Metal Sludge Thickening Characteristics:
Particle Density Considerations

by

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Thesis submitted to the Faculty of the

Virginia Polytechnic Institute and State University

in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

in

Environmental Sciences and Engineering

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March, 1984
Blacksburg, Virginia

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(ABSTRACT)

Since 1901, when G. G. Stokes published his equation for drag force on a uniform sphere settling in a liquid, researchers have searched for a modification of this equation that would predict particle settling behavior in concentrated suspensions such as sludges. During the 1960's, researchers such as Michaels and Bolger (1962) and Javaheri and Dick (1969) developed an index of the relative amount of water associated with a floc particle that appeared to have broad application in predicting sludge settling behavior. The objective was to attempt to apply various direct laboratory methods of wet particle density to the analysis of sludge flocs. The second objective was to use the analysis of Javaheri and Dick (1969) to evaluate the thickening characteristics of four non-biological sludges including the relevance of particle size, density, and water content to thickening rates. Aggregates of sludge flocs do not appear to retain their integrity during mechanical dewatering, although they do over the linear portion of the batch thickening curve. It appears that the floc density rather than the aggregate density may represent the highest bulk

density achievable by mechanical dewatering. The aggregate volume
index (AVI) of Javaheri and Dick (1969), which can be used to
determine aggregate size and density, predictively described the
settling behavior of the four chemical sludges evaluated. This
indicates that the analysis of Javaheri and Dick (1969) may be
the first mathematical description of settling of slurries broadly
applicable to the settling of water and wastewater treatment sludges.

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INTRODUCTION

Since 1901, when G. G. Stokes published his equation
for drag force on a uniform sphere settling in a liquid,
researchers have searched for a modification of this equa-
tion that would predict particle settling behavior in con-
centrated suspensions. Settling of this nature is an
essential separation mechanism, not only for water treat-
ment but for many domestic and industrial wastewater treat-
ment processes as well. Most of the work in this area has
centered on two types of slurries: non-porous particles
such as glass beads, tapioca and emory; and flocculated,
porous suspensions of kaolin or microbial cells such as
activated sludge. Additionally, the main emphasis of this
research centered around the relationship between inter-
facial settling velocity and the relative volume of parti-
cles as the descriptive parameters in the settling of
slurries. Up until the 1960's there was no empirical or
theoretical analysis developed that was predictive of
settling behavior for slurries of uniform particles over
broad concentration ranges, much less for highly diverse
slurries such as sludge.

During the 1960's, researchers (Michaels and Bolger,
1962; Javaheri and Dick, 1969) developed an index of the

relative amount of water associated with a floc particle that appeared to have broad application in predicting sludge settling behavior. This index, referred to as C_{ak} by the first authors and the Aggregate Volume Index (AVI) by Javaheri and Dick (1969), was calculated based upon a mathematical analysis of interfacial settling velocity data. Values of AVI could also be used to estimate values for sludge particle density. To date there has been no verifiable direct laboratory measurement for sludge particle density with which to compare these calculated values.

The objectives of this research were twofold. The first part of this research was aimed at reviewing the applicability of various direct laboratory measurements of wet particle density to the analysis of sludge flocs. The second objective of the research was to utilize the mathematical techniques of Javaheri and Dick (1969) to evaluate the thickening characteristics of several chemical sludges. Included in this objective was an evaluation of the role of particle size, density, and floc water content (quantified by the aggregate volume index) in determining chemical sludge thickening rates.

Chapter II

LITERATURE REVIEW

This chapter reviews, in historical order, highlights
in the research investigating particle settling in the in-
fluence of other particles. This research culminates in
the analysis of Javaheri and Dick (1969). A brief review
of fundamental sludge work since 1969 follows this analy-
sis. A final section related to the use of density gradi-
ents for particle density determinations has also been
included.

2.1 DEVELOPMENT OF PARTICLE SETTLING EQUATIONS

In 1901, G. G. Stokes published the equation for the
drag force on a spherical object in an infinite fluid.
Based on the assumption of laminar flow at low Reynolds
numbers such that inertial forces are negligible and vis-
cous forces predominate, Stokes related particle drag force
and settling velocity:

$$F_D = 3\mu VD \tag{2-1}$$

where F_D = drag force

μ = viscosity of the liquid

v = velocity of the particle

D = particle diameter

When this drag force and the force of gravity are equal to
the buoyant force on the particle, a terminal settling

velocity (V_o) is attained (Pao, 1961). Stokes equation re-written in terms of this terminal velocity becomes:

$$V_o = \frac{D^2 g (\rho_p - \rho_w)}{18\mu}$$ (2-2)

where   g = gravitational force

ρ_p = particle density

ρ_w = density of the fluid

In slurries where buoyant and viscous forces are influenced by the presence of other particles, researchers have modified Stokes' equation, theoretically and empirically, to account for observed particle settling behavior. As sludges are generally slurries of this sort, this type of analysis is essential to understanding sludge particle behavior and characteristics.

2.1.1   Interfacial Settling Equations for Non-Flocculated Suspensions

In 1911, Einstein, as referenced by Kynch (1952), pro-posed a formula to describe particle settling in a suspen-sion of particles where the density of the particles is small and their distance apart much greater than their radii. For non-porous, spherical particles, the settling velocity of the particles was quantified as:

$$V = V_o (1 - K\Phi_p)$$ (2-3)

where V_o = Stokes settling velocity for the particle

V = settling velocity for a particle in a suspension

Φ_p = volume fraction occupied by the particles

K = shape factor for non-porous spheres (= 2.5)

Other authors had obtained the same formula from observations of similar systems, but no one had satisfactorily obtained a formula for particle settling behavior in concentrated suspensions of spheres.

In 1944, Steinour (1944a) studied slurries of uniform spheres of tapioca settled through oil and microscopic glass spheres settled through water. When conditions were such that isolated spheres fell according to Stokes law and the distribution of spheres in the medium was fairly uniform the settling rate could be represented by Stokes law multiplied by a function of concentration only. For the interfacial settling velocity V_s, of this system, the following equation was determined:

$$V_s = V_o \frac{\varepsilon^3}{1 - \varepsilon} \Theta \qquad (2-4)$$

where ε = porosity or the volume fraction of water

   Θ = factor determined by the shape of interparticle
       flow spaces, determined empirically to be 0.123
       for ε below 0.7

Steinour concluded that increasing the number of spheres in the medium primarily affected the buoyant force in that the hydrostatic pressure of any layer in the medium would be determined by the density of the mixture rather than the density of the medium alone.

Working with angular particles of uniform-sized emory settled through water, Steinour (1944b) modified his suspension thickening expression to the following:

$$V_s = 0.176V_o \frac{(\epsilon - 0.168)^3}{1 - \epsilon} \qquad (2\text{-}5)$$

This equation was validated for suspensions with a porosity up to 0.75. Steinour postulated that the difference between equations 2-4 and 2-5 was due to water associated with the emory particles that travelled with the particles as "immobile water". Whether the water was bound to the particles or just "stagnant" within cracks in the emory, its effect was unrelated to the nearness of other particles, only to the number of particles. A proportionality constant was assigned to the amount of water and Equation 2-5 was rewritten:

$$V_s = \frac{0.123(\epsilon - \omega_i)^2}{(1 - \omega_i)(1 - \epsilon)} V_o \qquad (2\text{-}6)$$

where ω_i = term used to quantify the immobile water fraction

By repeating these experiments with three sizes of emory particles, Steinour determined that ω_i was independent of particle size and, therefore, shape dependent. This would suggest that the immobile water was stagnant rather than bound water. The work was also repeated with flocculated suspensions of emory and Equation 2-6 was found to be valid when ω_i was increased to account for the

lowered values of V_s observed.

In 1947 and 1948, Brinkman sought to derive mathematically rather than empirically, a formula for interfacial settling velocity of a concentrated suspension of uniform spherical particles. Flow around a single particle was described with a modified Navier-Stokes equation; flow outside the region of the particle was represented by Darcy's empirical equation for flow through a bed of particles. Since these two equations could not be solved mathematically, Brinkman made a detailed analysis of the different areas of flow to arrive at Equation 2-7 (Brinkman, 1947).

$$V_s = V_o [1 + 3/4 \Phi_p (1 - \sqrt{\frac{8}{\Phi_p} - 3})] \qquad (2-7)$$

In a later paper, Brinkman (1948) developed an equation relating settling rates to suspension porosity and permeability:

$$V_s = V_o \frac{4\pi r}{3\varepsilon} q^2 \qquad (2-8)$$

where r = particle radius

    $q = r/k^2$

    k = permeability

Settling tests verified the validity of the equation (2-8) for porosity values greater than 0.4.

In 1954, Richardson and Zaki published work critical of Steinour and others for assuming that the effective buoyant force was dependent on the density of the suspension

(Richardson and Zaki, 1954). These authors concluded that this assumption could not be true for suspensions of uniform particles. In such a suspension, particles should displace their volume in liquid. Only in suspensions with great disparities in particle sizes would the larger particles displace the suspension of other particles. Restriction of flow space between particles with increasing concentrations caused drag forces, not buoyant forces to be the primary consideration. From theoretical considerations, Richardson and Zaki developed the following equation to describe the settling velocity of uniform non-porous spheres.

$$V_s = V_o \epsilon^{4.65} \qquad (2\text{-}9)$$

With the addition of a dimensionless shape factor, this relationship was found to hold for non-spherical particles of uniform size also. Experimental data validated the predictive nature of this equation.

In 1958, Happel developed a purely mathematical model of particle settling which was tested against the laboratory data previously presented in the literature. Happel cited a need for a model based on a minimum of simplifying assumptions rather than the "semi-empirical" assumptions of previous authors. The need for a straightforward theoretical treatment that was predictive over the complete range of particle concentrations (ϵ = 0.2 to 1.0) was also

emphasized (Happel, 1958). The model depicted a particle surrounded by an envelope of liquid such that the fluid/particle ratio reflected that of the entire suspension. This equation, derived for uniform-sized, non-porous spheres, is:

$$V_s = V_o \frac{(3 - 9/2\,\Phi_p + 9/2\,\Phi_p^5 - 3\,\Phi_p^6)}{3 + 2\,\Phi_p^5} \qquad (2\text{-}10)$$

For the range of high particle concentrations, including packed beds (ε = 0.2 to 0.6), the equation predicted settling results within the experimental accuracy for data reported in the literature. In the intermediate range (ε = 0.6 to 0.95) the values predicted by Equation 2-10 agreed with special cases of fluidization. Particle agglomeration in this range was suggested to account for the poor correlation with other data. In the dilute range (ε greater than 0.98) settling rates calculated by the use of Equation 2-10 were 2 to 15% lower than recorded values. At ε = 0.999, close agreement between experimental results and predicted values was again obtained.

Aiba et al., (1965) in a discussion of separating microbial cells from industrial fermentation products, started with another author's equation for drag on a sphere during hindered settling and added a shape factor. The term, α, was developed which accounted for particle shape, a function of the volume fraction of particles (Φ_p), and

geometrical arrangement of the particles. The resulting equation was:

$$V_s = V_o \frac{1}{1 + \alpha \Phi_p} \qquad (2\text{-}11)$$

Because no previous author's work had described settling over the entire range of particle concentrations, Aiba et al., replotted their data in terms of α versus Φ_p and determined empirically that α was equal to $1.305 \Phi_p^{2.84}$ for irregularly shaped particles at Φ_p between 0.15 and 0.5. For spherical particles in dilute suspensions (Φ_p less than 0.15) α was approximately 1 to 2. For Φ_p between 0.2 to 0.5, α was equal to $1 + 229 \Phi_p^{3.43}$. These values could be used to determine V_s in both gravitational and centrifugal fields.

As these equations evolved and were modified for specific uses, particularly as they were modified to describe the settling of particles such as microbial cells and other irregular particles, they became descriptive of particle settling in certain types of sludges. However, none of the equations, (except for Equation 2-6) dealt with the type of flocculent suspension common to many water and wastewater treatment facilities. Section 2.1.2 deals with how these equations were further modified to describe flocculent settling. Table 1 summarizes the equations discussed in these sections for comparison.

TABLE 1

Equations for Interfacial Settling Velocity

EQ. #	Equation	Source
Non-Flocculated Suspensions:		
(2-4)	$V_s = V_o \dfrac{\varepsilon^3}{1 - \varepsilon} 0.123$	Empirical formula for sphere(Steinour, 1944a)
(2-5)	$V_s = V_o \dfrac{(\varepsilon - 0.168)^3}{1 - \varepsilon} 0.176$	Empirical formula for angular particles (Steinour, 1944b)
(2-7)	$V_s = V_o \left[(1 + 3/4(1 - \varepsilon))(1 - \sqrt{\dfrac{8}{(1 - \varepsilon)} - 3}) \right]$	Theoretical formula for spheres(Brinkman, 1947)
(2-9)	$V_s = V_o \varepsilon^{4.65}$	Theoretical formula for spheres(Richardson & Zaki, 1954)
(2-10)	$V_s = V_o \left[\dfrac{3 - 9/2(1 - \varepsilon) + 9/2(1 - \varepsilon)^5 - 3(1 - \varepsilon)^6}{3 + 2(1 - \varepsilon)^5} \right]$	Theoretical formula for spheres(Happel, 1958)
(2-11)	$V_s = V_o \dfrac{1}{1 + \alpha(1 - \varepsilon)}$	Empirical formula for bacterial suspensions (Aiba et al., 1965)
Flocculated Suspensions:		
(2-6)	$V_s = \dfrac{0.123(\varepsilon - \omega_i)^2}{(1 - \omega_i)(1 - \varepsilon)} V_o$	Empirical formula for flocculated angular particles(Steinour, 1944b)
(2-12)	$V_s = \dfrac{g(\rho_a - \rho_w)D_a^2 \varepsilon^{4.65}}{18\mu}$	Theoretical formula for angular particles and activated sludge(Michaels & Bolger, 1962; Javaheri & Dick, 1969)

2.1.2 Settling Equations for Flocculated Suspensions

Steinour (1944b) examined suspensionf of flocculated emory particles, as discussed in Section 2.1.1, and concluded from Equation 2-6 that the decrease in V_s resulting from flocculation of the particles was due to an increase in immobile water associated with the flocculated particles. However, this was an empirical observation as Steinour developed no theoretical model that would predict the observed results.

By using a theoretical approach, Michaels and Bolger (1962) demonstrated with flocculated kaolin suspensions that V_s was a function of just two variables: floc volume concentration, and the strength of the attractive forces between flocs.

The keolin studied had relatively uniform particle size; aggregation of the particles was accomplished by controlling pH. Flocs were defined as small clusters of primary particles plus the enclosed water. Because of an inherent mechanical strength they retained their identity under the mild surface shear of gravity settling. Having been formed under the severe agitation of a blender, their final shape was spherical and of fairly uniform size. Clusters of flocs were termed aggregates. Microscopic examination during settling showed that at dilute concentrations, aggregates settled as roughly spherical, individual

units. At higher concentrations the aggregates formed chains and networks. Michaels and Bolger (1962) also observed that when a standard mixing procedure was used in preparing the floc suspensions, the aggregates approached a fairly uniform, reproducible average equivalent spherical diameter, D_a. Using Equation 2-9 the aggregate settling rate in a dilute solution was expressed as follows:

$$V_s = \frac{g(\rho_a - \rho_w)D_a^2}{18\mu}(1 - \Phi_a)^{4.65} \qquad (2\text{-}12)$$

where ρ_a = density of the aggregates

Φ_a = volume fraction of the aggregates

or the expression could be written:

$$V_s = V_0(1 - \Phi_a)^{4.65} \qquad (2\text{-}13)$$

where V_0 = the Stokes settling velocity for an aggregate rather than for a discrete particle

Since ρ_a and Φ_a could not be measured directly, the following materials balance was used:

$$\rho_b - \rho_w = \Phi_a(\rho_a - \rho_w) = \Phi_k(\rho_k - \rho_w) \qquad (2\text{-}14)$$

where ρ_b = bulk density of the suspension

ρ_k = density of the dry keolin

Φ_k = volume fraction of solids

The terms ρ_b, ρ_k, and ρ_w could all be measured directly, leaving Φ_k to be calculated. This allowed Equation 2-12 to be rewritten:

$$V_s = \frac{g(\rho_k - \rho_w)D_a^2}{18\mu C_{ak}}(1 - C_{ak}\Phi_k)^{4.65} \qquad (2\text{-}15)$$

where $C_{ak} = \Phi_a/\Phi_k$

The Stokes settling velocity, V_o, for a single aggregate became:

$$V_o = \frac{0.349D_a^2}{C_{ak}} \qquad (2\text{-}16)$$

Based on these equations, Equation 2-15 could be written in the equation of a line:

$$V_s^{1/4.65} = V_o^{1/4.65}(1 - C_{ak}\Phi_k) \qquad (2\text{-}17)$$

A plot of $V_s^{1/4.65}$ versus Φ_k should yield a straight line such that V_o and D_a can be determined from the y-intercept and C_{ak} can be determined from the x-intercept. Data from dilute slurries of flocculated kaolin verified Equations 2-15 and 2-17.

In calculations such as this, direct measurements of only a few parameters can be made. Terms like V_s, g, μ, ρ_w, ρ_k, and ρ_b can be measured or determined directly. Terms like D_a, Φ_a, Φ_p, Φ_k, ρ_a, ρ_p, ρ_f (floc density) and terms derived from these terms must all be measured indirectly or derived from indirect measurements. Direct measurements of density, diameter or volume of flocs, aggregates or particles would greatly simplify the use of these equations as well as eliminate any uncertainty associated

with the graphical derivations.

In 1966, Mueller et al., published an article regard-
ing the measurement of floc diameters in activated sludge
for the purpose of determining oxygen transfer in the floc.
This article summarized some of the work of other authors
(Schroepfer et al., 1955; Schroepfer and Ziemke, 1957; Fin-
stein, 1965; and Morand, 1964) who likewise attempted to
measure floc diameter, density and volume. Schroepfer
et al., (1955) dropped digester wastes into salt solutions
and photographed the solids to estimate the floc diameters
by measuring their photographic images. Finstein (1965)
dried filamentous sludges on microscope slides and measured
their projected images. Morand (1964) solidified sludge
flocs in agar on microscope slides and measured flocs with
an optical micrometer. Mueller et al., (1966) were not
satisfied with these measurements because of the variability
of floc size within a slurry and because of the arbitrary
nature of the diameters chosen for optical measurements.
Instead, these authors calculated a nominal diameter based
on wet floc density, dry floc density, medium density, floc
counts and mixed liquor suspended solids (MLSS).

In the development of a wet floc density measurement,
Mueller et al., (1966), noted that water may be associated
with the floc in any or all of the following ways: a)
water adsorbed onto the surface; b) interstitial water or

water adsorbed within the floc but retaining the colligative properties of water; and c) bound water in colloidal micells with properties other than those of group b). This associated water caused difficulty in floc density determinations.

Schroepfer et al., (1955), who settled flocs through salt solutions, measured their dimensions (as previously described) and used Stokes' law to calculate floc density. In other work, Schroepfer and Ziemke (1957) placed flocs in salt solutions of varying densities and recorded the floc density as that of the salt solution which just suspended the floc. Mueller et al., (1966) noted the mass transfer of water out of the floc into the salt medium as a major limitation of either procedure. To reduce this mass transfer effect, Mueller et al., air-dried centrifuged activated sludge solids on filter paper until its cohesion formed small spheres of 2 to 3 mm in diameter. These were then placed into potassium bromide solutions of varying densities. Spheres that did not settle immediately were considered to have densities less than or equal to that of the medium. The spheres were assumed to contain only interstitial and bound water. No verification of this assumption was tested experimentally.

In 1968, Lagvankar and Gemmell, as part of a study of floc formation and floc characteristics, developed a

measurement of floc density based on solutions of sucrose of known density. As in the work of Mueller et al., (1966), density of sludge flocs was determined by whether or not flocs settled or floated in sucrose solutions of given densities. Flocs were taken directly from their original suspensions and placed in tubes of sucrose without the air-drying procedure used by Mueller et al. Lagvankar and Gemmell (1968) did not report comparing this technique with other techniques to verify its accuracy.

In 1969, Javaheri and Dick, using the work of Richardson and Zaki (1954) and Carmen (1937) developed a mathematical model for the thickening of activated sludge which they used to study the displacement of liquids from sludge aggregates. As in the work of Michaels and Bolger (1962), flocs were considered to be made up of primary particles plus the liquid trapped between the agglomerated primary particles. Aggregates were said to be made up of the constituent flocs and the liquid between them. The porosity of the suspension was determined by the interstices between aggregates. Javaheri and Dick (1969) defined two flow patterns for the purposes of their study; interaggregate flow, or flow between aggregates, and intraaggregate flow, or flow through aggregates. It is interaggregate flow which is of interest in particle settling.

Starting with Richardson and Zaki's (1954) equation
for uniformly sized, non-porous particles, (Equation 2-9)
Javaheri and Dick modified the expression in an attempt to
relate activated sludge settling rates to the effective
diameter, D_a, of the sludge aggregates. Laminar flow was
assumed and wall effects were ignored to rewrite Equation
2-9 as:

$$V_s = V_o(1 - \Phi_a)^{4.65} \qquad (2-18)$$

V_o was expressed in terms of Stokes' law as:

$$V_o = \frac{g(\rho_a - \rho_w)D_a^2}{18\mu} \qquad (2-19)$$

This was essentially the equations of Michaels and Bolger
(1962). Javaheri and Dick (1969) also rearranged the mater-
ials balance equation of Michaels and Bolger (1962) (Equa-
tion 2-14) to the form:

$$\rho_a - \rho_w = \frac{\Phi_k}{\Phi_a}(\rho_k - \rho_w) = \frac{1}{AVI}(\rho_k - \rho_w) \qquad (2-20)$$

where   AVI = Aggregate Volume Index

The term AVI was equivalent to the term C_{ak} of Michaels and
Bolger (1962) and was a measure of the water associated
with the dry solids in a sludge aggregate. The final equa-
tions were likewise similar to Michaels and Bolger (1962)
rewritten in terms of AVI. The linear form of the final
equation was:

$$V_s^{1/4.65} = V_o^{1/4.65} - V_o^{1/4.65} (AVI) \Phi_k \qquad (2-21)$$

When plots of $V_s^{1/4.65}$ versus Φ_k were made, the slope of the line was $V_o^{1/4.65} (AVI)$ and the y-intercept, $V_o^{1/4.65}$. If the plot was curved, then the aggregates themselves were considered to have released water during settling, causing a corresponding change in the AVI. Values of AVI and V_o could be determined by taking tangents to the curve and noting the slope and y-intercept of the tangents. Values of V_o could then be used to calculate D_a in Equation 2-19 or aggregate densities could be calculated from Equation 2-14 using AVI. Javaheri and Dick (1969) found that the simplifying assumptions made in developing these equations did not create major errors in the model when batch settling tests on activated sludge were performed.

2.1.3 Settling Theory Studies Since Javaheri and Dick

Since 1969, Javaheri and Dick's work has remained largely undeveloped. Little additional work has been done to further modify Stokes' law to explain settling in slurries. Some of the work which has continued in basic settling theory relating to the settling of slurries is summarized in this section.

In 1973, Dollimore et al., published a study done with particles of sulfur precipitate based on the work of Steinour (1944a). These authors were able to rewrite Steinour's

basic equations to determine a mean particle size for a given slurry based on one simple laboratory settling test. The effect of particle size and shape on the final settled volume of sludge was also examined.

In 1977, D. C. Dixon published an analysis of free particle settling based on a "momentum-balance" or an analysis of forces on a settling particle rather than a mass balance as in the case of authors previously cited. Dixon's argument was aimed primarily at Kynch's (1952) theory of settling which described layers of increasing concentration moving upwards from the bottom of a column during settling. From this momentum analysis, Dixon concluded that until particles struck the sediment accumulating at the bottom of the column, there was no change in concentration in the free settling particles above. If the slurry was compressible, then concentration layers might move upward in the sediment itself, but would not affect the area of settling above the sediment. Dixon's further analysis seemed to invalidate analyses previously developed which were based on the assumption that the velocity of the settling interface in the slurry was a function of solids concentration alone. However, Dixon was unable to develop experimental data to verify the analysis.

Perhaps the most interesting departure from the conventional analysis of particle settling in slurries was the

recent publication by Lawler et al., (1982). The authors
demonstrated that coagulation and differential sedimenta-
tion occurred during settling. This was in direct contrast
to previous analyses which assumed a constant terminal
settling velocity for all particles in the slurry as indi-
cated by the terminal interfacial settling velocity, V_s.
These authors studied slurries of SiO_2 of varying particle
size and analysed changes in particle size distribution
during settling with and without coagulation. From these
studies, three implications to the settling of sludges were
made. The first was that the concentration at which an
interface formed was dependent on the degree of particle
size uniformity in the original suspension. The second was
that, since the settling velocity of the interface was shown
to decrease as the larger particles dropped out, the addi-
tion of larger particles to a slurry might aid in increas-
ing interfacial settling velocities. The third implication
drawn by the authors was that increasing solids concentra-
tion on the bottom layers of a sediment may not be due sole-
ly to compression but may also be due to differential sedi-
mentation, thereby affecting current understanding of the
phenomenon. No mathematical analysis or predictive equa-
tions were presented.

2.2 DENSITY GRADIENTS FOR THE MEASUREMENT OF PARTICLE DENSITIES

In the previous section, Mueller et al., (1966) described the work of Schroepfer and Ziemke (1957) in which they used salt solutions of varying densities to determine the density of sludge flocs. In Section 3.2.5, a similar method, that of Lagvankar and Gemmell (1968) (also described briefly in Section 2.1.2) is described in more detail using sucrose as the density medium. Problems associated with these test methods are also described. Due to the limitations of these tests, a more precise, verifiable particle density measurement was sought. This section describes the use of density gradients for particle density determinations in biochemistry.

The techniques of step and continuous gradients are commonly used in the study of biochemistry for the separation of viruses, organelles, and macromolecules. Gravity sedimentation through such gradients could be used to separate larger particles; however, when particles are so small as to be dispersed by Brownian motion, a centrifugal force must be applied to overcome this dispersion (Morris and Morris, 1963). Centrifugation of larger particles may not be required to overcome dispersion, but can be a useful tool in accelerating the sedimentation process.

There are two methods of particle separation based on centrifugation of particles through a gradient medium. The

first method, called rate-zonal centrifugation or gradient-differential centrifugation, is based on separation of particles by their mean sedimentation coefficients. This method is kinetically based and the rate of sedimentation is a function of both particle density and particle size (Morris and Morris, 1963; Pharmacia, 1982). The second method, called isopycnic-gradient or isopycnic-zonal centrifugation, is commonly used to measure the buoyant density of molecules such as DNA (Lehninger, 1975). This method is an equilibrium method in that centrifugation through the gradient is continued until sedimentation stops. During centrifugation, particles migrate through the medium to their isopycnic point, or the point at which the density of the medium equals the density of the particle (Morris and Morris, 1963). Separation of particles by isopycnic sedimentation is independent of particle settling rates or particle diameter and is a function of density alone. For this reason it appeared to be the method of choice for this research.

A variety of substances have been used for the production of gradients. In biochemistry, sucrose has been most widely used because of its low cost, ease of preparation and stabilizing effect on subcellular particles. Morris and Morris (1963) discussed some of the important properties of an ideal medium. Apart from its density, the ideal medium

should exert a minimum osmotic effect on the particles; should cause minimum changes in viscosity, ionic strength, and pH of the diluent; should not be surface active; and should not cause denaturation or destructive changes in the particles. In Section 3.1.3 the properties of the medium Percoll are discussed. It had two major advantages over sucrose for density gradients in that it exerted little osmotic pressure and was essentially iso-osmotic throughout the gradient. Also, it could form self-generated continuous gradients in a centrifugal field which sucrose could not.

Generally, a gradient sedimentation experiment is undertaken in three steps. The first is the making of the gradient. Step gradients and continuous gradients are described in Section 3.2.5. Gradient formation can occur by layering, as in the step gradient, mixing in a continuous gradient maker, or self-generation in a high speed rotor. The second step is the sedimentation step in which the particles are layered on top of the gradient and then sedimented through it. For any of the gradients mentioned, this could be done at low speeds in a centrifuge with a swinging bucket rotor, but for certain media, a fixed angle rotor is required. Particles could be allowed to settle by gravity for as long a period as the gradient could maintain its integrity against diffusion. The third step is the

determination of particle density at the isopycnic point. This could be done in the case of Percoll by the use of calibrated density marker beads as described in Section 3.1.4. The gradient may be fractionated and characteristics of the fractions, such as refractive index, which is a function of medium density, may be determined.

Because of their long history of use in biochemistry, it was hoped that these highly accurate and reproducible methods could be successfully adapted to the direct measurement of wet particle density of sludge flocs.

Chapter III

MATERIALS AND METHODS

The techniques used to obtain the experimental data
for this research evolved through optimization studies of
various test methods, some of which were adapted from other
disciplines. While measurements such as total solids, bulk
density, vacuum dewatering, sludge settling and sludge
thickening by centrifugation may commonly be performed in
an environmental engineering laboratory, other procedures
such as particle settling in various media and the develop-
ment of step and continuous gradients for particle density
determinations required the use of techniques used primari-
ly in biochemistry. The following discussion emphasizes
the background principles, materials, and protocols asso-
ciated with many of the techniques.

3.1 MATERIALS

3.1.1 Sludges

Various sludges were used during the methods develop-
ment part of this research, including conditioned and raw
alum sludge (alum I), copper hydroxide sludge, nickel hy-
droxide sludge (nickel hydroxide I) and waste biological
sludges all of which were generated in the laboratory. The
four sludges used for experimental data collection included
an iron hydroxide sludge, alum sludge (alum II), lime

sludge, and nickel hydroxide (nickel hydroxide II) sludge.
The iron hydroxide sludge sample was collected from the
Town of Blacksburg, Virginia, drinking water treatment
plant. The alum (II) sludge sample was collected from the
water treatment facility at the Radford Arsenal in Radford,
Virginia. The lime softening sludge was collected from the
Fort Wayne, Indiana, municipal water treatment facility.
The nickel hydroxide (II) sludge was prepared in the lab-
oratory by dissolving nickel carbonate in acid solution,
then precipitating the nickel with sodium hydroxide.

3.1.2 Standard Sucrose Solutions

Sucrose solutions of varying densities were prepared
on a percent weight/volume basis. For example, a 10% su-
crose solution was made by placing 10 grams of sucrose into
a 100 ml volumetric flask and adding distilled water to
produce the desired final volume. Pycnometers were used to
verify the densities of the solutions used. Density values
for the solutions of sucrose used are summarized in Table 2.

3.1.3 Solutions of Percoll

Percoll (a registered trademark of Pharmacia Fine
Chemicals AB of Sweden) is a modified colloidal silica that
was developed specifically for the purpose of providing an
ideal medium for density gradient centrifugation for the
separation of biological particles. Percoll is colloidal

TABLE 2

Density Values of Standard Sucrose Solutions

Percent Sucrose(weight/volume)	Solution Density(grams/milliliter)
2	1.0079
6	1.0229
8	1.0308
10	1.0384
12	1.0443
15	1.0557
20	1.0743
30	1.1237
40	1.1487
60	1.2240
80	1.2924

silica coated with polyvinylpyrolidone (PVP) to increase
the stability of the colloid and decrease its toxicity in
biological work. It also has a very low osmotic pressure
which makes it iso-osmotic throughout the gradient. It can
be used to form gradients in the range of 1.0 to 1.3 grams
per milliliter (g/ml) and can form self-generated gradients
under mild centrifugation. The physical properties of Per-
coll are summarized in Table 3.

Percoll is supplied as a 23% weight/weight (w/w) col-
loidal sol with a density of 1.130 g/ml. It can be diluted
in a variety of media including water, saline and sucrose
although dilute solutions of Percoll in water may remove
PVP from the surface of the silica. Solutions of Percoll
of various densities up to 1.130 g/ml can be obtained using
Equation 3-1.

$$V_p = V_t \frac{(\rho - 0.1\rho_{10} - 0.9)}{\rho_0 - 1} \qquad (3-1)$$

where V_p = volume of Percoll required

V_t = total volume desired

= final density desired

ρ_{10} = density of dilution medium: 1.000 g/ml water

1.058 g/ml 0.15M NaCl

1.316 g/ml 0.25M sucrose

ρ_0 = density of Percoll as supplied by the manu-
facturer

TABLE 3

Physical Properties of Percoll (Pharmacia, 1982)

Property	Specifications
Composition	silica sol with non-dialysable PVP coating
Density	1.130 ± 0.005 g/ml
Conductivity	1.0 mS/cm
Osmolality	20 mOs/kg H_2O
Viscosity	10 ± 5 cP at 20^OC
pH	8.9 ± 0.3 at 20^OC

Concentrating Percoll beyond 1.130 g/ml (as it is supplied)
requires dialysis or prolonged centrifugation.

3.1.4 Density Marker Beads

Density marker beads are color coded derivatives of
Sepahadex (cross-linked dextran) that are modified to have
a specified density in gradients of Percoll. They are used
as an exterior marker to monitor the gradient shape and
range. They are packaged freeze-dried and their density is
calibrated to ± 0.0005 g/ml for solutions of Percoll dilut-
ed in saline or sucrose. The beads must be recalibrated
for use with Percoll diluted in another medium such as
water. This is accomplished by forming a gradient in the
desired medium with the marker beads present, fractionating
the gradient and using a refractometer to measure the re-
fractive index of the fractions containing the marker beads.
This is possible because the relationship between Percoll
density and refractive index is linear. Table 4 summarizes
the densities of the ten marker beads used in this study as
a function of the type of suspending medium utilized.

3.2 METHODS

3.2.1 Solids Determination

Solids concentration was determined as a precentage on
a weight/weight basis. The determination was made by ac-
curately weighing a quantity of sample in an aluminum pan

TABLE 4

Density of Calibrated Density Marker Beads in Gradients of Percoll
Diluted with Saline, Sucrose or Water

| Bead # | Color | Percoll Density Medium Diluted with | | |
		0.15M Saline	0.25M Sucrose	Water
1	blue	1.017	1.038	<1.030
2	yellow	1.033	1.049	--
3	green	1.048	1.054	1.046
4	red	1.062	1.068	1.051
5	blue	1.075	--	1.060
6	orange	1.087	1.096	1.077
7	green	1.097	1.108	1.085
8	red	1.121	1.133	1.091
9	violet	1.143	1.142	<1.093
10	blue	--	1.084	1.053

All values in g/ml

of known weight and drying to constant weight at 103°C.
Percent solids (%S) was calculated according to Equation
3-2.

$$\%S = \frac{\text{net weight dry sample}}{\text{net weight wet sample}} \times 100 \qquad (3-2)$$

3.2.2 Settling Velocity

Settling velocities for various concentrations of
sludges were determined using two liter graduated cylinders
and a stop watch in a standard batch settling test. A
slurry of a given concentration of sludge was added to the
two liter mark of the cylinder which was also graduated in
centimeters (cm) from the two liter mark. The slurry was
mixed in the cylinder. After mixing, the sludge formed a
visible interface at all but the most dilute concentrations.
The stopwatch was used to time the rate of fall of the in-
terface. Plots were made of settling time versus interface
height data. The interfacial settling velocity (V_s, in
m/hr) was calculated from the slope of the linear portion
of the plot. These measurements were repeated for several
concentrations of each sludge. Figure 1 shows data for
several initial solids concentrations of nickel hydroxide
(II) sludge.

3.2.3 Vacuum Dewatering

Vacuum dewatering of sludge samples was accomplished
on a standard vacuum filtration apparatus as depicted in
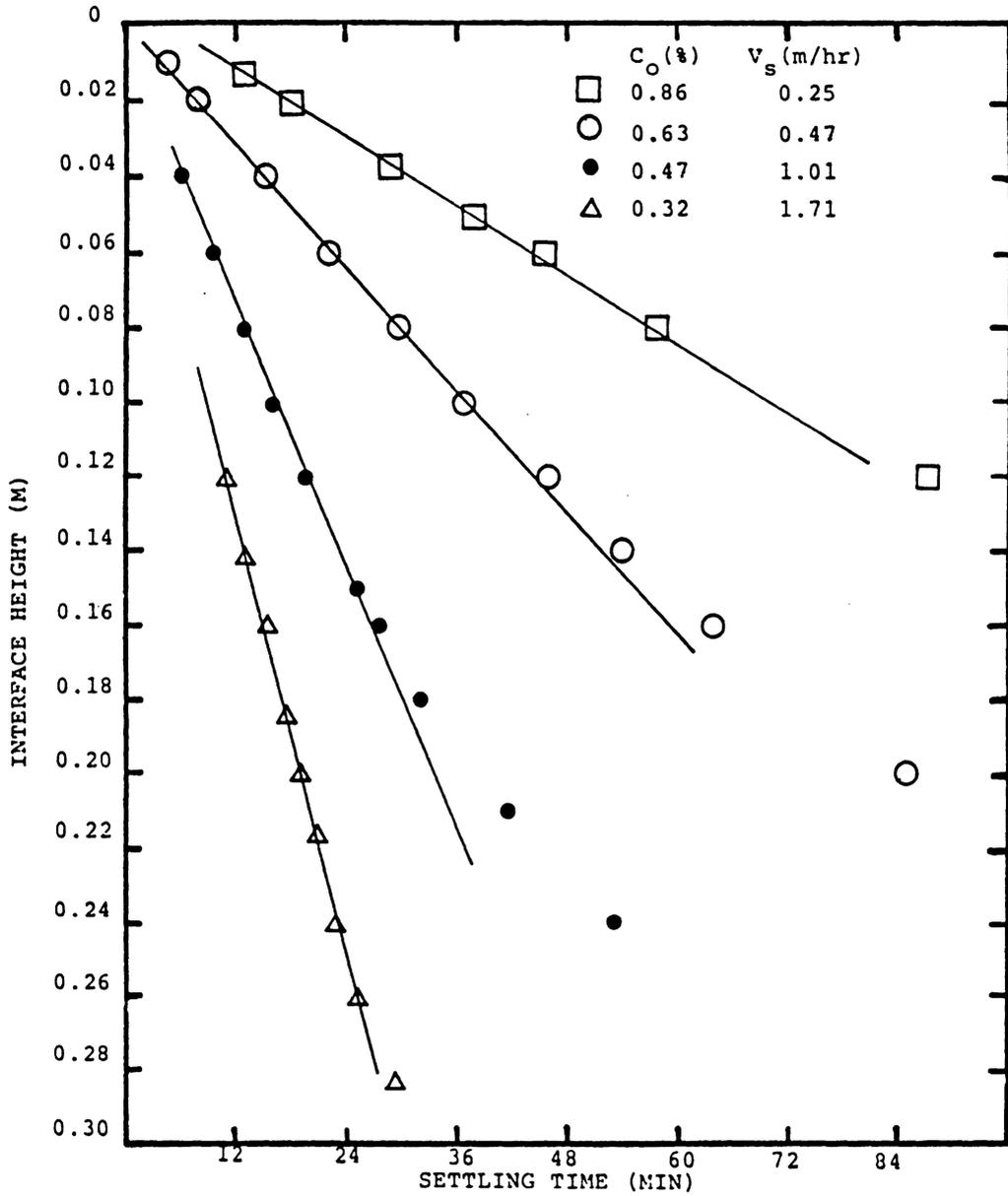
Figure 1:   Interfacial settling velocity determinations
            for the nickel hydroxide (II) sludge sample

Figure 2. Samples of sludges to be evaluated were placed in the Buchner funnel on ashless filter paper and dewatered to increasing levels of solids concentration. At each level of dewatering, samples of the sludge were collected for determination of solids concentration, bulk density and particle density.

3.2.4 Bulk Density

Measurements of bulk density were made using aluninum pycnometers such as the one presented in Figure 3. A pycnometer is a constant volume chamber designed specifically for accurate determinations of density. The chamber was first overfilled with water. The cap was locked in place with the sealing ring and excess water wiped off of the cap as it escaped through the opening in the top of the cap. Once an accurate value for the weight of water the chamber would hold had been made, the chamber was then dried and filled with sludge. The weight of the chamber filled with sludge was then determined.

During the methods development portion of the research, certain of the sludges, especially the alum sludges, had a high dissolved solids concentration in the supernatant. Bulk densities were determined using the value of the supernatant instead of the value of water in Equation 3-3. Supernatant was filtered twice through filter paper before its weight was measured in the pycnometer. Bulk densities
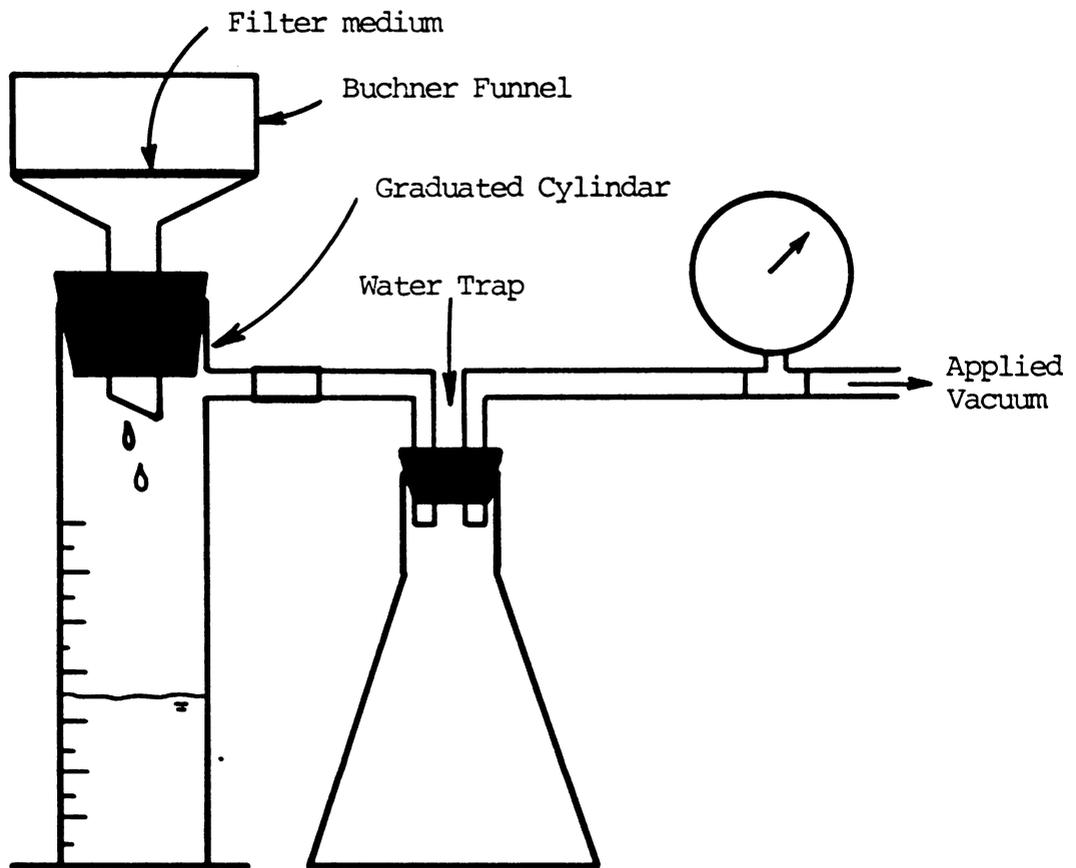
Figure 2: Testing apparatus used for vacuum dewatering
sludge samples

ELEVATION VIEW

Sealing Ring

Cap

Threads

Chamber

PLAN VIEW

Cap

Sealing Ring
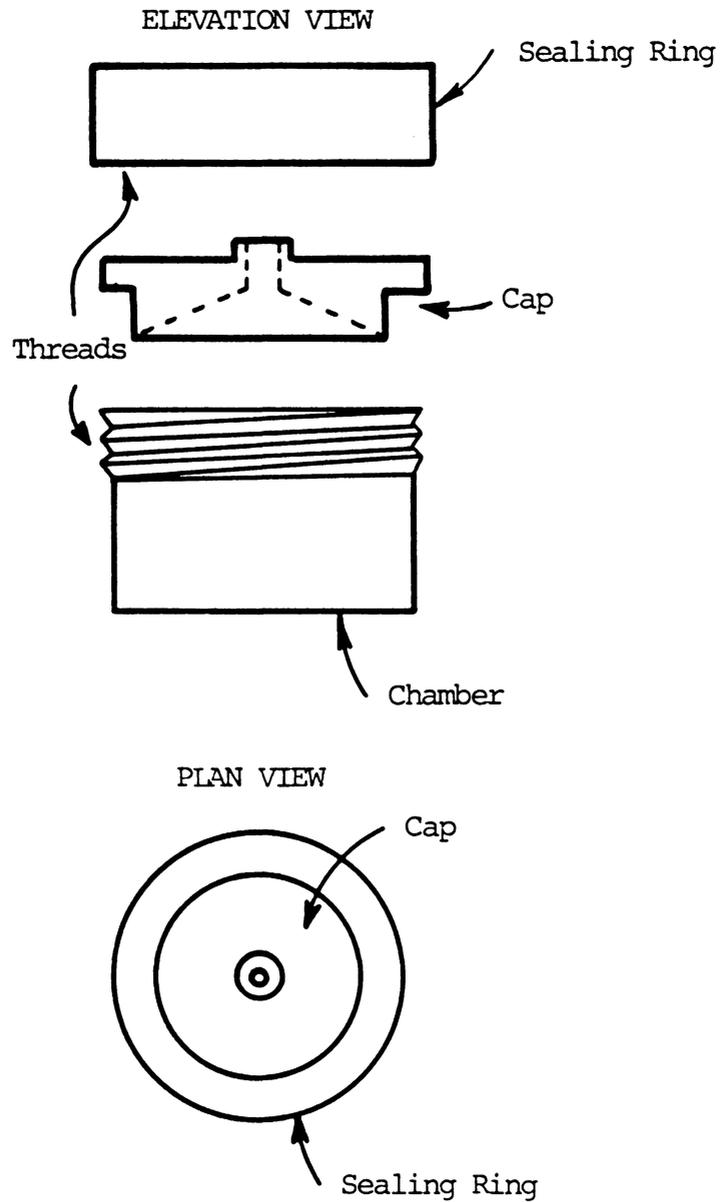
Figure 3: Schematic diagram of an aluminum pycnometer
used in determination of sludge bulk density
values

using supernatant (or filtrate) were denoted ρ_{bf} as opposed to densities determined using water, ρ_{bw}. Where bulk density is stated as ρ_b, water is assumed.

Because of the high pH of the metal sludges, the aluminum pycnometers were subject to pitting and required polishing with steel wool from day to day. Pitting prevented a tight seal between the cap and the chamber. This pitting allowed water to enter the threads of the sealing ring, causing erroneous values for the weight of water in the chamber. Polishing with first coarse, then fine steel wool eliminated the leaks caused by pitting.

Equation 3-3 was used to calculate the bulk density of the sludges.

$$\frac{\text{net weight of sludge(g)}}{\text{net weight of water(g)}} \times \text{unit density water} = \rho_{bw}, \text{ g/cm}^3$$

$$(3-3)$$

where unit density water = 1 g/cm^3 at 20°C

3.2.5 Particle Density Measurements

Several attempts were made at direct measurement of sludge particle density using a variety of methods. Among the methods attempted were step gradients, continuous gradients and settling tests using discrete tubes of media in a series of densities.

Step gradients were formed by layering media, such as sucrose or Percoll solutions, of known density in a

centrifuge tube. The medium was gently dripped down the side of the tube, the highest density solution first, then each lower density in succession. Each successive layer formed an interface with the previous layer which was marked and labelled on the tube. A thin layer of sludge was then applied to the top layer of the medium and the tube placed in a low speed centrifuge with a swinging basket rotor. After brief centrifugation, the particles in the sludge came to rest at the interface above the first layer of medium of greater density than the particles.

Continuous gradients in sucrose were formed by use of a continuous gradient maker as depicted in Figure 4. Continuous gradients between two densities were made by making solutions in sucrose of the highest and lowest density in the range desired. The high density solution was added to the left chamber of the gradient maker and the low density solution placed in the right chamber. The low density solution was allowed to fill the tubing leading to the centrifuge tube. The stirrer in the right chamber was turned on and the level of liquid in the right chamber adjusted to match the height in the left chamber. Both stopcocks (valve 1 and 2) were opened simultaneously and adjusted so the liquid fell at the same rate in each chamber. A thin layer of the sludge to be tested was applied to the top of the gradient. This tube was centrifuged at low speed in either a swinging basket or fixed angle rotor.
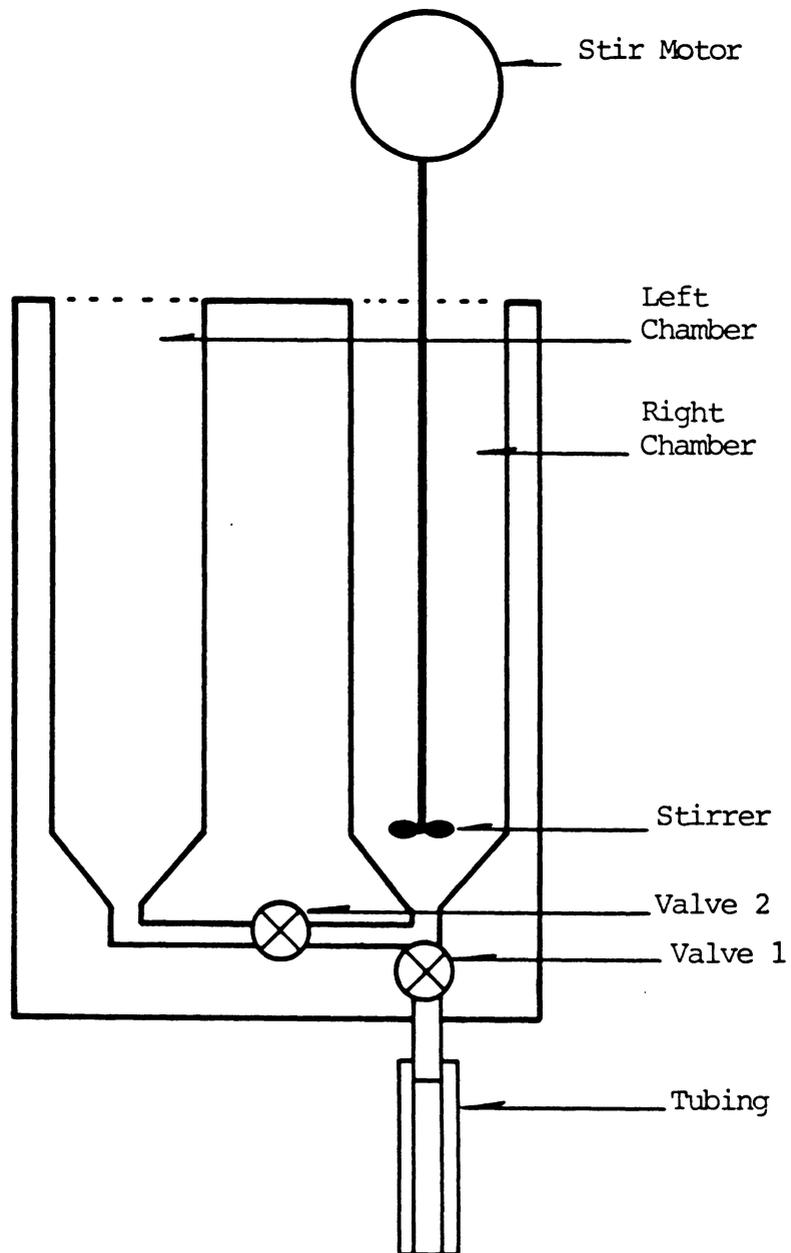
Figure 4: Schematic diagram of a plexiglas continuous
gradient maker used to prepare continuous
sucrose gradients for particle density de-
terminations

Continuous gradients can also be formed in Percoll by use of a continuous gradient maker. High speed centrifugation can be used to form self-generated gradients with Percoll but not with sucrose. When a solution of Percoll is centrifuged at 10,000 g_{av} (in an angle head rotor, g_{av} refers to the average g-force or the force exerted at the mid-point of the tube) in an angle head rotor a gradient forms isometrically around the starting density and becomes steeper with time. The shape of the gradient is approximately linearly related to the total g-force and time. It is not possible to use swinging basket rotors for gradient generation (Pharmacia, 1982). Density marker beads can be used to define the shape of the gradient as demonstrated in Figure 5.

To form a gradient, Percoll was diluted to the desired midpoint density as described in Section 3.1.3. Enough was made to fill two centifuge tubes. Density marker beads were placed on top of one of the tubes and both tubes were centrifuged at 10,000 g_{av} for the desired length of time. A thin layer of sludge was applied to the tube without the marker beads and an equivalent layer of water or saline (or whatever medium was used to dilute the Percoll initially) was layered on the tube containing the beads. The tubes were centrifuged again at 400 g_{av} for fifteen to twenty minutes. The bead densities were plotted versus their
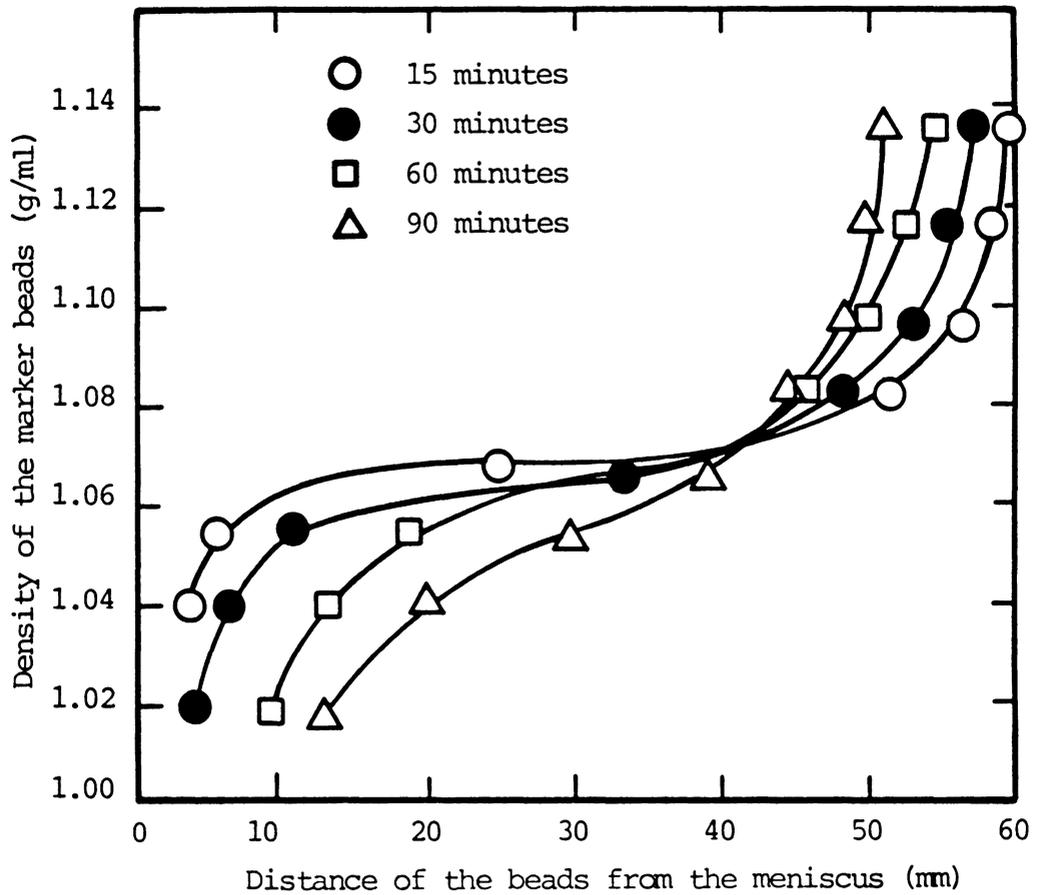
Figure 5:   Location of calibrated density maker beads
defining the shape of a self-generated density
gradient in Percoll. The curves were formed
by centrifuging 1.07 g/ml Percoll at 20,000
g_{av} in an angle head rotor for increasing
lengths of time. (Pharmacia, 1982)

distance in millimeters from the meniscus. This plot was used to determine the density of particles banding in the opposite tube.

The particle settling test used was a simplified version of the test of Lagvankar and Gemmell (19868) and parallels the step gradient in that it involved a series of solutions of discrete density. In this case, however, the solutions were placed individually in a series of small test tubes. A small drop of sludge flocs was released midway into a given test tube and a notation made as to whether the flocs rose or fell within a twenty to thirty second time period. The sludge particle density was said to fall within the range of densities between the lowest density tube in which the flocs rose and the highest density tube in which they sank.

Many variations of this test were attempted using both sucrose and Percoll solutions. The test was very sensitive to turbulence in the test tube created by the release of the flocs and also excess water carried over with the sludge flocs. Attempts were made to transfer sludge flocs to the test tubes with a variety of implements in order to overcome these sensitivities. The implements included finely drawn Pasteus pipettes, syringes with needles of different bores, and bacteriological inoculating needles. Other limitations of the test included limited accuracy in

that results are reported as a range of densities and
also, that the test had not been verified by comparison
with other methods.

3.2.6 Concentration of Percoll

The manufacturers of Percoll suggest several methods
for concentrating Percoll to densities greater than the
1.130 g/ml at which it is supplied. In order to conserve
as much Percoll as possible, concentration by dialysis was
selected. Several feet of 1 inch dialysis tubing filled
with Percoll at 1.130 g/ml were placed in trays containing
Carbowax 300 brand of polyethylene glycol (PEG) flakes and
covered with PEG. Dialysis tubing was prepared by boiling
for ten minutes in distilled water containing a small
amount of sodium carbonate. One end of the tubing was
tied, the Percoll added, and the open end likewise tied.
The tubing was then completely covered with PEG for several
hours. When the tubing was removed from the PEG, it was
lightly rinsed with distilled water, one end clipped, and
the contents recovered. Bulk density measurements by
pycnometer revealed the Percoll had been concentrated to a
density of 1.214 g/ml. As the Percoll at this density was
nearly gelatinous, no additional concentration was attempt-
ed.

Chapter IV·

RESULTS AND DISCUSSION

This chapter reviews and evaluates the laboratory data
and observations resulting from this research. Two ap-
proaches to this research were undertaken. The first was
the evaluation of laboratory methods related to the mea-
surement of wet particle density. The second approach was
to examine the mathematical techniques of Michaels and
Bolger (1962) and Javaheri and Dick (1969) for a better un-
derstanding of particle characteristics and behavior during
sludge settling.

4.1 LABORATORY MEASUREMENT OF PARTICLE DENSITY

4.1.1 Particle Density Determination by Density Gradient
       Techniques

The first attempts at density gradient sedimentation
of sludge particles were made with continuous gradients in
sucrose. Gradients ranging in concentration between 2%
(1.0079 g/ml) and 15% (1.0557 g/ml), 15% and 30% (1.1237
g/ml), 30% and 60% (1.2240 g/ml), and 20% (1.0743 g/ml) and
60% were made. Alum (I) sludge was layered on top of the
various gradients and the tubes were centrifuged in various
centrifuges at different speeds. A high speed Beckman cen-
trifuge (Beckman Instruments, Inc., Fullerton, CA, model
J-21C with rotor type JA-20) with a fixed angle rotor was
tried first at 10,000 rpm (approximately 8000 g_{av}) for

45

fifteen minutes, then at 5000 rpm (approximately 2000 g_{av})

for five minutes. In either case, the alum formed pellets

in the bottom of the centrifuge tube or formed smears down

the side of the tube, even in gradients with the highest

densities. An IEC (International Equipment Company, Divi-

sion of Damon, Boston, MA, Model CS) centrifuge with a

swinging basket rotor was tried at 2000 rpm for five min-

utes with similar results obtained. A tabletop clinical

centrifuge (International Equipment Company, Division of

Damon, Boston, MA, International Clinical Centrifuge model

CL) with a swinging basket rotor was tried at approximately

2000 rpm (half speed or approximately 1000 g) for two min-

utes with similar results. This alum sludge had a bulk

density as determined by pycnometer of 1.029 g/ml versus

water and 1.014 g/ml versus filtrate from the sludge. Be-

cause of the low bulk densities, it was unexpected that the

alum would have a particle density of greater than the

highest density sucrose solution used (1.224 g/ml). To de-

termine if the sucrose was interacting in some way with the

alum to change the particle density, two experiments were

undertaken.

The first was to take a sample of biological sludge

from a laboratory-scale activated sludge reactor with a

much lower bulk density (1.003 g/ml) and centrifuge the

sample under mild conditions in a sucrose gradient of 20%

(1.0743 g/ml) to 60% (1.2240 g/ml). At approximately 2000 rpm in the clinical centrifuge after two minutes, the biological sludge formed a band in the tube. To confirm that the isopycnic point had been reached, the tubes were centrifuged again at twice the initial speed for ten minutes. This time, the biological sludge had formed a pellet in the bottom of the tube.

The second experiment was to take two test tubes of 60% (1.2240 g/ml) sucrose and place into each tube a Pasteur pipette containing a few drops of either the alum (I) sludge or a few drops of the biological sludge. As the sucrose rose in the Pasteur pipettes, the sludge particles rose with the sucrose and floated near the top. The tubes were allowed to stand over night. In the morning, both sludges had settled to the bottom of the tubes.

The conclusions reached from these two experiments were that either the wet particles in both sludges were substantially denser than expected from bulk density measurements or the sucrose was removing or replacing the water in the sludge particles and thereby altering their densities.

In order to eliminate the possibility of the osmotic pressure of sucrose drawing water out of the particles, studies using Percoll diluted in water were initiated. Density marker beads (as described in Section 3.1.4) were

obtained along with a liter of Percoll. The beads are cal-
ibrated by the manufacturer for biological use. That is,
they are calibrated for solutions of Percoll diluted with
physiological saline (0.15M) or sucrose (0.25M) so that the
osmotic pressure of the medium is compatible with living
cells. In order to use the marker beads in solutions of
Percoll diluted in water the beads had to be recalibrated
in water as described in Section 3.1.4. Initial studies
utilized Percoll diluted in physiological saline. These
initial studies were done primarily to become familiar with
the use of the medium and marker beads as the manufacturer
had no product performance data for the use of these
products in water.

Continuous gradients using Percoll in saline were
formed by self-generation at high speeds in the Beckman
centrifuge. When the tubes containing Percoll at an aver-
age density of 1.100 g/ml were centrifuged at 14,000 rpm
(15,000 g_{av}) for forty five minutes, satisfactory resolu-
tion of the marker beads was obtained. When the alum (I)
sludge and biological sludge (density of 1.0004 g/ml for
that day) were layered on top of the preformed gradient of
Percoll in saline and centrifuged for twenty minutes at
3000 rpm in the Beckman centrifuge (as per the manufactur-
er's recommendation) the alum formed a smear down the side
of the tube. However, the biological sludge formed a broad

isopycnic band which did not move substantially even after spinning at 14,000 rpm for an additional ten minutes. The particle density for the biological sludge was determined to be between 1.042 g/ml and 1.045 g/ml. Figure 6 shows how this value was determined by plotting the location of the sludge band with respect to the density marker beads.

Upon the suggestion of the manufacturer, glass centrifuge tubes were obtained to determine if the alum was sticking to the tube wall rather than passing through the medium. The use of glass tubes did not improve the performance of alum (I) sludge in Percoll diluted with saline. Sometime later, glass tubes were sprayed with various lubricants including spray silicone, WD-40, and vegetable lecithin. A sucrose gradient between 20% (1.0743 g/ml) and 80% (1.2924 g/ml) was placed in the tubes and centrifuged at 3000 rpm in the Beckman centrifuge for fifteen minutes with alum (I) sludge layered on top. Although a sucrose medium had not been previously successful with alum (I) sludge or biological sludge, 80% sucrose was selected for this experiment because it was the highest density medium available. It was hoped that the lubricant would prevent sticking of the sludge to the tube walls so that an isopycnic band could form. None of the lubricants worked in this regard. Conditioning the alum (I) sludge likewise did not prevent sticking. Because of the failure to obtain
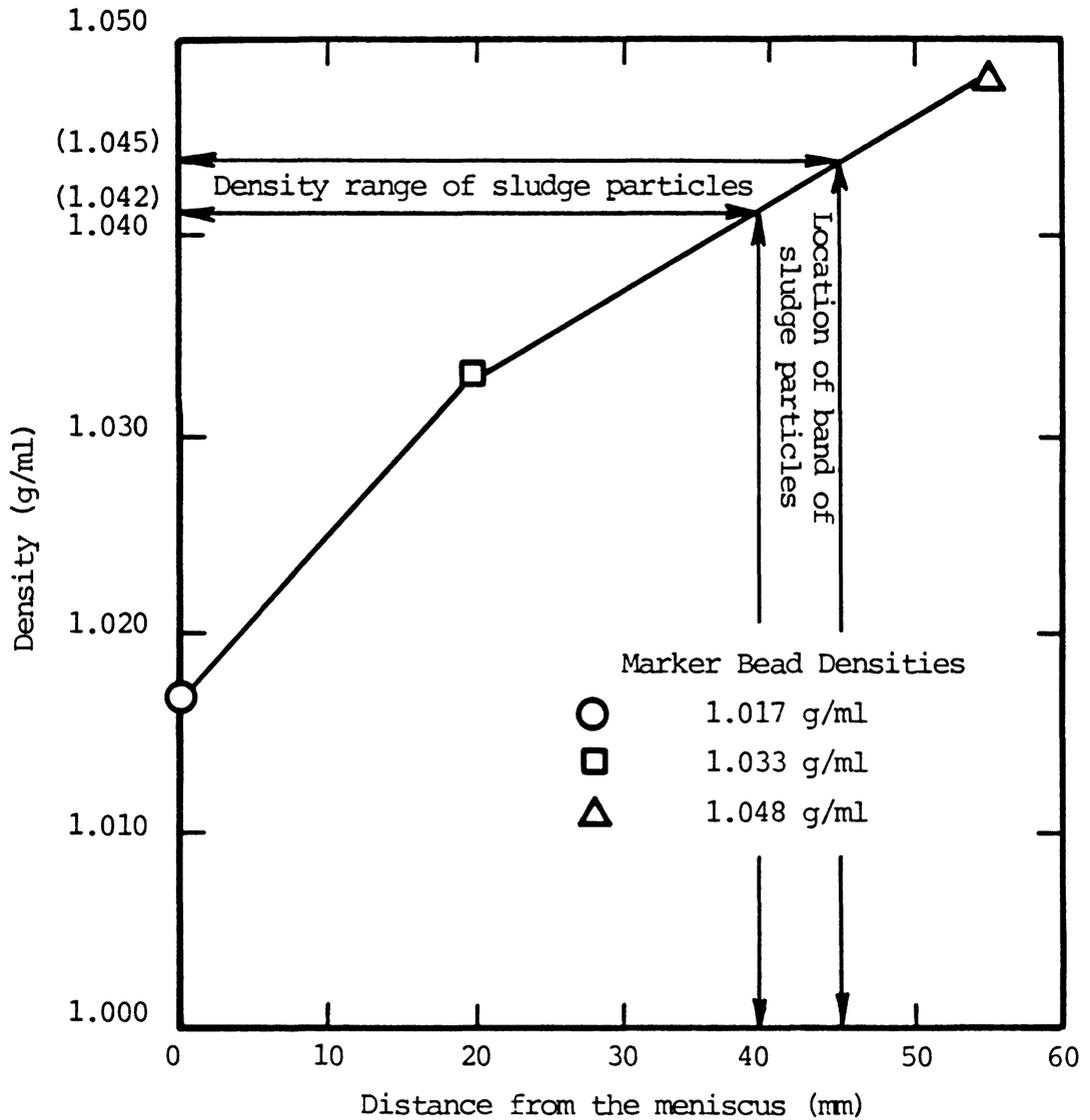
Figure 6: Density of biological sludge particles determined using density marker beads in a continuous gradient of Percoll diluted in saline

an isopycnic band for conditioned or unconditioned alum
sludge in either medium under the variety of circumstances
investigated even though reproducible bands for biological
sludge were obtained under certain circumstances, it was
concluded that the problem was the use of an angle head
rotor. During centrifugation in an angle head rotor, the
gradient layers form parallel to the walls of the tube and
return to horizontal when the rotor stops. Apparently,
alum sludge adhered strongly to the walls of the centrifuge
so that it did not return to a horizontal band with the
gradient.

At this point, studies using Percoll diluted in water
were initiated. The first experiment was done by making a
step gradient with steps between 1.010 g/ml and 1.130 g/ml
at 0.010 g/ml intervals. This step gradient was made for
two reasons: first, to get an approximate idea where the
density marker beads would statify in this medium; second-
ly, to see if the sludges would form bands in a step grad-
ient more readily than in the continuous gradients pre-
viously attempted. Biological sludge, copper hydroxide
sludge, nickel hydroxide (I) sludge, and conditioned and
unconditioned alum (I) sludge as well as the density marker
beads were evaluated. The tubes containing the step grad-
ients were centrifuged under much milder conditions than
the continuous gradients (360 g) in a swinging basket

rotor. All of the sludges except the biological sludge formed pellets in the bottom of the tubes. Biological sludge formed a band at the interface of steps 1.050 and 1.060 g/ml.

The values of sludge particle density indicated by the studies to this point were all higher than anticipated and were also much higher than values indicated by the modified settling test of Lagvankar and Gemmell (1968). There was concern that as sludge particles passed through the medium, water in the particles was being replaced by the medium, causing the overall density of the particles to increase. This seemed to be supported by an observation of the particle settling test that particle movement in the test tubes needed to be observed within the first twenty seconds after introducing the particles into the medium. This was because all particles sank after twenty to thirty seconds even in tubes of the highest density and regardless of the medium used (Percoll or sucrose).

In order to determine if centrifugation caused media to enter the sludge particles, two tests were performed. As density marker beads are discrete beads of Sephadex, they do not have water associated within them as sludge particles or flocs do. In the first test, sludge particles and density marker beads were allowed to stand over night in settling test tubes of Percoll in water at densities which

caused the particles and beads to float initially. Upon standing, the sludge particles sank after about thirty seconds while the density marker beads remained floating overnight.

The second test involved making particle settling test measurements of four sludges before centrifugation. Then, all four sludges were centrifuged through water and their particle densities measured by the same settling test. Two of the sludges were then centrifuged through Percoll in water and the particle densities measured again by the settling test. If centrifugation through water caused an increase in particle density, then the particles were being collapsed by the centrifugal force. If, on the other hand, particle densities were increasing because of gradient media penetrating the sludge particle, the particle density should increase after centrifugation through Percoll in water and not after centrifugation through water alone. Centrifugation was carried out in the Beckman centrifuge at 3000 rpm (322 g_{av}) for twenty minutes. The results are summarized in Table 5. Particle densities were also determined by the modified settling test of Lagvankar and Gemmell (1968) using Percoll diluted with water.

Because the particles appeared heavier after centrifugation through Percoll the results of these tests indicated that some part of the water in the sludge particles was

TABLE 5

Tests to Determine Media Penetration of Sludge Particles

Sludge	Particle Density, g/ml[*]		
	Initial	After Centrifugation through	
		Water	Percoll in water
alum(I)	1.030-1.040	1.030-1.040	
conditioned alum(I)	1.020-1.030	1.020-1.030	1.120-1.130
copper hydroxide	1.120-1.130	1.120-1.130	
nickel hydroxide(I)	1.010-1.020	1.010-1.020	1.110-1.120

[*] Particle densities determined by the modified particle settling test of Lagvankar and Gemmell(1968) as described in Section 3.2.5.

being replaced by the density medium during centrifugation. The isopycnic point reached by biological sludge centrifuged through Percoll suggested that this might be some fixed portion of the water.

The last experiment in this series was an attempt to determine if non-biological sludges could reach an isopycnic point in an isotonic medium such as Percoll diluted in water if the Percoll could be concentrated above the 1.130 g/ml as received from the manufacturer. A description of the method used to concentrate the medium is given in Section 3.2.6. A maximum density of 1.214 g/ml as determined by pycnometer was achieved.

Because the density marker beads are not useful for densities above 1.14 g/ml and also because of the small volume of Percoll obtained after concentration, a continuous gradient was not practical. A step gradient with the steps at 0.010 g/ml intervals between 1.130 g/ml and 1.210 g/ml was used. Samples of the four sludges used (alum (II) sludge, lime sludge, iron hydroxide sludge, and nickel hydroxide (II) sludge) were layered onto the gradient after the sludges had been conditioned to help prevent sticking to the tube walls. The tubes were placed in the clinical centrifuge and centrifuged at approximately 1000 rpm for sixty seconds. The level of the sludges was checked and the sludges were centrifuged twice more to be certain that

the isopycnic interface had been reached. The final particle densities for each of the sludges were as follows: iron, between 1.130 and 1.140 g/ml; alum (II), between 1.150 and 1.160 g/ml; nickel hydroxide (II), between 1.160 and 1.170 g/ml; and lime, greater than 1.210 g/ml.

4.1.2 Particle Settling Tests on Sludges after Thickening and Mechanical Dewatering

Several methods were used to explore the meaning and validity of the particle settling test described in Section 3.2.5. Particle densities determined by this test were compared to bulk density measurements made by pycnometer, they were made after sludges had been thickened and mechanically dewatered, and they were evaluated by comparison to other methods of particle density determination, particularly the mathematical determination of Javaheri and Dick (1969).

The first evaluation made was an approximate comparison of bulk density versus water (ρ_{bw}) and particle density by settling in sucrose tubes (ρ_{ps}) and settling in tubes of Percoll in water (ρ_{pp}). These results are summarized in Table 6. The range of sucrose solutions in this test included 2% (1.0079 g/ml), 6% (1.0229 g/ml), 8% (1.0308 g/ml), 10% (1,0384 g/ml), 12% (1.0443 g/ml), 15% (1.0557 g/ml), 20% (1.0743 g/ml) and 30% (1.1237 g/ml). The Percoll solutions in water were regular steps of 0.010 g/ml from 1.010

TABLE 6

Preliminary Comparison of Bulk Density Determinations with Particle Density Data Obtained from Particle Settling Tests using Sucrose and Percoll as the Density Media

Sludge	Solids Conc.(%)	ρ_{bw}, g/ml [*]	ρ_{ps}, g/ml [**]	ρ_{pp}, g/ml [***]
conditioned alum (I)	3.15	1.028	1.023-1.044	1.020-1.030
alum (I)	4.12	1.033	1.023-1.031	1.030-1.040
copper hydroxide	17.60	1.144	1.074-1.124	1.120-1.130
nickel hydroxide (I)	2.45	1.020	1.008-1.023	1.010-1.020
biological	0.23	1.002	<1.008	<1.010

[*]   Bulk density determined versus water
[**]   Particle density determined by particle settling tests in sucrose
[***] Particle density determined by particle settling tests in Percoll

g/ml to 1.130 g/ml. Although the ranges in the settling tests were broad, it was felt that the settling test using Percoll in water paralleled the sucrose test closely enough that the Percoll settling test could be used for the remainder of the experimental study.

The next evaluation was that of comparing the bulk densities of sludges after mechanical dewatering to their initial particle densities as determined by the particle settling tests in sucrose or Percoll. Theoretically, the density of the particles should be the maximum bulk density achievable by mechanical dewatering. If mechanical dewatering removed primarily the water between floc particles, then at maximum mechanical dewatering, only particles with their associated density would remain. In the initial study, two sludges (nickel hydroxide I and lime) were dewatered using the vacuum dewatering apparatus described in Figure 2. Lime was also dewatered using the Beckman centrifuge. The data are summarized in Table 7; the data for lime are also presented in Figure 7. At this point, work with biological sludges was discontinued. Denitrification was unavoidable during the time required to take bulk and particle density measurements. The resulting gas bubbles made these determinations impossible.

The data in Table 7 demonstrate that the sludge bulk densities produced after mechanical dewatering easily

TABLE 7

Relationship between Sludge Bulk Density, Solids Concentration,
and Particle Density obtained before/after Vacuum Filtration
or Centrifugation

Sludge	Dewatering Method		Solids Conc.(%)	ρ_{bw}, g/ml [*]	ρ_{pp}, g/ml [**]
nickel hydroxide (I)	vacuum filtration	initial	2.45	1.011	1.010–1.020
		final	26.20	1.168	---
lime	vacuum filtration	initial	12.37	1.083	1.080–1.090
		final	44.58	1.361	---
	centrifugation	initial	12.37	1.083	1.080–1.090
		final	44.75	1.327	---

[*] Bulk density determined versus water
[**] Particle density determined by particle settling tests in Percoll
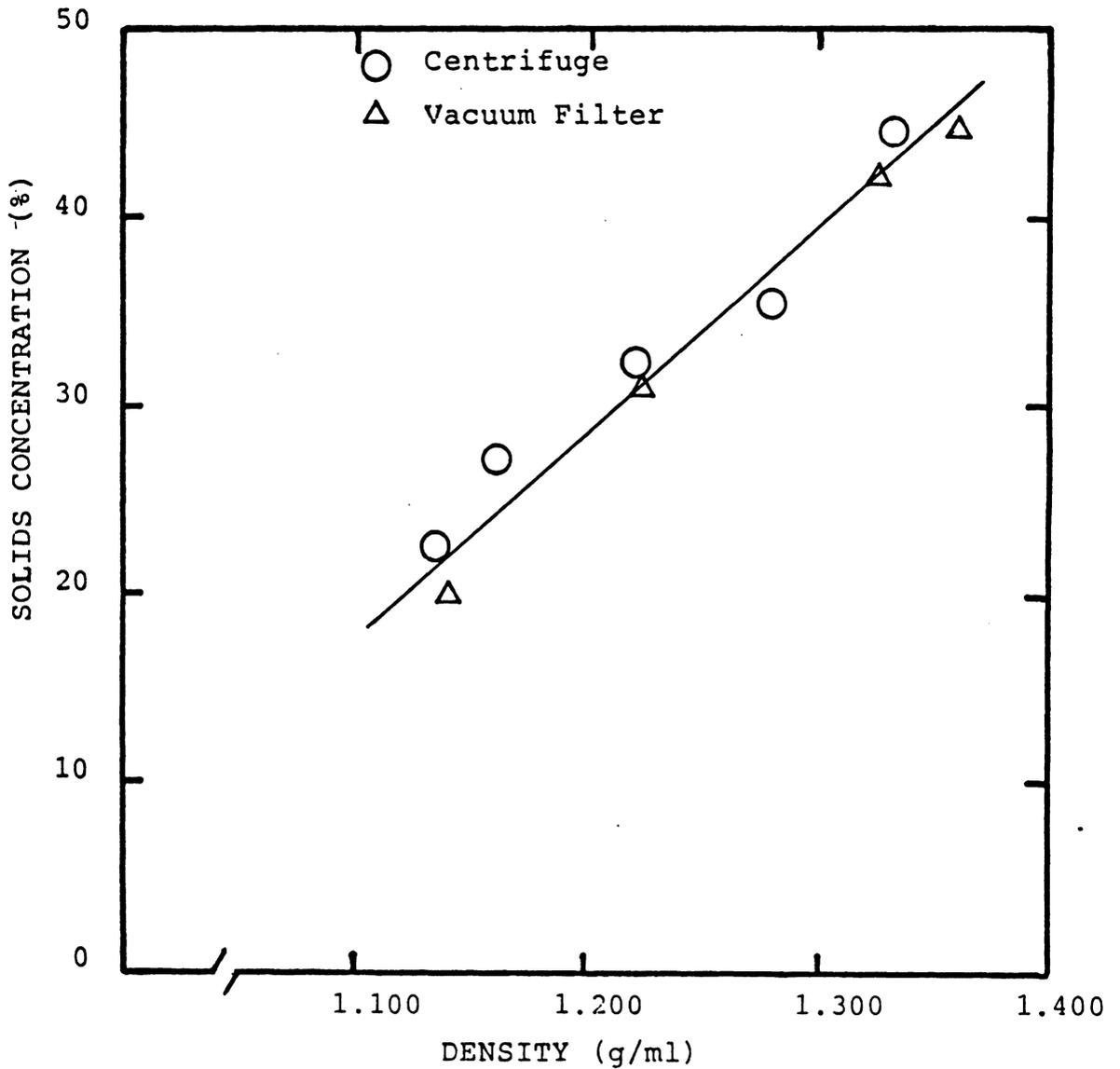
Figure 7: Comparison of lime sludge bulk density after
varying degrees of mechanical dewatering by
vacuum filtration and centrifugation

exceeded the original particle densities as determined by
the particle settling tests. Two possible explanations for
this phenomenon were considered. The first possibility was
that the density values from the particle settling tests
were really bulk densities rather than wet particle densi-
ties and that the true wet particle densities were much
higher than the settling test revealed. The second possi-
bility was that the sludge particles released water during
dewatering, resulting in an increased particle density. If
this were true, then particle settling tests done along
with dewatering should demonstrate a change in particle
density.

In order to evaluate these two explanations, two ex-
periments were undertaken. First, bulk and particle den-
sity determinations were made before and after gravity
thickening of sludges. Second, vacuum dewatering was per-
formed on different sludges to increasing solids concen-
trations using the same apparatus as before. At each in-
creasing level of solids concentration, bulk and particle
density determinations were performed. It was felt that
appreciable changes in the floc particle structure should
not take place under the mild conditions of gravity thick-
ening, even if mechanical dewatering had the effect of
collapsing the floc. Table 8 summarizes the results of
the bulk and particle density determinations before and

TABLE 8

Comparison of Bulk and Particle Densities Before and
after Gravity Thickening

Sludge	Solids Conc.(%)	ρ_{bw}, g/ml [*]	ρ_{pp}, g/ml [**]
Stock sludge solutions, undiluted, before settling:			
nickel hydroxide (II)	1.82	1.011	1.010-1.012
nickel hydroxide, fresh precipitate	2.00	1.017	1.014-1.016
lime	14.35	1.091	1.090-1.092
alum (II)	5.32	1.036	1.032[a]
iron hydroxide	2.33	1.016	1.016

Sludge	% Stock in mix	Hours settled	Solids Conc.(%)	ρ_{bw}, g/ml [*]	ρ_{pp}, g/ml [**]
nickel hydroxide (II)	75	12	2.35	1.017	1.014-1.016
	50	12	2.25	1.015	1.014
	35	12	2.12	1.016	1.014
nickel hydroxide, fresh precipitate					
	100	4	3.00	1.025	1.022-1.024
	100	12	3.30	1.028	1.026-1.028
lime	60	12	—	1.109	1.110
	40	12	—	1.095	1.090-1.092
	20	12	—	1.088	1.082-1.084
alum (II)	75	12	3.12	1.018	1.018
	35	12	3.01	1.020	1.018
	15	12	3.21	1.021	1.018-1.020
iron hydroxide	50	12	2.62	1.017	1.016-1.018
	30	12	2.79	1.018	1.016-1.018
	20	12	2.91	1.017	1.016

[*]   Bulk density versus water
[**] Particle density determined by particle settling tests in Percoll
[a]   In a particle settling test, when a particle neither rose nor fell during the 20 seconds of the test, the particle was considered to have the density value of the medium in that test tube rather than being assigned a range

after 12 hours of gravity thickening of several sludges at various initial concentrations. Additionally, freshly precipitated nickel hydroxide sludge was allowed to thicken. This was done to determine if repeated thickening and redilution of the stock sludges had changed the thickening properties significantly from those of fresh sludge.

The data in Table 8 strongly suggest that the particle settling test is actually a coarse measurement of bulk density, either this, or the sludge particle density so closely parallels bulk density this test is not sensitive enough to distinguish the two. The results of the vacuum dewatering tests summarized in Table 9 parallel the results of the gravity thickening tests. The changes in particle density so closely parallel the changes in bulk density that the conclusion was made that the wet particle settling test is not a conclusive measurement of particle density.

4.2 ANALYSIS OF METAL SLUDGE THICKENING DATA

In this part of the research the thickening and density data from four sludges (iron hydroxide sludge, nickel hydroxide II sludge, aluminum hydroxide II sludge, and lime sludge) were analysed using the mathematical techniques of Michaels and Bolger (1962) and Javaheri and Dick (1969) as described in Section 2.1.2. Results obtained from these studies on all four sludges are summarized in this section; sample calculations of the mathematical techniques are

TABLE 9

Comparison of Bulk and Particle Densities after
Mechanical Dewatering by Vacuum Filtration

Sludge	Solids Conc.(%)	ρ_{bw}, g/ml [*]	ρ_{pp}, g/ml [**]
iron hydroxide	2.33	1.016	1.016[a]
	2.71	1.017	1.016-1.018
	5.17	1.033	1.032
nickel hydroxide (II)	1.82	1.011	1.010-1.012
	2.37	1.018	1.014
	2.84	1.020	1.018
	3.35	1.024	1.024
	--	1.028	1.030
	--	1.064	1.068-1.072
	--	1.074	1.078-1.082

[*]   Bulk density determined versus water
[**] Particle density determined by particle settling
      tests in Percoll
[a]   In a particle settling test, when a particle neither
      rose nor fell during the 20 seconds of the test, the
      particle was considered to have the density value of
      the medium in that test tube rather than being
      assigned a range

presented only for the aluminum hydroxide sludge studied.
All pertinent data for the four sludges studied are con-
tained in Appendix B.

A typical set of batch thickening curves for the alum-
inum hydroxide (II) sludge is given in Figure 8. For each
batch thickening test, the initial solids concentration
(C_o) and bulk density (ρ_{bw}) were measured. The interfacial
settling velocity (V_s) was calculated from the slope of the
linear portion of the batch thickening test using a linear
regression analysis. The dry particle solids density (ρ_k)
was calculated using bulk density and settled solids con-
centration data by use of the following mass balance equa-
tion:

$$\frac{100}{\rho_{bw}} = \frac{(100 - x)}{w} + \frac{x}{\rho_k} \qquad (4-1)$$

where   x = mass solids concentration (%)

  (100 - x) = sludge water content (%)

  ρ_w = water density (1.0 g/cm^3)

Using this method, ρ_k values were calculated for each of
the four sludges tested. The corresponding values are as
follows: aluminum hydroxide (II) sludge, 2.87 g/ml; iron
hydroxide sludge, 2.60 g/ml; nickel hydroxide (II) sludge,
3.77 g/ml; and lime sludge, 2.47 g/ml.

The sludge thickening data were initially analyzed
according to standard semi-logrithmic graphical techniques
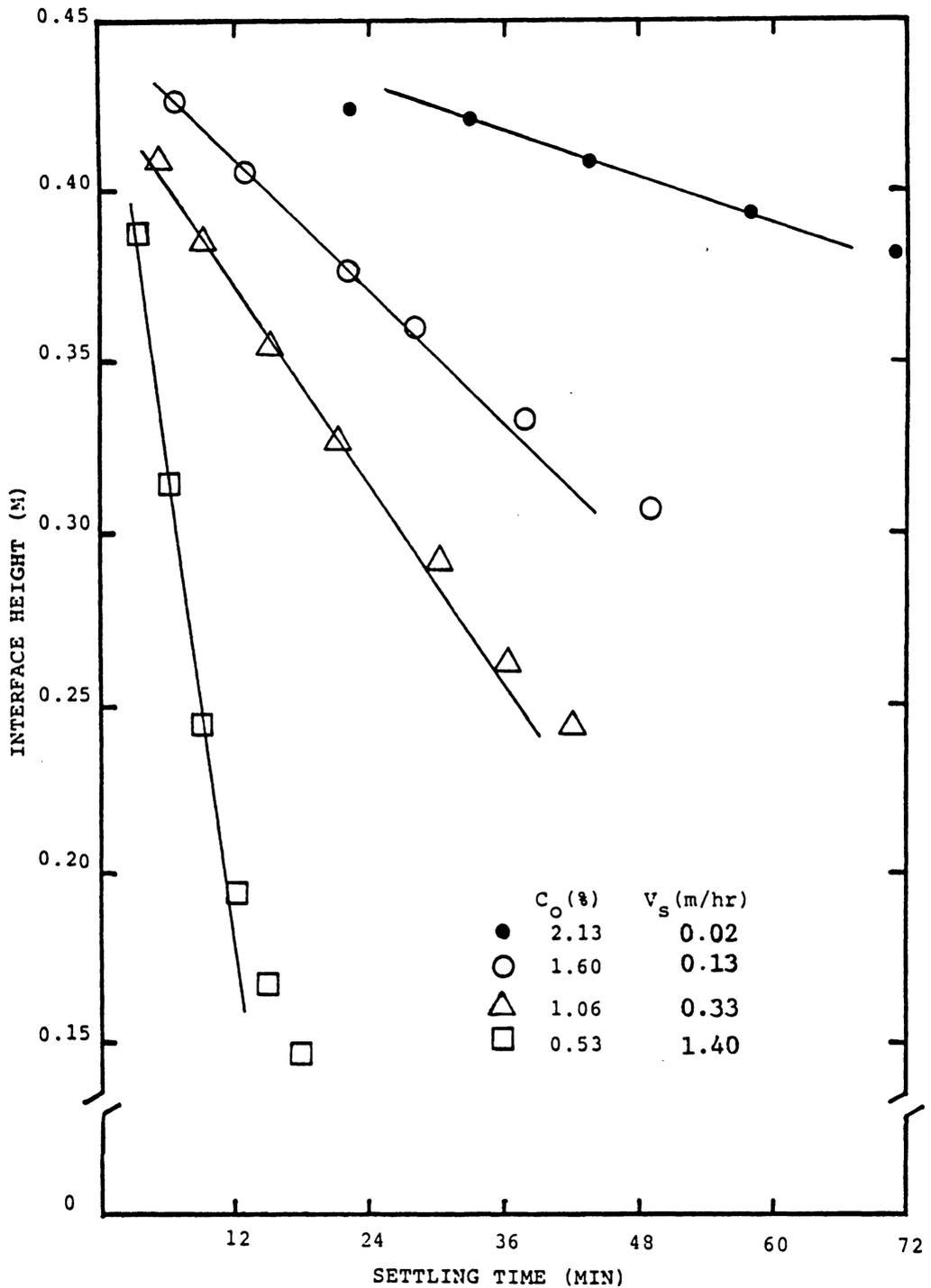
Figure 8: Determination of interfacial settling veloci-
ties for aluminum hydroxide (II) sludge from
batch thickening tests

as shown in Figure 9. The sludges tested resulted in four distinct graphs with varying slope and y-intercept values. Analysis of the slope values showed that, as the aggregate density of the sludge particles increased, the corresponding slope of the graph decreased (aggregate density values are given in Table 11), implying an inverse relationship between the two parameters.

An attempt was made to also analyze the data on a floc volume fraction basis using the techniques of Michaels and Bolger (1962) and Javaheri and Dick (1969). The use of these techniques required the conversion of all sludge solids concentration data from a mass basis to a volume basis. Using calculated values for ρ_k and initial solids concentration data, it was possible to convert the mass solids concentration values to corresponding dry solids volume fraction values (Φ_k) by:

$$\Phi_k = \frac{C_o}{\rho_k} \qquad (4-2)$$

Corresponding values of Φ_k and interfacial settling velocity (expressed as $1/V_s^{4.65}$) were determined (see Table 10 for aluminum hydroxide II sludge data) and plotted for each sludge tested. Representative data of this type are plotted in Figures 10 through 13.

By use of Equation 2-21, it was possible to analyze the data plotted in Figures 10 through 13 to obtain
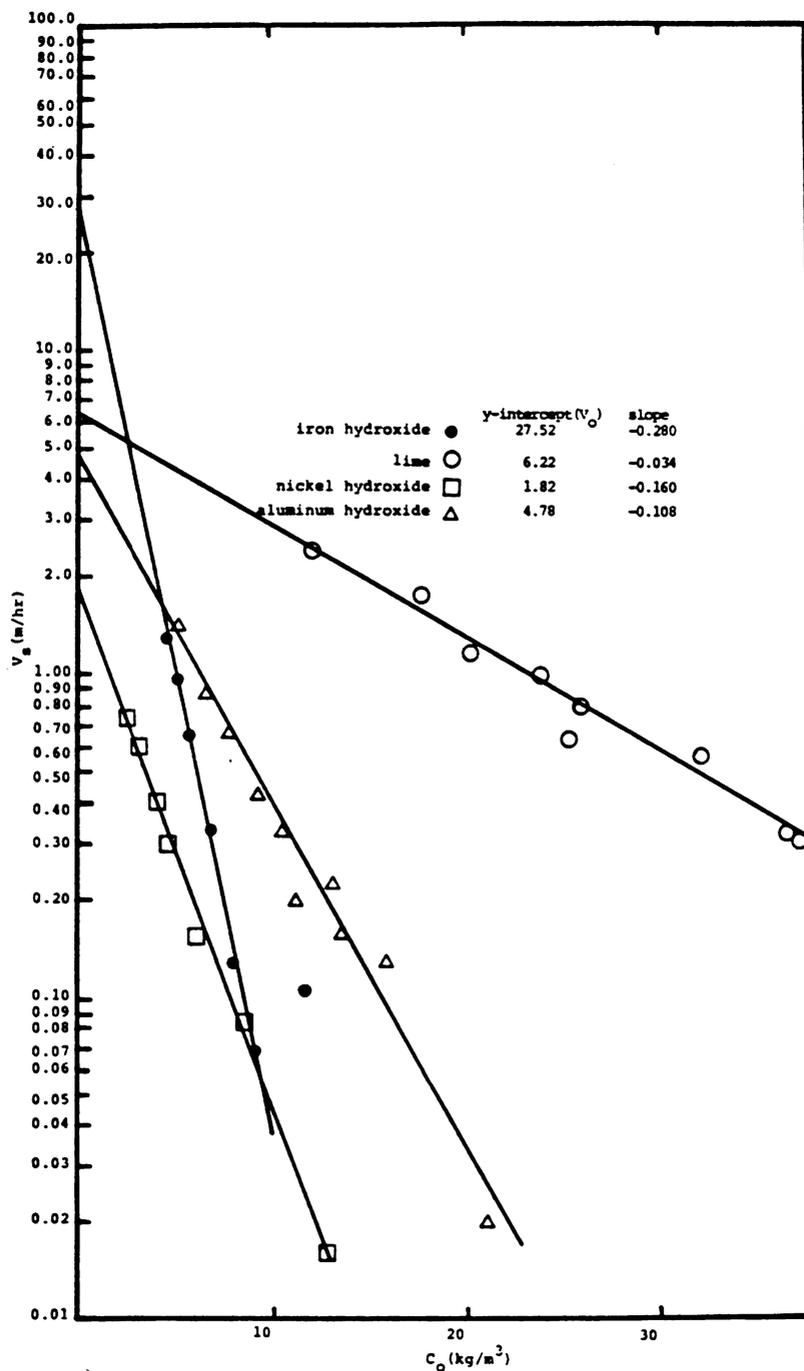
Figure 9: Plots of interfacial settling velocity (V_s) versus initial solids concentration (C_o) for four sludges

TABLE 10

Values of Solids Concentration (C_O), Interfacial
Settling Velocity (V_s), and Volume Fraction of
Solids (ϕ_k) for Aluminum Hydroxide (II) Sludge

Solids Concentration (C_O = kg/m^3)	V_s (m/hr)	$V_s^{1/4.65}$	ϕ_k (1 x 10^{-3})
5.3	1.405	1.08	1.87
6.7	0.883	0.97	2.36
8.0	0.674	0.92	2.82
9.4	0.420	0.83	3.31
10.6	0.327	0.79	3.73
11.4	0.204	0.71	4.01
13.3	0.227	0.73	4.68
13.7	0.156	0.67	4.82
16.0	0.133	0.64	5.63
21.3	0.020	0.43	7.50
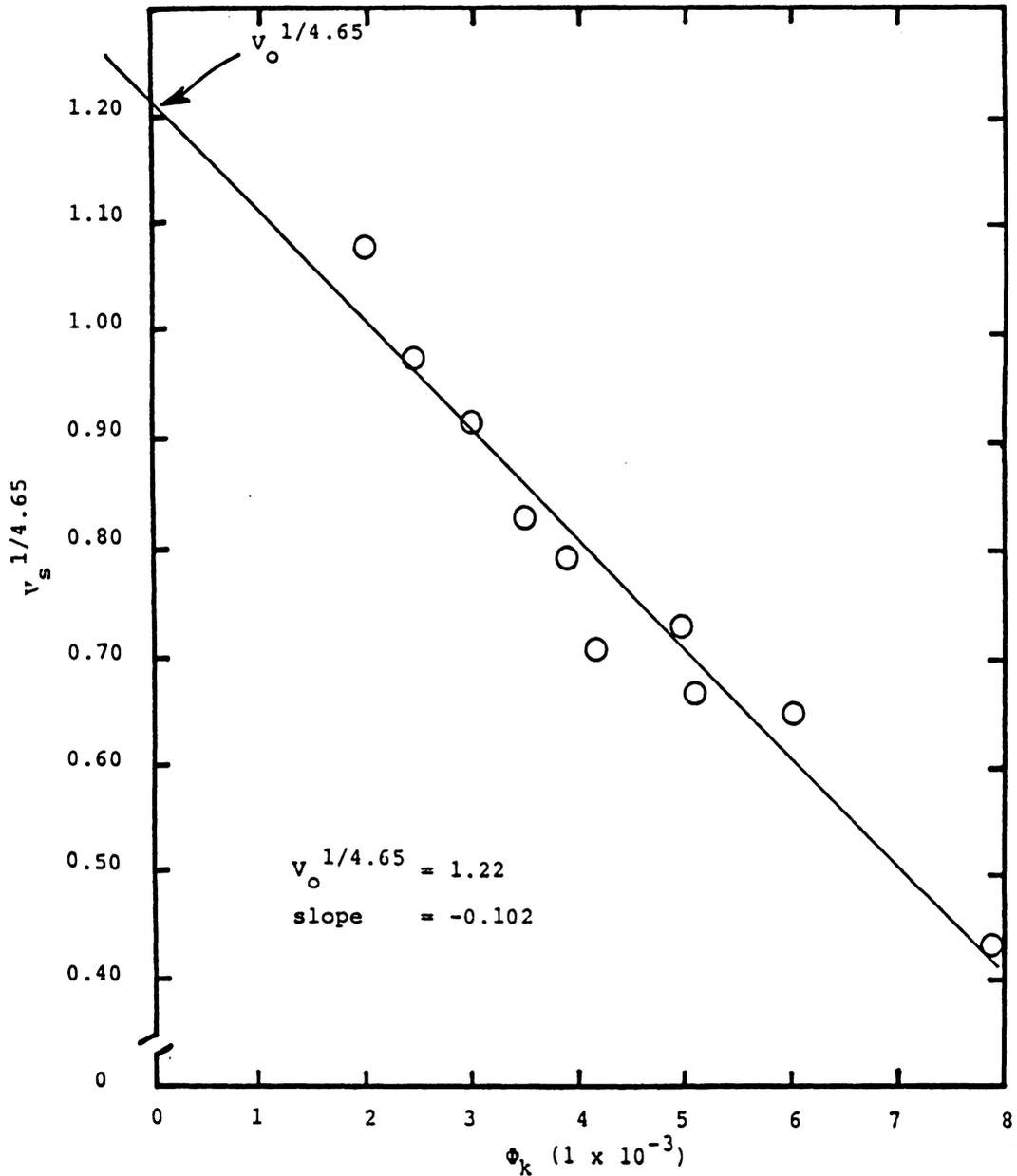
Figure 10: Plot of interfacial settling velocity expressed as $V_s^{1/4.65}$ versus volume fraction of dry solids, $\Phi_k (1 \times 10^{-3})$, for aluminum hydroxide (II) sludge
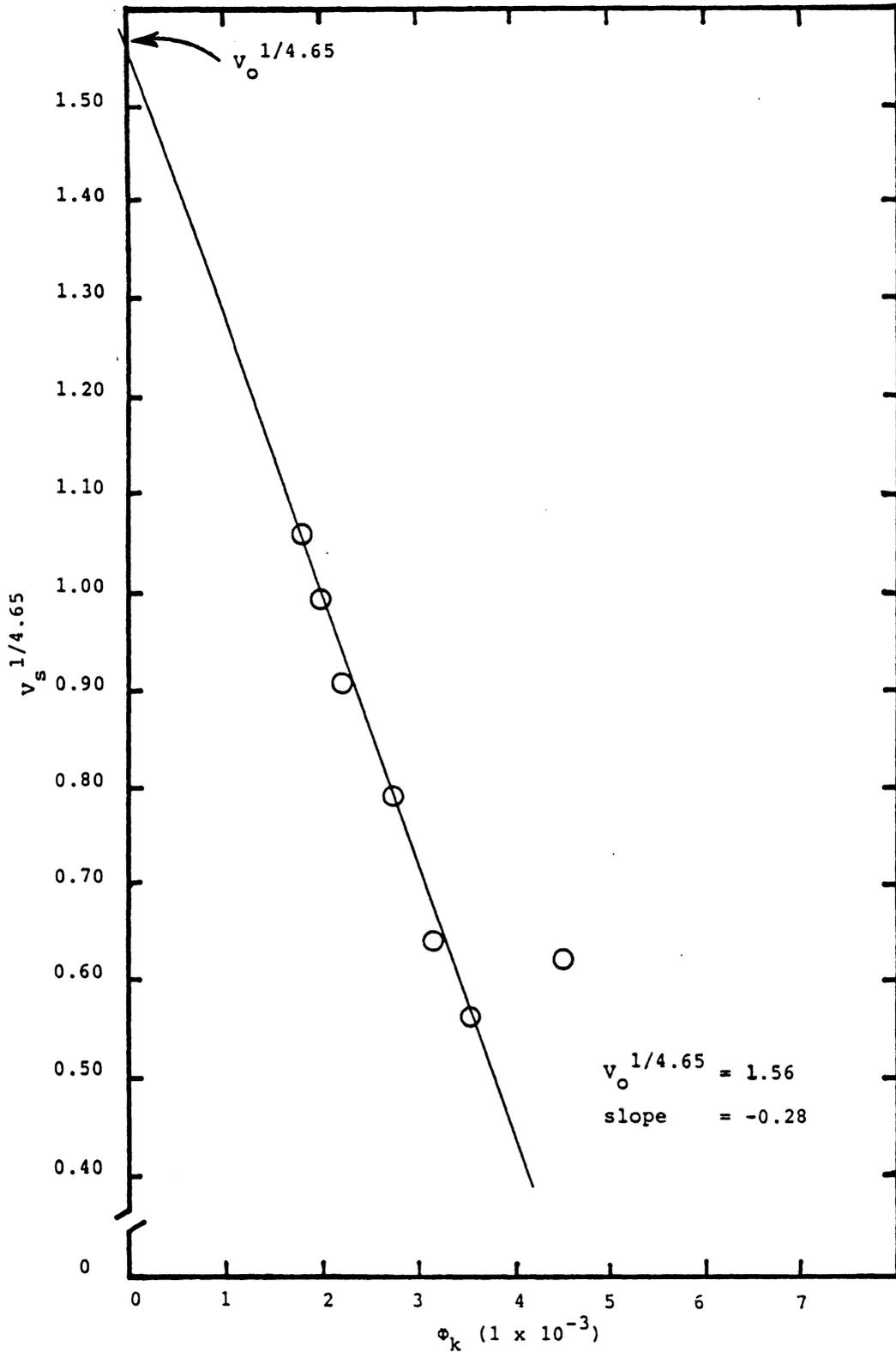
Figure 11: Plot of interfacial settling velocity expressed
as $V_s^{1/4.65}$ versus volume fraction of dry
solids, $\Phi_k(1 \times 10^{-3})$, for iron hydroxide sludge
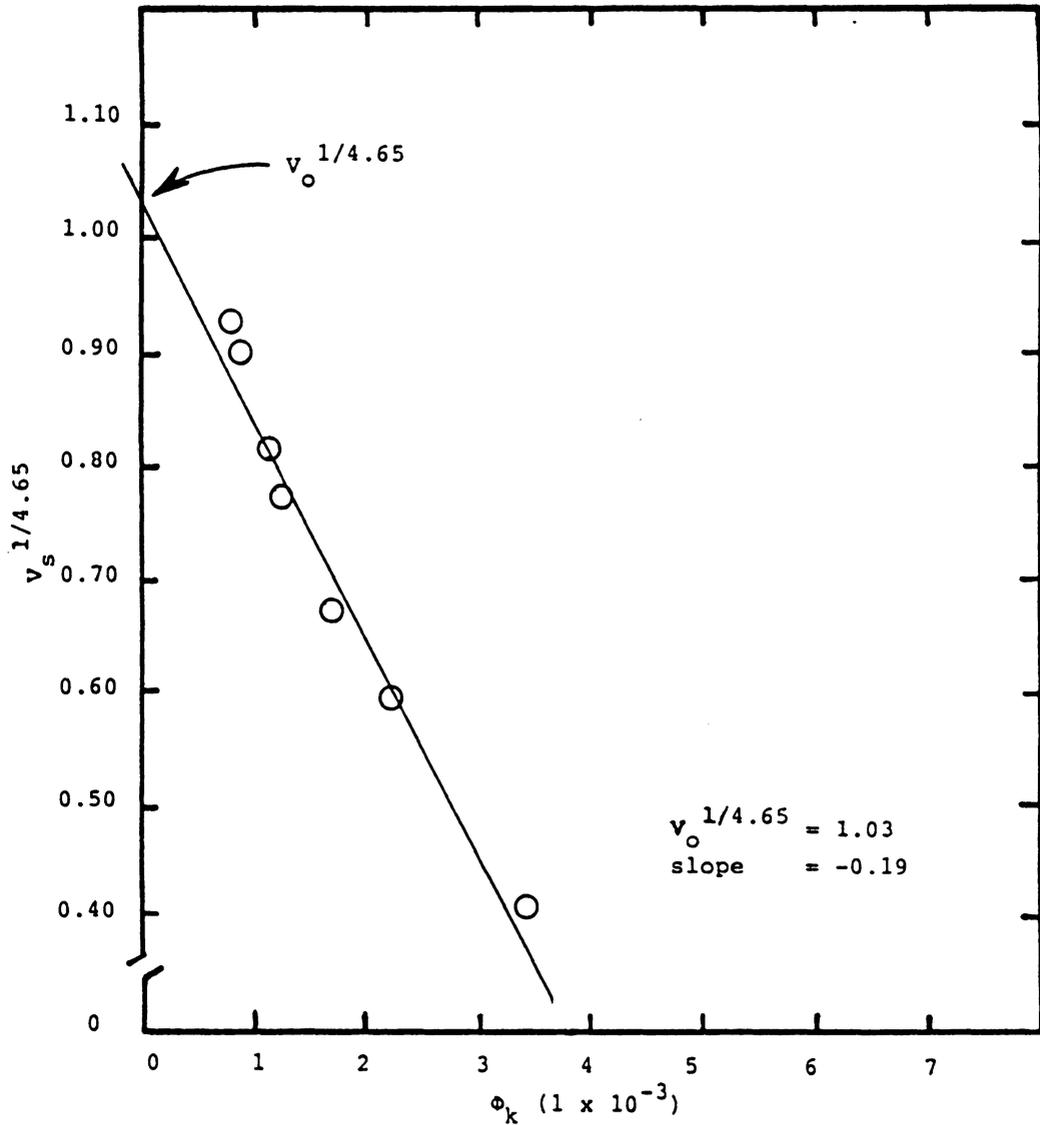
Figure 12: Plot of interfacial settling velocity expressed as $V_s^{1/4.65}$ versus volume fraction of dry solids, $\Phi_k (1 \times 10^{-3})$, for nickel hydroxide (II) sludge
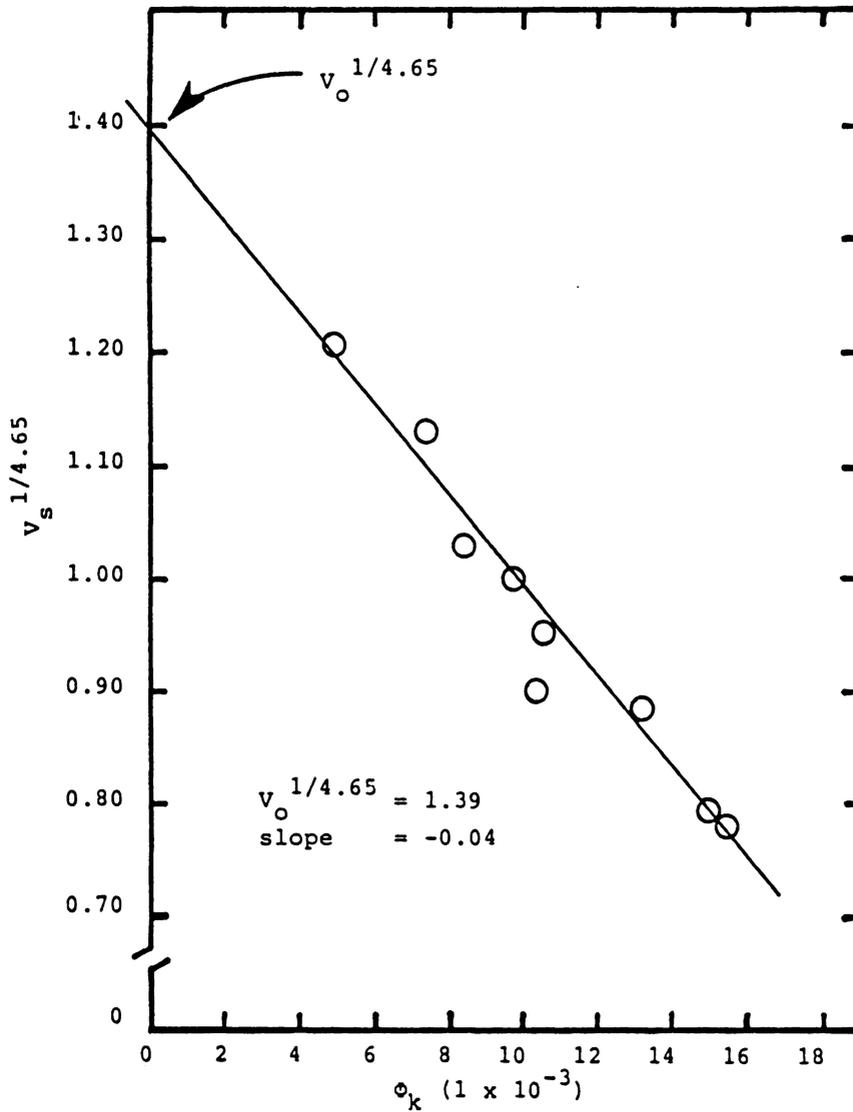
Figure 13: Plot of interfacial settling velocity expressed as $V_s^{1/4.65}$ versus volume fraction of dry solids, $\Phi_k (1 \times 10^{-3})$, for lime sludge

estimates for both the Stokes' law settling velocity value
(V_o) and the AVI for each sludge. Also, Equation 2-20 was
used to estimate the density of aggregates contained in
each sludge suspension. These calculated values are sum-
marized in Table 11. A review of the values presented in
Table 11 allows for certain interesting observations. For
example, there is an inverse relationship between aggregate
density and the aggregate volume index, a result predicted
from theoretical considerations. A large value for the AVI
would indicate an increased aggregate water content; thus,
one would expect a corresponding decrease in observed ag-
gregate density. The calculated Stokes' law velocity
values may also be used to estimate the relative size of
aggregates present in each sludge. For example, the iron
hydroxide sludge was apparently characterized by aggregates
of large size but extremely low density.

It should be noted that, with each of the four sludges
studied, the graphs presented in Figures 10 through 13 were
assumed to reasonably approximate straight line relation-
ships. Thus, the calculated values for V_o and AVI would
not change as a function of the extent of thickening. This
result is different than that observed by Javaheri and Dick
(1969) in their work with biological sludges. The authors
found that, as biological sludges were thickened into the
area of transition and compression thickening, there was an

TABLE 11

Values of AVI, V_o and ρ_a Derived for Four Metal Sludges from Plots of $V_s^{1/4.65}$ versus Φ_k

Sludge	AVI	V_o (m/hr)	ρ_a (g/ml)
aluminum hydroxide (II)	77.0	2.33	1.024
iron hydroxide	181.1	7.91	1.009
nickel hydroxide (II)	157.5	1.76	1.018
lime	30.1	4.78	1.049

observed curvature in the graphs of $V_s^{1/4.65}$ and ϕ_k. By taking the slope of tangents to these plots at various locations, Javaheri and Dick (1969) were able to calculate the changes in both the V_o and AVI values. They concluded that their results indicated a release of significant amounts of water from the aggregates during the later stages of thickening, resulting in both a decrease in aggregate size and a corresponding increase in aggregate density. This phenomenon was not observed with the four metal sludges tested during this study. However, the only data included in the mathematical analysis was that which corresponded to the zone settling portion of the thickening process. It is possible that a similar trend to that noted by Javaheri and Dick (1969) may have been observed had the sludges been analyzed completely through the compression phase of thickening.

Figure 14 summarizes the observed thickening velocity data as a function of the total sludge volume occupied by sludge aggregates. The ϕ_a values were calculated for each sludge by a knowledge of mass solids concentrations and the associated AVI value. Figure 14 shows that the data develop a series of four nearly parallel lines with vertical displacement being directly related to the size of aggregates present in each sludge. These results allow for insight into the thickening process. For example, in
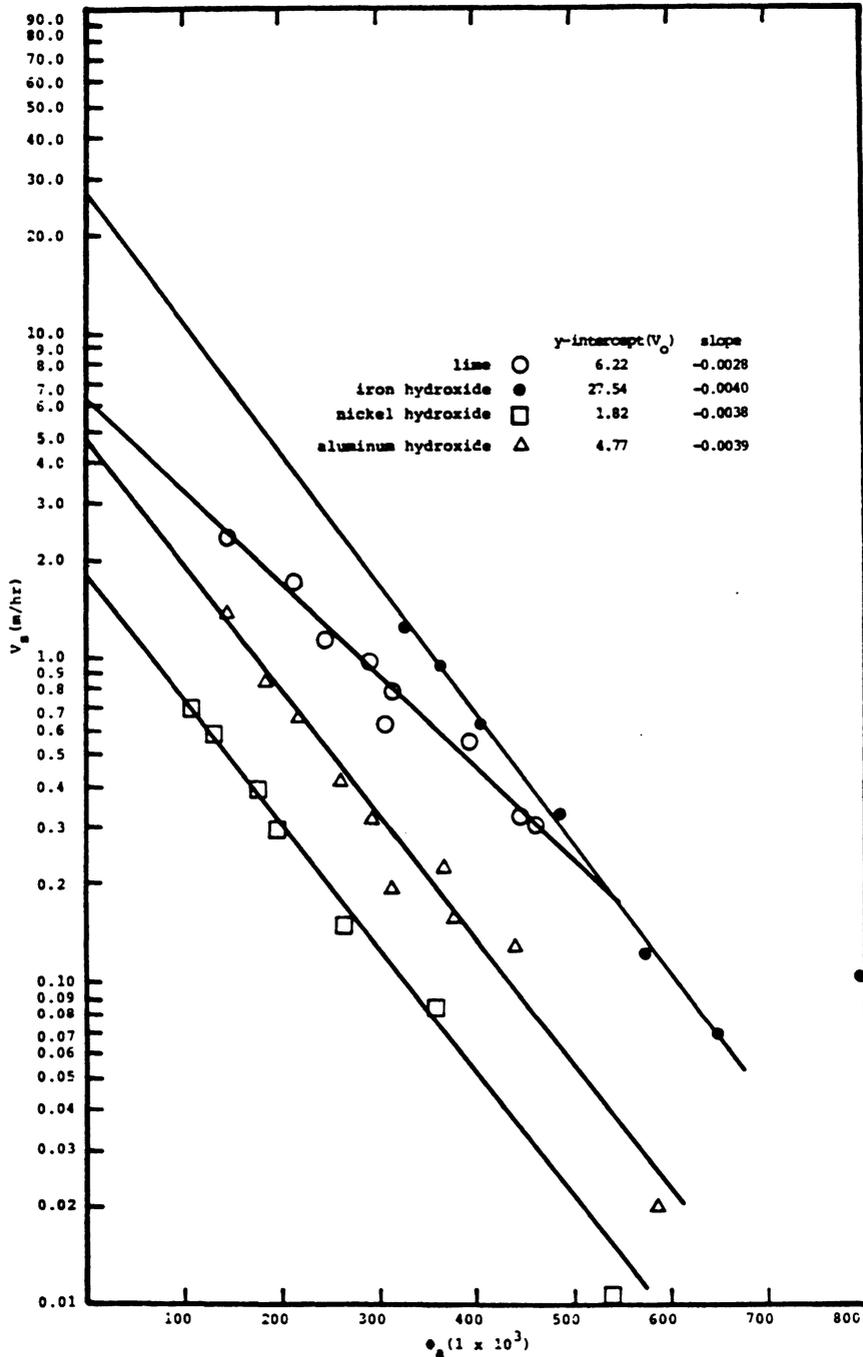
Figure 14: Plots of interfacial settling velocity versus aggregate density for four metal sludges showing vertical displacement directly corresponding to the variations in aggregate size between sludges

reviewing Figure 9, it is possible to rationally explain
the observed difference when plotting the data on a mass
solids basis. The reason that the iron hydroxide sludge
has such a greater slope than the lime sludge is that, for
a given change in mass solids concentration, the iron hy-
droxide sludge has a much greater increase in aggretate
volume fraction due to the greater amount of water included
in the iron hydroxide aggregates. Figure 14 shows that,
for any given value of Φ_a, the iron hydroxide sludge set-
tles fastest because of the fact that it has the largest
size aggregates. The observed characteristic of better
thickening of the lime sludge is simply a reflection of the
increased density of the lime aggregates.

Up to this point in this discussion, the terms "parti-
cle", "floc" and "aggregate" have been loosely interchange-
able and have all been used to refer to the largest dis-
crete entity in a suspension that would settle as a unit.
This "looseness" of terminology is not uncommon in the lit-
erature but served as a source of confusion in the inter-
pretation of the laboratory data obtained in this research.
As noted by Michaels and Bolger (1962) (summarized in Sec-
tion 2.1.2) the three terms refer to three separate entit-
ies, each with its own characteristics, particles being a
subunit of flocs and flocs being a subunit of aggregates.
In reviewing all of the laboratory data, particularly the

direct determinations of wet particle density, returning to the definitions of Michaels and Bolger (1962) helped to better interpret all of the laboratory data obtained. These definitions will be adhered to for the remainder of this discussion.

One last aspect of the sludge analysis was an attempt to better understand the association of water with sludge aggregates. This would also aid in evaluating the type of water which may and may not be removed by various dewatering methods. Table 12 summarizes the density values determined by the various direct laboratory methods and compares them to the aggregate density values determined by the analysis of Javaheri and Dick (1969). In Section 4.1.2, referring to the data presented in Table 8, a conclusion was drawn that the particle density settling tests carried out in tubes of Percoll (ρ_{pp}) might represent rough estimates of bulk density rather than being a true "particle" (or aggregate) density. In Table 12, when these density values are compared to the aggregate densities determined from the analysis of Javaheri and Dick (1969), ρ_a, a different interpretation can be made. The third column in Table 12, ρ_b, indicates the maximum bulk density to which the four metal sludges could be mechanically dewatered. Theoretical considerations stated in Section 4.1.2 predicted that the maximum bulk density achievable by mechanical

TABLE 12

Comparison of Sludge "Particle" Densities obtained by
Various Methods for Four Metal Sludges

Sludge	ρ_k	ρ_p *	ρ_b **	ρ_{pp} ***	ρ_a ****
aluminum hydroxide (II) ·	2.87	1.150-1.160	1.123	1.032	1.024
iron hydroxide	2.60	1.130-1.140	--	1.016	1.009
nickel hydroxide (II)	3.77	1.160-1.170	1.205	1.012-1.014	1.018
lime	2.47	>1.214	1.433	1.090-1.092	1.049

All density values given in g/ml
*   Particle density determined by step gradient
**   Bulk density determined after maximum mechanical dewatering
*** Particle density determined by settling in tubes of Percoll
**** Aggregate density determined by the analysis of Javaheri and Dick (1969)

dewatering would be the density of a "particle" of sludge
and this "particle" density should be the same as the
"particle" density measured by the "particle" settling test
in tubes of Percoll (ρ_{pp}). The ultimate densities obtained
after mechanical dewatering, ρ_b, were substantially higher
than the values of ρ_{pp}. A new interpretation of these data
might be made thus: that as ρ_{pp} and ρ_a are somewhat simi-
lar, it may be that the settling test in tubes of Percoll
actually measures (roughly) aggregate density. This is a
reasonable assumption as the results of the test are based
on observation of the largest discrete units visible to the
unaided eye. The ρ_b values of Table 12 could be inter-
preted as corresponding to floc densities after aggregates
had collapsed and released the water between flocs. This
would mean that under the gentle circumstance of gravity
thickening (for metal sludges such as the four tested),
aggregates are the largest discrete units of sludge in sus-
pension. Under the stress of mechanical dewatering, fragile
aggregates decompose to their floc subunits, and the sludge
floc becomes the largest discrete unit of sludge. The
second column in Table 12, ρ_p, indicates the values of
"particle" density obtained from a step gradient in Per-
coll. As these values are similar to the values of ρ_b in
Table 12, the interpretation might be made that the step
gradient is a direct measure of floc density; that the
Percoll medium has replaced the water between flocs in the

aggregate, but has not penetrated the more tightly bound floc. The column in Table 12 for ρ_k is the calculated dry density values for chemical precipitates with all water removed. Obviously, much additional data is required to firmly establish these interpretations, but they do serve as possible explanations for the different "particle" density values obtained by these various methods.

It is possible to develop a series of mass balance equations to interrelate the density values given in Table 12 so that the relative amount of water contained in the aggregates, flocs and chemical precipitates may be quantified. The following equations were used:

$$\rho_{bw} = \Phi_k(\rho_k) + (1 - \Phi_k)\rho_w$$
$$= \Phi_f(\rho_f) + (1 - \Phi_f)\rho_w \qquad (4-3)$$
$$= \Phi_a(\rho_a) + (1 - \Phi_a)\rho_w$$

where Φ_f = volume fraction of flocs

ρ_f = density of the flocs

Bu rearranging the expression in Equation 4-3, the following equations were developed:

$$(\rho_a - \rho_w) = \frac{\Phi_k}{\Phi_a}(\rho_k - \rho_w) \qquad (4-4)$$

$$(\rho_f - \rho_w) = \frac{\Phi_k}{\Phi_f}(\rho_k - \rho_w) \qquad (4-5)$$

$$(\rho_a - \rho_w) = \frac{\Phi_f}{\Phi_a} (\rho_f - \rho_w) \qquad (4\text{-}6)$$

The ratio of aggregate volume fraction (Φ_a) to dry
solids volume fraction (Φ_k) has been previously defined as
the overall aggregate volume index (AVI) of Javaheri and
Dick (1969). This term would indicate the total water con-
tent of sludge aggregates. The ratio of the aggregate
volume fraction to floc volume fraction (Φ_a / Φ_f), would be
indicative of the amount of water which has been removed by
mechanical dewatering methods. Likewise, the floc volume
fraction to dry solids volume fraction ratio, (Φ_f / Φ_k),
would indicate that portion of water which is tightly bound
within the floc, either due to physical forces or chemical
bonding. This remaining water would be considered not
available for removal by mechanical dewatering.

These relative volume fraction ratios were calculated
for each of the four sludges tested using the median value
for ρ_p reported in Table 12 for the value of ρ_f. In the
case of lime sludge where the floc density exceeded the
maximum density of the medium in the step gradient, the
value of 1.433 g/ml was used. These calculated results are
reported in Table 13. Examination of these values allows
for certain observations to be made. In each case, a sig-
nificant amount of aggregate water was removed by mechanical
dewatering, resulting in a significantly higher density

TABLE 13

Ratios of Aggregate to Solids, Aggregate to Floc,
and Floc to Solids Volume Fractions for
Four Metal Sludges

Sludge	Φ_a/Φ_k *	Φ_a/Φ_f	Φ_f/Φ_k
aluminum hydroxide (II)	78	6.5	12
iron hydroxide	178	15	12
nickel hydroxide (II)	160	10	16
lime	30	8.8	3.4

* This expression is equivalent to AVI

value for floc particles. Thus, although past research has
often concluded that mechanical dewatering removes mostly
interparticle water, these results would indicate that sig-
nificant amounts of intraparticle (intraaggregate) water is
removed also. When considering the water remaining within
the floc after dewatering, the conclusion may be reached
that precipitation of metals as metal hydroxide solids re-
sults in the incorporation of large amounts of water.
Table 13 demonstrates that lime sludge contained much less
water within the flocs than the metal hydroxide sludges.
The water content of the lime sludge flocs was probably due
in great part to the co-precipitation of $Mg(OH)_2(s)$ in this
sludge. Perhaps if this sludge had been from a plant
which practiced calcium removal only (yielding a pure
$CaCO_3(s)$ sludge) the floc water content would have been
even more reduced.

Chapter V

SUMMARY AND CONCLUSIONS

The purpose of this research was to study the settling behavior of four metal sludges in light of work done by Michaels and Bolger (1962) with flocculated suspensions of keolin, and Javaheri and Dick (1969) with activated sludge. Both of these analyses of particle settling in slurries involved using interfacial settling velocities from batch settling tests to calculate an index of the relative amount of water associated with slurry aggregates as they settle. Javaheri and Dick (1969) demonstrated that this analysis described settling behavior in activated sludge and could be used to determine relative aggregate size and aggregate density. There were two main objectives to this research:

1. To develop a direct laboratory measurement of wet sludge particle density, and

2. To analyze four metal sludges to determine if the index of Javaheri and Dick (1969) could be more broadly applied to non-biological sludges to predict settling characteristics and to determine sludge particle densities.

From the results of the research described herein, the following conclusions seem warranted:

A. With regard to Objective 1:

    1. The modified particle settling test of

        Lagvankar and Gemmell (1968) used in this work may

be a measurement of bulk density rather than sludge particle (or aggregate) density. If it in fact a measure of sludge aggregate density, then aggregates collapse during the mechanical dewatering of sludges.

2. Aggregates of sludge do not retain their integrity in density gradient media in that a portion of the water within the aggregates may be replaced by the medium even at very low osmotic pressures. The density values obtained, however, may represent a direct measurement of floc density in tests such as step gradients.

B. With regard to Objective 2:

1. The aggregate volume index (AVI of Javaheri and Dick (1969)), when used to determine aggregate settling velocity, V_o, which is an indicator of aggregate size, and aggregate density, ρ_a, predictively describe the settling behavior of the four metal sludges evaluated. This indicates that the analysis of Javaheri and Dick (1969) may be the first mathematical description of settling of slurries broadly applicable to the settling of water and wastewater treatment sludges.

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APPENDIX A

Symbols

AVI	Aggregate Volume Index
C_{ak}	Φ_a / Φ_k
C_o	Solids concentration in g/l
D	Particle diameter
D_a	Average spherical particle diameter
F_D	Drag force
g	Gravitational force
g_{av}	Average g in an angle head rotor
K	Proportionality constant
k	Permeability
q	r/k^2
r	Radius
%S	Solids concentration as a percent
V	Particle settling velocity in a suspension
V_a	Aggregate settling velocity
V_o	Terminal (Stokes) settling velocity
V_p	Volume of Percoll
V_s	Interfacial settling velocity
V_T	Total volume
α	Factor accounting for particle shape
Φ_a	Volume fraction of aggregates
Φ_f	Volume fraction of flocs

Φ_k	Volume fraction of solids
Φ_p	Volume fraction of particles
ε	Porosity or the volume fraction of water
μ	Viscosity
ρ_a	Aggregate density
ρ_b	Bulk density
ρ_{bf}	Bulk density calculated versus filtrate
ρ_{bw}	Bulk density calculated versus water
ρ_f	Floc density
ρ_k	Solids density
ρ_p	Particle density
ρ_w	Fluid density
Θ	Flow space shape factor
ω_i	Immobile water

APPENDIX B

Raw Data on Four Metal Sludges

I. Nickel hydroxide (II) sludge

V_s (m/hr)	$V_s^{1/4.65}$	C_o (kg/m^3)	Φ_k (1 x 10^{-3})	Φ_a (1 x 10^3)
0.016	0.41	12.9	3.42	538.7
0.085	0.59	8.6	2.28	359.1
0.154	0.67	6.3	1.67	263.0
0.30	0.77	4.7	1.25	196.9
0.60	0.90	3.2	0.85	133.9
0.40	0.82	4.2	1.11	174.8
0.72	0.93	2.6	0.69	108.7

II. Lime sludge

V_s (m/hr)	$V_s^{1/4.65}$	C_o (kg/m^3)	Φ_k (1 x 10^{-3})	Φ_a (1 x 10^3)
2.40	1.21	12.1	4.90	147.5
1.75	1.13	17.7	7.17	215.8
1.16	1.03	20.4	8.26	248.6
0.99	1.00	23.9	9.68	291.4
0.63	0.90	25.4	10.28	309.4
0.80	0.95	26.0	10.53	317.0
0.56	0.88	32.3	13.08	393.7
0.33	0.79	36.7	14.86	447.3
0.31	0.78	38.0	15.38	462.9

III. Aluminum hydroxide (II) sludge

V_s (m/hr)	$V_s^{1/4.65}$	C_o (kg/m^3)	Φ_k (1 x 10^{-3})	Φ_a (1 x 10^3)
0.02	0.43	21.3	7.61	586.0
0.23	0.73	13.3	4.75	365.8
0.67	0.92	8.0	2.86	220.2
1.40	1.08	5.3	1.89	145.5
0.88	0.97	6.7	2.39	184.0
0.33	0.79	10.6	3.79	291.8
0.13	0.64	16.0	5.71	439.7
0.16	0.67	13.7	4.89	376.5
0.20	0.71	11.4	4.07	313.4
0.42	0.83	9.4	3.36	258.7

IV. Iron Hydroxide sludge

V_s (m/hr)	$V_s^{1/4.65}$	C_o (kg/m^3)	Φ_k (1 x 10^{-3})	Φ_a (1 x 10^3)
0.107	0.62	11.7	4.50	817.7
0.129	0.64	8.2	3.15	572.4
0.069	0.56	9.3	3.58	650.5
0.335	0.79	7.0	2.69	488.8
1.29	1.06	4.7	1.81	328.9
0.645	0.91	5.8	2.23	405.2
0.968	0.99	5.2	2.00	363.4

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