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Evaluation of Inertial Filter for Dissolver Solution Clarification

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ABSTRACT

The performance of some types of solvent extraction contactors is adversely affected by a high concentration of entrained solids in the process feed streams. Therefore, during reprocessing of spent reactor fuels it is desirable to separate undissolved solids and insoluble fission product residues from dissolver solutions.

A series of statistically designed filtration tests were conducted to evaluate the separation efficiency of a Mott inertial filter for the removal of sub-micron particulate matter from dissolver slurries following centrifugation. Slurries used for testing consisted of 0.1 weight percent solids (needle-shaped yellow Fe_2O_3, rhombehedral-shaped red Fe_2O_3, or SiC hulls from HTGR fuel spheres) dispersed in water. Particle diameters ranged from less than 0.1 micron to 10 microns. The operating conditions varied temperature, inlet and outlet pressures, backwash intervals and particle shape. Measured separation efficiencies were greater than 90% indicating that all particles larger than 0.2 microns were removed.

INTRODUCTION

An ideal nuclear fuel reprocessing situation would be one in which all solids were dissolved into a liquid phase, then separated chemically. Since the ideal situation does not exist, solid-liquid separation becomes necessary. The undissolved, radioactive fission products, which can be mechanically entrained with the products, make the solvent extraction step less efficient. These particles range from less than 0.1 micron to relatively large agglomerates. Centrifugation can remove particles larger than $\sim$4 microns, but the smaller particles must be removed by filtration.

The vertical centrifuge tests carried out in the General Atomic (GA) pilot plant separated all particles greater than 4 microns (1). Although standard filtration methods, such as microfilters and membranes, can remove smaller particles, they are slow, require maintenance, and may even break down in a radiation field, making them unsuitable for nuclear fuel reprocessing plant application. For this reason, an inertial filter, made by Mott Metallurgical Corporation, was chosen for evaluation. A statistical experiment design was used to identify the operating variables having the greatest measured effect on the filter separation efficiency.

SYSTEM DESCRIPTION

The Mott inertial filter is a continuous cross-flow filter, which uses a high-velocity inertial effect and subsurface membrane development to achieve high filtrate purity. Figure 1 is a photograph of the inertial filter system.

The slurry is pumped through a sintered stainless steel tube at a linear velocity of $\sim$213 cm/s (7 ft/sec). The sintered tube has 0.5-micron pores and is centered inside a solid stainless-steel pipe. Together, these form an annulus. A valve on the downstream side creates a differential pressure across the porous tube. Some liquid slurry passes through the porous tube into the annulus and exits through a filtrate outlet.

Figure 2 shows a schematic diagram of the system. The slurry is returned to a holding tank and recirculated through the system, becoming more concentrated with each pass.

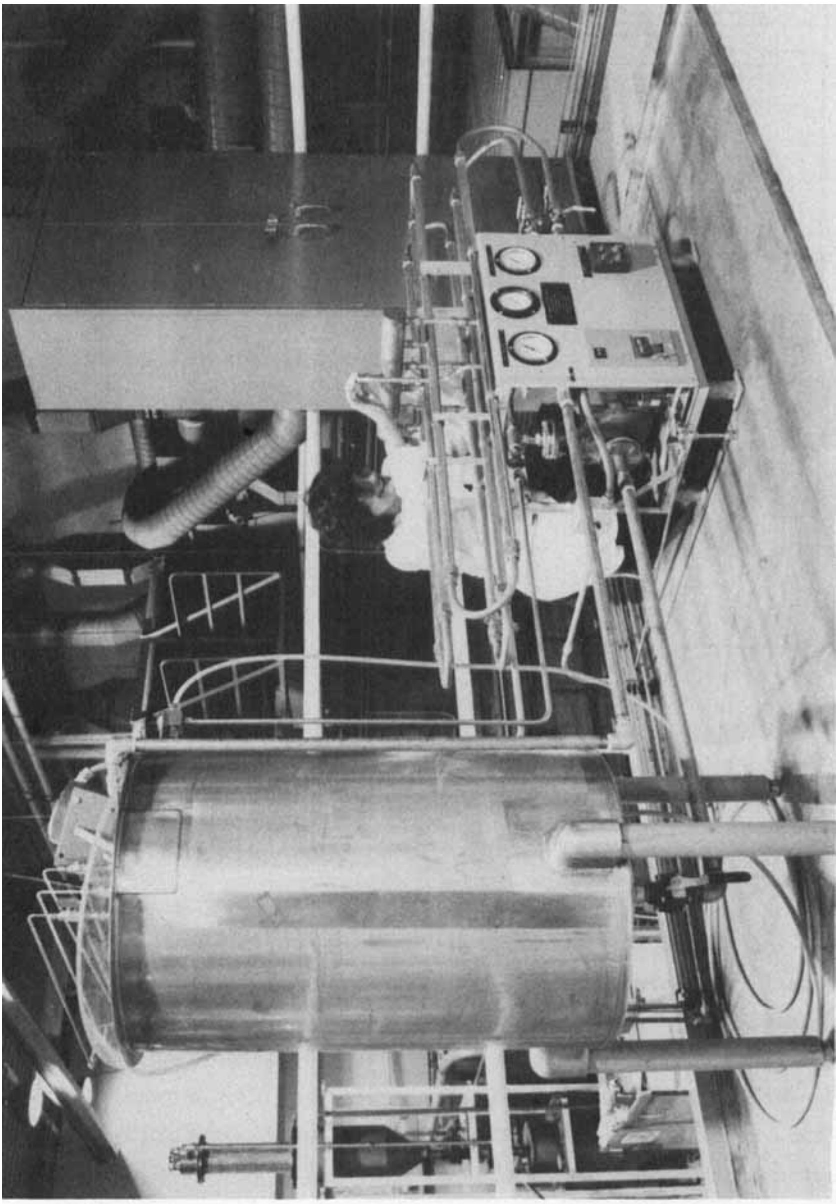

FIGURE 1. Mott-Inertial Filter System

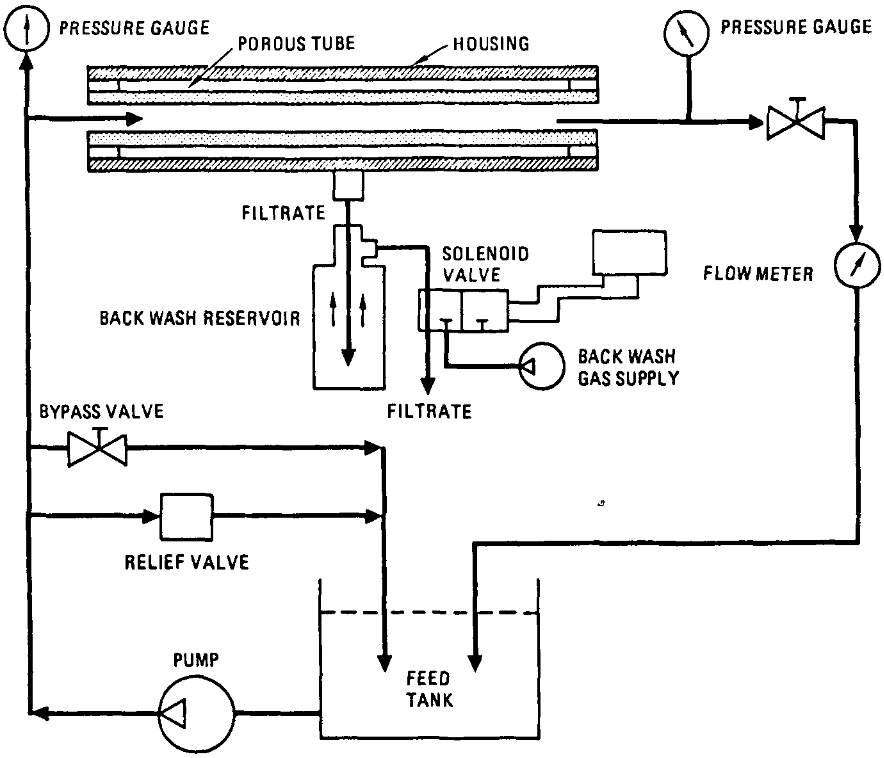

FIGURE 2. Inertial filter system schematic

As flow continues, a layer builds up on the inner tube wall. This layer increases the filter efficiency by keeping smaller-than-nominal-size particles from passing through the filter pores. A backflush of clear effluent can remove this layer when necessary. Gas at 652 kPa (80 psig) forces the effluent from a reservoir back through the porous tube. Particles are forced back into the slurry stream. Backflush interval and duration can be set within 0.1 s to 1 h. Figure 3 illustrates how backflushing can increase filtrate flow for a given interval.

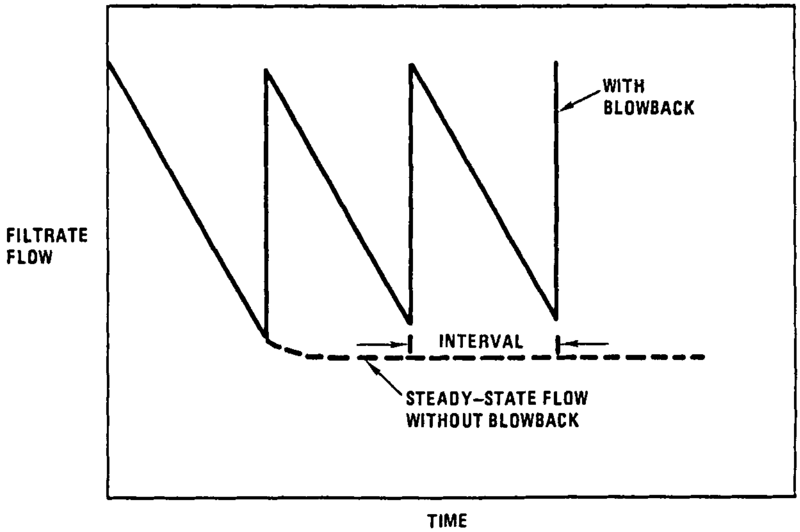

FIGURE 3. Filtration Rate Curves

EXPERIMENTAL

Shakedown Tests

Twenty-four shakedown tests were made to determine the operating range and capacity of the filter. The first five tests filtered an 0.1 wt % slurry of zirconium hydroxide in a basic medium. Filtration of the zirconium slurry was very slow, because the particles form a gelatinous layer on the tube wall and prevent the liquid from passing. Therefore, further testing of the filter with this slurry was not done.

Iron oxide in water slurry was used for 19 of the shakedown tests. Inlet and outlet pressure, blowback interval and duration were varied randomly. Filtration rates were determined, and the results of the shakedown were used to plan the variable limits for the statistical tests.

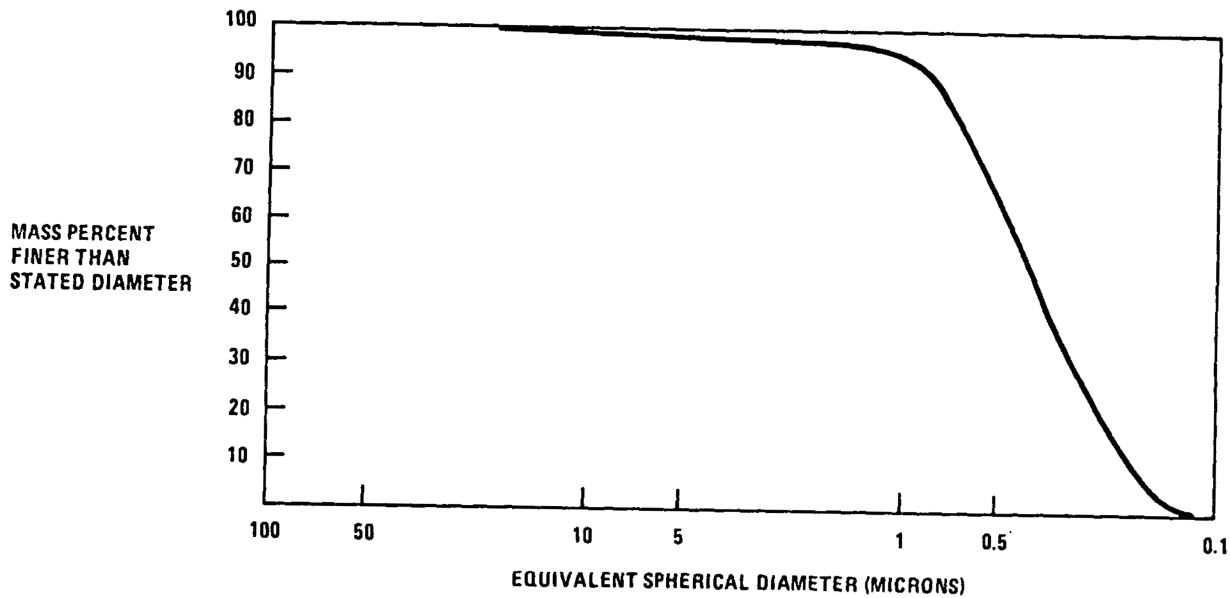

FIGURE 4. Particle Size Distribution Yellow Fe_2O_3

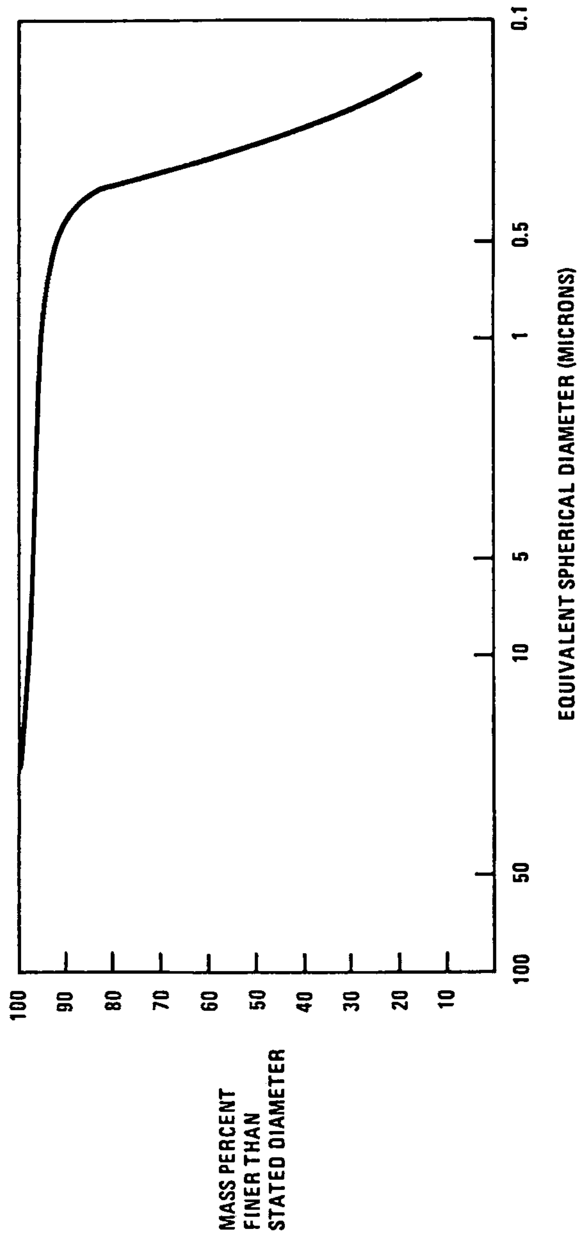

FIGURE 5. Particle Size Distribution Red Fe_2O_3

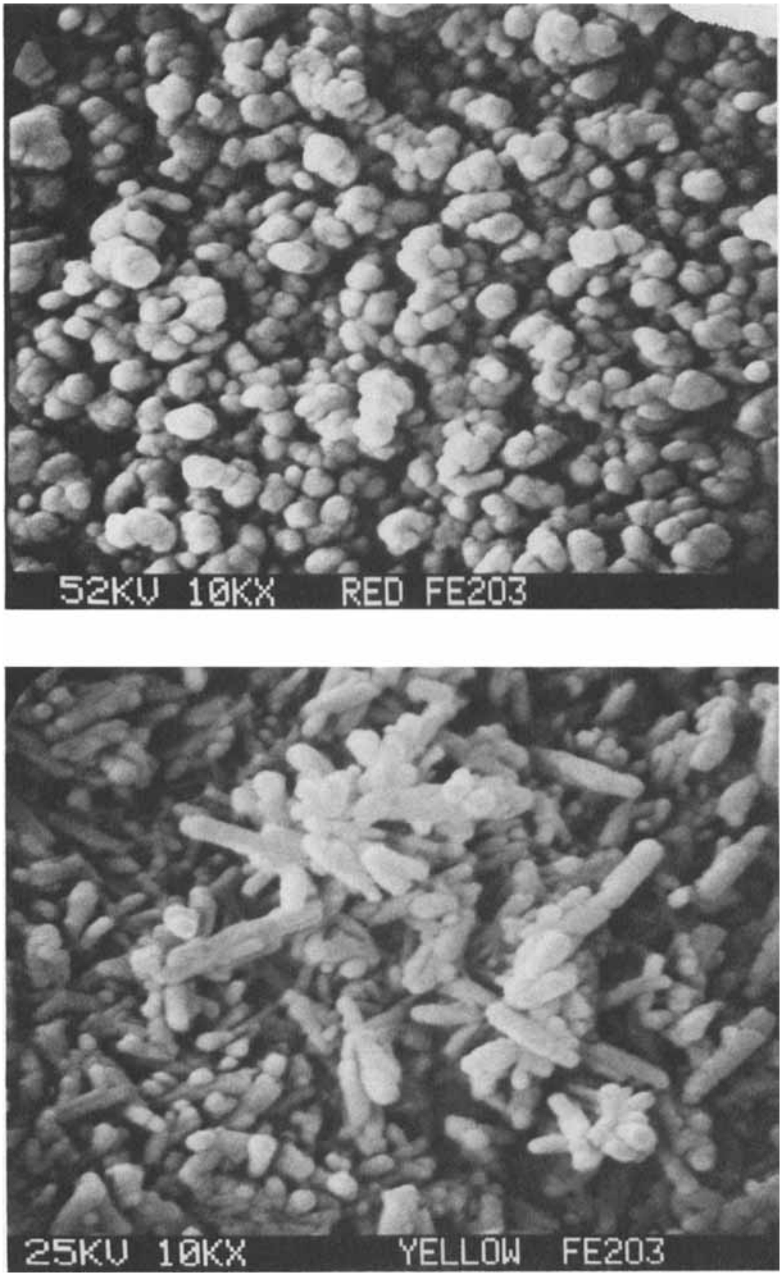

FIGURE 6. Microscopic View Of Iron Oxide Particles Used In Filtration

TABLE 1
Statistical Block Design Format - Inertial Filter Tests

Run	Temp.	Particle Shape	Blowback Interval	Outlet Press.	Inlet Press.	Y	Col. 1	Col. 2	Col. 3	Naive Estimates	Alias Structure of Estimates
M-12	-	-	-	-	+	99.76	199.66	391.06	776.54	Avg $\bar{y}$ = 97.07	
M-13	+	-	-	+	-	99.90	191.40	385.48	5.56	ℓ_A = 1.39	A + BE
M-5	-	+	-	+	-	94.70	199.48	2.14	-21.74	ℓ_B = -5.44	B + AE
M-1	+	+	-	-	+	96.70	186.00	3.42	4.84	ℓ_{AB} = 1.21	E + AB + CD
M-11	-	-	+	+	+	99.63	0.14	-8.26	-5.58	ℓ_C = 1.40	C + DE
M-14	+	-	+	-	-	99.85	2.00	-13.48	1.28	ℓ_{AC} = 0.32	AC + BD
M-6	-	+	+	-	-	91.40	0.22	1.86	-5.22	ℓ_{BC} = -1.31	BC + AD
M-2	+	+	+	+	+	94.60	3.20	2.98	1.12	ℓ_{ABC} = 0.28	D + CD
M-10	-	-	-	-	-	99.81	199.71	396.70	781.67	Avg $\bar{y}$ = 97.71	
M-15	+	-	-	+	+	99.90	197.00	384.97	10.32	ℓ'_A = 2.58	A - BE
M-7	-	+	-	+	+	98.80	199.36	-0.51	-16.46	ℓ'_B = -4.12	B - AE
M-3	+	+	-	-	-	98.20	185.61	10.83	9.26	ℓ'_{AB} = 2.32	- E + AB + CD
M-9	-	-	+	+	-	99.46	0.09	-2.71	-11.73	ℓ'_C = -2.93	C - DE
M-16	+	-	+	-	+	99.90	-0.60	-13.75	11.34	ℓ'_{AC} = 2.84	AC + BD
M-8	-	+	+	-	+	87.61	0.44	-0.69	-11.04	ℓ'_{BC} = 2.76	BC + AD
M-4	+	+	+	+	-	98.00	10.39	9.95	10.64	ℓ'_{ABC} = -2.66	D - CE

Evaluation Tests

A fractional factorial design was chosen to limit the number of tests required for the filter evaluation. This statistical design is used to generate relative values for the degree of influence that each operating variable has on the separation efficiency. Sixteen tests were needed to examine the effects of the five operating variables in this 2_{IV}^{5-2} saturated fractional factorial. Upper and lower limits were selected for each variable, and a statistical block diagram was made. Table 1 shows the design format.

Solids Composition

Red and yellow iron oxides were used as the solids in the filtration tests. Figures 4 and 5 show a size distribution of the equivalent spherical diameters of the oxides. The particles, although similar in size, differ greatly in shape. Figure 6 shows a microscopic view of the two particle types. The red is rhombehedral, and the yellow is needle shaped. For the statistical analysis, an arbitrary value of 10 was assigned to the needle-shaped particles and a value of 1 to the rhombehedral.

Testing

Samples of the effluent and slurry streams were taken every 4 min. When blowback was used, samples were taken just prior to the backflush. Slurry samples were taken from the slurry recycle stream. Effluent samples were taken at the filtrate outlet.

The initial slurry for each run consisted of 0.1 wt % solids dispersed in water. Iron concentrations in the feed slurry and effluent streams were determined by dissolving the iron oxide in sulfuric acid and performing a colormetric analysis.

RESULTS AND DISCUSSION

Equation 1 was formulated from the results of the inertial filter tests and can be used to estimate the Mott filter separation

TABLE 2
Operating Conditions For Inertial Filter Tests

Run	Temp (°C)	Inlet Pressure [kPa (psig)]	Outlet Pressure [kPa (psig)]	Flow Rate [l/min (gal/min)]	Blow-back (min)	Average Filtrate [l/min·cm^2 (gal/min·ft^2)]	Solids Removed (%)	Predicted Solids Removed (%)	Particle Type
M-1	50	342 (35)	135 (5)	19.5 (7.8)	0	0.0011 (0.28)	96.70	86.06	Red
M-2	50	342 (35)	204 (15)	25.7 (6.8)	4	0.0005 (0.12)	94.60	94.76	Red
M-3	50	273 (25)	135 (5)	25.7 (6.8)	0	0.0003 (0.08)	98.20	92.74	Red
M-4	50	273 (25)	204 (15)	15.5 (4.1)	4	0.0011 (0.26)	98.00	96.88	Red
M-5	25	273 (25)	204 (15)	16.7 (4.4)	0	0.0006 (0.16)	94.70	94.71	Red
M-6	25	273 (25)	135 (5)	21.2 (5.6)	4	0.0007 (0.17)	91.40	95.05	Red
M-7	25	342 (35)	204 (15)	24.6 (6.5)	0	0.0007 (0.16)	98.80	93.04	Red
M-8	25	342 (35)	135 (5)	27.6 (7.3)	4	0.0008 (0.19)	87.61	92.60	Red
M-9	25	273 (25)	204 (15)	16.3 (4.3)	4	0.0011 (0.26)	99.46	90.35	Yellow
M-10	25	273 (25)	135 (5)	24.6 (6.5)	0	0.0007 (0.18)	99.81	86.21	Yellow
M-11	25	342 (35)	204 (15)	24.2 (6.4)	4	0.0011 (0.27)	99.63	88.35	Yellow
M-12	25	342 (35)	135 (5)	29.5 (7.8)	0	0.0009 (0.23)	99.76	79.66	Yellow
M-13	50	273 (25)	204 (15)	16.7 (4.4)	0	0.0011 (0.27)	99.90	88.05	Yellow
M-14	50	273 (25)	135 (50	25.0 (6.6)	4	0.0009 (0.23)	99.85	88.39	Yellow
M-15	50	342 (35)	204 (15)	24.2 (6.4)	0	0.0015 (0.37)	99.90	81.77	Yellow
M-16	50	342 (35)	135 (5)	29.5 (7.8)	4	0.0012 (0.31)	99.90	83.47	Yellow

TABLE 3
Main Effects (By order of importance)

Operating Variable	Statistical Definition	Measured Main Effect
Particle shape	$B = (\ell_B + \ell'_B)/2$	-4.78
Blowback interval	$C = (\ell_C + \ell'_C)/2$	-2.17
Temperature	$A = (\ell_A + \ell'_A)/2$	1.99
Inlet pressure	$E = (\ell_{AB} - \ell'_{AB})/2$	-1.19
Outlet pressure	$D = (\ell_{ABC} + \ell'_{ABC})/2$	-0.56

efficiency within the range of variables tested:

$$y = 97.07 + \left(\frac{A - 37.5}{12.5}\right) - 2.39\left(\frac{B - 5.5}{4.5}\right) - 1.09\left(\frac{C - 2}{2}\right) - 0.28\left(\frac{D - 30}{5}\right) - 0.60\left(\frac{E - 10}{5}\right) - 0.30\left(\frac{B - 5.5}{4.5}\right) \times \left(\frac{E - 10}{5}\right) - 0.33\left(\frac{A - 37.5}{12.5}\right)\left(\frac{E - 10}{5}\right) + 0.39\left(\frac{D - 30}{5}\right) \times \left(\frac{E - 10}{5}\right) + 0.74\left(\frac{C - 2}{2}\right)\left(\frac{E - 10}{5}\right), \quad (1)$$

where y = separation efficiency,

A = temperature (°C),

B = particle shape (assigned between the two arbitrarily assigned limits),

C = blowback interval (min),

D = outlet pressure (psig), and

E = inlet pressure (psig).

Equation 1 can be algebraically reduced to give

$$y = 104.4 + 0.13A - 0.04B - 1.29C - 0.21D - 0.47E - 0.0053AE - 0.0133BE + 0.074CE + 0.016DE \quad .$$

Table 2 lists the operating conditions and both the empirical and experimental separation efficiencies for the 16 statistical tests.

The insoluble residues expected from the dissolution include irregularly shaped fission product oxides smaller than 5 microns (2). The shape of the iron oxide particles studied simulate these fission product oxides. The iron oxides have small size range differences and provide the shape variations expected.

The measured and empirically determined separation efficiencies differ by an average of 11.67%, with the measured separation consistently higher.

All but one test gave separation efficiencies greater than 90%, indicating that all particles greater than 0.15 microns were removed. The equation predicts that 4.58% more of the yellow particles will be removed than the red; the experiments obtained a value of 4.78%, suggesting good consistency of the empirical equation.

Table 3 lists the main effects in their order of importance.

CONCLUSIONS

1. The Mott inertial filter, with 0.5 micron porous tubes, can remove all particles greater than 0.15 microns.

2. Simplifying assumptions made in developing the empirical equation give conservative results.

3. Separation efficiency decreases as frequency of blowback increases.

4. Particle shape has a very large impact on separation.

5. In general, the greatest separation will be obtained above 25°C without blowback.

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