst particles with respect to the particle size and liquid properties is required.

Specific Comments

- (1) As a requirement of the downstream wax refining process, the solids content of the slurry F-T product wax should be limited to a few ppm, preferably below 2-5 ppm.
- (2) Difficulties in slurry F-T catalyst/wax separation arise from two major facts:
 - a. In high-wax mode operation, the slurry reactor wax has a high viscosity in the range of 4-8 cP (-200°C), and
 - b. In normal synthesis runs, the iron catalyst particle size in the slurry reactor is very small, below ~3 microns and down to submicron size.
- (3) The F-T slurry catalyst/wax separation can be conducted inside or outside the synthesis reactor. Internal filters, such as those immersed in the slurry reaction medium, have not been satisfactory. A filter as a section of the reactor wall, though feasible for bench- or pilot-scale units, is not practicable in commercial-size reactors. In general, internal filters are subject to plugging risks, which may cause unscheduled plant shutdown and are therefore not a reliable means for industrial use. The high gradient

magnetic technique may be feasible for internal catalyst/wax separation, for instance in a recycle line attached to the reactor.

- (4) Vacuum distillation of the reactor slurry and thermal cracking of the vacuum bottoms are by no means catalyst/wax separation techniques. Recovery of solids-free wax by vacuum distillation is impossible due to the high boiling point and cracking tendency of waxes. Thermal cracking of reactor wax vacuum bottoms is not a good way to upgrade waxes either, since the converson is limited by solids deposition and the products are not of good quality.
- (5) Very little work has been done on chemical separation of F-T iron catalyst from reactor wax, and results so far have not been successful. Chemical methods are generally expensive and sometimes create environmental problems.
- (6) Gravity sedimentation or settling is simple and inexpensive but takes a long time (ususally hours) to achieve efficient separation. Hydroclone operation is also inexpensive, but its separation efficiency needs to be improved. Centrifuges are usually employed for deactivated catalyst separation from reactor liquid medium. These methods are technically feasible and solvent dilution of the slurry may assist and speed up the separation, which is worthy of testing.
- (7) High gradient magnetic separation shows promising results on slurry wax/catalyst separation, it may also be used as an internal separation device, such as a filter matrix in a recycle leg attached to the reactor. The matrix is energized by a toroid electric magnet surrounding it. It should be further studied to improve the separation efficiency and technical feasibility.
- (8) It may be reasonable that a combination of more than one, probably two techniques, be adopted to obtain a very clean wax product and complete catalyst recovery. Gravity settlers or hydroclones can be used in the primary stage to recover most of the catalyst fines and a clarified wax, and the latter is subject to the second stage of separation or finishing, by pressure filtration or HGMS, for obtaining a solids-free wax product.

XIV. RESEARCH RECOMMENDATIONS

Based on the above discussions, we suggest that the following techniques be studied for their application to slurry F-T catalyst/wax separation:

- (1) gravity sedimentation,
- (2) pressure filtration,
- (3) hydroclone separation, and
- (4) high gradient magnetic separation.

Basic and engineeering research, as well as economic studies need to be performed. The basic research should include:

- (1) laboratory study on settling characteristics of slurry F-T catalyst/wax system, with parameters such as catalyst density and particle size, wax density and viscosity, the effect of solvent such as F-T naphtha, and the use of appropriate agglomerants or coagulants, and
- (2) laboratory study on HGMS with respect to magnetic field strength, filament matrix material and structure and parameters including slurry flow rate, solids content of the slurry, particle and liquid properties, etc.

The engineering research should include

- (1) bench-scale screening tests on
 - a. gravity settlers with different inserts,
 - b. commercial hydroclones, studying separation efficiency under different conditions,
 - c. pressure filters of different structures, and
 - d. high gradient magnetic separator, conducting cyclic operations with slurries of different solids contents, and identifying its efficiency.
- (2) pilot-scale and PDU engineering tests on
 - a. satisfactory techniques selected from the screening tests would be further studied to improve their cost-efficiency. Prototype designs will be tested in this stage for sophistication.
 - b. Combinations of different techniques would be composed and investigated to achieve required solids content of the purified wax down to below 2 ppm, as recommended in this report. Catalyst recovery of more than 99% should be attempted.
 - c. Incorporation of the technically proven and economically viable catalyst/wax separation equipment into a PDU slurry F-T process operation.

Economic studies need be made on above techniques, singly or in combination.

The investigation may proceed in two phases, embracing research items defined above:

Phase I Study:

- 1. Basic research,
- 2. Bench-scale screening tests, and
- 3. economic study.

Phase II Study

- 1. Pilot-scale tests, and
- 2. PDU runs with the incorporation of pilot-scale demonstrated catalyst/wax facility.

The basic research must precede, or be in parallel with, the bench-scale screening tests which are followed by the pilot-scale engineering research. The economic study should proceed prior to the pilot plant tests. A successful PDU run with incorporated catalyst/wax separation system will conclude the research.

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