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TECHNICAL NOTE

A Note on Flow and Separation in a Centrifugal Spectrometer

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ABSTRACT

Results of preliminary tests with a prototype machine show excellent fractionation of a mixture according to settling velocity, and they establish the concept of centrifugal spectroscopy as broadly applicable and commercially feasible. Since operation of the centrifuge is truly nonstop, a completed system will efficiently process the large volume of fluid typically involved in a technological application that requires a mixture to be both separated and sorted according to size or density differences with a high, assured degree of purity.

INTRODUCTION

The centrifugal spectrometer (1, 2) is a device by which particles of any size can be separated from a mixture and sorted according to sedimentation velocity. Since operation of the centrifuge is truly nonstop, it can efficiently process the large volumes of fluid encountered in certain bioprocesses, or any technological operation that requires quantity but precise fractionation with a high, assured degree of purity.

The central idea of the spectrometer is the utilization of a continuous flow of mixture cascaded through narrow channels formed by a stack of slotted cones, the number of which is related to the purity desired. In a steady, axial flow of a mixture, various particle species will completely settle out of the stream at distinctly different distances along the channel length (Fig. 1) because the sedimentation time of a particle decreases with increasing mass or size. Many old and new separation techniques (3, 4)

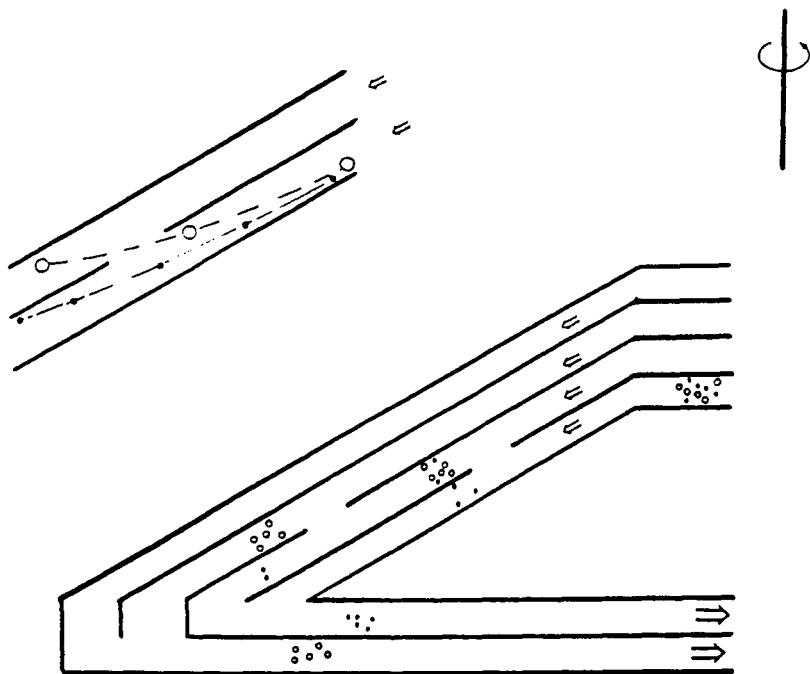


FIG. 1 Top: Particle settling in a centrifugal field. Bottom: The spectrometer concept: separation, fractionation, and an internal cascade.

are based on this observation, but particles in the spectrometer are also diverted to an outer channel through a slot at an appropriate location. This enables complete capture or retention in the inner channel of only particles smaller than an assigned cutoff value, while some are also unavoidably diverted along with all the larger particles to the outer passage-way. By repeating this cut many times in the conical disk stack, in effect producing an internal cascade, the final result will be a two-part fractionation of the original size distribution having diameters above or below the cutoff value. (The operating procedure can be modified to purify and harvest either or both the larger or smaller sizes, entirely internally or by external recycling.)

THE SPECTROMETER CENTRIFUGE

The spectrometer is a very complex multichannel flow which was a challenge to construct. For budgetary reasons, only a two-stage centrifuge

was built which necessitated the substitution of the complete internal cascade by the processing of the exiting fluid and its return as input, as many times as necessary to achieve the purification desired. (The condensing centrifuge must still be constructed to complete the feedback loop required for continuous operation as shown in Fig. 2, a schematic of the entire experimental system.)

In essence, the device consists of three main components—the inlet manifold, the centrifuge bowl with exit manifold, and the collection assembly—as shown in Fig. 3.

A brief description of the flow is as follows. Five flow streams enter the device and are combined internally so that only two streams emerge. Of the entering channels, two are used to bring the mixture to the desired volume concentration but with the particles also placed most advantageously for separation. A third channel provides pure fluid to accept the particles that pass through the slot in the conical wall, and the last two wash-fluid channels of pure fluid assist in removing the two particle-laden streams from the center of the centrifuge.

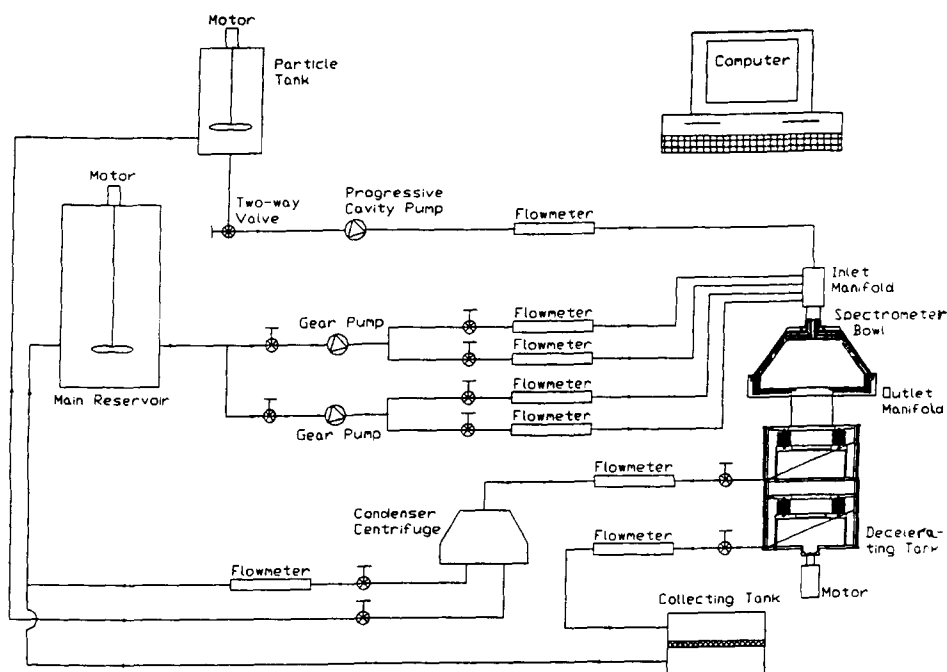


FIG. 2 Schematic of the spectrometer experiment.

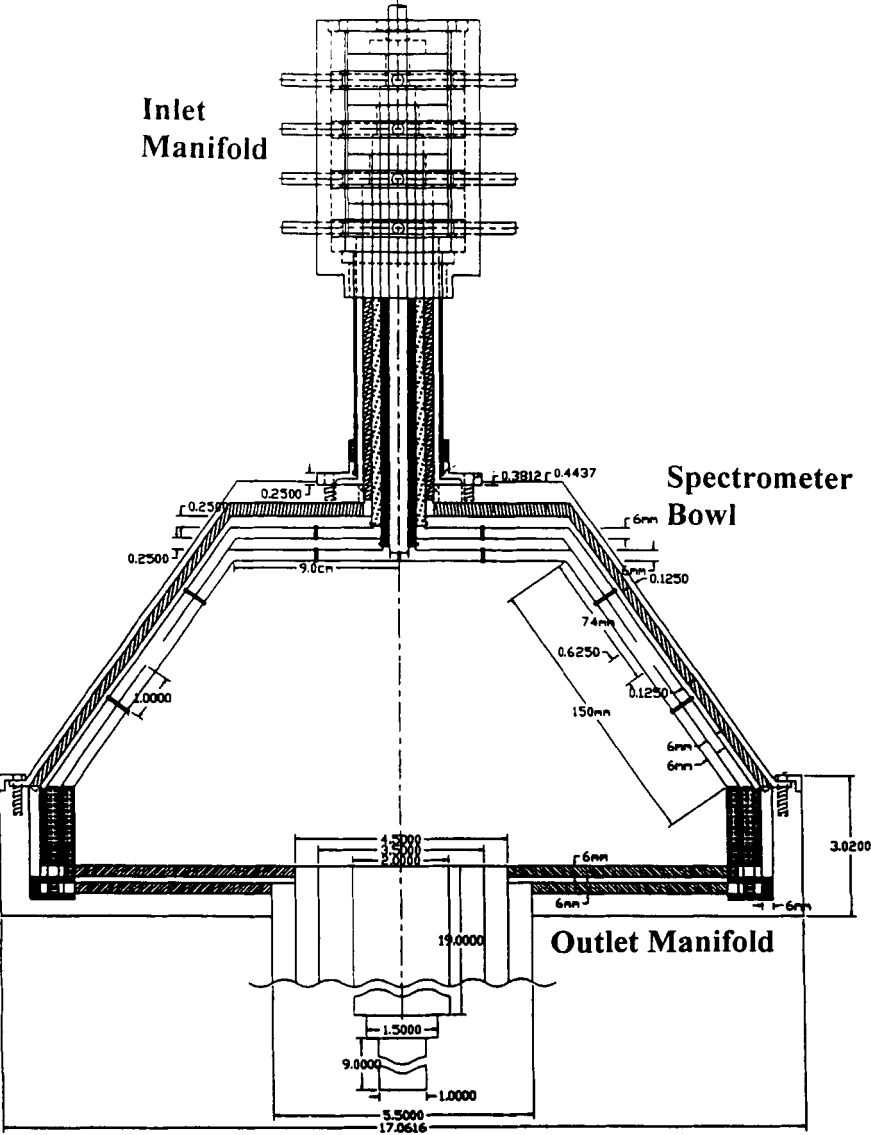


FIG. 3 Drawing of the main components: inlet and outlet manifolds, and spectrometer bowl.

The inlet manifold is a more or less straightforward construction of vertical channels with appropriate seals and exit slots.

The outlet manifold unites the various channels into two streams in a manner that can be generalized for a true multistage machine. The design, patent pending, mitigates the effects of the Coriolis force and produces an advantageous velocity field while preventing particle accumulation due to the induced retrograde rotation.

Constant volume pumps maintain the proper flow rates in each channel, and flowmeters monitor conditions around the circuit. The entire process (pumps, motors, meters, etc.) is computer controlled; the machine is conservatively designed to operate over a wide range of conditions and parameter settings.

In the experiments underway, the fluid employed is polyethylene glycol of density 1.04 g/cm^3 and viscosity $2.5 \text{ cm}^2/\text{s}$, and the particles are polystyrene spheres of density 1.05 g/cm^3 with diameters in the range of $300\text{--}400 \text{ }\mu\text{m}$. A mixture of fluid with a 1% volume concentration of particles is fed into the centrifuge set to rotate at 600 rpm so that only particles with settling velocities below an assigned cutoff value can remain in the inner channel after passing the diversion slot. These particles are harvested by passing the effluent from this channel through collection screens. The purified fluid is returned to the reservoir and the mixture emanating from the second channel would be reprocessed by a new type of condenser centrifuge into two streams—a 10% volume concentration flow returned as feed input and pure fluid returned to the reservoir. The cycle could then be repeated until assays of the emergent streams indicate that all particles with settling velocities below the cutoff value have been collected. Let r be the fraction of desired particles harvested in each cycle, then after n passes, or in a cascade of n stages, only $(1 - r)^n$ of the original number in this size range would still remain in the waste effluent. A value of r as close to 1 as possible is the ultimate goal. If all the particles had the same density, which they certainly do not, the settling velocity would correlate exactly with diameter, and the harvested particles would all be below a cutoff size. Density deviations were held to $\pm 0.005 \text{ g/cm}^3$ by repeated flotation tests but still represented a major source of error in the results of fractionation *by size*. Sorting by settling velocity, the operating principle of the spectrometer, is a much more precise indicator of success.

EXPERIMENTAL RESULTS

The separative efficiency and sorting capability have been evaluated in a single-stage prototype spectrometer by simply comparing the volume fractions and size distributions of the input and output streams (i.e., the

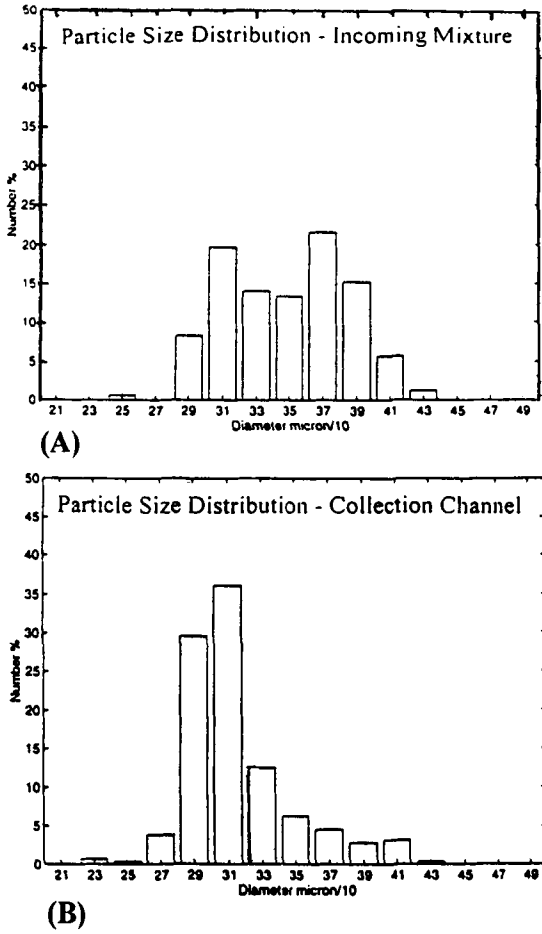
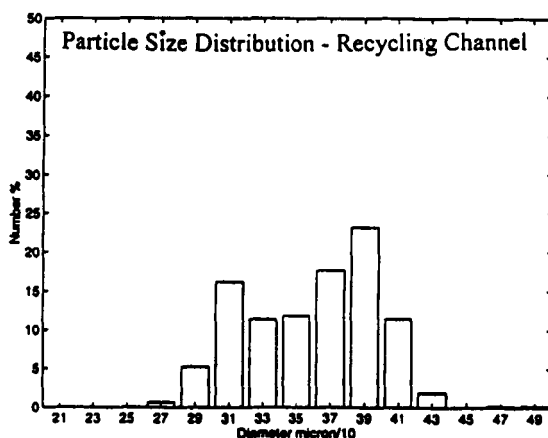


FIG. 4 Experimental results with angular velocity of 600 rpm; fluid density = 1.04 g/cm³; fluid viscosity = 2.5 cm²/s; particle diameters in the 300 to 400 μ m range; particle density = 1.05 \pm 0.005 g/cm³. Diameters of 1000 particles were measured microscopically and confirmed by analysis with a Coulter counter. (A) Particle size distribution in a mixture entering the spectrometer centrifuge. (B) Particle size distribution in flow from the collection channel. (C) Particle size distribution of mixture in the outer channel to be recycled (the second stage of the cascade).

first stage of the cascade process). The results were extremely gratifying because the centrifuge produced an excellent fractionation by settling velocity and very good sorting by size, sharper than that presently obtainable in bulk from commercial sources! In a low speed test (600 rpm and a



(C)

FIG. 4 Continued

collection channel volume flux of $10 \text{ cm}^3/\text{s}$) designed to examine the fractionation of an arbitrary sample of particles in the diameter range of $300\text{--}400 \mu\text{m}$, the machine was set so that only particles below some definite settling velocity, corresponding roughly to an effective diameter of $340 \mu\text{m}$, would be collected from one port (the inner channel). Another outer channel returned the remaining particles for reprocessing to simulate the internal cascade, although this test had just a single pass through the centrifuge for about 90 minutes duration. Figure 4 shows that almost all of the particles collected from the inner channel had diameters below $340 \mu\text{m}$, the approximate diameter calculated from the trajectory of a single particle in a rotating channel flow. Examination of some of the seemingly aberrant, larger particles included in this group showed that each indeed was lighter than the average and actually had a settling velocity that fell below the cutoff value. (Particle diameters were measured under a microscope; densities and settling velocities were determined using solutions of glycerine and water.) Fractionation according to settling velocity was confirmed, and the concept of centrifugal spectroscopy was established as commercially feasible and broadly applicable.

The original goal was to prove the feasibility of the concept by developing a machine and a system that would separate and fractionate a mixture of particles with diameters roughly between $300\text{--}400 \mu\text{m}$ into 5, possibly more, precise size groups, in the ranges of $300\text{--}320 \mu\text{m}$, $320\text{--}340 \mu\text{m}$, etc. This objective awaits completion of the condensing centrifuge. In the

meantime, the prototype is employed to test performance with a number of different fluids and particle size distributions and to identify optimal operating conditions.

Although the fractionating capability of the spectrometer centrifuge is very good, a conclusion that is anticipated to hold in general, retrieval of the separated product can be substantially improved. The problem in the prototype seems to be accentuated by any short vertical surface where the sediment is particularly difficult to move. Design studies coupled with some exploratory but fairly simple experiments reveal the required modifications for greater yields. Several other changes will be made to improve the practical utility of the instrument.

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