Fractionation of Fine Particle Slurries Using Periodic Flows in a Spinning Helix

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An unusual method of separating or fractionating fine-particle slurries has been proposed and partially explored by Landin (1980), Papanu (1983, 1986), Menon (1983), Lennartz (1984), and Lennartz et al (1987). A helically coiled tube spinning steadily on its major axis is connected to two agitated reservoir pumps which cycle a slurry bidirectionally through the helix as shown in Figure 1. With appropriate pumping steps, slurries can be concentrated or fractionated. The operation can be made continuous by adding feed at F and withdrawing products at I and II.

Research is motivated by the prospect of achieving sharp separations with fine particles smaller than about 10 μm. The technique is inherently multistage; the number of theoretical stages is approximately equal to the helix tube volume divided by the fluid volume pumped per cycle. The number of theoretical stages increases with reduced pumping volume.

In this work, the method is further explored for fractionation. Early investigators found that separation was hindered by material accumulation in the helix, caused by ineffective particle resuspension, an essential step of each cycle. A new equipment design provides more effective particle resuspension by reorienting sedimented particles in the centrifugal field. A mathematical model is developed to describe and simulate the process. Data from a batch fractionation experiment using a sand/kaolin-clay slurry is reported.

Fractionation—Six-Step Sequence

A slurry containing two types of particles, A and B, with different settling rates, is fractionated by convecting particle-free fluid, slurry containing B, and slurry containing both A and B, at different times, toward separate reservoirs. The helix spins steadily throughout the entire fractionation sequence. The faster-settling species, A, is sedimented in step 1, leaving substantial amounts of slower-settling B, in suspension. In step 2, a small volume of fluid and B particles is convected to the right (B-rich) reservoir. Step 3 allows all A and B particles to completely sediment. A larger volume of clear fluid is pumped slowly to the left (A-rich) reservoir in step 4. By the end of this step, secondary flows are generated, due to an imbalance of centrifugal forces in the helix cross-section. These secondary flows resuspend sedimented solids in step 5. If these secondary flows are inadequate for particle resuspension (the particles collect at a stagnation point), the helix cross-section may be reoriented in the centrifugal field to move sedimented solids back into the fluid stream during step 5. Figure 2 shows these secondary flows, axial flows, and centrifugal forces, for cases where the fluid flow opposes or complements the spinning direction. In step 6, resuspended A and B particles are convected leftward with fluid, toward the A-rich reservoir. The sum of volumes pumped left in steps 4 and 6 equals the volume pumped right in step 2; no net fluid movement occurs. At the end of the sequence, there has been a net movement of B particles toward the right reservoir and A particles toward the left reservoir. Repeating the sequence indefinitely, increases the degree of fractionation until steady state is achieved with balance axial dispersion and axial separation effects.

Model

The six-step fractionation cycle is modeled by material balances for the suspended and the sedimented particles. Since the helix fluid velocity profiles are not known precisely, the model detail is limited to a one (space) dimensional axial dispersion-convection approach. A material balance on the ith species of

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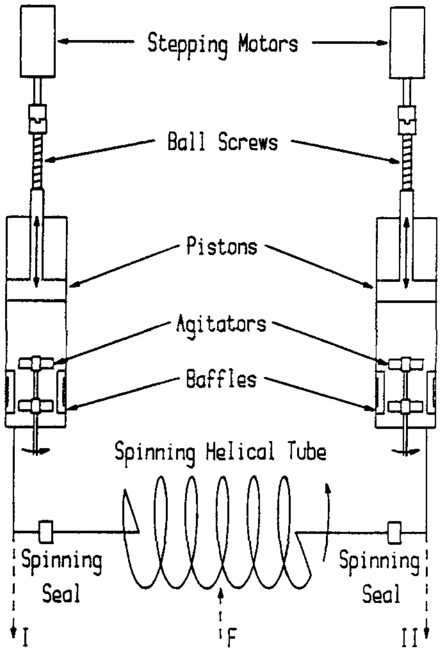

Figure 1. Separation apparatus.

suspended particles is:

$$\frac{\partial C_i}{\partial t} = D\frac{\partial^2 C_i}{\partial x^2} - u\frac{\partial C_i}{\partial x} - \delta(t - n\tau_1) f_{pi} C_i' - \delta(t + n\tau_3) f_{ti} C_i' + \delta(t - n\tau_5) f_{ri} S_i' \quad (1)$$

with boundary conditions for flow to the right ($u > 0$):

$$uC_{Li}(t) = uC_i(0^+, t) - D\frac{\partial C_i(0^+, t)}{\partial x}$$

$$\frac{\partial (V_R C_{Ri})}{\partial t} = uAC_i(L^-, t) - DA\frac{\partial C_i(L^-, t)}{\partial x}$$

$$\frac{\partial V_R}{\partial t} = uA$$

and boundary conditions for flow to the left ($u < 0$):

$$\frac{\partial V_L}{\partial t} = -uA$$

$$\frac{\partial (V_L C_{Li})}{\partial t} = -uAC_i(0^+, t) + DA\frac{\partial C_i(0^+, t)}{\partial x}$$

$$uC_{Ri} = uC_i(L^-, t) - D\frac{\partial C_i(L^-, t)}{\partial x}$$

A material balance on the ith species of sedimented particles is:

$$\frac{\partial S_i}{\partial t} = \delta(t - n\tau_1) f_{pi} C_i' + \delta(t - n\tau_3) f_{ti} C_i' - \delta(t - n\tau_5) f_{ri} S_i' \quad (2)$$

The model can be used to simulate experimental results. Equa-

The model can be used to simulate experimental results. Equations 1 and 2 are finite-differenced and solved on a computer. Experimental parameters are specified, including pumping speeds and volumes, as well as helix, reservoir, and connecting tube volumes, and operation cycles. The f_{pi} and f_{ti} factors can be estimated from Stokes' law, but f_{ri} and D are hard to estimate *a priori*. The f_{ri} are assumed, and the appropriate value of D (via axial dispersion Peclêt number) is determined by fitting the model to data.

Equipment

Hardware consists of an outer cylindrical sleeve and an inner concentric drum with a polyethylene tube helix in the annular space between them. Two subhelices are nested in parallel and connected together at one end of the assembly. Coil cross-sections are reoriented by axially displacing the outer sleeve. Centrifugal force fields up to 1,500xg, are achievable. The helix volume is 390 mL. Connecting tube and rotary seal volumes were minimized to between 28 and 110 mL, depending on the experiment, since Lennartz (1984) showed that such "dead" volumes inhibit separation. Computer controlled stepping motors turn ball screw assemblies to power the 200 mL reservoir/pumps equipped with agitators and baffles, as shown in Figure 1. The entire apparatus is interfaced to a personal computer, giving flexibility for changing many experimental parameters with software. Experiments can be run in either batch or continuous mode.

Optional rotation of the helix tube cross-sections can aid particle resuspension. Rotation of up to 90 degrees moves the particle stagnation point into a position of more vigorous secondary flow; rotation between 90 and 270 degrees reverses the direction

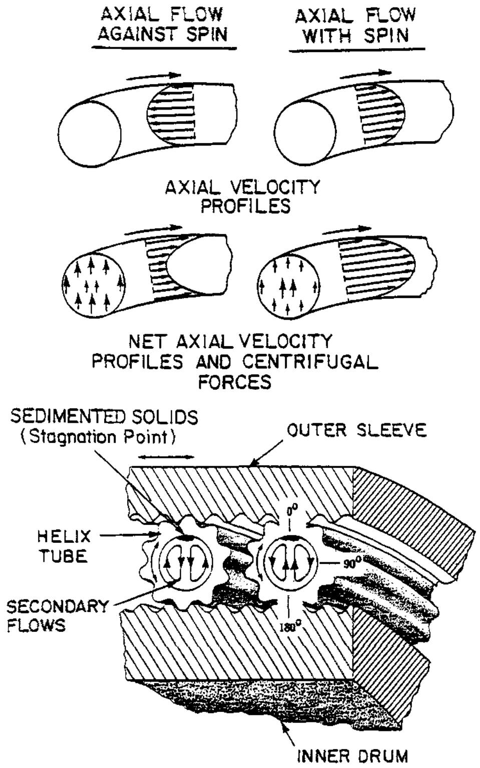

Figure 2. Secondary flows in a spinning helix.

of the centrifugal force, as shown in Figure 2 (bottom). When the outer sleeve is axially displaced, the helix tube cross-sections all rotate simultaneously so the helix tubing is twisted only at the ends.

Experimental Study

Mixtures of silica sand (specific gravity ~2.68) and kaolin clay (specific gravity ~2.60) were investigated. Coulter-counter particle size analyses (data appears in the Supplementary Material) show that the sand and kaolin size distributions overlap only slightly, at about 44 μm, so wet sieving at 325 mesh is a practical and simple method for analysing sand and kaolin fractions.

Slurry is prepared in the apparatus with equal weighed amounts of sand and kaolin in an alkaline surfactant. Moderate agitation is maintained in the reservoirs throughout the experiment, to prevent kaolin particle flocculation to and provide uniform reservoir concentration. The helix is brought to the desired spinning speed, and the specified number of six-step separation cycles are executed. Analysis is done gravimetrically. Slurry is carefully flushed out of separate parts of the apparatus; sand is sieved out (325 mesh and above), washed, dried, and weighed. The kaolin and washings are also dried and weighed.

Results and Discussion

A variety of operating conditions were used in many experiments (see Supplementary Material). One is presented here in detail as an illustrative example. Operating conditions for the six-step cycle, and final-over-initial concentration results appear in Table 1. The data show fractionation with enrichment of species.

The computer model was used to approximate these results. The sand and kaolin fractions sedimented in step 1 were calculated as follows. At 800 rpm, particles experience 109xg acceleration in the helix. Using Stokes' law and the "initial" size ranges from Figures 4 and 5, settling velocities are 103 to 653 cm/s for sand, and 0.038 to 9.74 cm/s for kaolin. The helix tubing is 0.635 cm inside diameter, so even the smallest sand particles completely sediment in the 0.25 seconds allowed for partial sedimentation in step one; hence, f_p for sand is 1. Assuming the resuspended kaolin particles are uniformly distributed throughout the helix cross-section, the fraction sedimented can be calculated, based on settling velocities. The calculation was done for each size of kaolin particles and weighted by volume percent to give the total fraction sedimented, $f_p = 0.43$, for kaolin. (Calculations appear in the Supplementary Material.) Enough time was allowed in step 3 for total sedimentation so that the $f_{tt} = 1$. The f_{rt} are assumed to be 1 since the centrifugal field is reversed for step 5.

The computer model was run with various axial dispersion Peclêt numbers to mimic experimental results. Figure 3, which closely approximates run 23 data, has an overall axial dispersion Peclêt number, $N_{Pe} = 300$. The model determines concentration profiles through the separation machine, and average concentrations are compared against experimental data in Table 1. Species "A" is sand and kaolin larger than ~5 microns, while species "B" is kaolin fines. (The model does not distinguish between materials by composition.) The model concentration profile shows promise for continuous separation, since net flows from both ends of the apparatus will disperse the concentration peaks in directions beneficial to the separation.

Particle size analyses of materials taken throughout the apparatus, provide additional insight. The sand separation was so thorough that insufficient sand for particle analysis remained in the kaolin-rich side of the machine. Sand in the sand-rich reservoir and in the sand-rich subhelix have similar particle sizes. Figure 4 shows that sand concentrates over its entire size range.

Particle size analysis of the kaolin feed material, reservoir, and helix samples are presented in Figure 5. Within the helix, the kaolin size distributions show fractionation occurring at about five microns. There is some overlap of particles near 5 μm in size, but this is expected from dispersion effects. Calculation of the fraction of particles sedimented (see Supplementary Ma-

Table 1. Run 23 Experimental Conditions, Results (60 Separation Cycles)

Rotation Speed	*Total Solid Conc.	Sedimentation Time, Step One	Sedimentation Time, Step Three	Helix Rot.	Volume Pumped, Step Two	Volume Pumped, Step Four	Volume Pumped, Step Six	Pumping Rate, Step Two	Pumping Rate, Step Four	Pumping Rate, Step Six
rpm	g/mL	s	s	deg	mL	mL	mL	mL/s	mL/s	mL/s
800	0.0228	0.25	1	180	149	102	46.9	30.1	3.2	77.2

	Sand Rich Reservoir	Sand Rich Helix	Kaolin Rich Helix	Kaolin Rich Reservoir	Dead Volumes: Sand Side	Dead Volumes: Kaolin Side
Total Equip. Vol. (mL)	200	245	226	206	49.6	37.0
Exp. Results (C/C_0)**						
Sand	2.02	1.75	0.004	0.11	—	—
Kaolin	0.23	0.64	1.49	1.59	—	—
Simul. Results (C/C_0)						
Sand	2.01	1.51	0.01	0.0	—	—
Kaolin	0.04	0.55	1.98	1.86	—	—

*Solids were always 50% sand and 50% kaolin.
**C_0 = initial uniform concentration of sand and kaolin.

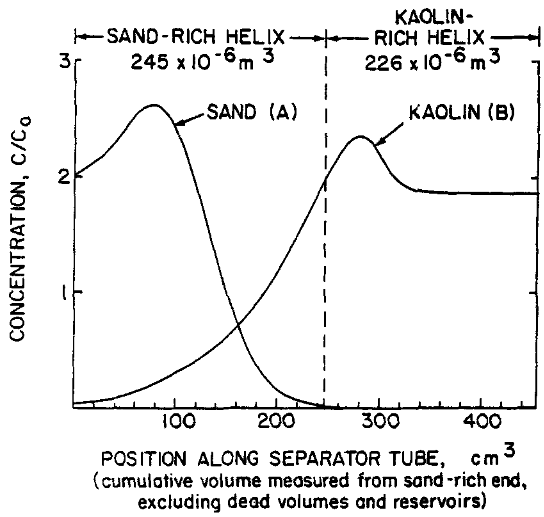

Figure 3. Run 23 simulation.

terial) shows that all particles larger than about five microns are totally sedimented in Step 1, while particles smaller than five microns are only partially sedimented. Hence kaolin smaller than ~5 microns is enriched in the kaolin-rich side of the apparatus, while kaolin larger than ~5 microns is enriched on the sand-rich side. Large kaolin particles became more concentrated in the sand-rich helix than the sand reservoir—evidence of the concentration peaks suggested by the model.

Conclusions

Sand and kaolin have been fractionated by settling velocity at a size of about 5 μm. Since most of the kaolin is smaller than five microns and virtually all the sand is larger than five microns, the sand-kaolin mixture was significantly fractionated, demonstrating fine-particle fractionation, using the reoriented centrifugal field.

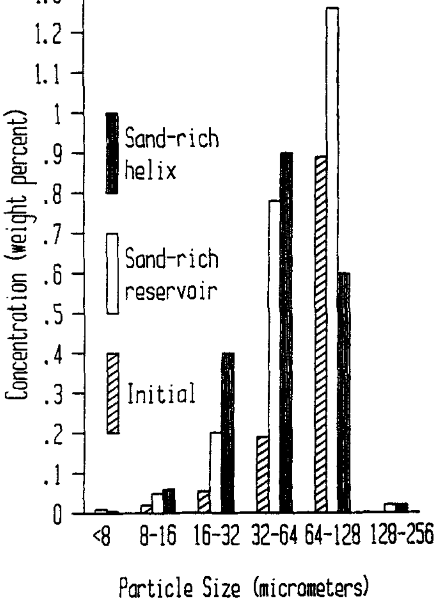

Figure 4. Run 23 results—sand.

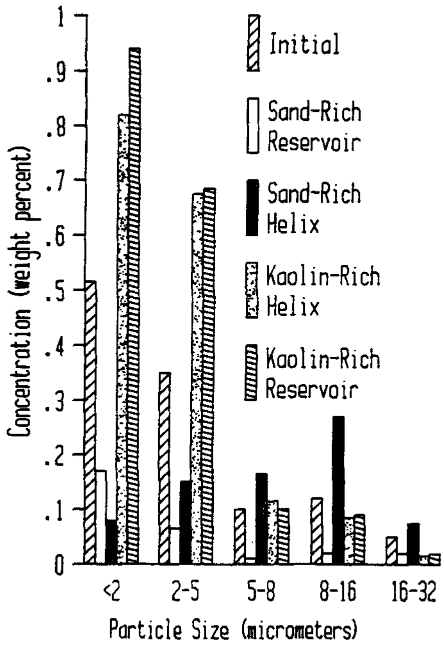

Figure 5. Run 23 results—kaolin.

Acknowledgment

Essential financial support was provided by C.E. Tyler, Incorporated, the National Science Foundation (C.P.E. 812096), and by Helipump Corporation. The authors also wish to acknowledge the assistance of Drs. Maurice L. Adams, Eugene Bahnuik, and Daniel K. Wright, as well as Messrs. Steve Bowen, Emil Palik, and Bob Russka, all of Case Western Reserve University.

Notation

A = cross-sectional area of helix tube
$C_i = C_i(x, t)$ = concentration of species, i, in suspended phase
$C_i' = C_i(x, t - \epsilon)$ = concentration of species, i, in suspension just prior to time, $t(\epsilon \rightarrow 0)$
$C_{Li} = C_{Li}(t)$, $C_{Ri} = C_{Ri}(t)$ = concentration of species, i, in left and right reservoirs
$D = D(t)$ = axial dispersion coefficient, finite during steps 2 and 6, zero during all other steps
f_{pi} = fraction of species, i, partially sedimented in step 1
f_{ti} = fraction of species, i, totally sedimented in step 3; usually = 1
f_{ri} = fraction of species, i, resuspended in step 5
L = length of helix tube plus connecting tubing
n = cycle number, 1, 2, 3, . . . , ∞
N_{Pe} = axial dispersion Peclêt Number = uL/D
$S_i = S_i(x, t)$ = pseudo-concentration of species, i, in the sedimented phase
$S_i' = S_i(x, t - \epsilon)$ = pseudo-concentration of species, i, in the sedimented phase just prior to resuspension ($\epsilon \rightarrow 0$)
t = time
$u = u(t)$ = axial flow velocity ($u > 0$ for flow to right, $u < 0$ for flow to left)
$V_L = V_L(t)$, $V_R = V_R(t)$ = volume of left and right reservoirs
x = axial displacement from left reservoir
$\delta(\xi)$ = Dirac Delta function, zero expect at $\xi = 0$
τ_1, τ_3, τ_5 = time from beginning of cycle to end of steps 1, 3, 5

Literature Cited

Landin, M. E., "Slurry Separation by Centrifugal Fractionation," MS Thesis, Case Western Reserve Univ., Cleveland, OH (1980).
Lennartz, J. W., "On the Separation of Fine Particle Slurries Using

Asymmetric Periodic Flows in a Centrifugal Field," MS Thesis, Case Western Reserve Univ., Cleveland, OH (1984).
Lennartz, J. W., M. B. Gorensek, and R. J. Adler, "Separation of Fine Particle Dispersions Using Periodic Flows in a Spinning Coiled Tube: II. Batch Fractionation Experiments," *AIChE J.*, **33**(3), 506 (Mar., 1987).
Menon, M. M., "Periodic Centrifugal Separations," MS Thesis, Case Western Reserve Univ., Cleveland, OH (1983).
Papanu, J. S., "Centrifugal Separation of Fine Particle Slurries Using Periodic Flows," MS Thesis, Case Western Reserve Univ., Cleveland, OH (1983).
Papanu, J. S., R. J. Adler, M. B. Gorensek, and M. M. Menon, "Separation of Fine Particle Dispersions Using Periodic Flows in a Spinning Coiled Tube," *AIChE J.*, **32**(5), 798 (May 1986).

Manuscript received March 28, 1988, and revision received Jan. 24, 1989.

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