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EFFECT OF ACCELERATION FORCE ON THE SETTLING RATES OF FLOCCULATED THORIA SUSPENSIONS

D. M. Eissenberg

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D. M. Eissenberg

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NOMENCLATURE

C	drag coefficient
D_e	effective floc diameter assuming smooth sphere
g	local acceleration in units of 32 ft/sec^2 = 1 g
h	length of thoria stringer caused by channeling
r	radius of thoria stringer caused by channeling
S	solidity (volume fraction solids) of suspension
S_c	solidity at the onset of compaction settling
S_e	effective solidity = $\frac{S}{S_{floc}}$
S_{floc}	solidity of a floc
U	settling rate observed
U_o	settling rate at infinite dilution as calculated from a solidity function equation
α	$\left(\frac{\text{volume of water}}{\text{volume of solid}}\right)_{floc}$
μ	viscosity of water = 1 cp
ρ_l	density of water = 1 g/cc
ρ_f	density of settling unit
ρ_s	density of thoria = 10 g/cc
$\phi(S_e)$	solidity function = $U/U_o = (1 - S_e)^2\, 10^{-1.82\, S_e}$ as determined by Steinour

EFFECT OF ACCELERATION FORCE ON THE SETTLING RATES OF FLOCCULATED THORIA SUSPENSIONS

D. M. Eissenberg

ABSTRACT

The effect of varying the acceleration force imposed on several sedimenting flocculated thoria suspensions was briefly studied. It was found that the Stokes' law prediction of a linear relation between the hindered-settling rate and the acceleration is not valid for the slurries tested, probably due to channeling of the escaping water. The settling rate in hindered settling was found usually to be proportional to the 0.65 power of acceleration.

Compaction-settling rates were initially independent of acceleration. The rate-controlling factor was probably the vibrations of the centrifuge during operation.

In Appendix II it is shown that channeling occurs in the hindered settling of thoria slurries at gravitational acceleration. It is believed that channeling is also involved in the deviation from Stokes' law of settling rates at high acceleration.

1. INTRODUCTION

Thermal breeder reactors require, for the fertile blanket region, a high concentration of thoria in a form which can be satisfactorily cooled and which can be removed periodically for processing. Thoria suspensions in high-temperature water provide one means for achieving a satisfactory blanket if the thoria can be maintained in suspension adequately. This can be accomplished generally by circulating a slurry of small particle size. However, it has been observed that for pure thoria the surface chemical nature of the thoria particles causes agglomeration and rapid settling-out of the suspension. Chemical dispersants in general do not prevent agglomeration (flocculation) at the proposed temperature of a suitable blanket (250°C). It is therefore essential that relationships be developed between the rate of sedimentation of unstable suspensions (suspensions that tend to agglomerate) and the environment to which the suspensions are subjected.

In this report the effect on the sedimentation rate of flocculated thoria of an imposed acceleration field is studied. The observations may be useful in clarifying the mechanism of sedimentation, as well as being directly applicable to predicting settling rates in equipment where centrifugal forces are applied.

Rates of sedimentation for many types of suspensions have been investigated experimentally and analytically. It has been found that three essentially different types of settling occur, depending on the volume fraction of the suspension which is occupied by the settling units. These settling units can be either discrete solid particles or flocs; the latter consist of loosely packed collections of discrete particles and all the non-free-draining (immobile) water in the interstices between particles.

For dilute suspensions, when the volume fraction occupied by settling units (effective volume fraction solids) S_e is less than some minimum value (on the order of 0.1), the units will settle individually at the expected rates based on the individual settling-unit parameters (size, density, shape, roughness, etc). This effect is utilized, for example, in the determination of particle-size distribution. In very dilute flocculated suspensions floc formation is hampered, causing most of the settling units to be the discrete particles rather than flocs.

At higher effective volume fraction solids (between 0.1 and 0.6), the expected distribution of settling rates is no longer apparent. Instead, all of the suspension settles at the same rate, dependent on the initial concentration, and in fact this rate is constant with time. This uniform-settling-rate region is designated as hindered settling and is caused by hydrodynamic interaction between settling units. That is, adjacent particles or flocs are sufficiently close to one another so that their velocity streamlines interact, the particles or flocs thus tending to drag each other down and tending to make the over-all terminal velocity greater than the terminal velocity of the discrete particles or flocs. The observed hindered-settling rate at this intermediate concentration is often reduced to less than the single-particle settling rate, however, in the case of settling in vertical tubes, by an upward flow of water. The water comes from two sources--water equivalent to the volume displaced by a settling unit, and water released from the interstitial volume of a floc when that floc is crushed at the bottom of the settling container by the weight of flocs above it.

When the volume fraction occupied by the settling units is increased to a sufficiently high value (approximately 0.60 in the case of uniform spheres, but higher for flocs, since the flocs are neither spheres nor rigid) hindered settling can no longer take place. For nonflocculated uniform suspensions, all settling soon ceases. For

flocculated suspensions, however, depending on the structural strength of the floc, further settling can take place. This settling of flocculated suspensions is called compaction settling, and is due to collapsing of the floc structure.

1.1 Hindered Settling

The hindered-settling rate of flocculated thoria slurries has been studied at room temperature and at elevated temperatures as a function of slurry properties (refs 1 and 2, for example). On the assumption that floc size and density do not vary with concentration, a method was developed[1] for calculating apparent floc size and density based on Stokes' law validity:

$$\frac{U}{U_o} = \phi(S_e) = (1 - S_e)^2 \, 10^{-1.82 \, S_e}, \qquad (1)$$

where $$U_o = \frac{D_e^{\,2}(\rho_f - \rho_l)g}{18\mu} = \frac{D_e^{\,2}(\rho_s - \rho_l)g}{18\mu(1 + \alpha)} .$$

If D_e and α are constant, it is possible to calculate them from settling rates at two concentrations.

This method uses the particular solidity function ϕ experimentally determined by Steinour,[3] which assumes:

1. no structural resistance (taken to mean no yield value) of the suspension,

2. settling-unit size and density constant with change in concentration.

1.2 Compaction Settling

No studies have been found which deal with compaction-settling rates. The following qualitative observations can be made regarding suspensions in compaction settling.

1. The concentration at the onset of compaction settling can be obtained readily from settling-rate experiments (see Fig. 6, Appendix II).

2. Slurries initially at concentrations close to the concentration at the onset of compaction settling exhibit nonreproducible settling-rate curves.

3. Slurries which are in hindered settling show visible floc structures, whereas slurries in compaction settling appear homogeneous.

4. Compaction-settling rates decrease as settling proceeds in contrast to hindered-settling rates, which are constant with time.

1.3 Effect of Acceleration

The purpose of this study was to elucidate the effect of acceleration (variation in g) on the settling rates of thoria suspensions. It is noted that for the correlation of hindered-settling rates presented in Sec. 1.1, the effect should be linear, provided that the settling units are in the Stokes' law regime.

In the experiments described in this report, thoria slurries were subjected to accelerations of up to 30 times the acceleration of gravity, both in the compaction-settling and hindered-settling regions.

2. DESCRIPTION OF EQUIPMENT

2.1 Centrifuge

The centrifuge (Fig. 1) was fabricated of steel pipe. It consisted of two arms of 1/4-in. sched-80 pipe directly coupled to the vertical shaft of a variable-speed universal gear motor (Bodine type CRGL-2). The extremities of the arms were hinged and were fitted with sample holders made from 12-in. lengths of 1-in. sched-40 pipe. The mean radius of the sample during centrifuging was 18 in. The motor and assembly were mounted on a tripod which was braced to the wall. The entire assembly was contained in a safety shield. Rotational speed settings were made by varying the input voltage to the motor with a Variac. The average rotational speed was calculated either by counting the number of rotations during the entire centrifuging period or by counting revolutions during portions of the run. The highest rotational speed that could be measured by this means was approximately 200 rpm, which was equivalent to 30 times the acceleration of gravity acting on the sample.

2.2 Sample Tubes

The settling rates were determined in glass tubes. Initially, 16-mm-ID test tubes were used, and the slurry-water interface height was measured by comparison with a ruler held next to the tube. The sample-tube holder was modified later to permit use of 10-ml graduated cylinders (approx. 12.5-mm ID).

The sample tubes were held in the holders by large rubber stoppers, which fit into the threaded end of the sample holders and could be removed instantly upon stopping the centrifuge for rapid reading of slurry interface location.

3. DESCRIPTION OF EXPERIMENTS

Two groups of experiments were performed. The first involved three slurries (Table 1). The concentration chosen for each slurry

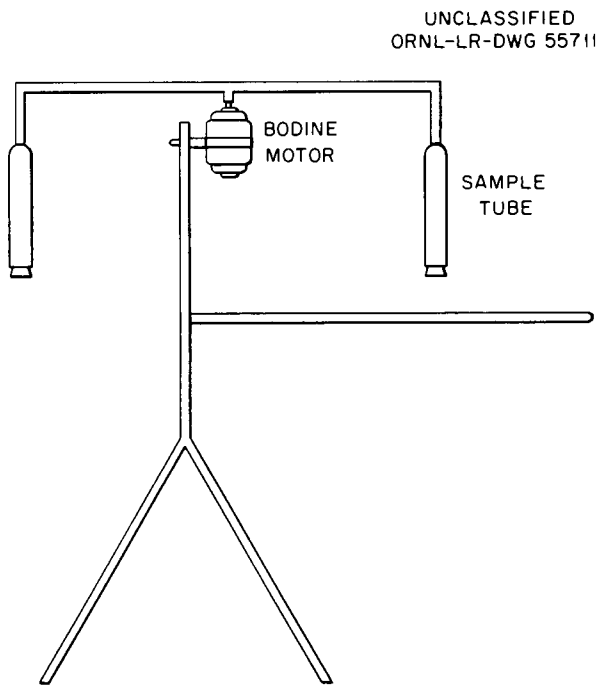

Fig. 1. Low-Speed Centrifuge.

Table 1. Slurry Properties

Slurry Designation	Particle Size (μ)	Firing Temp. (°C)	Comments
D-41	7	800	Oxalate precipitated; umpumped
LO 35-37	1.8	1600	Oxalate precipitated; umpumped
Cubes	3-5	----	See ref 4; umpumped
MO-56, 57	3.5	1050	Pumped 880 hr

(approx. 800 g of ThO_2 per liter) was sufficiently high so that the settling was assumed to be in the compaction region. The subsidence of the interface was followed for approximately 1 hr at each rotational speed.

Before starting an experiment, the slurry sample tube was put into one arm of the centrifuge and the opposing centrifuge arm was filled with sand so that the centrifuge rotated with no serious vibration. Then the Variac was adjusted to give the proper rotational speed, measured by counting revolutions in a given time. The centrifuge was stopped; the sample was removed, shaken vigorously, and replaced; and the centrifuge was started immediately. The rotational speed was checked periodically during a run. Readings were made by stopping the centrifuge rapidly, removing the sample, reading the interface height with a ruler, replacing the sample, and continuing centrifuging. Since the normal (1 g) settling rate was low compared to the centrifuge settling rates, the time taken for readings was ignored. The results are plotted in Fig. 2 as interface subsidence vs time.

The second group of experiments performed involved slurry MO-56, 57 (Table 1) at two concentrations (533 and 478 g of ThO_2 per liter), which were considered to be initially in the hindered-settling region. Since in this region the settling rates were much faster than in compaction settling, the methods of measurement were different. After initial balancing and speed adjustments, the centrifuge was started by turning on the motor and rotating the centrifuge arms by hand to increase speed quickly. Rapid stopping was accomplished by hand braking of the shaft. The rotational speed was calculated by counting the total number of revolutions in the total run time. If, because of variations in hand-starting torque, there was a different total number of revolutions in the predetermined run time, then that run was discarded. Immediately upon stopping the centrifuge, the sample tube was removed and the interface location was determined. The sample tubes were 10-ml graduated cylinders to facilitate rapid reading of the slurry-water interface. The maximum centrifuging time for each acceleration was chosen so that the slurry-water interface did not fall below a predetermined value, which was chosen to keep the slurry in the hindered-settling region.

In order to determine settling rates, duplicate experiments, except for variation in time of centrifugation, were carried out for each rotational speed. The settling rate was taken as the difference in the final height of the slurry-water interface divided by the difference in time of centrifugation for two runs of differing time. The mean height of at least three runs was used for each time interval, and the time intervals were chosen so that the interface height after centrifuging was within the same specified limit for all the rotational speeds. The time required for starting and stopping the centrifuge and for removing the sample tube and reading the interface height was taken to be constant throughout a run; therefore, assuming that the

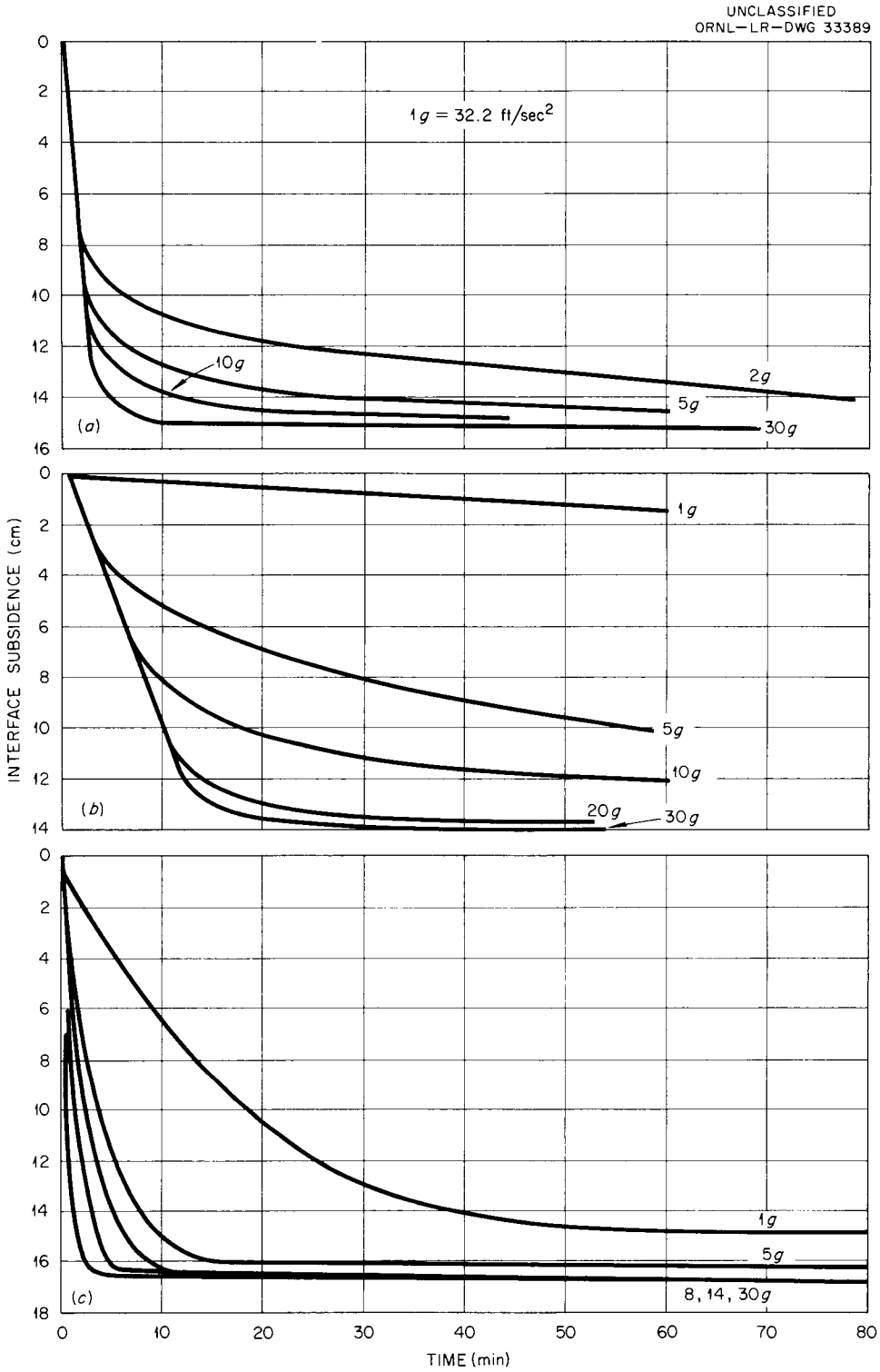

Fig. 2. Effect of Acceleration Forces on Initial Settling Rate of Three Thoria Slurries. (a) D-41 slurry; 800°C-fired; initial concentration, 790 g of ThO_2 per liter; particle size, 7 μ. (b) LO 35-37 slurry; 1600°C-fired; initial concentration, 790 g of ThO_2 per liter; particle size, 1.8 μ. (c) 3- to 5-μ cubical particles; initial concentration, 795 g of ThO_2 per liter.

slurry was in the hindered-settling region, the error due to settling during this time was canceled out by subtracting interface heights at different times of centrifugation.

The results of these experiments are plotted in Figs. 3 and 4 as settling rate vs acceleration.

4. DISCUSSION OF RESULTS

The results of the five experiments are presented in two forms. The compaction-settling data are plotted as interface height vs time with acceleration as a parameter. The hindered-settling data are plotted as acceleration vs settling rate. It is noted that the data for the ThO_2 cubes, originally considered to be compaction-settling data, can be plotted as acceleration vs settling rate (Fig. 5). It seems probable that this slurry was in reality in the hindered-settling region, even though the concentration of slurry was relatively high. This could have been due to a lower degree of flocculation of this slurry. These data will therefore be considered with the hindered-settling data.

4.1 Hindered Settling

The hindered-settling rate vs acceleration plots for the three hindered-settling slurries show similar linear relationships when plotted on log-log paper. However, for the correlation presented in Sec. 1.1, the log slope of the settling rate should be unity, whereas the observed values are less than 0.65. To account for this, two possibilities exist. First, the floc parameters (density and diameter) may themselves be functions of the settling rate such that the Stokes' law may be valid, but with varying settling-unit size. This, however, seems unlikely since if the Stokes' law assumptions were valid, the shear at the floc surface would be always low, and the influence on the floc size small.

The second possibility is that the Stokes' law assumptions are not valid for the conditions of this experiment. This would occur if the effective size of settling unit were sufficiently large. For the slurries of this experiment, in order to account for an observed 0.625-0.65 power dependence of settling rate on acceleration, assuming the settling units were randomly distributed spheres, and of a uniform size, the Reynolds number would have to be approximately 140 (Appendix I). The settling-unit diameter necessary to account for this Reynolds number is approximately the vessel diameter.

To account for this large an effective settling unit, it is necessary to consider channeling (see Appendix II). If channels were present, the settling unit would be essentially the space occupied by solids between channels, and would be equivalent to stringers of high

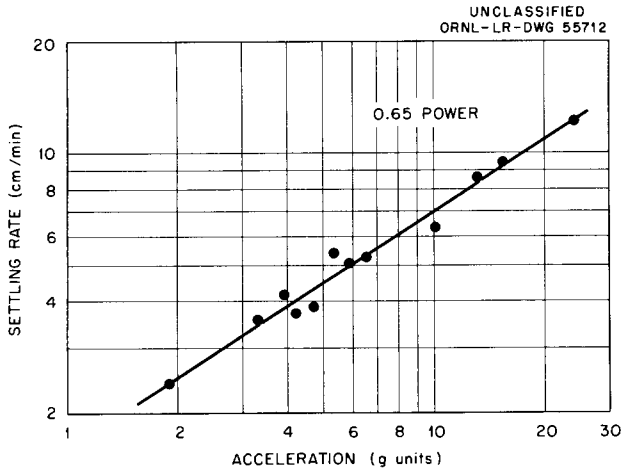

Fig. 3. Settling Rate of SM-6 Slurry at Various Accelerations, 478 g of ThO_2 per Liter.

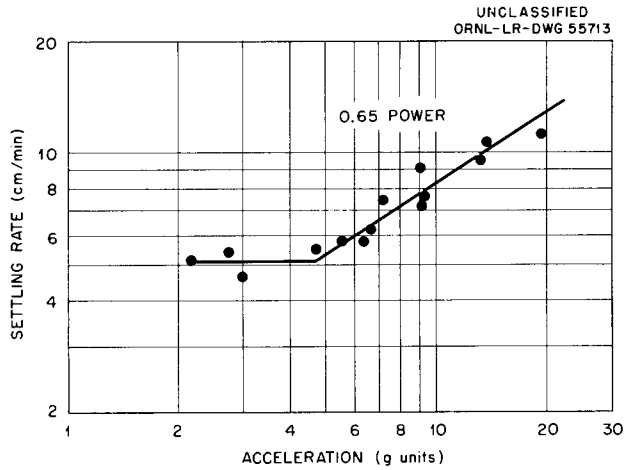

Fig. 4. Settling Rate of SM-6 Slurry at Various Accelerations, 533 g of ThO_2 per Liter.

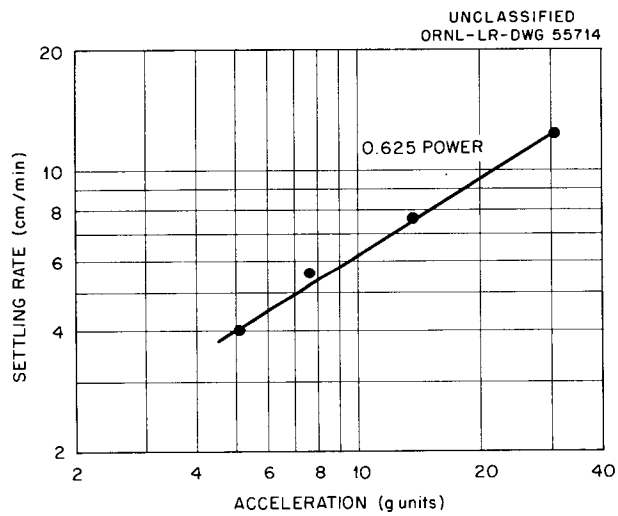

Fig. 5. Settling Rate of 3- to 5-μ Thoria "Cubes"; 795 g of ThO_2 per Liter, Tube Diameter, 2.21 cm.

aspect ratio. Calculations shown in Appendix I indicate reasonable diameters of stringers necessary to account for the observed deviation. Stringers due to channeling are clearly visible in slurries in the hindered-settling region. These stringers are approximately of the size calculated.

It is noted that with one slurry sample (Fig. 4), the log slope of the acceleration vs settling-rate curve was not constant. The slope above 4 g is consistent with that of the other two slurries. There is a possibility that this slurry was close to the region of compaction concentration so that at the low accelerations the results corresponded to compaction settling (see Sec. 4.2). This suggests the likelihood that if the acceleration were increased sufficiently high, the slurries described in Sec. 4.2 might also exhibit an effect of acceleration on initial settling rate.

4.2 Compaction Settling

The two slurries in compaction settling behaved in a similar manner--that is, the effect of applied acceleration was to prolong an initial, constant settling rate--but the settling rate was independent of acceleration in this experiment.

This implies that the settling rate is not initially controlled by the rate of water extrusion, since this would vary as the 0.5 to 1.0 power of the acceleration. Instead, it is likely that the initial settling is controlled by the rate of rearrangement of particles to positions of greater density within the compacted bed. This is controlled in turn by the vibrations received during centrifuging. The water released from the shrinkage of the compacted bed percolates or channels out of the bed. At sufficiently high concentrations (after compaction settling has proceeded sufficiently long), the controlling rate is determined by the balance between the acceleration driving force and the frictional drag on the escaping water, which is a function of concentration. This accounts for the variations in location of the break in the compaction-settling curves.

This picture is confirmed in part by the observation that any equilibrium settled bed of flocculated slurry, if settled without vibrations, can be made to settle further by applying sustained mild vibrations. Here again, the controlling condition is the ability to rearrange the particles to positions of greater density.

5. CONCLUSIONS

The use of a low-acceleration centrifuge has revealed an unexpected deviation from Stokes' law behavior for flocculated thoria suspensions. The results have been explained by assuming appreciable bypassing of flow (channeling), resulting in fluid turbulence. Channeling can also account for the shape of the settling-rate-concentration curve.

As a result, correlations based on flow behavior through beds of nonagglomerating suspensions should not be directly applied to flocculated suspensions without experimental determinations of their validity.

The use of a low-acceleration centrifuge provides such a test of the use of correlations involving the Stokes' law assumption of creeping flow.

Appendix I

CALCULATION OF EFFECTIVE REYNOLDS NUMBER BASED ON UNIFORM SPHERES

The data presented in Fig. 3, for example, can be expressed analytically as

$$g = K.U^{1.54}. \qquad (2)$$

If this relationship is inserted into the equation for the drag coefficient,[5]

$$C = \frac{4(\rho_f - \rho_1)g\ D_e}{3U^2\ \rho_1}\ (1 - S_e)^2. \qquad (3)$$

Then $$C = K_2 U^{-0.46}, \qquad (4)$$

and a plot of the drag coefficient vs Reynolds number would have a slope of -0.46 at the conditions of the experiment. This slope appears at a Reynolds number of 140, assuming that the settling units are smooth spheres.

However, if the assumption is made that the settling units are long cylindrical stringers, an average cylindrical diameter can be calculated by using a plot based on sphericity parameters such as in ref 5.

For example, assume a cylinder such that the height is 20 times the radius. The sphericity is then 0.580, and the value of the Reynolds number based on a volume equivalent sphere is 20. The mean velocity of liquid past solid is $U/(1 - S_e)$, since U alone is equivalent only to a superficial velocity. Thus

$$20 = \frac{D_e\ U_\rho}{\mu(1 - S_e)},$$

and $U = 7.0$ cm/min at 10 g for example,

$\rho_1 = 1$ g/cc,

$\mu = 0.85$ cp,

$S_e = 0.64$ based on $C_{floc} = 750$ g of ThO_2 per liter,

Thus the diameter of the volume equivalent sphere is $D_e = 0.525$ cm.

Since we assumed that h = 20 r, we can solve for a radius of cylinder with a volume equivalent to a sphere of 0.505 cm diameter.

$$\frac{4}{3}\pi\left(\frac{0.525}{2}\right)^3 = \pi r^2 \cdot 20\ r$$

r = 1.1 mm, h = 2.2 cm.

Thus, to account for the observed deviation from Stokes' law, we need to postulate channeling such that the mean distance between channels is approximately 2 mm and the length of channel is approximately 2 cm. These are considered to be reasonable values.

The estimated Reynolds number is in a range where one would expect turbulent, rather than Stokes' law, settling behavior. The physical size of the settling units is also reasonable on the basis of visual observations.

Appendix II

INFLUENCE OF CHANNELING ON HINDERED-SETTLING RATE

As noted in Sec. 1.1, the hindered-settling rate of a nonflocculated suspension can be correlated by means of the solidity function:

$$\frac{U}{U_o} = \phi(S_e) \qquad (1)$$

Many investigators[6] have calculated or experimentally determined the form of the solidity function. The particular function used in these calculations[3] was chosen because it falls approximately in a central position when compared with the other forms:

$$\phi(S_e) = (1 - S_e)^2 \; 10^{-1.82\, S_e} \qquad (1a)$$

To apply this equation to thoria suspensions, it is necessary to estimate a value of S_e.

In Sec. 1, it was observed that for flocculated suspensions, compaction settling can occur as a result of crushing of the flocs. It seems likely then, that the concentration corresponding to the onset of compaction settling is an estimate of the concentration within the flocs, since when that concentration is reached, there is no longer free water through which the flocs can settle. However, the concentration at the onset of compaction will be biased, tending to be higher than the floc concentration, since some of the flocs (near the bottom of the container) will be crushed by the time the top layer of slurry has reached the onset of compaction.

For the purpose of estimating the effective volume fraction solids, however, we shall use the mean value of the concentration at the onset of compaction as determined at several initial concentrations.

Then

$$S_e = \frac{S}{S_c},$$

where $S_e = \dfrac{\text{volume of settling units (including immobile water)}}{\text{total volume}}$.

Thus, if we find that the settling rate vs concentration curve experimentally determined has the form of Eq. (1a), we can infer that the assumptions of constant floc size and density are reasonable. Figure 6 is a typical plot of interface height vs time for slurry 200A-21A (an oxalate-precipitated 24-hr-digested 800°C-fired thoria with a 1-micron mean diameter) which had been pumped at elevated temperatures for over 2000 hr.

Figures 7 and 8 are plots of initial settling concentration vs settling rate for slurry 200A-21A and for slurry 200B-4B (an oxalate-precipitated 1600°C-fired thoria with a 1.8-micron mean diameter) which had been pumped for over 3000 hr. On the same plots are also shown the variation, with initial concentration, of the concentration at the onset of compaction for these slurries. If we choose as the floc concentration, the value of the onset of compaction concentration for the dilute suspensions, which appears to be constant, then the solidity function for each slurry can be calculated. This appears on each figure as a dashed line. Since the line represents ratios of settling rates, its slope is fixed, but not its intercept. It is observed that the solidity function matches the dilute-suspension settling rate for both suspensions. This also is the region in which the onset of compaction concentration was constant.

In the portion of the curve where the solidity function is followed, it is possible then to calculate an effective floc diameter, assuming the floc density previously calculated, and the extrapolated value of settling rate at infinite dilution. For the slurry shown in Fig. 7, the floc diameter is 120 microns, and for Fig. 8, 213 microns. Visual observation (Figs. 9 and 10) qualitatively confirmed these values. The deviation which occurs at concentrations above approx. 200 g of ThO_2 per liter can be attributed to channeling, which provides a low-resistance path for the escaping fluid. The channeling is analogous to channeling in aggregative fluidization, since in that case, also, the predicted superficial fluid velocity is much less than the observed. Thus hindered settling with channeling of flocculated (structured) suspensions can be considered analogous to aggregative fluidization in the same manner as hindered settling of nonflocculated suspensions in analogous to particulate fluidization.[7] Since the flocs tend to congeal in the regions bypassed by the channels, the effective flow-unit size and density increase with increasing concentration. At sufficiently high concentrations, the Stokes' law assumptions will not be valid, and the deviation obtained with the centrifuge would be expected.

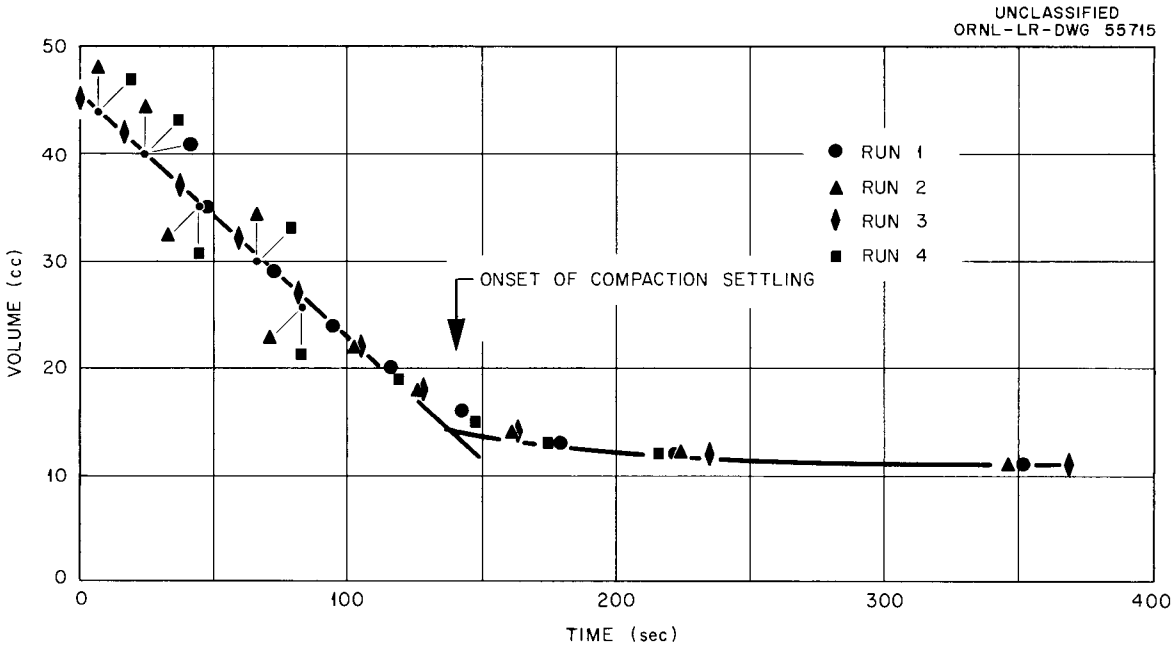

Fig. 6. Sedimentation Curves of 200A-21A Slurry at a Concentration of 183 g of ThO_2 per Liter. Four duplicate runs are shown. There are 3.145 cc per centimeter of height.

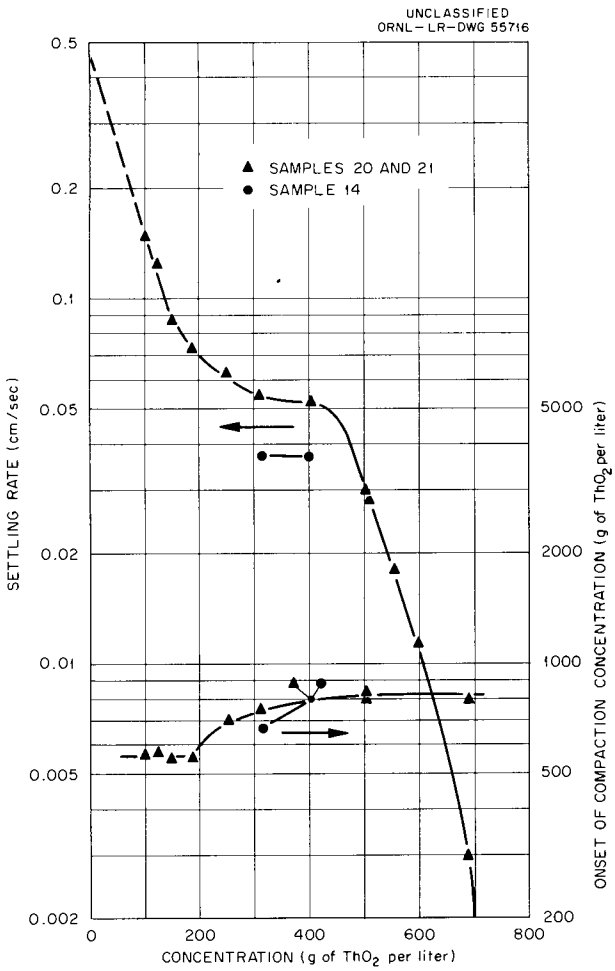

Fig. 7. Effect of Initial Concentration on Settling Rate and Onset-of-Compaction Concentration for 200A-21A Loop Samples. Dashed line represents extrapolation based on

$$S_e = \frac{\text{concentration}}{556} .$$

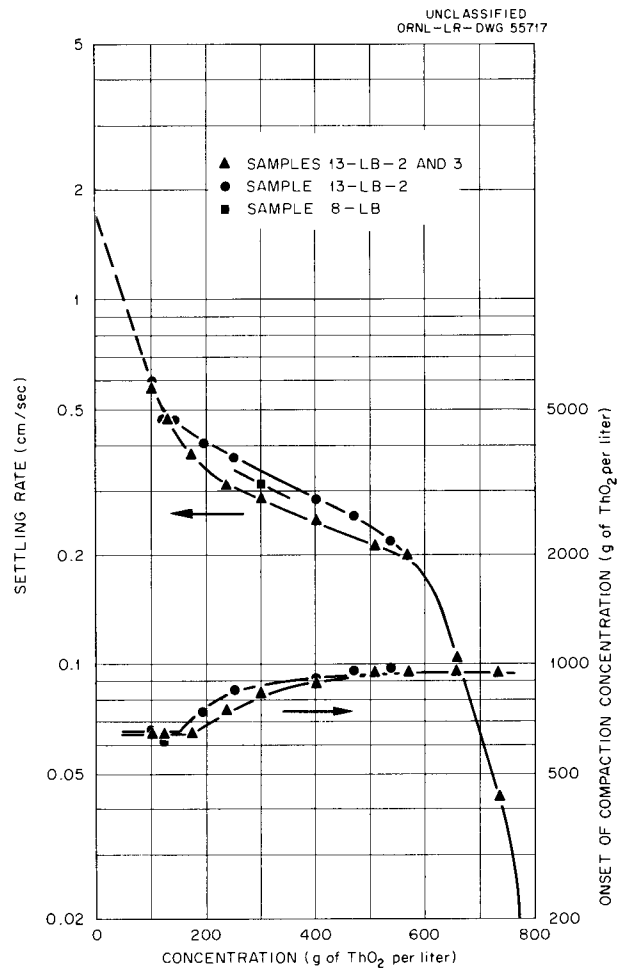

Fig. 8. Effect of Initial Concentration on Settling Rate and Onset-of-Compaction Concentration for 200B-4B Loop Samples. Dashed line represents extrapolation based on

$$S_e = \frac{\text{concentration}}{650} .$$

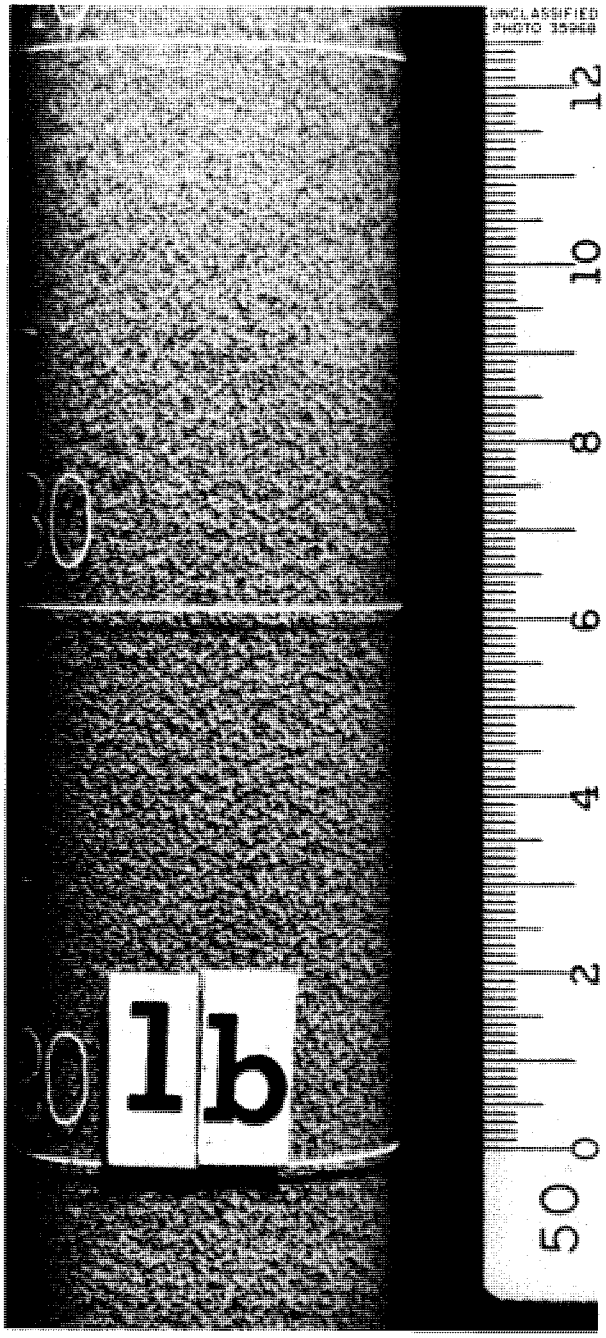

Fig. 9. Settling Flocs; 200B-4 Slurry at 100 g of ThO_2 per Liter. Floc diameter is 213 μ.

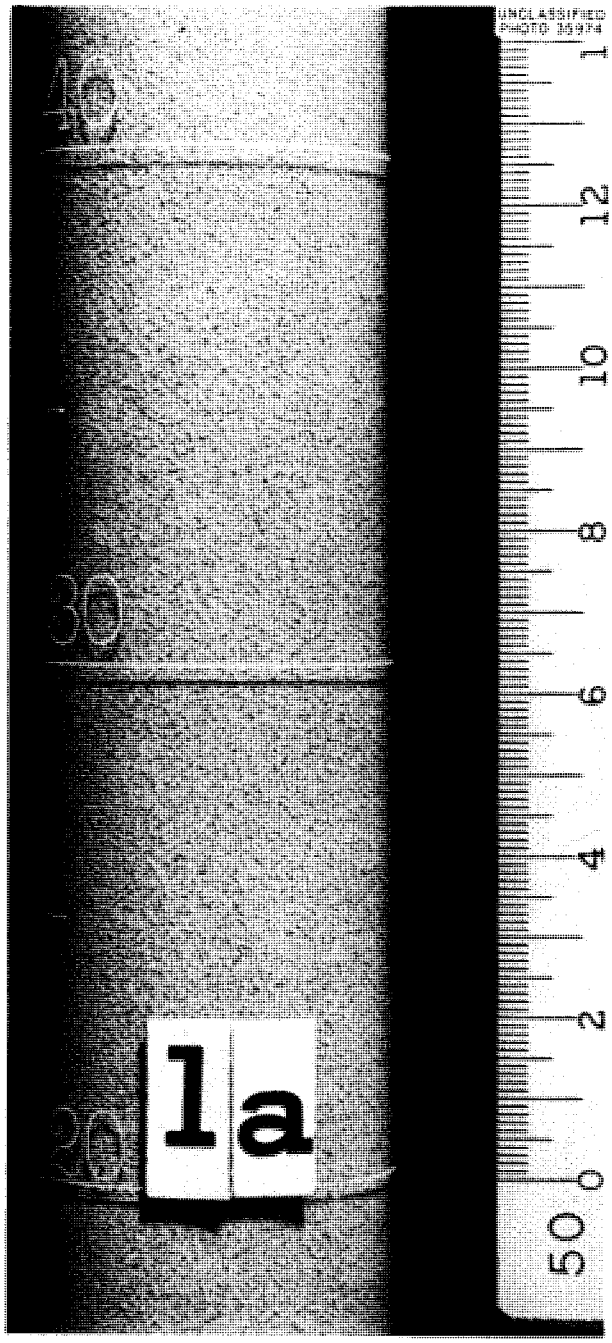

Fig. 10. Settling Flocs; 200A-21 Slurry at 100 g of ThO_2 per Liter. Floc diameter is 120 μ.

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