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Recovery and Reconstitution of Ferromagnetic Fluids

J. Farkas; B. Hargitay[a]
[a] TECHNICON INSTRUMENT CO., TARRYTOWN, NEW YORK

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Recovery and Reconstitution of Ferromagnetic Fluids

J. FARKAS

8 WALLACE DRIVE
SPRING VALLEY, NEW YORK 10977

B. HARGITAY

TECHNICON INSTRUMENT CO.
TARRYTOWN, NEW YORK 10591

Abstract

A potentially large-scale application of ferromagnetic fluids (FMF) lies in the tunable sink/float separation of materials of different specific gravities. In this application the separated particles emerge from the sorter covered with FMF from which they have to be liberated and the valuable FMF recycled. Encouraged by the reported partial success achieved by the U.S. Bureau of Mines in washing aluminum and copper wires from kerosene-based FMF with water, it was attempted to extend the technique to finer particles such as sand (<0.5 mm). Because the water-wash of such fine particles proved to be quite inefficient, a countercurrent solvent spray-wash was evaluated. The utility of such a process depends on the cost effectiveness of the subsequent recovery of FMF and kerosene from the spent wash solution. Two competing methods, distillation and ultrafiltration, have been analyzed and compared for a pilot size of about 25 L kerosene per hour recovery, in which also a twentyfold concentration of FMF (from 1 to 20 milliTesla) occurs to recover the valuable separation medium. While the investment costs are comparable for the two methods, the operational cost of the UF plant is only a small fraction of the one using distillation.

BACKGROUND

Ferromagnetic fluid, FMF, is a stabilized colloidal suspension of magnetite in a carrier fluid. FMF has properties which are common to fluids and is also attracted by a magnetic field. Because it translates magnetic field gradients into fluid pressure and/or a change in apparent density, its use in densitometric separations such as the sink/float type is evident. This process

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has been discussed earlier (*1*) and a practical method for the manufacture of FMF has also been covered in another paper (*2*). The densitometric separations yield products with entrained FMF. Because FMF is a relatively valuable density medium (about $1/L for 20 milliTesla (mT) fluid: 1 Tesla = 10^4 Gauss), there is an economic and an ecologic incentive to recover it from the dilute wash fluid. This paper explores two competitive methods for the recovery and reconcentration of dilute FMF from densitometric separations.

After it was shown by the U.S. Bureau of Mines (*3*) that washing with water could remove adhering FMF from some particles (5- and 16-gauge aluminum and 12- and 26-gauge copper wire), it was attempted at Union Carbide Corporation's Sterling Forest Research Center to extend the method to mineral particles in the 0.425 to 0.710 mm range. The rationale for exploring this technique is the simple fact that oil-based FMF is not miscible with water and therefore it could be reused in the sink/float process directly after decantation. The simplicity and economy of such a procedure would make a water-wash very attractive. In the following section, the limitations of this technique are presented as it applies to mineral particles and an alternative method using a nonaqueous wash is explored.

RECOVERY BY WASHING WITH WATER

Equipment

The washing equipment used is detailed below. The spray washer in Fig. 1 consists of:

1. Feeder Table. The feeder table used is made by Eriez Magnetics, Model 40A, Style 26. Full load power consumption is 30 W. It has been modified by lifting up the tray by 50 mm so that liquid outlet tubes could be connected. The tray edges have also been modified for acceptance of a hood incorporating the spray nozzles.

2. Screens. The screens consist of commercially-available screens with openings of 2.36 mm (8 mesh), 1.40 mm (14 mesh), 0.85 mm (20 mesh), and 0.425 mm (40 mesh). They are of rectangular pattern and have bending tabs along the sides for a snug fit on the feeder tray edges.

3. Nozzles. The spraying nozzles are Model 3.6 SQ by Spraying Systems, Inc. These nozzles disperse a solid square-based pyramid of mist. This particular model has been found to be very effective with the countercurrent kerosene spray system.

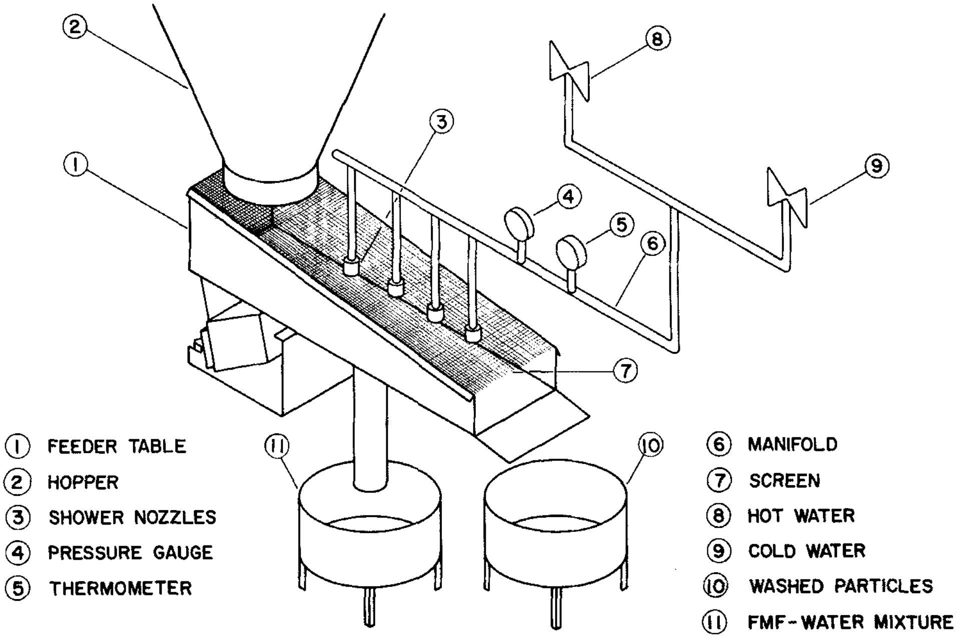

FIG. 1. Spray washer equipment as used with water.

4. *Instrumentation.* The thermometer is a Weston dial-type instrument with a scale range of 0 to 100°C. A "T" manifold fitting allows the stem of the thermometer to be in the flowing stream. Leak tightness is achieved by a compression-type fitting. The pressure gauge is a standard hydraulic gauge reading from 0 to 608 kPa.

5. *Miscellaneous.* The other equipment consists of various pipes, valves, and connecting equipment available off the shelf.

Operation

The materials coming from the sink/float separator, from which the FMF has to be removed, are fed into the hopper. The feed rate of the particles through the spray washer (the residence time) depends on the particle size and shape for the relatively "coarse" particles. The "finer" particle residence times were not determined because washing efficiency is too low to be of importance. The pressure and temperature of the water are adjusted to the

TABLE 1
Water Washing Experiments[a] and Comparison with USBM Data

	No. 1	No. 2	No. 3	USBM
Input				
FMF on sample (mL/kg)	230	230	230	84
Feed rate (kg/h)	30	15	15	41
Water temperature (°C)	25	70	70	Not mentioned
Water pressure (kPa)	101.3	101.3	405.3	172.2–4154.3
Water flow rate (L/h)	340	340	500	180
Output				
Dragout: FMF on sample after wash (mL/kg)	145	180	220	50
Water consumption:				
$\frac{\text{L water used}}{\text{L FMF removed}}$	400	1020	2500	54
FMF removal efficiency (mL FMF removed/mL FMF initially on sample) (%)	37	22	4.4	96.3

[a] Sand of 0.425–0.710 mm (−25 + 40 mesh) was used as the sample.

desired conditions. The spray nozzles require a minimum of 50 kPa to operate efficiently. The highest safe pressure is around 4000 kPa. The flow rate of water is determined separately as a function of temperature and pressure, and the obtained correlation is then used in calculating the water consumption used in Table 1. The feeder table is slightly inclined forward to facilitate material flow and to prevent screen clogging. The cleaned material is collected in a bucket, whereas the FMF-water mixture draining through the screen is allowed to settle and is then decanted. The amount of FMF removed by spray-washing is then determined volumetrically and is used to determine the efficiency of the washing procedure.

Feed Requirements

The particles to be liberated from FMF are discharged from the separator first onto a drainage screen. This minimizes the amount of FMF to be washed off. Porous particles cannot be efficiently washed since the surface tension of FMF is lower than that of water, thus water cannot displace FMF from within the pores and concave surfaces.

The water can be ordinary hot and cold tap water. There is no need to use distilled or deionized water; however, the use of lake or river water is not recommended. Strong acids and organic contaminants should be avoided because they may upset the colloidal nature of FMF.

Performance of Spray-Washer

The performance of the process was measured by comparing the efficiency of removing FMF from the particles with the consumption of water and the energy required to produce the result. For this purpose 2 kg of particulates (e.g., sand) were saturated with FMF of 20 mT strength and left to drain by gravity on a screen prior to feeding into the spray washer. The amount of FMF imbibed by the particulates had to be known precisely prior to the washing operation by substrating the drained FMF from the amount used to saturate the sand.

After passing through the spray-washer, the amount of FMF entrained by the wash-water was measured volumetrically after decantation and the FMF persistently adhering to the particles evaluated by extraction with a small amount of kerosene (300 mL max). The volume of the extract was then measured and its saturation magnetization determined from which the "drag-out" FMF was calculated.

The results comparing the efficiency of the process run with varying parameters (temperature, water pressure or flow rate and particle feed rate) are summarized in Table 1.

It can be seen that contrary to expectations, high water flow rate and elevated temperature are *not* advantageous in liberating sand from adhering FMF. Also, for sand the highest efficiency measured is only 37%, too low indeed for an economical process. This compares unfavorably with the results of USBM obtained on "wire scrap." The comparison of values of the surface per particle (see Table 2 and Fig. 2) indicates that a spray-washing

TABLE 2
Particle Sizes Used for Water Washing Experiments

Material	Size D (mm)	Particle surface area (mm^2)	R[a]
5-gauge Al wire,[b] USBM	4.62	91.6	96
12-gauge Cu wire, USBM	2.05	32.3	34
16-gauge Al wire, USBM	1.29	18.8	20
26-gauge Cu wire, USBM	0.40	5.3	5.5
Sand, UCC	0.55	0.95[c]	1.0

[a] Ratio of particle surface area to that of sand.
[b] Each piece of wire was 4 mm long and of cylindrical shape.
[c] Sand particles are considered as perfect spheres.

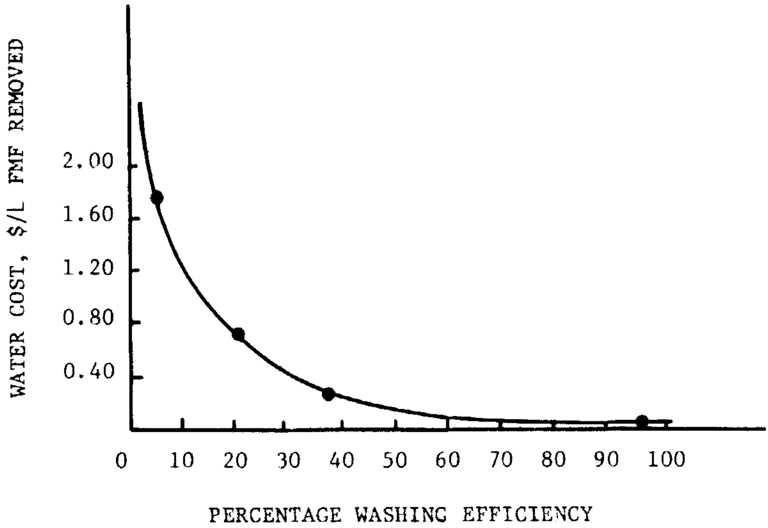

FIG. 2. Water cost as a function of washing efficiency.

process using water does not seem useful for particles smaller than about 0.5 mm.

In addition to the purely economic arguments (Table 3), the following observed drawbacks argue against the use of water-wash for small particles: 1) inadequate phase separation between FMF and water, 2) sludge formation in the three component system (sand/water/kerosene) which is very difficult to handle, and 3) clogging of the screens used by the sludge formed with fine particles.

TABLE 3
Water Costs at the Obtained Efficiencies

Exp. no.	Water consumption: vol water / vol FMF removed	Economy: cost of water $ / L FMF removed	Washing efficiency: vol FMF removed / initial FMF on sample
1	400	0.275	37
2	1020	0.70	22
3	2500	1.72	4.4
USBM	54	0.037	96.3

SOLVENT-WASH TECHNIQUE

A qualitative manual check has proven that kerosene can wash even porous particles free of FMF to any desired degree of cleanliness. The washer of Fig. 1 was modified for solvent wash in such a way that each nozzle was fed by an individual pump with the wash liquid drained off the adjacent shower (Fig. 3). The wash liquid was led in countercurrent to the particles to be washed. The solvent draining from the particles just entering the system contained enough magnetite for recycling to an FMF of 20 mT by a ten- to twentyfold reconcentration. As neither kerosene nor magnetite is lost during the process, the economic analysis of the solvent washing method simplifies to evaluating the energy efficiency of the process separating FMF from kerosene and the cost of the equipment necessary for the purpose.

While the water-wash scheme proved to be *technically* not feasible for small (<0.5 mm) particles, the solvent-wash is technically feasible and its merit depends on its *economic* feasibility. Therefore, two competing separation schemes, ultrafiltration (UF) and distillation, are compared and their respective operating and investment costs determined.

Ultrafiltration

Superparamagnetic fluids are stable colloidal solutions. The ones under consideration consist of iron oxide particles of colloidal dimensions (10 to 20

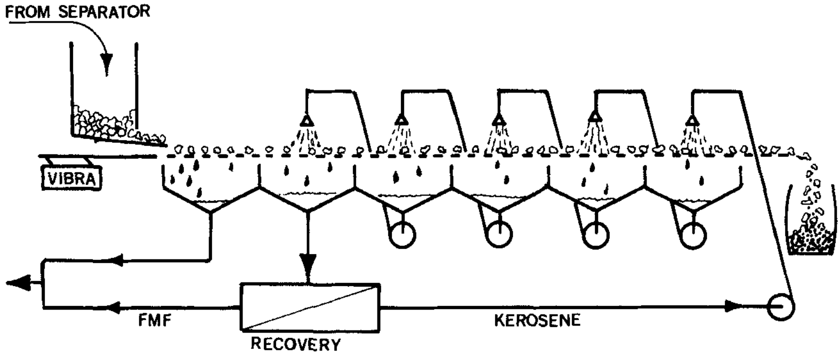

FIG. 3. Liberation of separated particles from entrained FMF by countercurrent solvent wash.

nm) suspended in an organic matrix, i.e., kerosene. The suspension is stabilized by fatty acids chemisorbed onto the oxide surface, thus preventing a direct contact between the inorganic particles. The low viscosity of these solutions as well as electron microscopy is convincing evidence that the particles are almost spherical.

The separation of particles of colloidal dimensions from the continuous phase should be possible by filtration through membranes, the pores of which are smaller than 10 nm. Unlike evaporation or crystallization, ultrafiltration does not involve phase changes and is therefore inherently more energy efficient and no extra engineering efforts are necessary to recover the energy associated with the phase changes.

In order to prove technical feasibility it was necessary to find a membrane of the proper size which can be subjected to the influence of organic solvents and can tolerate elevated temperatures as well. Such a separator was found to be the UCARSEP ultrafiltration system which is now described briefly.

The crucial component of the system is a porous tube (6 mm i.d., 10 mm o.d.) made from essentially pure, only slightly graphitized, carbon. The pore size distribution, as determined by mercury intrusion porosimetry, peaks in the neighborhood of 100 nm and no through-pores are present larger than 500 nm (by bubble point determination). These relatively large pores permit a fast flow of filtrate through the 2-mm thick wall. The strength of the tube is sufficient to withstand routinely well over 3×10^6 Pa (450 psi or 30 atm) internal pressure.

In order to make this tube reject particles of 10 nm, a layer of refractory metal oxides (usually ZrO_2) is deposited on its internal surface by a proprietary process. This very thin, coherent layer constitutes the working filter while the carbon tube lends the necessary rigid but highly permeable mechanical support to the system.

Both the carbon and the refractory metal oxide are chemically inert toward acids, bases, oxidants, and organic solvents and, consequently, can be easily cleaned from fouling deposits, an advantage not matched by other ultrafiltration systems.

During filtration the concentration of the rejected species tends to build up next to the permeation barrier, i.e., the surface of the filter. In order to prevent caking, which would inordinately slow the rate of filtration, turbulence is generated by pumping the fluid at high tangential rate through the tube. Reynolds numbers in excess of 10^4, mostly over 2×10^4, are usually maintained. Even at such high circulation rates, concentration polarization is not completely eliminated and, as will be shown, it contributes importantly to the selectivity of the filtration system.

Method and Laboratory Equipment

In order to establish feasibility of a UF process for recovery of FMF and to determine the most important parameters, a single tube (0.60 m) laboratory UF "loop" was used (as shown in Fig. 4). The precoated carbon tube (CT) was mounted in a 12.7 mm (1/2") o.d. stainless steel shell (S) to receive the permeate. The tangential flow was generated by a magnetically coupled centrifugal pump (M) (Micropump, Model 101-841-00B) capable of delivering up to 0.5 L/s at no head or able to generate 138 kPa (20 psi) at a 0.25-L/s circulation rate. The driving pressure was generated via a pressure vessel (PV, Amicon RS4) by nitrogen from a forepressure regulator (R) on a gas cylinder. In this simplest arrangement, the filtration rate was determined by collecting (at F) permeate for 60 s in a graduated cylinder. The filtration rate in mL/s divided by the internal surface area of the tube (11×10^3 mm^2) gave the specific filtration rate in $mm^3/mm^2 \cdot s$ but is conveniently expressed by units of mm/day, or the linear velocity by which the liquid front of permeate advances through the filter. The rate of circulation was in general expressed in terms of pressure drop along the filter tube (P_1–P_2).

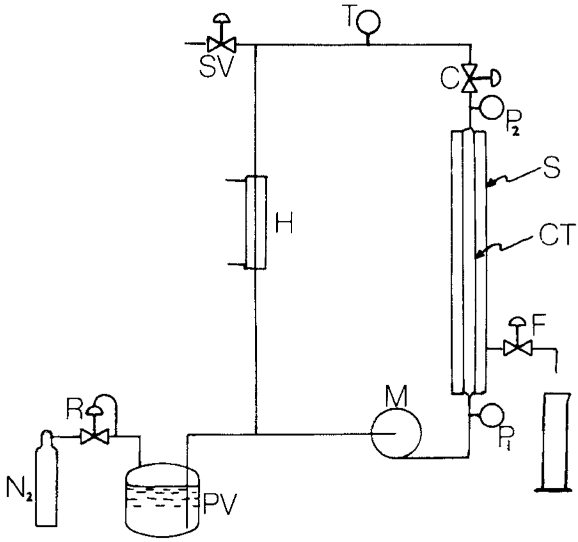

FIG. 4. Layout of the laboratory scale ultrafiltration loop. The components are described in the text.

The concentration (magnetic strength) of the "retentate" (or concentrate) was routinely measured by determining the increase of buoyancy of a tungsten cylinder probe immersed in the FMF when brought into a standard magnetic field. The weight of the probe immersed in FMF (W_F) and its weight (W_M) in FMF between the poles of a strong permanent magnet were measured. A calibration curve of ($W_M - W_F$) vs magnetite concentration was established by dilution of a known FMF standard and was used to characterize the samples withdrawn from the loop.

In order to maintain a desired temperature in the loop, a heat exchanger (H) was used through which water from a thermostat was circulated. Due to the energy dissipated by the pump (M), the temperature tended to rise without cooling.

The course of a typical run follows. The pressure vessel was filled with the dilute FMF solution to be concentrated. While opening the sampling valve (SV) and closing the filtrate valve (F), the dilute solution was forced into the loop by nitrogen pressure. When completely filled, the sampling valve was closed and the centrifugal pump motor (M) was started. The pressure within the loop was brought up to the desired value ($P_1 + P_2$)/2 by manipulating the pressure regulator (R) and the cross-flow was regulated by adjusting the valve (C) to obtain the desired pressure differential $P_1 - P_2$. After the desired temperature was reached, the filtrate valve was opened and the filtration flux rate periodically measured. This flux rate was then correlated with the concentration of the FMF in the loop as determined on samples drawn simultaneously through the sampling valve (SV).

Results

As predicted by the classical theory of UF based on gel polarization (*4*, *5*), the rate of filtration decreases with increasing concentration of the rejected species. Unlike the osmotic effect observed in reverse osmosis separations, the concentration polarization by colloidal particles acts by increasing the hydrodynamic resistance across the boundary layer, thereby reducing the flux.

With the FMF studied, the "gel-polarization" is strictly reversible, indicating that no adsorption, aggregation, or precipitation occurs. The selectivity of rejection is strongly dependent on the pressure used during filtration and increases dramatically with pressure. On the other hand, with increasing pressure the filtration rate increases only at quite low trans-membrane pressures, after which it levels off and even tends to decrease slightly. This goes hand in hand with the increase of rejection, indicating that a more compact gel layer is being created by increasing the pressure.

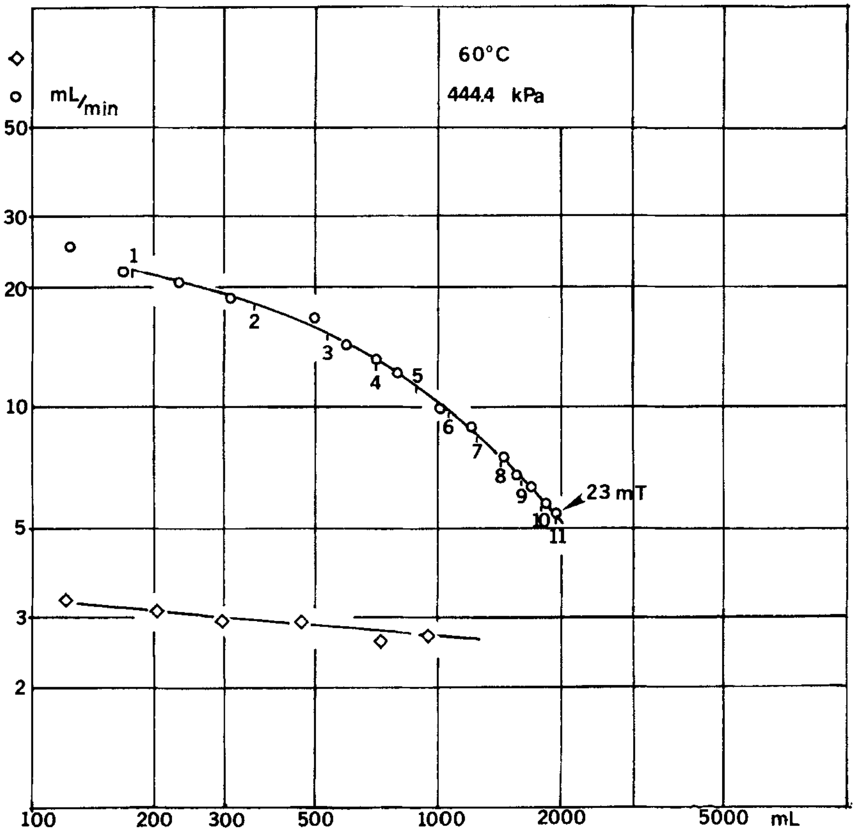

FIG. 5. Filtration rate (flux) as a function of the volume filtered. The numbers along the line indicate where samples were taken. Upper curve (○), coated tube; lower curve (◇), uncoated (bare) carbon tube. Points to the left of the *y*-axis indicate the initial flux.

Figure 5 shows the actual flux observed as a function of volume filtered using the laboratory loop. As long as the FMF is very dilute, the decrease is gradual but drops precipitously in the later stages. In Fig. 6 the same result is plotted against the concentration of FMF in terms of magnetic flux density at saturation in milliTesla (= 10 Gauss) units. Extrapolation past the 23-mT level indicates that it is likely that around 70 mT, the filtration rate would drop to zero. It is interesting to note that this correlates well with what would be expected from geometry considerations taking into account a monolayer of oleic acid covering the magnetite surface. No excess solvent kerosene would be present at about 70 mT, and this conclusion has also been reached

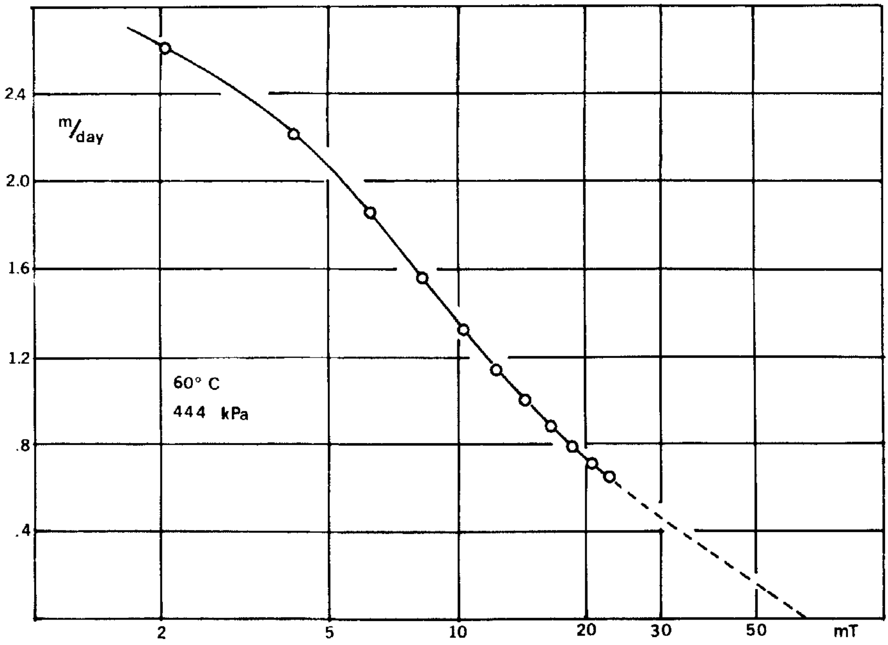

FIG. 6. Flux data from Fig. 5 replotted against the magnetic strength of concentrate samples taken at Points 1 through 11.

by Kaiser (*6*) from the viscosity behavior of similar FMF. It is predictable that using somewhat larger magnetite particles and/or somewhat shorter surfactants would allow the manufacture of lower viscosity FMF for a given magnetic saturation or higher saturation for a given viscosity. In fact, FMF is available today at about 90 mT.

The behavior observed when varying the transmembrane pressure differential fully corresponds to what theory predicts and is shown in Fig. 7. Pure kerosene follows the ideal behavior of flux proportionality to pressure. As the concentration of the colloid increases, deviation from this ideality occurs at lower and lower concentration, indicating the formation of a compressible gel-like structure in the boundary layer enriched by concentration polarization. The severity of this gel polarization is decreased by increasing temperature, as can be expected. Part of the doubling flux rate by raising the temperature from 60 to 93°C is due to the decreased viscosity of kerosene but the magnitude of the pressure where deviation from ideality occurs is indicative of the compacting of the gel layer at lower temperatures.

The color of the permeate is indicative of its purity which can be followed colorimetrically. On opening the filtrate valve the first permeate is very dark and barely distinguishable from the contents of the loop. However, in a matter of minutes the color of the filtrate changes to paler brown and at the same time the rate of flux decreases. The most striking effect observed is the strong dependence of permeate purity on transmembrane pressure. Below about 2×10^3 kPa the permeate remains quite dark even after prolonged filtration. With increasing pressure the permeate becomes paler and above 6×10^3 kPa it is essentially indistinguishable from pure kerosene (see Fig. 8). The color of a 0.1-mT saturation magnetization FMF is clearly straw-colored compared with essentially colorless kerosene. This effect is absolutely reversible to a degree never observed in other UF systems.

If the filtrate valve is closed for a few seconds until the pressure in the shell builds up to that within the loop, the filtration is halted in the absence of a transmembrane pressure differential and the gel layer on the filter surface is swept away by the turbulent flow within the tube. On reopening the filtrate valve, the first permeate is dark again and the flux is high until, within seconds, the new gel layer develops.

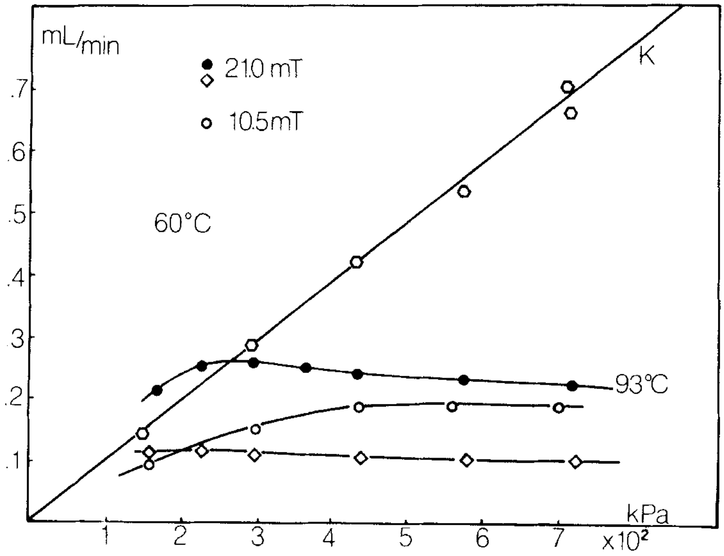

FIG. 7. Dependence of flux on transmembrane pressure. Curve K (⬡) obtained with pure kerosene. Open symbols refer to 60°C.

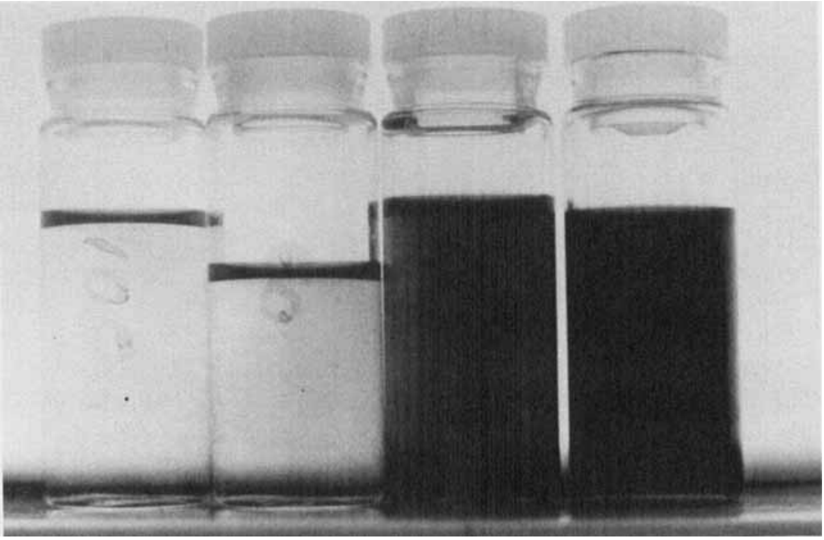

FIG. 8. Photograph taken of permeates obtained at 60°C at transmembrane pressures of 100, 60, 40, and 20 psi (left to right).

Remembering that the pore size of the carbon tube is about 10 times larger than the magnetite particles, it cannot be expected that an uncoated tube would act as a filter. However, it is observed that a "bare" carbon tube can produce as pure kerosene as a coated one but at a rate which is about one-tenth for dilute FMF and which decreases much less with concentration. This behavior is apparent in Fig. 5 where the upper curve is obtained with a coated and the lower with an uncoated carbon tube under identical conditions. It is evident that in the case of a bare tube, the gel layer forms *in the pores* of the tube and is consequently up to 2 mm thick. In contrast, the smaller pore size of the metal oxide coating causes the formation of the gel layer on the inner *surface* of the tube, where its thickness is limited to about 1 μm by the turbulence maintained in the tube.

The fact that under low pressure the filtrate contains magnetite (i.e., poor selectivity) shows that the pore size of the oxide layer used is not sufficiently small to prevent *by itself* magnetite permeation but that it is effective in keeping the gel layer, which becomes the actual filter, limited to the surface.

A plot of log flux against the reciprocal absolute temperature (Fig. 9) shows that the slope (i.e., the free energy of activation for flux) is higher for FMF (at 10.5 mT) than for pure kerosene and that it increases further with concentration. The measured flow is that of the filtrate (i.e., essentially pure kerosene) and not of FMF; therefore, if the matrix through which flow occurs were unchanged, the enthalpies of activation should be the same. The fact that they are not indicates that the resistance (compactness and thickness) of the gel layer increases with concentration.

The purity of the filtrate as a function of temperature is as expected, i.e., highest purity at low temperature. This dependence, however, is minor and at high transmembrane pressure, even at 90°C, a filtrate of 0.1 to 0.2 mT can be produced. The gain in filtration rate by far outweighs the loss in purity. Because the recovered kerosene is destined to be reused as the washing medium, its light straw color is of no consequence.

The UF process can also be used to effect solvent (or matrix) exchange in FMF. When the desired magnetite concentration is reached in the loop, the feed (in the pressure vessel) can be switched to another solvent, e.g., toluene. As filtrate is discharged from the UF system, an equivalent volume of the new solvent is drawn into the loop. By following the refractive index of the filtrate, the solvent exchange can be stopped at any desired composition. In this way toluene- and benzene-base FMF have been obtained which are somewhat less stable than the kerosene-base FMF. In particular, a strong magnetic field can induce irreversible aggregation in them. Prolonged

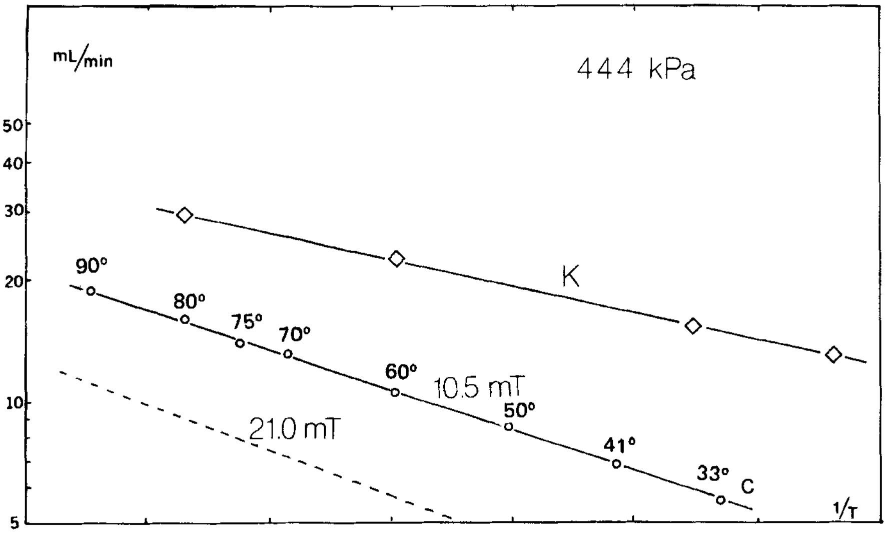

FIG. 9. Arrhenius plot (log flux vs $1/T$) for three FMF concentrations. The zero concentration line coincides with the plot of the reciprocal viscosity of kerosene.

diafiltration of FMF with pure solvents leads to a slow loss of some stabilizing surfactant, presumably molecules that are only adsorbed rather than chemisorbed to the magnetite surface. However, if UF-recovered kerosene is used for the washing process, the leaking surfactant is recycled with the solvent and no net loss of it results.

Scale-Up to Pilot Plant

Based on the experience gathered with the laboratory loop, a pilot-plant unit was constructed. It is capable of automation and recovers from a low grade effluent (~1 mT) reusable FMF (20 mT) and kerosene ($\ll$0.1 mT) for recycling into the washing operation. The salient features of the pilot unit are its capability of performing ultrafiltration 1) at elevated temperature (>60°C), 2) at a transmembrane pressure of 3 to 6×10^3 kPa, 3) at high Reynolds numbers (>10,000), 4) automatically, and 5) economically (*7*, *8*).

Because the productivity of the UF plant increases with temperature, no attempt was made to remove the heat generated by the circulation pump. To the contrary, the loop's conduits were thermally insulated and the heat contained in the purified kerosene leaving the system was recovered in a countercurrent heat exchanger used to preheat the feed stream. This energy recovery also yielded the added benefit of lowering the temperature and the vapor pressure of the product kerosene, thereby significantly reducing air pollution and fire hazard.

The UCARSEP system (Figs. 10 and 11) used in the pilot plant contained a filtration module of 19 carbon tubes in parallel 1.2 m in length. The tubes were held in a shell-and-tube manner in a symmetrical arrangement in two stainless steel headers by Buna N O-rings in countersunk grooves. (A smaller 7-tube module made of ordinary gas-pipe fittings has also been used successfully to prove that no expensive materials of construction are necessary in a nonpolar matrix-based system.)

Circulation in the system was maintained by a magnetically coupled March pump (Model TE-7S-MD) with stainless steel housing. The feed was delivered from a 210-L drum by a positive displacement pump (Fluid Metering RPID-CSC) with ceramic piston and housing. Its capacity of 7.5 mL/s was higher than the expected maximal filtration rate and it could deliver feed against a head in excess of 700 kPa. A variable pressure relief valve (Hoke 6534L4Y) in parallel with the feed pump prevented over-pressurizing the UF loop and allowed choosing the operating system pressure.

Temperature was measured by a Weston (Model 4302; 0-100°C) bimetal

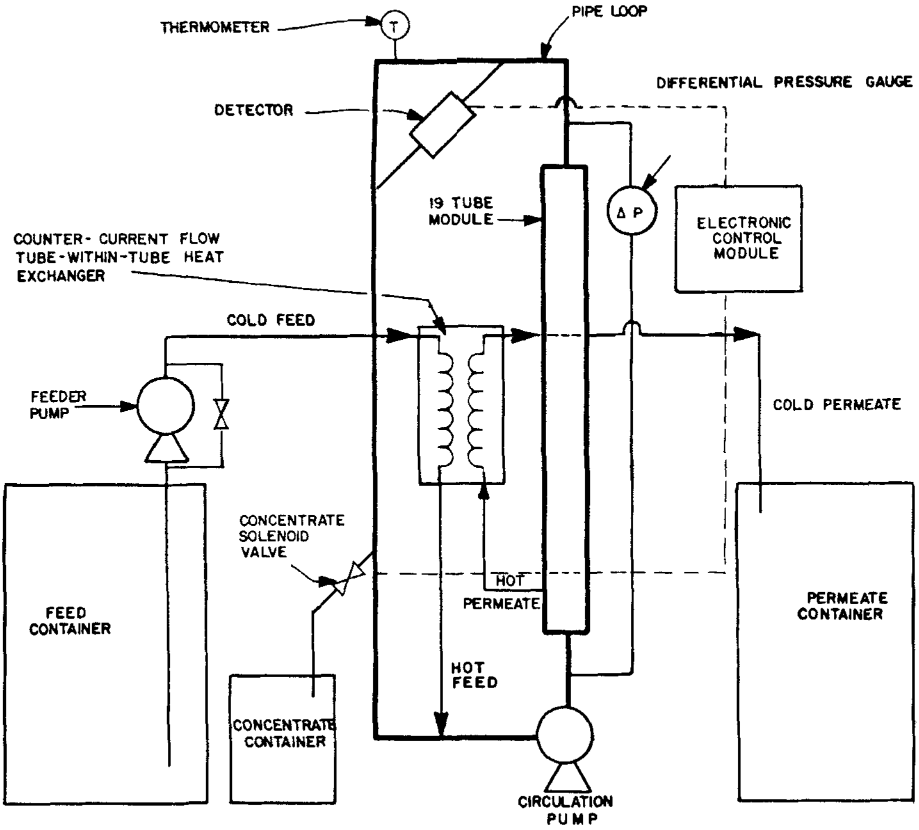

FIG. 10. Layout of the 19-tube UF pilot-plant unit.

thermometer, and the pressures (upstream and downstream of the module) were read on a dual gauge (Weksler GP2-17-3).

The automatic operation was controlled by an instrument which measured the magnetic susceptibility of the loop-content on a slip-stream contained in a Teflon tubing (12.5 mm i.d.) surrounded by two adjacent coils. The primary coil received an ac signal from a Heathkit SG1X71 signal generator while the secondary coil was connected to the amplifier stage of a Simpson 344XA, 0–10 VAC (1000 Ω/V) voltmeter. The two coils around the Teflon tubing acted as a variable transformer, the voltage output of which depended on the magnetic flux generated in its core, i.e., in the FMF contained in the tubing. This instrumentation was calibrated by FMF solutions, the strength of which was determined by the method described in Ref. *9*.

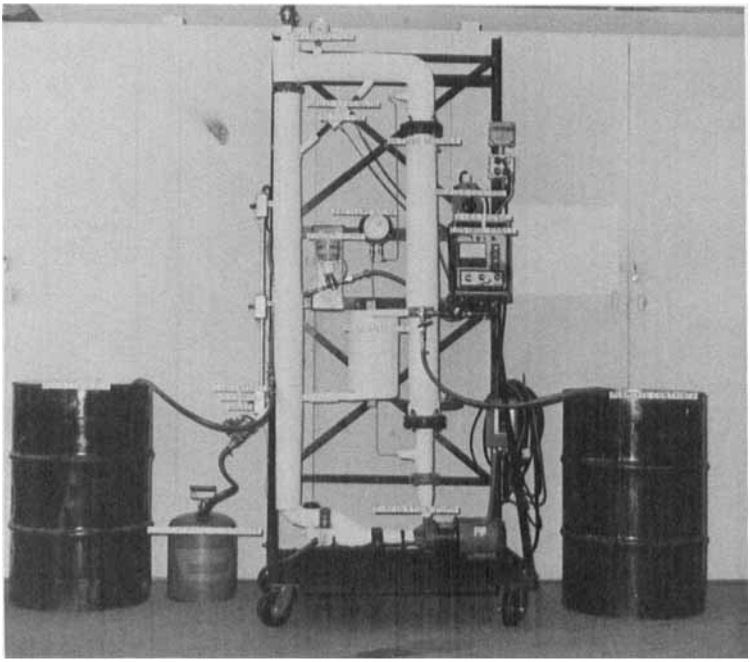

FIG. 11. Photograph of the 19-tube UF pilot-plant unit.

The voltmeter had a noncontact type match-needle relay which was used to activate a timer device controlling the solenoid valve on the concentrate discharge line of the UF loop. The timer (Automatic Timing Controls type 319) had selectable "On" (0 to 3 s) and "Off" (0 to 100 s) times.

The hot filtered product kerosene was evacuated from the system through a tube-within-tube (copper, DHTC-CU-BZ by Parker-Hannifin Corp.) heat exchanger in countercurrent with the cold feed entering the system.

In order to protect the pumps, valves, and the coating of the UCARSEP tubes from abrasion, the intake of the feed line is covered by a fine screen to prevent particulates (e.g., sand) to enter the system.

Operation of the Pilot Plant

With the concentrate valve manually opened, the loop was filled with the dilute FMF (feed). When completely full, the concentrate valve was closed and the pressure built up until it reached the value preset by the pressure relief valve at the intake. As soon as the loop was filled, the circulator pump was started and the produced filtrate was temporarily recycled into the feed reservoir until its color indicated good selectivity (about 15 min). After that point the permeate was collected in the kerosene recovery tank, a 210-L drum (Fig. 11).

The temperature of the system increased spontaneously and with it the filtration rate. For short runs the heating was helped along with a heating tape incorporated into the insultating sleeves around the conduits of the loop. If the controller was set to 20 mT, it took 10 to 20 loop volumes of filtration before the first concentrate emerged.

When the controller opens the concentrate valve for a preset short "On" time (usually 0.5 s), the pressure in the loop instantly drops to essentially atmospheric pressure but the continuous feed reestablishes the operating pressure within about 10 s. After the preset "Off" time (e.g., 40 s), the valve opens again for another short burst and the cycle repeats itself until the loop concentration has dropped below the selected threshold value. Then the concentrate valve stays closed until enough kerosene has left the loop through the filter to restore the desired high (e.g., 20 mT) FMF concentration.

The feed to the UF system is, if the wash is conducted in countercurrent, usually 1 to 2 mT in strength. The reconstitution to the original, useful concentration (i.e., ten-to twentyfold) is well within the economical range of the UF system.

Performance of the Pilot Plant

In order to visualize the performance of the UF system, the operational parameters under steady-state condition are shown in Table 4 for a typical

TABLE 4
Steady State Operational Parameters of 19-Tube Pilot Unit

Total filter surface (19 tubes)	0.43 m^2 (= 4.62 ft^2)
Feed concentration	1 mT (saturation magnetization)
Feed temperature	20°C
Average system pressure	608 kPa (88 psi)
Pressure drop along module	64 kPa
Concentrate valve timing	0.5 s open/40 s closed
Kerosene product rate	22.8 L/h
Permeate flux	53 L/h·m^2 (= 31.2 GFD)
Temperature	65–67°C
Concentrate rate during discharge	43 mL/0.5 s
Concentration of FMF produced	20 mT

run. The first group of variables is preselected or predetermined by the spray-washing unit. The second group mirrors the result of the operation in the 19-tube UCARSEP system.

OPERATIONAL COSTS

In principle, operational costs include the costs of utilities, raw materials, and labor necessary to run the equipment. However, in both the UF and the distillation systems under comparison, there is no raw material used up because both are strictly reconstituting from a dilute stream of FMF a small volume of FMF concentrate and a large volume of solvent, both to be reused in the sink/float and washing processes. Also, because both systems are automated to a large extent, labor is involved only during start-up, shutdown, and maintenance. For continuous operation, these are negligible and therefore the utility costs are the deciding factors in comparing the economy of the two systems.

UF System

Electrical energy is the only utility used in the UF system. Measurement of the energy consumed under steady-state conditions (see Table 5) yielded 0.606 kWh/22.8 L permeate or 26.6 Wh/L. The "by-product" of some 50 mL FMF does not consume extra energy. Using the cost of electric power prevailing in 1976 (*10*), the cost of 19 L of kerosene plus 1 L of (20 mT) FMF recovery from (1 mT) wash fluid amounts to 19 × 26.6 Wh × 4.19¢/kWh = $0.0212.

TABLE 5
Energy Consumptions for a 19-Tube UCARSEP Unit[a]

Device	Power consumption (W)
Circulation pump	559
Feed pump	30
Solenoid valve (while open)	17
Total power	606

[a] This calculation does not count an initial 30 min warm-up period with the heaters.

Distillation

It was shown, using ordinary laboratory-scale glass equipment, that distillation at atmospheric pressure can recover the desired 20 mT FMF from 1 mT wash fluid without difficulty. Rather than building a pilot plant for evaporative recovery, a paper evaluation was made based on data given by a supplier (*11*) of such equipment. A 28-L kerosene/h still was considered. According to the manufacturer, 238 Wh are required for the distillation for 1 L of kerosene. Therefore, for the standard production of 19 L kerosene plus 1 L of (20 mT) FMF, the energy requirement is

$$19 \times 238 \text{ Wh} = 4.552 \text{ kWh or } \$0.1895$$

No cost is attributed to the cooling necessary for the condensation of the kerosene vapor because it occurs in a heat exchanger used to preheat the feed to the still.

CONCLUSIONS AND RECOMMENDATIONS

Based on the experience presented, it can be stated that for particles smaller than 0.5 mm, washing with water is technically not feasible; however, using the base liquid of the FMF for washing in countercurrent yields clean particles and a dilute FMF that can be reconstituted. For very small particles in the micron range which cannot be handled with conventional screens, a filter-cake type washing process would be more appropriate.

The reconstitution of 20 mT FMF from the dilute wash liquid by a 10- to 20-fold concentration is technically feasible by either an evaporative process (distillation) or by ultrafiltration. Both yield reusable FMF of the proper strength and clean kerosene to be reused in the spray-washing process.

An analysis of the investment and operational costs for the two kinds of recycling processes was made (Table 6). The investment costs for both systems in the envisioned size range (23 to 28 L kerosene/h) are quite comparable if carbon steel construction is used (~$11,000), while if made of stainless steel (which is mandatory if used with water-base FMF), the distillation equipment is almost twice as expensive as the corresponding UF loop ($22,000 vs $11,250).

Comparison of the operating costs for the two systems reveals a much greater discrepancy, namely 2.1¢ (UF) versus 19¢ (distillation) for each liter (20 mT) FMF recovered. The 19 liters of kerosene generated are a useful "by-product" at no extra cost. This almost tenfold economy in favor of the

TABLE 6
Comparison of Capacity and Costs for FMF Concentration by Distillation and UCARSEP Ultrafiltration System

Concentration method	Capacity, (L kerosene/h)	Operational cost[a] (¢/L of 20 mT FMF)	Capital investment ($, F.O.B.)
19-tube UF system	22.8	2.1	10,000 (carbon steel) 11,250 (stainless steel)
Distillation unit	28	19.0	11,600 (carbon steel) 22,000 (stainless steel)

[a] Labor costs have been excluded because all methods are automated and require a minimum of labor. Energy costs assumed at 4.19¢/kWh, September 1976.

UF system is expected to be amplified in the case of water-base FMF. The energy consumption in distillation depends on the latent heat of vaporization which for water is over 5 times that of the solvents in question. The energy dissipation in UF is related to the viscosity of the fluid, and in the temperature range envisioned the viscosity of water is comparable to that of the solvent.

On scaling up the recycling equipment, the operational costs increase in proportion of the throughput. The investment costs are likely to rise faster for the UF system because of the modular nature of the equipment, while there may be economy of size for the distillation installation. However, in view of the overwhelming ratio in operating costs, only very large installations based on evaporation are likely to be able to compete with the UF system. Considering that the production cost of 20 mT FMF is not likely to be lower than $1/L (*2*), it appears that the recovery and reconcentration of dilute FMF is highly advantageous even using the less economical distillation process.

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