Separation of Fine-Particle Dispersions Using Periodic Flows in a Spinning Coiled Tube Part II: Batch Fractionation Experiments

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Introduction

The fractionation of fine particles, defined here as having effective diameters less than 10 μm, has numerous industrially significant applications. Among these are beneficiation of mineral ores, coal washing, and removal of fines from synfuels. Recently, a novel method was proposed for separating fine particles in suspension through the interaction of secondary flows with the centrifugal field in a spinning coil (Papanu et al., 1986); the results of proof-of-principle experiments for batch concentration were also presented. This paper reports the results of additional experiments that successfully demonstrated the batch fractionation of a binary mixture of fine particles.

Concept

A detailed description of the new method has been presented by Papanu et al. (1986). The present work is limited to the five-step batch fractionation cycle proposed in that paper. For simplicity, the objective is taken to be the separation of a bidisperse suspension of fine particles in which each of the two species has a distinct, different sedimentation velocity.

Separation occurs in a coiled tube spinning rapidly and continuously about its helical axis, as represented in Figure 1. Rotation is fast enough to ensure a strong centrifugal field across the tube. The steadily rotating coil is connected at both ends to stationary ports by means of a spinning seal assembly. Each port leads to a reservoir, allowing flow through the coil in either direction, from one reservoir to the other. The system is initially filled with the fine-particle suspension that is to be separated. A periodic, five-step sequence of back-and-fourth flows and rest periods is then imposed. Particles of one species accumulate in one reservoir, while the second species is concentrated in the other reservoir.

The five-step sequence is illustrated in Figure 2. It presumes two particle species, A and B, with A having a larger sedimenta-

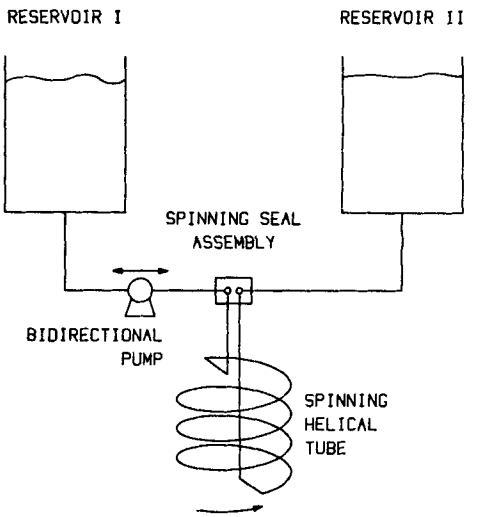

Figure 1. Batch fractionation apparatus.

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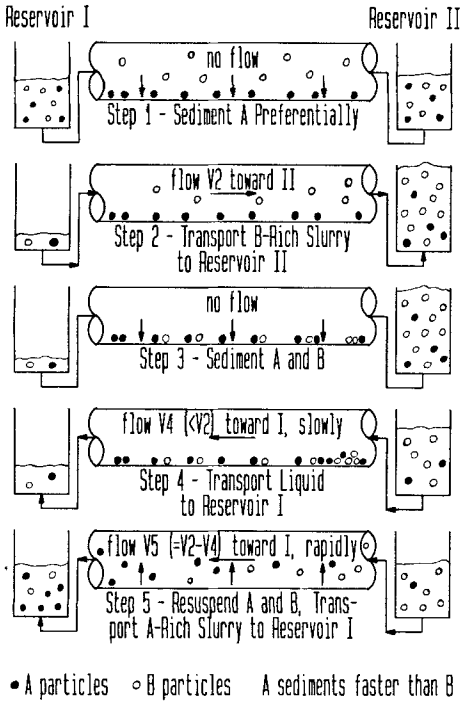

Figure 2. Five-step batch fractionation sequence.

tion velocity than *B*. In the first step, the fluid in the coil is allowed to sediment for a time long enough so that *A* drops out of suspension completely, but short enough so that a substantial portion of *B* remains suspended. In step 2 the coil contents are slowly displaced toward the right at a flow rate such that the sediment is undisturbed. The only particles moved are those of species *B*. Step 3 is a complete sedimentation step in which all of the suspended particles drop out. The fourth step is a displacement to the left of a fraction of the volume of the coil at a flow rate slow enough that the sediment stays undisturbed. No particles are moved. Step 5 is a rapid displacement to the left of the remainder of the coil volume at a flow rate large enough to ensure complete resuspension of the sediment, restoring the reservoirs to their original levels. The net effect is to move *A* to the left (step 5) and *B* to the right (step 2 minus step 5).

Experimental Design

The prototype apparatus used in earlier experiments (Papanu et al., 1986) was substantially upgraded for this work. Figure 3 is a diagram of the equipment as presently configured.

The rotating coil assembly was essentially unchanged. It is driven by an IEC centrifuge (model UV with a modified speed controller) capable of precise (±2 rpm) speeds in the range 0–2,000 rpm. The coil itself is comprised of 5½ turns of Teflon tubing (4.4×10^{-3} m ID, 6.6×10^{-3} m OD) wound inside a 0.204 m ID stainless steel bowl that is mounted on the centrifuge rotor. A spinning seal assembly at the top of the bowl provides stationary flow connections to both ends of the coil.

Pumping ability and reservoir capacity are incorporated in a pair of synchronized pumping assemblies. Each assembly consists of a 350×10^{-6} m^3 displacement cylinder equipped with a piston and plunger. Both plungers are precisely driven by computer-controlled stepping motors. Rotation of each motor shaft is converted into axial piston motion by means of a screw turning through a ball nut fixed to the plunger. Both cylinders are

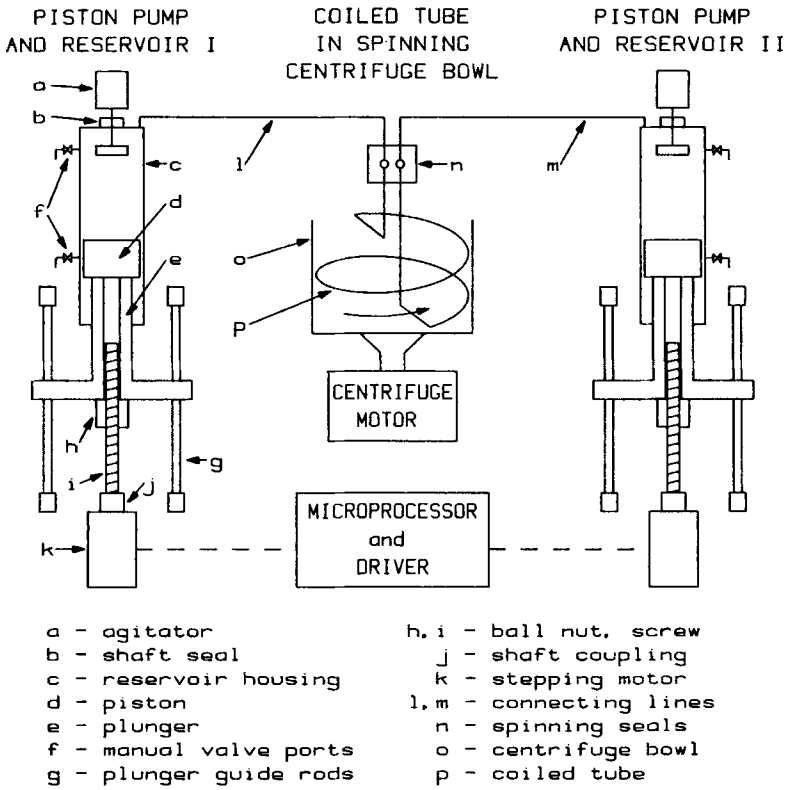

Figure 3. Experimental apparatus.

equipped with an agitator to prevent settling, and ports for sampling and flushing.

The coiled tube has a total volume of 54×10^{-6} m^3. Each reservoir-to-coil connecting line holds 6×10^{-6} m^3, while the spinning seal assembly has a volume of 9×10^{-6} m^3.

Dilute aqueous suspensions of ferric oxide (Pfizer, Pigments Div., R-9998) and zirconium oxide (Harshaw Chemicals Co., Colors Dept., D-420) were chosen for the proof-of-principle demonstration. This system is described in Table 1. The existence of particle size distributions, although not extreme, did render the system nonideal. Significant overlap in settling rates existed between the two populations. Laboratory measurement of settling rates confirmed the upper size limit for each material. Composite analyses of mixtures were made through filtration and selective dissolution of the ferric oxide using concentrated hydrochloric acid.

Results

Conditions under which the ferric oxide and zirconium oxide particles would be sedimented or resuspended were separately determined in a series of preliminary batch-concentration experiments. (See Papanu et al., 1986, for a description of the batch-concentration mode of operation.) Based on these results, a tentative five-step fractionation sequence was devised. Further refinements were made in subsequent experiments. Table 2 lists the final set of operating conditions that was developed for separating the two oxide powders.

The results of a batch fractionation experiment conducted according to these conditions are illustrated in Figure 4. Separation was achieved to a large degree. Final reservoir concentrations indicate an enrichment-to-dilution ratio of 3.6 for the ferric oxide and 4.8 for the zirconium oxide. Complete separation of the mixture could not be expected due to the overlap in settling rates noted earlier. However, nearly 80% of the amount of each component initially present throughout the apparatus was transferred to the desired reservoir after 100 cycles. Further processing would be expected to cause additional transfer, mostly of ferric oxide. Although the system had not achieved steady state operation, these results clearly represent a success-

Table 1. Experimental Particle System

Properties	Ferric Oxide Fe_2O_3	Zirconium Oxide ZrO_2
Density, kg/m^3	5.15×10^3	5.75×10^3
Shape	Nearly spherical	Nearly spherical
Size,* μm		
50 wt. %	3–4	2–15
80 wt. %	1–5	0.5–20
95 wt. %	0.5–10	0.3–25
100 wt. %	<20	<30
Settling rate,** m/s $\times 10^2$		
50 wt. %	20–36	10–580
80 wt. %	2.3–57	0.65–1,000
100 wt. %, calc.	<900	<2,300
100 wt. %, meas.	<1,200	<2,600

*Size range of particles comprising given wt. % of distribution, based on manufacturers' data and optical measurement.

**Gravity settling rate of particles comprising given wt. % of distribution, calculated from Stokes' law, in distilled water containing 0.05 wt % dispersant (Alconox laboratory detergent).

Table 2. Experimental Conditions for Batch Fractionation Experiment

System Geometry	
Initial reservoir volumes	290 mL each
Connecting line volumes	6 mL each
Coil volume	54 mL
Seal assembly volume	9 mL
Coil	5½ turns TFE tubing Tube radius = 0.22 cm Coil radius = 10.2 cm
Initial Concentrations	
Fe_2O_3 = 0.0030 g/mL everywhere	
ZrO_2 = 0.0032 g/mL everywhere	
Procedure	
Coil rotation rate	160 rpm
Centrifugal field strength	2.86×10^3 cm/s^2 ($2.92 \times$ g)
Cycle sequence	
Step 1	2.5s pause
Step 2	20 mL pumped toward II at 1.1 mL/s
Step 3	90s pause
Step 4	5 mL pumped toward I at 1.1 mL/s
Step 5	15 mL pumped toward I at 56 mL/s
Directions of rotation	Rotation and flow toward reservoir I coincide
Duration of experiment	100 cycles
Sampling	25 mL removed from each reservoir every 25 cycles

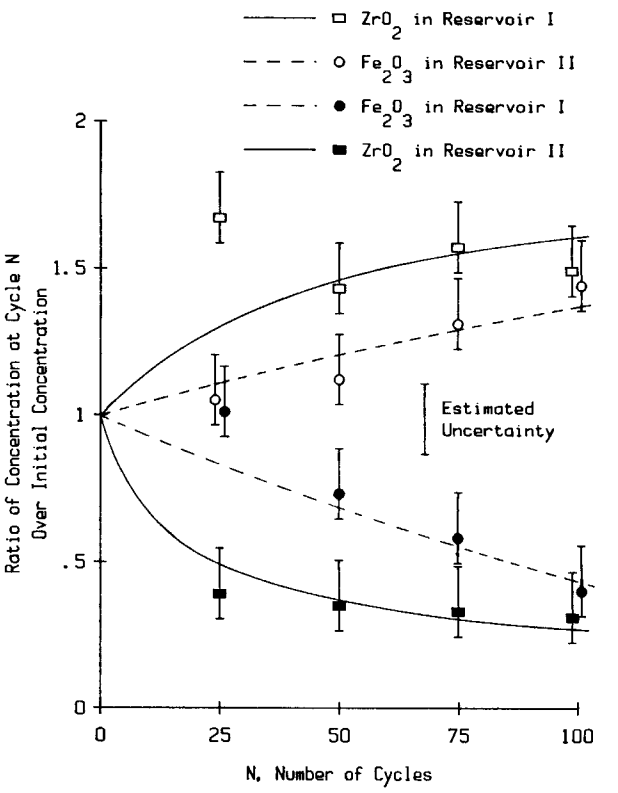

Figure 4. Results of batch fractionation experiment.
Curves sketched only to aid visualization

ful separation of a mixture of fine particles. Thus, the new method can be used to fractionate fine-particle mixtures.

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Literature cited

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 (1986).

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