A Radioisotopic Tracer Method for Measurement of Solids Concentration in a Settling Bed of Solids

A new and improved radiotracer technique provides information on both the particulate concentration and the movement during sedimentation of monodispersed and binary mixtures. In studies of sedimentation with 44 and 62 μm ion exchange particles, several solids concentrations, three fluid viscosities, and three mixtures with different ratios of the two size particles, the results all lie close to a correlation proposed by Richardson and Zaki.

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SCOPE

Sedimentation of particulates is an important operation in a number of industrial processes in which particles must be separated or removed from fluids. Theories and correlations used to analyze sedimentation operations have been based largely upon data obtained by observing a single interface between clarified fluid and the settling suspension. A new and improved radiotracer technique permits quantitative and continuous measurements of solids concentration within the slurry at different positions. This provides important new information on the concentration and movement of particles within the suspension. For instance, with binary suspensions the concentrations and settling velocities of both particles within the suspension can be inferred from radiotracer data. Such results are useful for testing and improving theories and correlations.

CONCLUSIONS AND SIGNIFICANCE

Although sedimentation is an important step in a number of industrial chemical processes, the theories and correlations used to design and model these systems are based largely upon simple experiments that follow the descending interface between settling particles and the particle-free supernate. An improved radiotracer technique has now been tested that can give additional insight into the sedimentation process by providing information on both the concentrations and movements of particles within the suspension. When studying sedimentation of binary mixtures, two settling regions could be observed: a lower region in which the larger particles were moving past the smaller particles, and an upper region which contained only smaller particles that had been passed by the larger particles. The concentration of particles could be determined in both regions. Studies with two sizes of ion exchange particles (44 and 62 μm dia.) settling individually and in three different mixtures showed settling rates which were very close to those predicted by a correlation proposed earlier by Richardson and Zaki. The agreement with the correlation held for both particle sizes and both settling regions, for solid volume fractions down to 0.4, and for viscosities between 1×10^{-3} and 5×10^{-3} Pa·s.

INTRODUCTION

Many industrial processes depend on sedimentation as the prime method to separate suspended solids from a slurry. Examples of sedimentation devices are static thickeners and clarification basins, as seen in coal preparation, wastewater treatment, coal conversion, and other industries. Since the beginning of the century, designs of sedimentation devices have been based on the classical theory developed by Coe and Clevenger (1916), later expanded and modified by Kynch (1952), and reviewed by Fitch (1979). According to this theory, it is assumed that four zones usually form (from the top downward):

Zone A, clear supernatant liquid
Zone B, initial concentration
Zone C, graded concentration
Zone D, sediment

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Both the Coe and Clevenger and the Kynch approaches are based on a force balance across a differential slice of thickener cross section. The classical assumptions used in simplifying the force balance equation are that local acceleration terms are negligible and that the solids stress is negligible. These assumptions lead to the equaton

$$U = U(C) \tag{1}$$

i.e., the settling velocity, U, is a function of solids concentration, C, alone. These considerations are part of what is normally referred to as flux theory.

Dixon (1977) proposed a theory of sedimentation that states that the local acceleration terms cannot be neglected in the force balance equation. Inclusion of these terms leads to the following functional relationship for the settling velocity:

$$U = U\left(C, \frac{DU}{Dt}\right). \tag{2}$$

Dixon concluded that there can be no flux-limiting region in the free settling zone of a thickener (zone B) and that the only zone in which concentration gradients can develop is zone D. Dixon has claimed that zone C, the zone of graded concentration proposed by Coe and Clevenger and supported by Kynch, cannot exist. To support this argument, Dixon et al. (1976) numerically implemented the force balance equation with and without the acceleration terms. The resulting simulation predicted Kynchian behavior when the acceleration terms were not included. However, when the inertial effects were included in the model, there was no indication of a zone of graded concentration.

A useful study would include measuring solids concentration profiles in batch settling tests to determine conclusively the nature of the solids in the settling zone. These tests should be performed under conditions where the characteristics of the particles (i.e., diameter, shape, and density) are controlled and known.

RADIOACTIVE TRACER TECHNIQUE

The classical method for acquiring sedimentation data is the batch settling test, in which a sample of slurry is allowed to settle while the zone A/zone B (A/B) interface height is recorded as a function of time. The recording yields the data necessary for using the Coe and Clevenger or the Kynch technique for thickener design. This method does not, however, provide information regarding the solids concentration profile throughout the tube during the test.

The method used in this study involves a radioactive tracer technique adapted to allow nonintrusive measurement of settling solids concentration. Similar techniques have been reported by Richardson and Shabi (1960) and Tory et al. (1978). Richardson and Shabi studied sedimentation of a polydisperse mixture of slate ground to a range of sizes between 0 and 17.5 μm dia. This material was subsequently irradiated to produce radioactive emissions between 0.75 and 1.0 MeV. Measurements were then made of solids concentration (proportional to radioactive count rate) as a function of time and height in a settling tube. Because a random sample of the solids was irradiated, measuring and differentiating the settling behavior of each particle size were not possible. Tory et al. presented a detailed mathematical analysis to examine the subtleties that may occur in measuring solids concentration with a collimated radiation beam. Their model was tested using an irradiated praseodymium oxalate slurry and a hard gamma signal, 1.57 MeV, emitted by ^{142}Pr. As with the Richardson and Shabi work, no attempt was made to differentiate the effects of particle shape and size on overall settling characteristics.

In the present study, the collimated-beam radioactive tracer technique was implemented with spherical particles of uniform density and either uniform diameter or a mixture of discrete diameters. The radioactive tracer was ^{57}Co, obtained as $CoCl_2$ in HCl solution from the Isotopes Sales Section at Oak Ridge National Laboratory.

The solids used to prepare the slurry for settling tests consisted of spherical Dowex 50W-X8 cation exchange resin prepared by Bio-Rad Laboratories. Ion exchange resins were particularly convenient for these studies because they are essentially perfect spheres, have uniform loading capacity for the radiotracer, and can accommodate any of several radiotracers. The ^{57}Co tracer was chosen for its gamma energy and decay half-life. The relatively low energy (0.12 MeV) was high enough to make self-shielding in the ion exchange resins and the sedimentation columns negligible, but it was low enough that the radiation could easily and effectively be shielded from the detector, except for the signal passing through the slit. The larger spheres were nominally 62 μm in dia. and the smaller particles were nominally 44 μm in dia.

EXPERIMENTAL

The experimental apparatus used to measure settling behavior with the radioactive tracer is shown schematically in Figure 1. The settling tube is a 75 cm long straight section of glass pipe having a 5.0 cm I.D., with a flared section welded onto the top and a fritted glass disc welded onto the bottom. Lead bricks are stacked between the column and a lead-shielded NaI-crystal detector. Slits (0.32 cm × 5.1 cm) are provided in some of the bricks at different positions. The slit provides a collimating port through which the NaI-crystal detector views the column. A slit 0.64 cm high × 5.1 cm long × 5.1 cm deep is also present in the lead shield surrounding the NaI crystal. By proper stacking of the lead bricks and correct positioning of the NaI crystal, essentially any position along the column axis can be viewed by the detector. The gamma-ray detection and counting equipment consists of a 7.62 cm dia. NaI crystal coupled to a photomultiplier tube. The signal from the photomultiplier tube is further processed in a multichannel analyzer (Tracor Company, model TN-1706).

The experimental technique is especially useful for studying *mixtures* of particles and, in principle, permits individual particles to be studied separately, with different tracers on each type of particle. However, when ion-exchange resins are used, only one tracer can be used on each type of resin (cation or anion). Although the concentration of electrolyte in the water can be very small, it cannot be eliminated completely. Thus, different tracer ions added to different resin components would

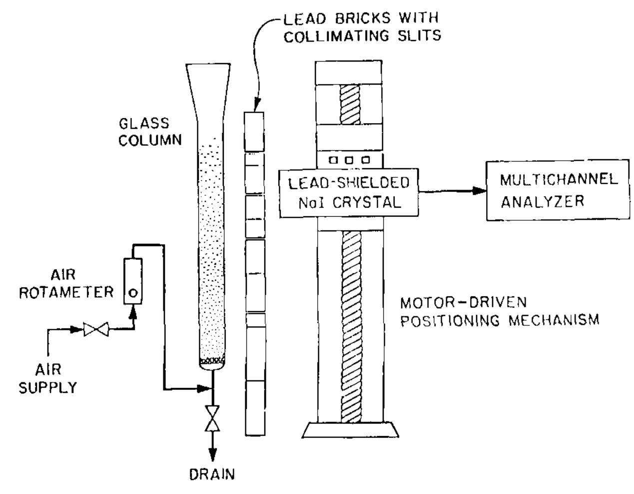

Figure 1. Apparatus for radioactive tracer studies of batch sedimentation.

slowly exchange, and eventually both tracers would be found on all resin particles. The two particle sizes had mean diameters of 62 and 44 μm. Three mixtures of these particles were studied: 10, 50, and 70 wt. % of the 62 μm particles (or 90, 50, and 30 wt. % of the 44 μm particles).

RESULTS

Initial Slurry Uniformity

To permit analysis of the sedimentation measurements, it was of prime importance to characterize the nature of the suspension at initial time (i.e., immediately after mixing air was stopped). To simplify the analysis, it was desirable to produce a uniform initial suspension with a minimal axial solids concentration gradient.

To measure the slurry uniformity, two different procedures were followed. The first method involved using the resin tagged with ^{57}Co. Slurry was mixed at a fixed air flow rate, and count-rate determinations were made immediately after air flow stopped. These measurements were repeated at several positions along the column axis and for three different total-solids concentrations. The second technique used nonradioactive particles in a column equipped with sample ports; after the air flow was stopped, samples were withdrawn from the sample ports and analyzed for solids content. Three sets of data were taken, corresponding to initial void fractions of 0.87, 0.93, and 0.95.

The results from both methods were in excellent agreement. Variations in solids concentration throughout the column were minimal ($\sim\pm7.5\%$ of the mean), with the expected slightly higher concentration near the bottom and the slightly lower concentration near the top. Within the accuracy of our measuring technique, there was no significant initial solids concentration gradient.

Solids Concentration Profile and Settling Velocity

Initial experiments were performed in which only one particle size was present in each test. The tests were made with the 44 μm dia. and the 62 μm dia. particles. For each particle size, three initial void fractions were tested (0.87, 0.93, and 0.95). Each combination of particle size and void fraction was tested at either five or six detector positions.

The results from one representative settling test are presented in Figure 2. The test was made with 62 μm dia. particles

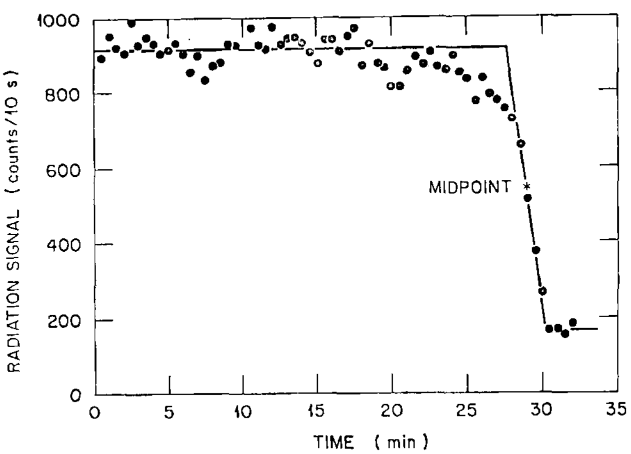

Figure 2. Sedimentation test for 62 μm dia. resin tagged with ^{57}Co, settling through water. Column height, 65 cm; void fraction, 0.95; detector positioned at 5.8 cm.

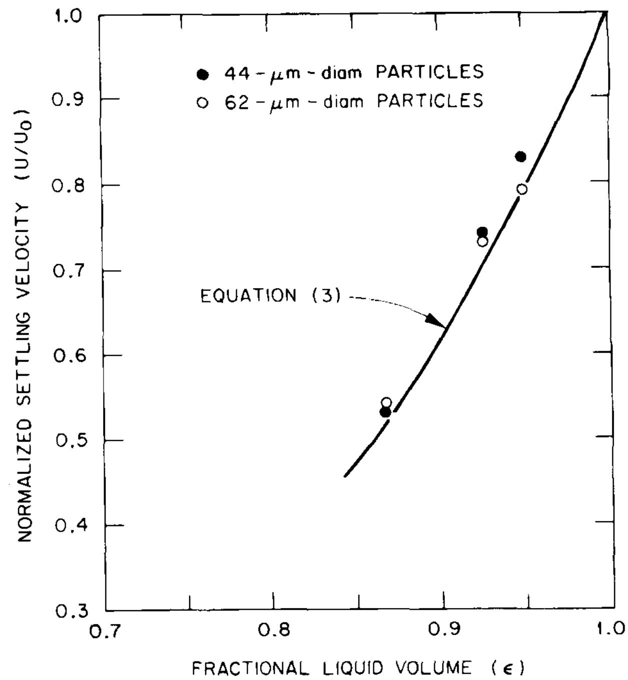

Figure 3. Settling velocity as a function of void fraction for 44 and 62 μm dia. particles compared with the Richardson-Zaki correlation.

at an initial void fraction of 0.95. The detector was positioned 5.8 cm above the bottom of the column. The 10 s count-rate determinations, which were made every 30 s, are plotted as a function of time. Qualitatively, the data in Figure 2 can be described as follows. The initial portion, from 0 to $\sim$25 min, is characterized by a constant value of count rate. Since the solids concentration is proportional to the count rate, the data in Figure 2 imply that the concentration of solids passing the detector did not change from time 0 up to 25 min. From 25 to 30 min, the count rate dropped rapidly from this constant value to the background value of 170 counts every 10 s. For time greater than 30 min, the count rate remained essentially unchanged at the background level (although this is not shown explicitly here). The curvature and finite slope in Figure 2 represent both the slight nonuniformity in particle diameters and the effective width of the collimating slit for the detector.

The location of the rapid drop in count rate can be used to calculate the settling velocity of the A/B zone interface. The time corresponding to the midpoint of this section is interpreted at the time at which the A/B interface midpoint passed the detector. Dividing this value into the distance from the detector to the initial slurry height yields the sedimentation velocity. For the data in Figure 2, the settling velocity was 2.03 cm/min. The same calculation can be performed for each detector position, which yields a series of values for settling velocity averaged over the appropriate portion of the column.

The initial data measured with particles of uniform size are summarized in Figure 3, where the measured values for settling velocity are plotted as a function of initial void fraction. The settling velocity was a nonlinear function of void fraction and is described well by

$$U = U_o \epsilon^{4.85}, \tag{3}$$

reported by Richardson and Zaki (1954) for settling of suspensions in the hindered settling regime. The sedimentation velocity remained constant, and there was no indication within the resolution of these data that the velocity decreased near the end of the settling period.

Mixtures of Two Particle Sizes

When a binary mixture was used, the particle motion could be inferred from count rate vs. time profiles such as the one shown in Figure 4. Note that there were three periods during which count rates were approximately constant: an initial period when a mixture of both the larger and smaller particles was settling past the detector, a (usually) shorter period when only smaller particles were passing the detector. These periods were separated by two interfaces. The first interface separated the mixture of larger and smaller particles from a region with only the smaller particles, and the second interface separated the region with only smaller particles from the supernatant above the settling solids. Conventional visual studies such as those described by Coe and Clevenger (1916) detected and followed only the second, or upper, interface. In more recent years sedimentation of binary mixtures of particles have been studied using particles with different colors, and the position of the intermediate interface can be estimated visually (Smith, 1965; Lockett and Al-Habbooby, 1973, 1974; Mirza and Richardson, 1979). The radiotracer technique used in this study provides additional quantitative information on the concentration of particles between the interfaces.

The time required for each interface to cross the detector determined the velocity of that interface. Measurements taken with the same mixture but with the detector located at different vertical positions showed that the velocity of each interface was constant.

Note that the initial horizontal region of Figure 4 indicates that the total solids concentration in the lower settling region (both larger and smaller particles) remained constant; this is an important point. The count rate is a measure of total-tracer and thus total-solids concentration, and the constant total-solids concentration and the constant settling rate in the lower region indicate that there was a constant solids composition in this region. Furthermore, this composition must have been the same as the initial mixture. Such information on the concentration of particles during the settling process is not available from earlier measuring techniques that only follow the movement of interfaces.

Obviously, the larger particles were settling through the slurry in the lower region faster than the smaller particles, but the composition in the region remained constant and uniform. The lower settling velocity of the smaller particles in this region means that some smaller particles were passed by the interface and formed the second (upper) region, which contained only smaller particles.

Measurements taken with the detector at different elevations show that the upper interface also moved at a uniform rate. (This is not obvious in Figure 4 because in this particular case the upper interface passes the detector relatively quickly after the lower interface.) The steady signal observed for this upper region also implies that the concentration of smaller particles was constant with time. The concentration of particles in the upper region can be estimated from the detector signal while that region passes before the collimated detector. As noted earlier, shielding is minimal and the signal is proportional to the solids concentration. For instance, in Figure 4 the count rate in the lower region is approximately 5,600 counts/10 s, and the count rate in the upper region is approximately 4,600 counts/10 s. Thus, the solids concentration in the upper region is 4,600/5,600 times the concentration in the lower region, and the concentration in the lower region is known since it is the same as that in the original suspension. When the solids concentrations are expressed as volume fractions, the void fraction in each region is simply one minus the solids concentration.

The settling velocity of smaller particles in the lower region in the presence of the larger particles can be determined by a mass balance on smaller particles across the lower (first) interface. Since the lower interface travels downward at velocity U_L and the smaller particles in the lower region settle at velocity U_s, the rate at which particles in the lower region are overtaken by the descending lower interface is $C_s(U_L - U_s)$. The upper

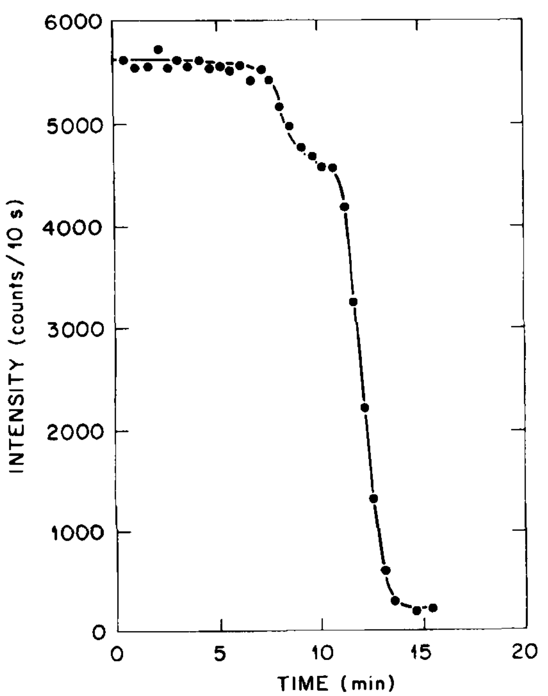

Figure 4. Radiation intensity at a fixed position for a 50:50 mixture of 62 and 44 μm dia. particles.

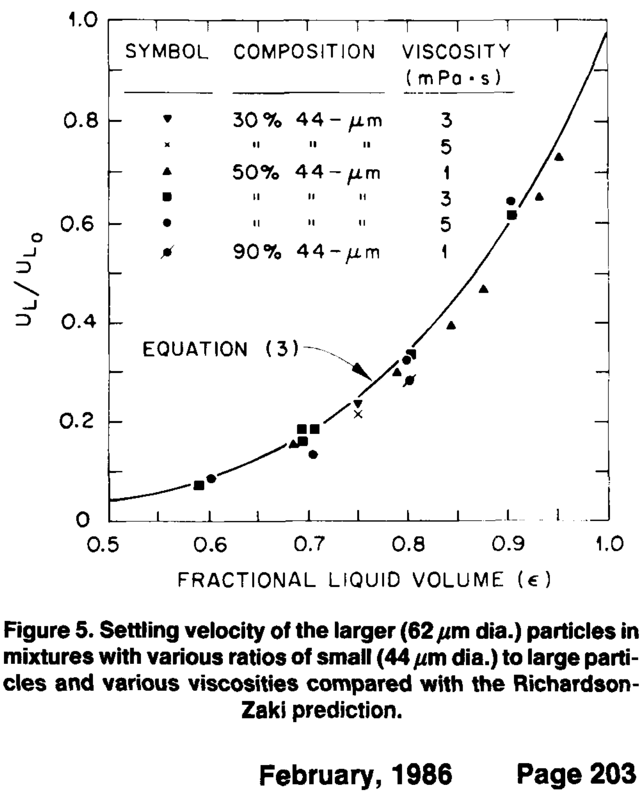

Figure 5. Settling velocity of the larger (62 μm dia.) particles in mixtures with various ratios of small (44 μm dia.) to large particles and various viscosities compared with the Richardson-Zaki prediction.

Table 1. Summary of Data and Results

Conditions and results	Run No. 1	2	3	4	5	6	7	8	9	10	11	12
% 44 µm	50	50	50	50	50	50	89.1	89.1	89.1	89.1	89.1	30
% 62 µm	50	50	50	50	50	50	10.9	10.9	10.9	10.9	10.9	70
μ (Pa·s×10^3)	0.914	0.914	0.914	0.914	0.914	0.914	0.906	0.906	0.912	0.914	0.914	2.91
ρ_s (g/cm^3)	1.205	1.205	1.205	1.205	1.205	1.205	1.205	1.205	1.205	1.205	1.205	1.256
ρ_f (g/cm^3)	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.090
ϵ_L	0.951	0.930	0.873	0.842	0.789	0.684	0.700	0.700	0.901	0.801	0.801	0.75
U_{s_o} (cm/s)	0.0237	0.0237	0.0237	0.0237	0.0237	0.0237	0.0237	0.0237	0.0237	0.0237	0.0237	0.00602
U_{L_o} (cm/s)	0.0470	0.0470	0.0470	0.0470	0.0470	0.0470	—	—	—	—	0.0470	0.0119
U_L (cm/s)	0.0342	0.0303	0.0217	0.0182	0.0149	0.0070	—	—	—	—	0.0132	0.00263
U_u (cm/s)	0.0192	0.0174	0.0133	0.0113	0.0092	0.0047	0.0041	0.0040	0.0151	0.0091	0.0091	0.00179
ϵ_u	—	0.963	0.915	0.895	0.875	0.784	—	—	—	—	0.811	0.784
U_s (cm/s)	—	0.0168	0.0106	0.0091	0.0083	0.00382	—	—	—	—	0.00903	0.0002
U_L/U_{L_o}	0.727	0.645	0.461	0.387	0.298	0.149	—	—	—	—	0.282	0.0221
U_u/U_{s_o}	0.810	0.739	0.563	0.479	0.387	0.197	0.170	0.167	0.635	0.384	0.385	0.291
U_s/U_{s_o}	—	0.708	0.448	0.384	0.351	0.161	—	—	—	—	0.369	0.015

Conditions and results	Run No. 13	14	15	16	17	18	19	20	21	22	23
% 44 µm	30	50	50	50	50	50	50	50	50	50	50
% 62 µm	70	50	50	50	50	50	50	50	50	50	50
μ (Pa·s×10^3)	5.15	3.04	3.04	3.04	2.99	2.97	5.22	5.18	4.26	4.64	3.27
ρ_s (g/cm^3)	1.269	1.256	1.256	1.256	1.256	1.256	1.269	1.269	1.269	1.269	1.256
ρ_f (g/cm^3)	1.120	1.090	1.090	1.090	1.090	1.090	1.121	1.121	1.121	1.121	1.090
ϵ_L	0.75	0.701	0.695	0.695	0.800	0.900	0.900	0.800	0.600	0.704	0.589
U_{s_o} (cm/s)	0.00305	0.00527	0.00527	0.00527	0.00587	0.00592	0.00332	0.00303	0.00368	0.00337	0.00535
U_{L_o} (cm/s)	0.00603	0.0115	0.0115	0.0115	0.0117	0.0117	0.00593	0.00602	0.00730	0.00668	0.0106
U_L (cm/s)	0.00130	0.00210	0.00212	0.00188	0.00378	0.00723	0.00380	0.00195	0.00058	0.00088	0.00078
U_u (cm/s)	0.00088	0.00143	0.00146	0.00135	0.00262	0.00473	0.00208	0.00132	0.00035	0.00070	0.00035
ϵ_u	0.781	0.725	0.744	0.747	0.857	0.947	0.942	0.862	0.633	0.731	0.620
U_s (cm/s)	0.00007	0.00087	0.00100	0.00100	0.00212	0.00460	0.00182	0.00108	0.00015	0.00055	0.0000
U_L/U_{L_o}	0.215	0.183	0.185	0.164	0.325	0.616	0.640	0.324	0.080	0.132	0.074
U_u/U_{s_o}	0.292	0.272	0.275	0.256	0.446	0.800	0.698	0.434	0.095	0.208	0.090
U_s/U_{s_o}	0.020	0.166	0.190	0.190	0.413	0.778	0.601	0.355	0.042	0.163	0.00

interface moves downward with velocity U_u. Thus, the upper region expands in length at a rate of $U_L - U_u$. Equating the rate at which small particles in the lower region are overtaken by the lower interface with the growth of the upper region gives

$$C_s(U_L - U_s) = C_u(U_L - U_u)$$

or

$$U_s = \frac{U_u C_u - U_L(C_u - C_s)}{C_s}. \qquad (4)$$

Thus, measurements such as those shown in Figure 4 can be used to determine three settling velocities: 1) smaller particles settling alone in the upper region; 2) larger particles in the lower region settling in the presence of smaller particles; and 3) smaller particles in the lower region settling in the presence of larger particles. Furthermore, the solids concentration and composition are known in both regions; this is the strength of the new radiotracer technique.

The results for three different mixtures of the two sizes of particles at various viscosities are tabulated in Table 1 and shown graphically in Figures 5 through 7. Viscosity was varied by adding glycerol to the settling medium, water. Table 1 shows the experimental conditions, the experimentally measured Stokes settling velocities (U_{s_o} and U_{L_o}), the original void fraction (ϵ) determined by the quantity of solids used in the experiment, the void fraction in the upper region (ϵ_u) determined from the count rates measured during the passage of the upper and lower regions, and the experimentally determined velocities of the lower and upper region interfaces. The settling rates for the larger particles in the lower region (U_L) and for the smaller particles in the upper region (U_u) were simply the velocities of the interfaces; the settling rates for the smaller parti-

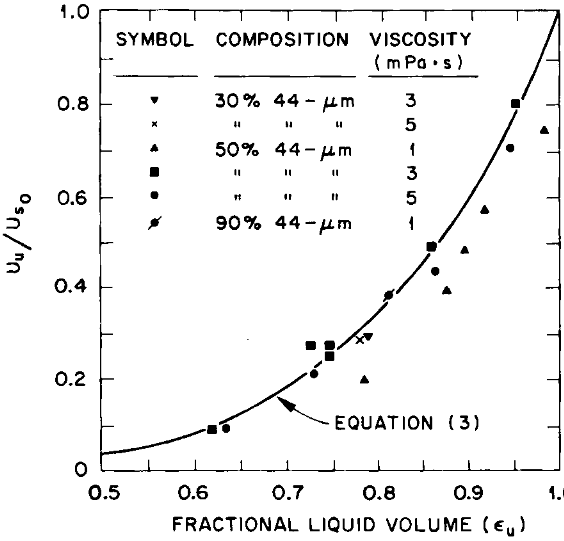

Figure 6. Settling velocity of the smaller (44 μm dia.) particles in the upper region, which has been cleared of larger particles, in bimodal mixtures compared with the Richardson-Zaki prediction (based on liquid volume fraction obtained from experimental measurements of solids concentration).

cles in the lower region (U_s) were calculated using Eq. 4.

Note that all the results could be fitted well with a Richardson-Zaki correlation (Eq. 3). This is in agreement with more recent results reported by Mirza and Richardson (1979). The most accurate data are shown in Figure 5. The velocity of the lower interface, U_L, was measured directly. This is also the velocity of the larger particles in the lower region. The steady signal of the lower region indicates that the solids fraction was the same as the original suspension.

The settling velocity in the upper region was also measured

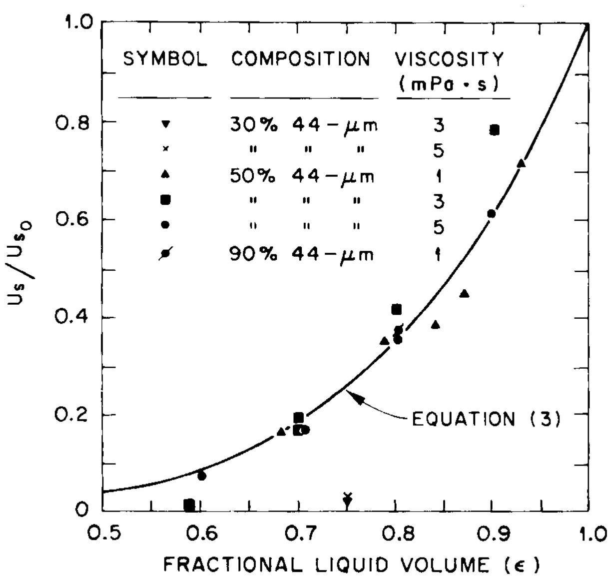

Figure 7. Settling velocity of the smaller (44 μm dia.) particles in bimodal settling region (lower) compared with the Richardson-Zaki prediction.

directly. However, the solids fraction, measured by the intensity of the signal, was not known as accurately, usually because the shoulder (second horizontal portion) of the signal (see Figure 4) often could not be measured as accurately. The shoulder was especially short with a low concentration of smaller particles and with the detector positioned closer to the top of the sedimentation column.

Settling velocities of smaller particles in the lower region (Figure 7) were most prone to error because they were obtained by material balance using Eq. 4. Errors in any of the measurements used in the balance would affect the results. Nevertheless, practically all the data fall along a single curve, which has the Richardson-Zaki form. Only three points deviate significantly from the curve.

CONCLUSIONS

A new experimental apparatus has provided interesting and useful data on velocities of uniform and nonuniform particles within a settling swarm. Agreement of the results from binary mixtures of spheres with the Richardson-Zaki equation is excellent for both uniform particles and nonuniform mixtures over the range of particle concentrations and compositions studied. If the relatively simple Richardson-Zaki correlation holds for more complex, polydispersed systems, new sedimentation design methods that supplement the traditional Coe-Clevenger (1916) approach will be possible. With uniform particles there was no indication of transition zones as suggested by Coe and Clevenger, but with binary mixtures two settling zones were observed. Thus, with many different particle sizes, a number of zones could be expected.

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NOTATION

- C = volume concentration of solids
- C_u = concentration of smaller particles in the upper region
- C_s = concentration of smaller particles in the lower region
- D = substantive derivative
- t = time
- U = settling velocity
- U_L = settling velocity of the larger particles in the lower region
- U_s = settling velocity of the smaller particles in the lower region
- U_{L_o} = settling velocity of the larger particles at infinite dilution
- U_{s_o} = settling velocity of the smaller particles at infinite dilution
- U_o = settling velocity at infinite dilution
- U_u = settling velocity of the smaller particles in the upper region (velocity of the upper interface)
- ϵ = void (liquid) fraction in the suspension

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