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FLOC DENSITY MEASUREMENT AND THE EFFECTS OF MICROPROPERTY VARIATIONS ON SLUDGE DEWATERING CHARACTERISTICS

by

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(ABSTRACT)

The dewatering characteristics of sludges produced by water and wastewater treatment plants bear heavily on the methods chosen to treat and dispose of the sludge, as well as on the costs associated with handling the large volumes of sludge produced at these facilities. This study investigated why different sludges dewater to different dry solids concentrations, how sludge structure affects dewatering, and how sludge structure changes during gravity thickening and during different types of mechanical dewatering.

It is generally thought that sludge can be described as having a three-tiered structure: (1) primary particles, (2) floc particles, and (3) aggregate particles. To investigate sludge structure in relation to sludge dewatering, this study has defined sludge structure using several sludge particle microproperties. A laboratory technique incorporating isopycnic centrifugation in gradients of Percoll® media was developed to measure one microproperty known as floc particle density. Six field and laboratory sludges were subjected to a series of dewatering tests: gravity thickening, centrifugation, and vacuum filtration. Each sludge was analyzed for macro- and microproperties through each stage of dewatering. It was concluded that improvements in thickening and

dewatering characteristics were heavily dependent upon increases in sludge floc density and decreases in aggregate water content.

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CHAPTER I. INTRODUCTION

The handling and disposal of water and wastewater sludges remains a major challenge to the designers and operators of water treatment facilities. The volume of sludge produced at these facilities is so great that the cost of sludge treatment and disposal often represents the largest single item in a treatment plant budget. An important task, therefore, lies in finding improved ways to reduce the volume of this solids and water mixture called sludge.

A number of methods are used to reduce the volume (i.e., dewater) of water and wastewater sludges, most of which fall under the categories of gravity thickening or mechanical dewatering. The objective of all sludge dewatering methods is to increase the sludge dry solids concentration by removing as much water as possible from the raw sludge in a timely and efficient manner. In view of this objective, many of the past dewatering studies focused only on so-called macroproperties of sludges, such as dry solids concentration and bulk density, when analyzing and comparing sludges. This has often led to conclusions about dewatering characteristics that were only applicable to a specific sludge or group of sludges. To explain observed dewatering phenomena more completely, sludge microproperties must be considered along with macroproperties.

These microproperties have been defined in studies of sludge structure. The microproperties must now be analyzed to determine how and to what degree they affect the extent of dewatering. This research has two main objectives:

1. Establish a method for measuring one particular sludge microproperty, floc particle density; and
2. Correlate variations in sludge macro- and micro-properties with sludge dewatering characteristics.

Chapter II. Literature Review and Theoretical Considerations

The theoretical structure of water and wastewater treatment sludges will be reviewed in this chapter, with emphasis on the various fractions of water and solids found in these sludges. Certain sludge properties and their reported effects on sludge dewatering will also be reviewed in conjunction with theories of gravity thickening and mechanical dewatering. Furthermore, there will be a discussion of analytical centrifugation, as used in biology and medical science, for its potential use in quantifying the wet density of sludge flocs.

Theoretical Sludge Structure

Sludge is a combination of dry solids and water. These two materials interact to form three different types of particles which constitute a sludge structure. The first type of particle, the primary particle, is comprised of dry matter and water which is tightly bound to the dry material. These particles are the smallest in size and the strongest, in terms of shear strength, of the three particle types. Floc particles constitute the second category of sludge particles and consist of primary particles with water entrapped between them. Floc particles, or flocs, are larger than primary particles and possess lower shear strength. Flocs attach to each other, entrapping water between them in the process, to form aggregates, the third type of particle. Aggregates are the largest and weakest of the sludge particles (1).

The distribution of water in the sludge solids depends upon chemical and physical forces that bind the water to the solids. At least three theoretical models have been proposed to define this water distribution. Vesilind (2) defined the following four categories of water sludges:

1. Free water - water that is unbound, i.e., not held to any sludge particles; removed by gravity thickening.
2. Floc water - water held within a floc and removed by mechanical dewatering.
3. Capillary water - water adhered to individual sludge particles and removed only when these particles are deformed and compressed.
4. Bound water - water which is held to individual sludge particles by chemical forces; not removed by mechanical dewatering.

The terminology in Vesilind's model differs from that used in this chapter in that he uses "floc" instead of "aggregate" and "individual particle" instead of "primary particle". Vesilind did not address the concept of a floc particle as it was described at the first of this section.

Vesilind suggested that high-speed centrifugation could be used to quantify the amount of water in each category. It was hypothesized that a plot of dry solid concentration versus centrifugal acceleration would reveal points of inflection, signifying transitions or breakpoints between stages of removal of various water types.

Moller (3) published a second theory of water distribution in which the importance of energy requirements was stressed for breaking the binding forces which are on the water molecule. Moller defined the following categories of water:

1. Interspace water - a type of external water having the weakest attachment to sludge particles and composing the major portion of water in an unthickened sludge.

2. Adhesion and capillary waters - external waters with intermediate binding forces which together account for the second largest category.
3. Adsorption water - most strongly bound external water; forms a protective layer around the particles (equivalent to primary particles) and constitutes only a small fraction of the total water present; can only be removed by thermal forces.
4. Internal water - particle moisture and chemically bound water that is removed by changing internal water to external water by biological or thermal means.

Arundel (4) offered a third model of sludge water distribution which also defines four categories of water, although they differ from the previous models' categories. These four categories are as follows:

1. Interspace/free water - water loosely bound between the sludge aggregates; its quantity is approximated by the amount of water removed during gravity thickening.
2. Interfloc/intra-aggregate water - water entrapped within the aggregate structure, yet outside the aggregates' constituent flocs; freed after disrupting the aggregate structure but prior to floc deformation; removed during centrifugation, vacuum dewatering, and the early stages of higher pressure mechanical dewatering.
3. Intrafloc water - water within a floc; removed only after floc deformation using the high mechanical dewatering pressures.

4. Adsorbed and internal water - water associated directly with primary particles; not removed by mechanical means.

Arundel's interspace/free water category compares to Vesilind's free water category and Moller's interspace water. Like Vesilind, Arundel has defined this category in terms of its method of removal. The interfloc/intra-aggregate water of Arundel's model compares to Vesilind's floc water category and to Moller's second category, adhesion and capillary waters. Arundel's intrafloc water category compares with Vesilind's capillary water, except for the types of particles used in each definition. There is no category in the other two models which parallels Moller's third category, adsorption water. The final category in Arundel's model, adsorbed and internal water, compares well with Vesilind's bound water and Moller's internal water.

Sludge Microproperties

The relative distribution of dry material and water among the three types of particles determines to a large extent the microproperties of a sludge. These microproperites include the dry particle density, floc density, and aggregate density. Comparisons can be made of these values with the corresponding density of the entire sludge, i.e., the bulk density.

The dry particle density (ρ_k) is a measurement of the weight per unit volume of the dry solids fraction present in a sludge. Its value may be calculated using the following relationship (5):

$$\frac{100}{\rho_b} = \frac{(100 - C)}{\rho_w} + \frac{C}{\rho_k} \qquad [1]$$

where C = dry solids concentration, percent by weight

ρ_w = density of water (1.0 mg/L)

ρ_b = bulk density of the sludge at C, g/mL

Using a technique presented in the next chapter, floc density (ρ_f), may be measured in the laboratory. However, the aggregate density (ρ_a), must be calculated as follows (5):

$$\rho_a - \rho_w = \frac{\rho_k - \rho_w}{AVI} \qquad [2]$$

where AVI is the abbreviation for Aggregate Volume Index (6). A characterization parameter which will be further discussed in a subsequent section. Finally, the bulk density (ρ_b) is defined as the weight per unit volume of the total sludge and may be measured in the laboratory using a pycnometer.

Sludge Dewatering Theory

Sludge dewatering processes utilize different means to produce a sludge with a higher percentage of dry solids. The process of thickening utilizes the force of gravity and the density of the sludge particles to thicken sludges while mechanical dewatering uses an applied mechanical force to extract water from sludge. Another dewatering method, sludge drying, utilizes the natural process of evaporation in conjunction with gravity (i.e., filtration). This section will review thickening and mechanical dewatering along with the cited effects sludge microproperties have on these dewatering processes.

Gravity Thickening. The two important aspects of gravity thickening are the extent of dewatering achieved and the rate of thickening. The extent of dewatering refers to the amount of water that is removed from the sludge or to the final dry solids concentration achieved by the process. Thickening rate relates to the subsidence of the interfacial plane between the sludge and the supernatant as a function of both time and solids concentration.

Michaels and Bolger (1) identified three categories or phases of gravity thickening: settling in dilute suspensions, settling at intermediate solids concentrations, and settling at high solids concentrations. While the sludges in these three categories were considered to consist of predominantly aggregate particles, these researchers concluded that the settling rates are primarily affected by the floc particles.

A dilute suspension was defined as having a solids concentration of less than 1.8% on a weight basis. Under these conditions, the aggregates were found to settle as roughly spherical, individual units and the settling rates were the highest among the three categories. As a result, Scott (5) termed this type of gravity thickening "free settling". It must bear in mind, however, the type of sludge being studied when defining the solids concentration limits of the settling phases. For instance, Michaels and Bolger (1) worked with flocculated kaolin which would tend to be a relatively low water content sludge and which, at a 1.8% dry solids concentration, would be a relatively dilute suspension. Other types of sludges having a much higher water content may not achieve 1.8% dry solids until after several hours of thickening.

In the intermediate sludge settling phase, at solids concentrations higher than those of dilute sludges, Michaels and Bolger (1) noted that the aggregates are initially interconnected, irregular spheres having tortuous fluid flow paths between them that are characterized by many contractions and expansions. These restrictive flow paths result in strong drag forces on the aggregates and low initial settling rates. Gradually, the aggregates tend to line up, enabling water to flow upward in vertical channels rising through the aggregate region and, thereby, allowing slowly increasing settling rates. During this phase of gravity thickening, interaggregate water (i.e., Arundel's interspace/free water) is released as the aggregates deform and move closer together.

Gravity thickening of sludges having high dry solids concentrations occurs at a much lower rate than in those with dilute or intermediate solids concentrations. The settling that does occur in this phase is due to the intra-aggregate water being slowly squeezed out as the aggregates' constituent flocs crowd closer together. Therefore, this phase of settling has been called the compression phase. Scott (7) noted that settling in concentrated sludges is controlled by the much lower permeability of the aggregates, which is associated with higher solids concentrations. Due to this lower permeability, the only fluid flow possible up through the sludge is between the particles making up the aggregates (i.e., floc particles). Javaheri and Dick (6) referred to this flow pattern as intraaggregate flow and found that it accompanies the split of aggregates into smaller constituent flocs. Scott (5) explained this splitting of the aggregates in terms of their low shear strength. He noted that aggregate particles can support only a limited quantity of overlying solids and when this quantity is exceeded, the aggregate structure collapses, releasing its internal water. The rate of this collapse and, thus the settling rate, depends upon the rate at which the freed water permeates upward through the voids within the aggregates.

Sludge settling rates depend on the permeability and size of the channels formed within the network of aggregate particles (1,5). These channels represent voidage within the sludge and, as such, are affected by solids concentration and the attractive forces between the aggregates' constituent floc particles. Settling velocity decreases with increasing solids concentration due to an associated decrease in voidage or porosity. Michaels and Bolger (1) noted that when the attractive forces between the flocs are strong, the diameter of the channels is larger and the settling rates are high.

The fraction of a volume of sludge made up of aggregate particles also affects gravity thickening rates. Michaels and Bolger (1) determined that, for five sludges studied, the ratio of the aggregate volume fraction to the floc volume fraction was approximately 3.8. This means that, unless the aggregates were confined and strongly compressed, the flocs always clustered in a way that provided a floc volume fraction within an aggregate close to 1/3.8 = 0.26. They concluded that at floc volume fractions less than 0.26, the aggregates could settle as free particles, but at values above 0.26 the aggregate volume concentration would be unity and there could be no free-fall settling.

The aggregate volume fraction (Φ_a) may be calculated by the following formula (1):

$$\Phi_a = AVI(\Phi_k) \qquad [3]$$

where Φ_k represents the dry solids volume fraction or concentration. The dry solids volume fraction may be computed using the following mass balance (6):

$$\rho_b - \rho_w = \Phi_k(\rho_k - \rho_w) \qquad [4]$$

or with this formula:

$$\Phi_k = C/\rho_k \qquad [5]$$

where C is the solids fraction of the total weight of the sludge. The floc volume fraction or concentration, Φ_f, may be calculated using the following mass balance (6):

$$\rho_b = \Phi_f \rho_f + (1 - \Phi_f)\rho_w \qquad [6]$$

The method for determining a sludge's AVI (Aggregate Volume Index) will be explained later in this section.

The initial basis for quantifying thickening rates may be found in Stokes' Law (6):

$$V_o = \frac{D^2 g(\rho_p - \rho_w)}{18\mu} \qquad [7]$$

where V_o = Stokes' settling velocity, meters/second (m/s)

D = average aggregate diameter, m

g = gravitational acceleration, m/s^2

ρ_p = particle density, $g\text{-}s^2/m^4$

ρ_w = fluid density, $g\text{-}s^2/m^4$

μ = solution viscosity, $g\text{-}s/m^2$

The above law assumes that a single spherical particle is settling in an infinite fluid under laminar flow. However, such an ideal state does not exist in sludge thickening, during which the normal conditions are nonspherical particles, particle-particle interference, turbulent and viscous flow, and wall effects.

A number of thickening rate expressions have been proposed to describe observed sludge subsidence phenomena. Richardson and Zaki (8) formulated the following equation, which was validated with experimental data, for the settling of uniformly-sized, non-porous particles:

$$V_s = V_o \varepsilon^{4.65} \quad [8]$$

where V_s = settling rate of sludge/supernatant interface, m/hr

V_o = Stokes' settling velocity, m/hr

ε = void fraction or sludge porosity

Two sets of researchers, Michaels and Bolger (1) and Javaheri and Dick (6), modified Equation [8] to the following form:

$$V_s = V_o(1 - \Phi_a)^{4.65} \quad [9]$$

Thus, Equation [9] expresses the importance of the size of sludge aggregate particles to gravity thickening rates. Figure 1 demonstrates the relationship between V_s, sludge porosity, and Φ_a.

Sludge particles may be characterized by their size and their water content. The Stokes' settling velocity, V_o, has been used as an indication of relative aggregate size while the AVI is an indirect measurement of aggregate water content (6). The following mass balance equation (partially appearing in Equation [4]) developed by Javaheri and Dick (6) is the basis of the AVI:

$$\rho_b - \rho_w = \Phi_a(\rho_a - \rho_w) = \Phi_k(\rho_k - \rho_w) \quad [10]$$

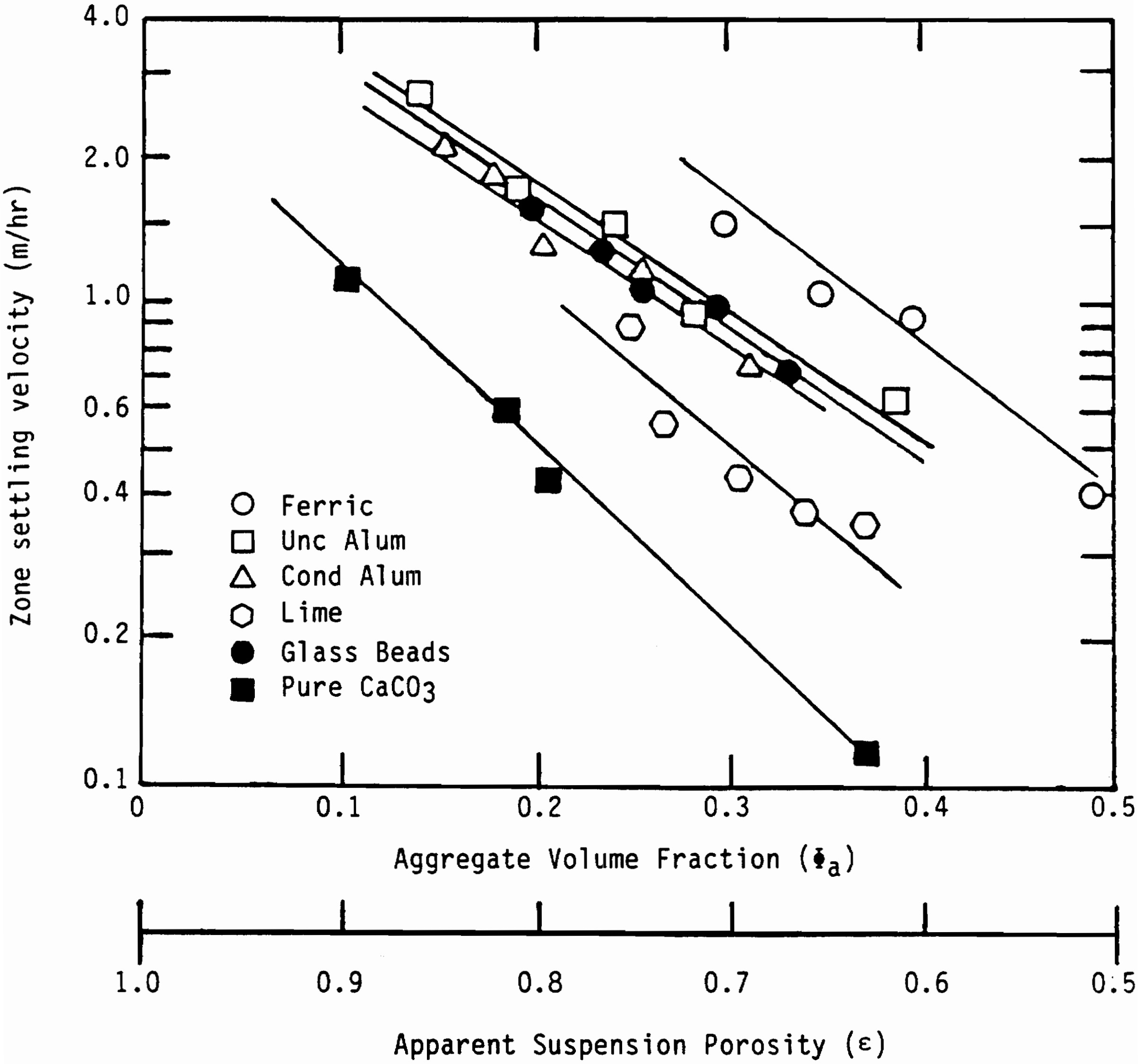

Figure 1. Relationship of sludge settling velocity (V_s) and sludge aggregate volume fraction for six sludges (4).

Rearranging these expressions yields:

$$\rho_a - \rho_w = \frac{\Phi_k(\rho_k - \rho_w)}{\Phi_a} = \frac{(\rho_k \quad \rho_w)}{AVI} \qquad [11]$$

Substituting for Φ_a and rearranging equation [9] yields (1):

$$V_s^{1/4.65} = V_o^{1/4.65} - V_o^{1/4.65}(AVI)\Phi_k \qquad [12]$$

Plots of $V_s^{1/4.65}$ versus Φ_k should yield a straight line with a y-intercept of $V_o^{1/4.65}$. The slope of this line may be used to mathematically estimate the AVI. If the AVI and the aggregate settling velocity vary with concentration, equation [12] will plot as a curve and any such curved segments of the plot signify compression of the aggregates with a corresponding release of interfloc/intra-aggregate water.

Finally, Knocke (9) reported these general expressions which he used to analyze the relationship between sludge settling velocity and solids concentration and aggregate volume fraction, respectively:

$$V_s = V_o(10^{-k_1 C}) \qquad [13]$$

and

$$V_s = V_o(10^{-k_2 \Phi a}) \qquad [14]$$

The k values represent the slope of the line resulting from a plot of $\log(V_s)$ versus C or Φ_a.

Mechanical Dewatering. The second type of sludge dewatering method reviewed is mechanical dewatering. The typical modes of mechanical dewatering are pressure filtration, centrifugation, and vacuum filtration. Laboratory scale equipment are used to study the extent and rate of dewatering that may be accomplished by each dewatering process.

The extent of dewatering that can be achieved by each mechanical system is highly variable. The U. S. Environmental Protection Agency (10) published the information in Figure 2 which shows the performance capabilities of various mechanical processes when handling raw primary and raw waste activated sludges. The data clearly indicate that filter presses (i.e., pressure filtration) normally remove more water from sludges than do centrifuges or vacuum filters.

Two major points are to be made concerning the data shown in Figure 2. First, the degree of water removed for each sludge type is apparently a function of the pressure applied to the sludge during dewatering. For example, the diaphragm filter press (high pressure filtration) resulted in much higher dry solids concentrations than did either the gravity belt concentrator or the dual cell gravity unit, both of which are low-pressure dewatering methods that employ gravity. A second important point to be made is that the type of sludge being dewatered (e.g., 100% WAS versus 100% primary versus various mixtures) evidently has a great effect on the extent of water removal observed in each dewatering system.

The water distribution models reviewed earlier reinforce the above discussion of Figure 2. Those models emphasize that there are different types of sludge particles and various categories of water within a sludge. The water is characterized by the type of particle it is associated with, which in turn affects the intensity of the force required to remove it from the suspension. Some of this water is basically free or loosely attached to sludge particles while other water is bonded tightly.

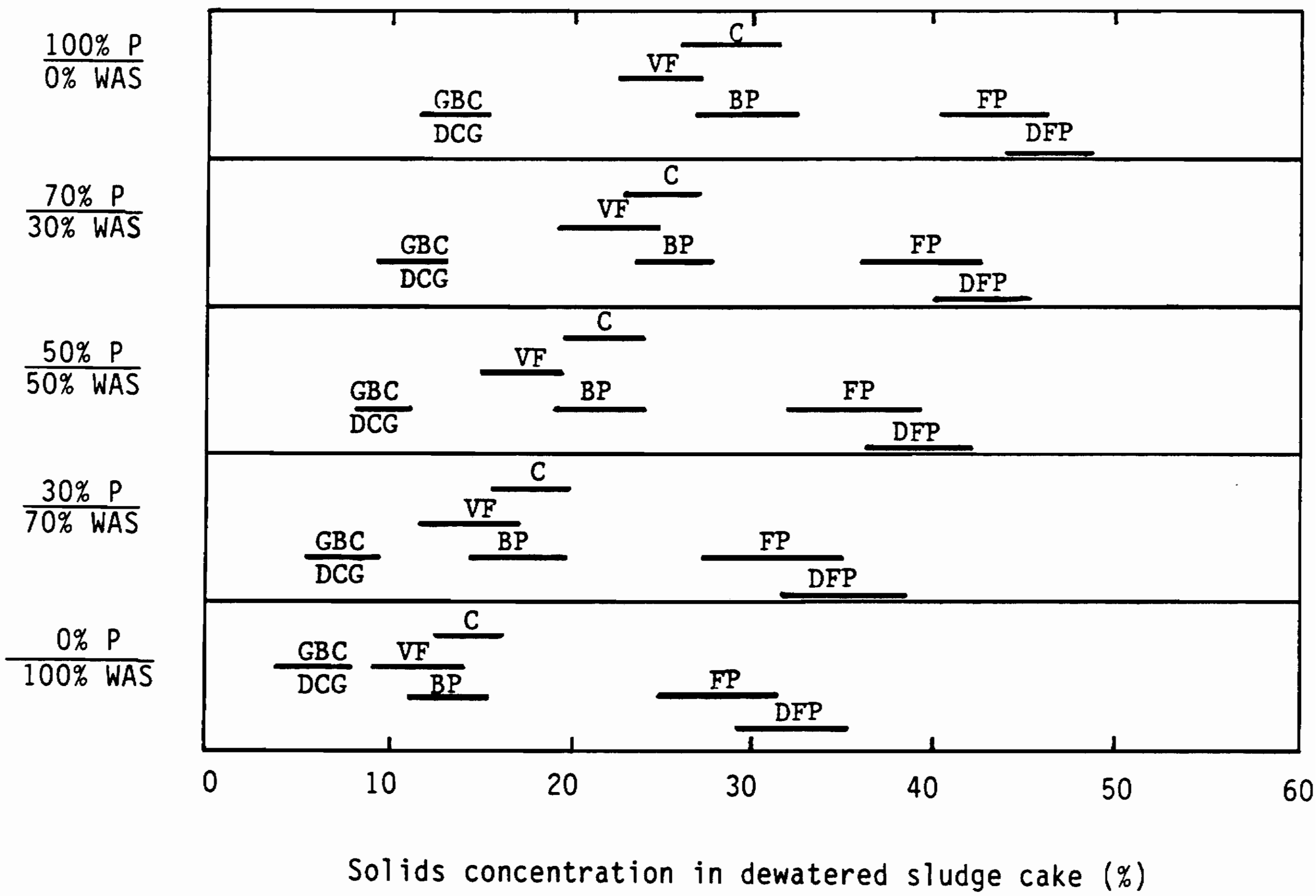

Figure 2. Sludge cake solids concentrations obtained from dewatering various mixtures of digested primary (P) and digested waste activated (WAS) sludges (10).

C = Solid Bowl Centrifuge
VF = Vacuum Filter
BP = Belt Press
FP = Filter Press
DFP = Diaphragm Filter Press
GBC = Gravity Belt Concentrator
DCG = Dual Cell Gravity Unit

Therefore, the amount of water that can be removed from a sludge and the energy required to release that water depends on the characteristics and the relative quantities of its three types of sludge particles. One major purpose of this study was to investigate these types of issues.

Sludge Structure Changes During Dewatering. Sludge dewatering processes have the potential of altering the structures of the three theoretical types of sludge particles (4). This is illustrated by the data included in Figure 3, a plot of percent mass solids concentration versus sludge bulk density measured following application of gravity thickening and different levels of mechanical dewatering. Gravity thickening resulted in bulk densities equal to or greater than the calculated aggregate densities, yet well below the measured floc density. Centrifugation and vacuum filtration dewatered the sludges to bulk densities that generally exceeded the aggregate densities, yet did not exceed the floc density. In high-pressure dewatering, the bulk densities surpassed the floc density but never approached the dry particle density. These results suggest the following changes in sludge structure during dewatering (4):

1. During gravity thickening, aggregates enter a zone of compression whereby pressure due to overlying water and solids causes aggregate deformation and release of internal aggregate water.

2. The intermediate dewatering pressures, exerted in this case by centrifugation and vacuum filtration, result in complete aggregate disruption and an undeformed hydrated floc. These dewatering methods remove water that is outside the floc structure; and little or no water is removed from within the floc.

3. During high-pressure dewatering, the floc structure begins to break apart and release its internal water. The primary particle structure is thought to be unaffected by even the most extensive dewatering methods.

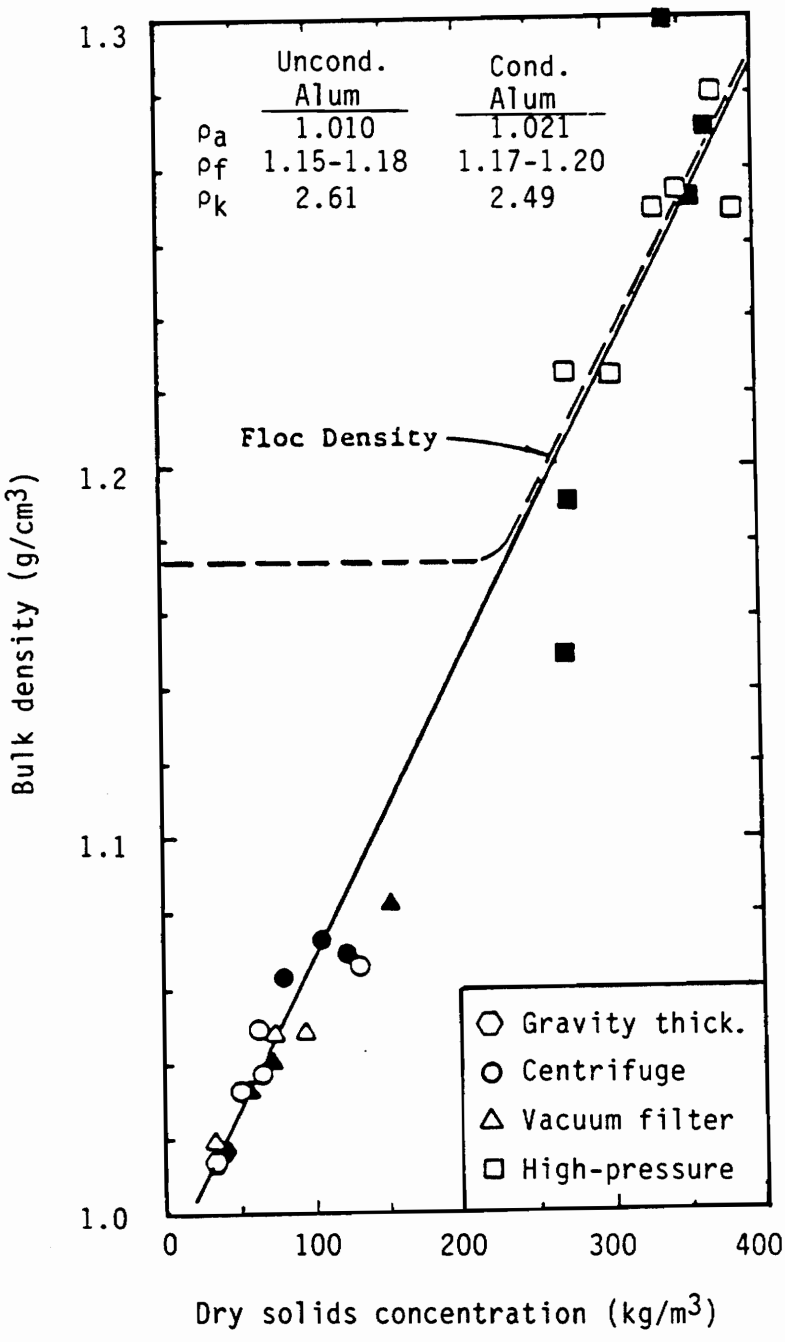

Figure 3. Relationship between measured bulk density and dry solids concentration during the dewatering of unconditioned alum (open symbols) and conditioned alum (closed symbols) sludges (4).

Theory of Analytical Centrifugation

Thus far, this chapter has described various sludge microproperties, general ways of dewatering sludge, and some relationships between microproperties and dewatering. Determination of these microproperties is a key to sludge research. Various methods of microproperty determination (eg. particle size measurements, dry solids density, AVI) are known and used in many sludge characterization studies. However, a method of measuring floc density demands further investigation, documentation, and simplification, as well.

The process of centrifugation has been used successfully for years in the biological and medical sciences for purifying, isolating, and characterizing various biological particles. It has been used to remove cells or other particles from their surroundings, to separate one cell type from another, to isolate viruses and macromolecules such as DNA and proteins or to determine these particles' physical characteristics, and to separate the various subcellular organelles from dispersed tissue (11).

The basic theory behind analytical centrifugation is that a particle suspended in a less dense liquid will migrate downward through the liquid under the force of gravity. Such a particle migration, however, will quite often not occur under the force of gravity alone when dealing with very fine particles because back-diffusion, resulting from a concentration gradient created by partial sedimentation of the particle, balances out the downward transport. Therefore, by increasing the sedimentation or downward force the diffusion or mixing force can be rendered negligible in comparison. Centrifugation provides this increased force, allowing separation of particles to occur much more rapidly than they would under the force of gravity alone (12).

There exist two major approaches to analytical centrifugation (11). One is called differential centrifugation and involves the stepwise removal of different classes of particles from a heterogeneous mixture by successive centrifugation at increasing relative centrifugal force. The other approach, density gradient centrifugation, is more suited to separating a family of particles having similar sedimentation properties. It is this latter approach that will be reviewed for its application to quantifying the density of sludge flocs.

The premise behind density gradient centrifugation is that particles layered onto the surface of a column of liquid that possesses increasing density from top to bottom (i.e., a density gradient) will, during centrifugation, migrate to that point in the liquid column that is of a density equal to their own. This point is termed the isopycnic point (11). Hence, if the density of the liquid at the isopycnic point is known, the density of the particle is known also.

Preparation and Use of Density Gradients. There are two types of density gradients: step gradients and continuous gradients. Step gradients are prepared by layering solutions of different densities in a centrifuge tube and then placing the sample to be tested on top of the last and least dense solution layer or step. A step density gradient may be formed either by carefully layering the steps on top of each other with a pipette (beginning with the densest solution at the bottom), or by placing the layers at the bottom of the tube with a narrow canula, beginning with the least dense step. The distinct changes in density between the steps eventually diminish somewhat due to diffusion; therefore, these gradients need to be used soon after they are prepared.

The density of the medium in continuous density gradients varies continuously from one end of the centrifuge tube to the other. Typically, continuous gradients are made using devices known as gradient makers whereby two different concentrations of the stock gradient solution are mechanically mixed to produce the desired smooth gradient. Another method is to select a concentration of the stock solution that is in the middle of the density range of interest and then centrifuge this liquid at high speed for a period of time. This liquid, initially of a uniform density, will then disperse to form a continuous density gradient. Precalibrated density marker beads are used in this procedure to signify the density of the medium at any point in the centrifuge tube (11).

Density gradient centrifugation is obviously a very useful analytical method, but in order to apply it, the parameters that impact on the test must be understood. These parameters are associated with the gradient medium or solution and the centrifugation conditions. The major characteristics of the gradient medium which impact centrifugation analyses are ionic strength and osmolarity, viscosity, and temperature. The parameters linked to the centrifuge are the type of rotor, the size of the gradient, and the angular acceleration of the centrifuge (11,12).

The ionic strength and/or osmolarity of a fluid can be translated into the osmotic pressure that it will exert on any particle that is within its boundaries. The greater the ionic strength or osmolarity, the higher will be its associated osmotic pressure. A density gradient medium can exert sufficiently high osmotic pressure to disrupt bonds within a particle; e.g., the bonds between a floc particle and its bound water. This disruption will alter the size and density of the particle (11,13).

The viscosity of the gradient medium is another important consideration. A highly viscous medium increases the difficulty with which different concentrations of the medium and the gradient itself are made. Furthermore, the rate at which a particle will reach its isopycnic point decreases with increasing viscosity of the surrounding solution (11,12).

Temperature impacts centrifugation analysis through its effect on the phenomena of convection and diffusion (11,12). Convection is the movement of solute or solvent within the gradient medium and can cause unwanted changes in density. This unwanted convection is most commonly due to temperature variations within the tube. The problem is compounded when the temperature of the density gradient differs appreciably from its environment because different regions of thes of the gradient will change temperature at different rates. Similarly, diffusion is the movement of the medium's solute particles from a region of higher concentration to one of lower concentration. This movement is a consequence of Brownian motion and is greater at higher temperature. Fortunately, temperature effects can be minimized due to the capability of controlling the temperature of the density medium.

Sheeler (11) set forth these general criteria for selecting a centrifugation medium when working with biological particles:

1. The solute should be adequately soluble to create the required range of densities.

2. The medium should not be highly viscous.
3. The solution should not exert too high an osmotic pressure.
4. The solution should possess a property that will permit convenient measurement of gradient density.
5. The medium should not react with the sample particles.
6. The medium should be affordable in the volumes required.

Lagvankar and Gemmell (13) described a method of floc density measurement using sucrose as the density medium. Their method, however, did not utilize centrifugation. Instead, they suspended the sample particle in a sucrose solution of known density. If the particle did not sink or rise in a particular solution, then that particle's density was recorded as being the density of that medium. This floc density method has two disadvantages. First, high osmotic pressures are associated with sucrose. Therefore, the release of intrafloc water would occur soon after placing a sample in the sucrose. Of course, this would result in an increase in the density of a sludge floc particle. A second disadvantage concerns the type of particle that was being analyzed. The researchers assumed that they were transferring individual floc particles into the sucrose density medium. The particles tested in the sucrose were carefully removed from their suspension and gently pipetted into the density medium without disrupting their structure. Therefore, since some force is required to break aggregates into their constituent flocs, it seems more likely that the samples contained aggregates, not flocs.

There are two major types of centrifuges, the swinging-bucket and the fixed-angle rotor, each of which have advantages and disadvantages associated with their use in particle density measurement. In swinging-bucket rotors, centrifuge tubes are held in buckets which reorient from the vertical the horizontal position during acceleration. In fixed-angle rotors, centrifuge tubes are held at a fixed angle to the horizontal plane. Convection resulting from sedimentation of the medium particles (particles which make up the density medium) during centrifugation, rather than from temperature, occurs in both types of rotors. During centrifugation in swinging-bucket rotors, the concentration of medium particles and, thus, the medium's density, increases at the tube walls and decreases at the center and particles move toward the bottom of the tube. The effect of this downward movement is greater in fixed-angle rotors because the medium particles sediment radially away from the axis of rotation and shortly encounter the tube wall. The result is an increase in medium density along one edge of the tube followed by convection of the particles toward the bottom. Consequently, swinging-bucket rotors are preferred when working with density gradients and when convection is a major concern.

Vollrath-Vaughn (14) noted another disadvantage of fixed-angle rotors in that certain sludge samples would smear against the outer side of tubes during centrifugation in fixed-angle rotors. This apparently occurred because the centrifugation tubes could not align with the horizontal plane of rotation, thereby forcing the sludge particles into the wall of the tubes.

There are two forms of the density gradient approach to particle separation: rate and isopycnic. In rate centrifugation, the sample particles are more dense than the gradient and, therefore, sediment to the bottom of the centrifuge tube. On the other hand, in isopycnic centrifugation, the density of the sample particles falls within the range of the density gradient, thereby enabling them to settle to their isopycnic points. Obviously, isopycnic centrifugation is the approach that is relevant to the floc density test.

For isopynic separations, the fixed-angle rotor is more efficient due to its reduced gradient path length, the elliptical shape of the gradient cross-section (increased gradient cross-sectional area), and the fact that convection occurring in fixed-angle rotors brings particles to their isopycnic point more quickly than in swinging-bucket rotors (11).

The length of the gradient and the speed (rpm) of the centrifuge affects the time required to reach the isopycnic point (11). A shorter gradient length will reduce this time because the sample particles will have less distance to travel; however, shorter gradients can reduce the test accuracy. At higher centrifuge speeds, sample particles experience greater g forces and, therefore, their velocity through the gradient will be more rapid, i.e., they will reach their isopycnic point quicker.

Centrifugation is a powerful analytical tool in the fields of biology and medicine and one can see its potential for the study of sludge floc particle density. Many questions must be answered about its use in determining this sludge microproperty. One goal of this research is to refine a centrifugation method for testing sludge floc density which will afford a more complete analysis of sludge microproperties and their role in sludge dewatering.

CHAPTER III. METHODS AND MATERIALS

Methods and materials used to collect data during this research are described in this chapter. These descriptions cover the test sludges, the analyses of various sludge properties, the development of a floc density test, and the test methods utilized for quantifying sludge dewatering characteristics.

Sludge Generation and Identification

The study incorporated ten different sludges, six of which were produced in the laboratory. The four field sludges are described as follows:

1. Alum I. This unconditioned alum sludge was obtained form the Blacksburg-Christiansburg-VPI Water Authority water treatment plant located near Blacksburg, Virginia.
2. Alum II and III. These unconditioned alum sludges were obtained from the water treatment facilities at the Radford Army Ammunition Plant near Radford, Virginia.
3. Lime. The source of the lime sludge was the Columbia, Missouri water treatment plant which is a lime softening facility that practices only partial magnesium removal.
4. Waste activated sludge I (WAS I). This sludge was obtained from the Lower Stroubles Creek Sewage Treatment Plant, a completely-mixed facility treating a relatively dilute, low-industry wastewater.

Four of the six laboratory-generated sludges were prepared using coagulant dosages which provided good color or turbidity removal. These dosages were determined using a jar test apparatus which was made to simulate rapid mixing, flocculation, and settling. The four sludges were as follows:

1. Humic acid + polymer. This sludge was prepared by coagulating 20 liters (L) of a humic acid solution (TOC = 25 mg/L) with a 40 mg/L dosage of Betz 1195 polymer.
2. Humic acid + alum. This sludge was prepared by treating 20 L of a humic acid solution (TOC = 8 mg/L) with a 75 mg/L dose of aluminum sulfate ($Al_2(SO_4)_3$ - 18 H_2O), i.e., alum, at pH 6.5.
3. Bentonite + polymer. To generate this sludge, Betz 1195 polymer was added at 2.5 mg/L to a 20 L solution containing 100 mg/L of bentonite clay.
4. Bentonite + alum. This sludge was produced by treating 20 L of a 100 mg/L bentonite solution with 30 mg/L of alum at pH 7.0.

The remaining two laboratory-generated sludges are described as follows:

1. Alum III + polymer. This sludge was prepared by conditioning the Alum III sludge with 105 mg/L of Percol 727 polymer. This sludge was prepared by Zoccola (15) during experiments to determine optimum sludge conditioning parameters based upon dewatering tests.
2. Waste activated sludge II (WAS II). This sludge was obtained from a laboratory continuous-flow stirred-tank reactor in the Department of Environmental Sciences and

Engineering at VPI & SU. The substrate being fed to the reactor was a solution of bactopeptone.

Determination of Sludge Properties

Solids Concentration. Solids concentration was determined on a mass percentage basis by weighing a sample of sludge in an aluminum pan of known weight and placing the pan and sample in a drying oven at approximately 103^{o}C for 24 hours or until the sample reached a constant weight. The solids concentration of dewatered sludge cake samples was determined by oven-drying at 103^{o}C for only one hour. The pan plus dried sludge was then re-weighed after cooling. The following equation was used to calculate the percent solids (C):

$$C = \frac{\text{net weight of dry sample}}{\text{net weight of wet sample}} \times 100 \qquad [15]$$

Bulk Density. The bulk density of the test sludges was evaluated using a glass pycnometer. The pycnometer was first filled with distilled water (temperature = 20 - 22^{o}C), and then weighed to the nearest 0.001 gram (g). The pycnometer was then emptied, thoroughly dried, refilled with sludge, and reweighed. Each weight was remeasured until consecutive values agreed within 0.001 mg. The bulk density could then be calculated as follows:

$$\rho_b = \frac{\text{net weight of sludge}}{\text{net weight of water}} \times \rho_w \qquad [16]$$

where ρ_b = bulk density, g/mL

ρ_w = density of water (assumed to be 1.0 g/mL)

Dry Particle Density. The dry particle density (ρ_k) for each sludge was calculated by substituting measured paired values of solids concentration and bulk density into the following equation:

$$\frac{100}{\rho_b} = \frac{(100 - C)}{\rho_w} + \frac{C}{\rho_k} \qquad [17]$$

The value for ρ_k was reported as the average of two to three replicate tests performed for each sludge.

Sludge Settling Velocity. Gravity thickening rates for different concentrations of each sludge were measured in either a one-half, one-, or two-liter graduated cylinder, depending upon the volume of sludge available and the required initial solids concentration. A volume of previously thickened sludge was placed in a cylinder and diluted with supernatant or tap water. After mixing the contents, a sample was taken for determination of solids concentration. An interfacial plane soon developed between settling sludge particles and clear supernatant. The height of this interface in centimeters (cm) was recorded versus time at one to two-minute intervals. The sludge settling velocity (V_s) was determined from plots such as those in Figure 4. This velocity, often called zone settling velocity, is represented by the slope of the linear portion of each plot. Zone settling generally corresponds to the first two of Michaels and Bolger's (1) categories of gravity thickening which were outlined in Chapter II, i.e., settling in dilute suspensions and at intermediate solids concentrations. At least four different initial solids concentrations of each sludge were evaluated. The resulting values of V_s were plotted versus C in a semi-logarithmic plot for comparison of the gravity thickening characteristics of the sludges. Work-

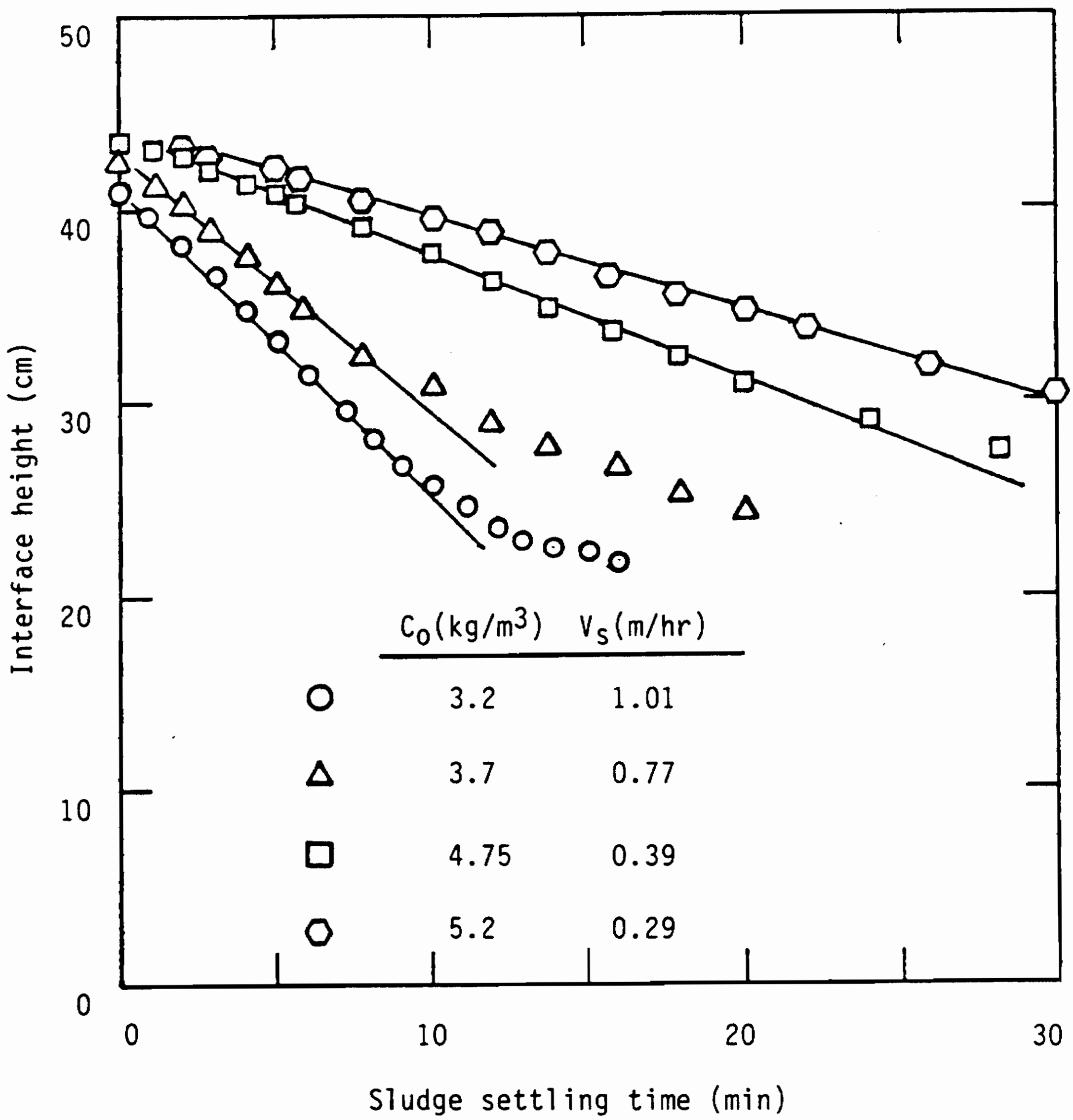

Figure 4. Determination of sludge settling velocity (V_s) for various initial solids concentrations of a sludge.

ing from the semi-logarithmic plot of V_s versus C, the characteristic value of k_1 (from Equation [13]) for each sludge was estimated as the negative slope of the plot. Also, values for Stokes' Law settling velocity (V_0) were represented as the y-intercept of these plots.

Aggregate Volume Index. The Aggregate Volume Index (AVI) was determined by plotting $V_s^{1/4.65}$ versus C/ρ_k (i.e., Φ_k). The AVI was then calculated by dividing the negative slope of the line by its y-intercept. The y-intercept represented V_0 raised to the power of 1/4.65. Figure 5 demonstrates the method for evaluating a sludge's AVI. For purposes of calculation and comparison, one AVI value was recorded for each sample, based on data taken only from the zone settling stage of thickening, during which the aggregate structure is considered undeformed. Using a sludge's AVI, its aggregate volume fraction (Φ_a) and aggregate particle density (ρ_a) were calculated. The k_2 values of Equation [14] were then derived for each sludge from the slope of the semi-logarithmic plots of V_s versus Φ_a.

Maximum Solids Concentration Achieved by Gravity Thickening. During the thickening tests, one concentration of each sludge was allowed to thicken for 24 hours. The concentration of this thickened sludge was calculated as follows:

$$C_{max} = C_o\,(H_o)/H_{min} \qquad [18]$$

where C_{max} = maximum solids concentration achieved by gravity thickening, %

C_o = initial solids concentration, %

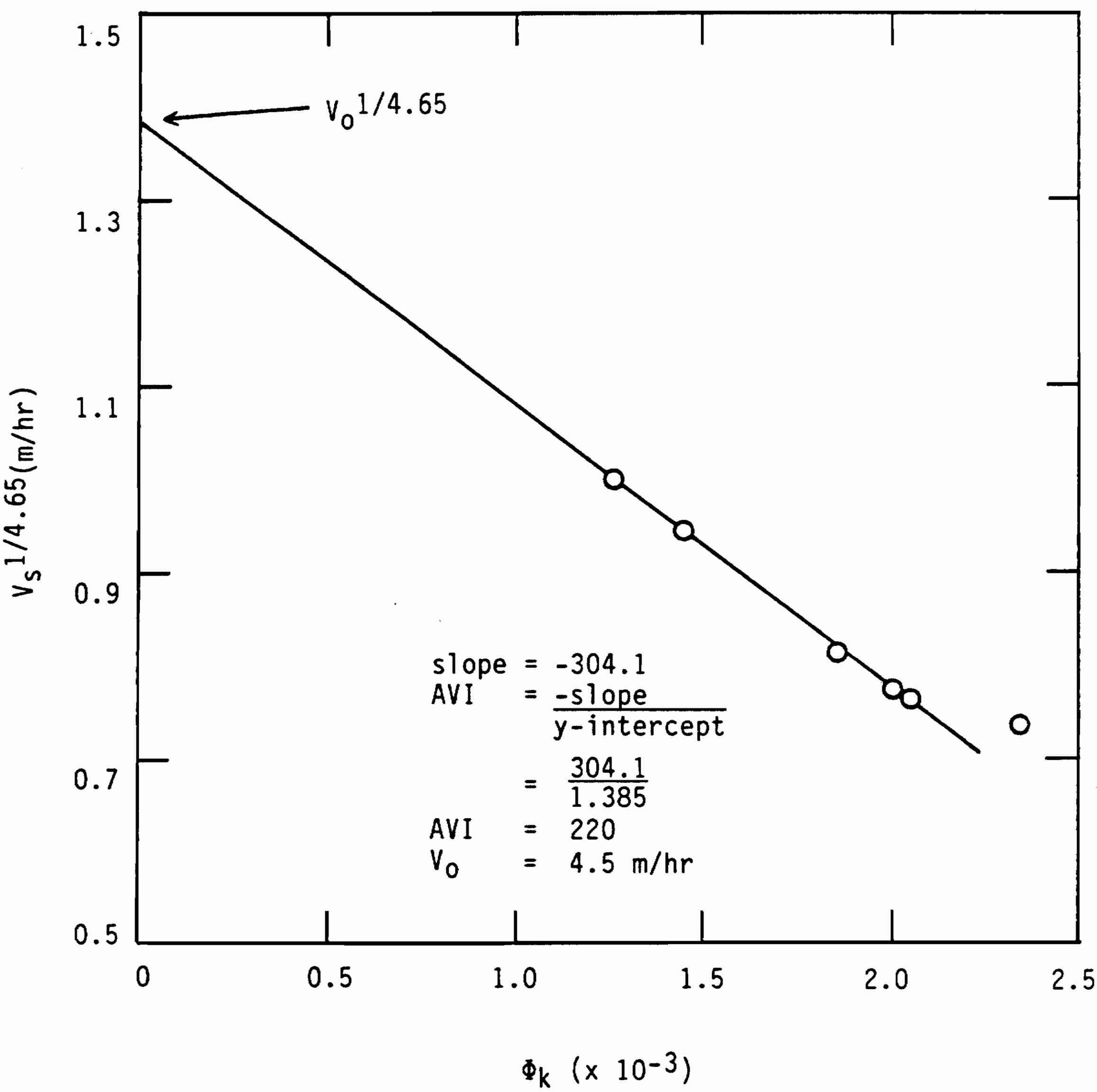

Figure 5. Typical relationship between sludge settling velocity (V_s) and volume fraction of dry solids.

H_o = initial height of unsettled sludge in graduated cylinder, cm

H_{min} = height of thickened sludge, cm

Development of a Floc Density Test

Using as foundations the work of previous researchers (4, 14, 16) and the literature cited in Chapter II, steps were taken to develop a method for measuring sludge floc density. Important variables in density gradient centrifugation were considered, including the gradient medium, method of gradient formation, method of sample addition, centrifugation force, centrifugation time, and temperature of the test environment. The methods used in evaluating these variables will be reviewed in this section while the results of this evaluation will be presented in Chapter IV.

Gradient Media. The media considered were cesium trifluoroacetate (CsTFA™), Percoll®, and sucrose solutions. CsTFA is marketed by Pharmacia Fine Chemicals AB of Sweden (17). Percoll, also marketed by Pharmacia, consists of colloidal silica particles which are coated with polyvinylpyrrolidine (18). Several properties of both of these products are listed in Table 1. A stock sucrose solution was prepared by adding 82.2 g sucrose to water to equal 100 mL total volume.

These three media were experimentally evaluated in terms of their osmotic pressure and viscosity. Osmotic pressure was investigated for its effect on the apparent floc density by comparing the floc density of a certain sludge as measured in each media. Depending on its ionic strength, a medium will exert a certain osmotic pressure. Therefore, stock Percoll was diluted with distilled water and with saline solution

Table 1. Physical properties of CsTFA and Percoll (18,19).

	Specifications	
Property	CsTFA	Percoll
Composition	134 g cesium trifluoroacetate per 100 mL of solution	silica sol with nondialysable PVP coating
Density	2.0 ± 0.05 g/mL	1.13 ± 0.005 g/mL
Conductivity	not available	1.0 mS/cm
Osmolality	19.8 Os/kg H_2O	0.020 Os/kg H_2O
Viscosity	1.4 cP at 20°C	10 ± 5 cP at 20°C
pH	4-9 (unbuffered)	8.9 ± 0.3 at 20°C
Refractive Index	1.376 at 25°C	1.354 ± 0.005 at 20°C

to create four sets of media having equal densities but being approximately 0.00%, 0.01%, 0.10%, and 1.0% saline, respectively. Figure 6 shows the relationship between density and osmotic pressure of each medium. The values of osmotic pressure for the various Percoll-derived media were calculated using Equations [19] through [22] as follows:

$$P_t = P_p + P_s \qquad [19]$$

where P_t = total osmotic pressure, pounds per square inch (psi)

P_p = osmotic pressure contributed by Percoll, psi

P_s = osmotic pressure contributed by saline, psi

and

$$P_p = S \times R \times K \times 14.7 \qquad [20]$$

where S = osmosity of the medium, mol/L

R = gas constant (0.082 atm-L/mol-^{o}K)

K = degrees Kelvin (295^{o})

Values for osmosity were found in the Handbook of Chemistry and Physics (19) by correlating them with the appropriate values of osmolality which were calculated by:

$$O_p = \frac{(\rho_p - 1)\ \rho_i\ O_i}{(\rho_i - 1)\ \rho_p} \qquad [21]$$

where O_p = osmolality of Percoll diluted with distilled water prior to dilution with saline, Os/kg

ρ_p = density of diluted Percoll prior to addition of saline, g/mL

ρ_i = density of undiluted Percoll, 1.13 g/mL in this case

O_i = Osmolality of undiluted Percoll, 0.020 Os/kg

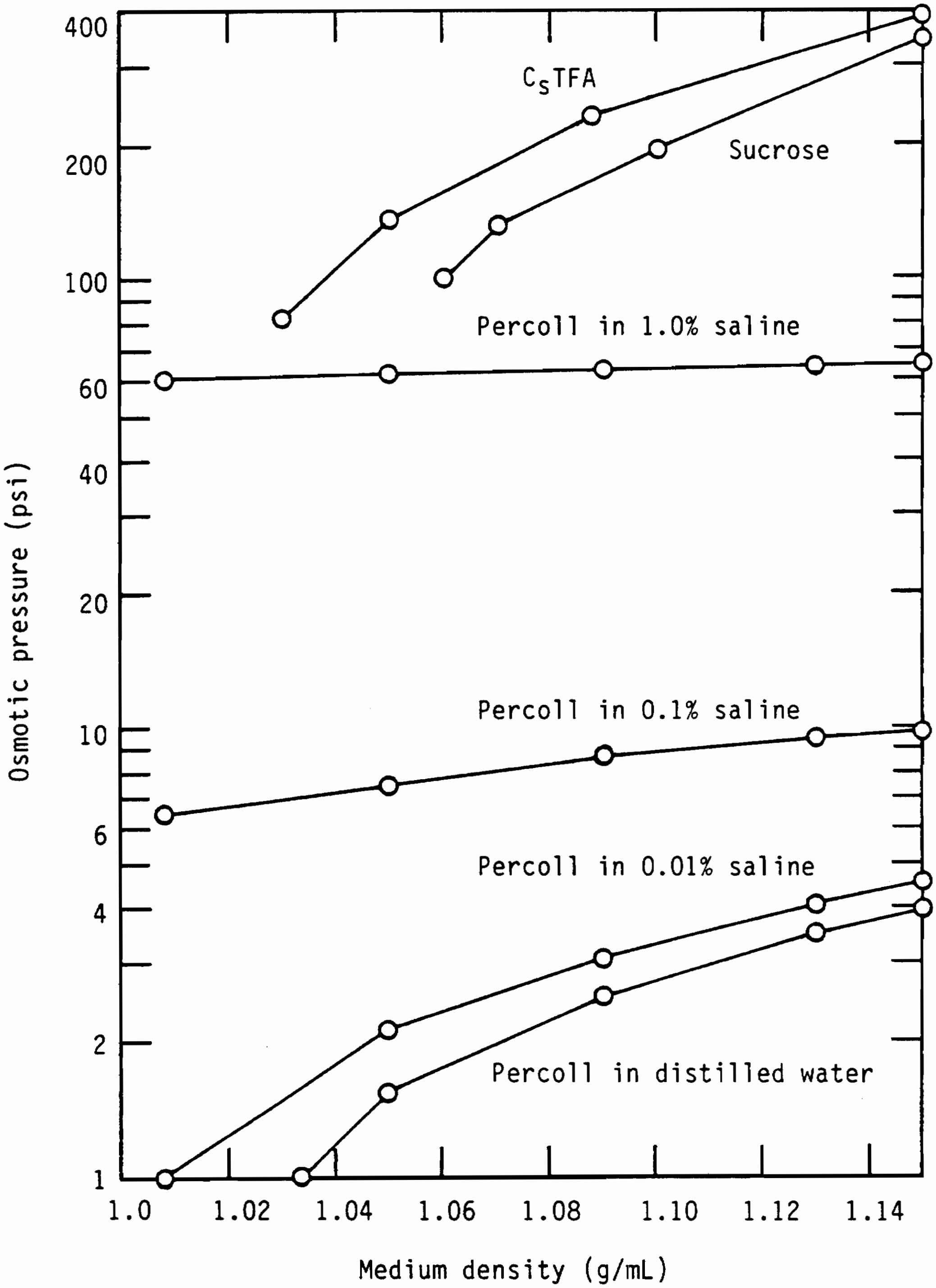

Figure 6. Relationship between medium density and osmotic pressure for various centrifugation media.

Values for P_s were determined using this equation:

$$P_s = M \times R \times K \times 14.7 \qquad [22]$$

where M = molarity of NaCl solution, mol/L,

1.0 % NaCl = 0.171 mol/L,

0.10 % NaCl = 0.017 mol/L, and

0.01 % NaCl = 0.0017 mol/L.

The osmotic pressures for CsTFA and sucrose were likewise calculated from known values of osmolality and osmosity.

Viscosity was viewed for its impact on ease of handling the media and on the rate at which a sludge sample reached its isopycnic point. Figure 7 shows the relationship between density and viscosity for the three media considered. Osmotic pressure and viscosity properties were not available for CsTFA; instead, information on these properties of cesium chloride was used because of this compound's similarity to CsTFA. Data on cesium chloride were obtained from the CRC Handbook of Chemistry and Physics (19).

Gradient Formation. As explained in Chapter II, there exist two types of density gradients: Step and continuous. This subsection will describe how each type was formed in the study.

Prior to making the gradients, various concentrations (i.e., densities) of each medium were prepared to cover the expected range of floc densities to be measured. These different densities were formed by dilution of stock or concentrated solution according to this formula:

$$V_i = \frac{V_F(\rho_F - \rho_D)}{\rho_i - \rho_D} \qquad [23]$$

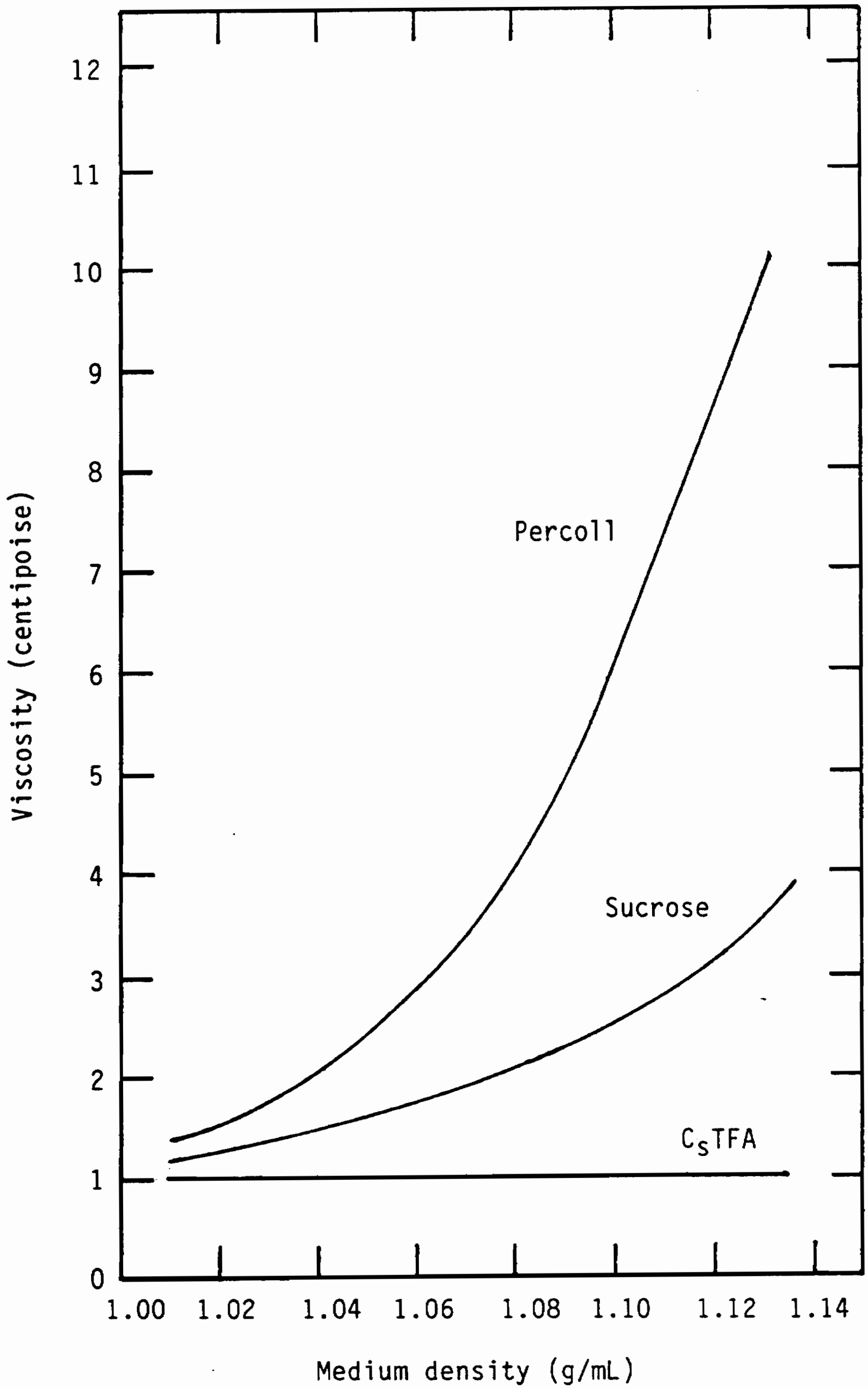

Figure 7. Relationship between medium density and viscosity for three centrifugation media (11,19).

where V_i = required volume of the undiluted medium, mL

ρ_i = density of the undiluted medium, g/mL

V_F = desired volume of the final working solution, mL

ρ_F = desired density of the final working solution, g/mL

ρ_D = density of the dilutant, g/mL,

distilled water = 1.000 g/mL,

0.01 % saline = 1.000 g/mL,

0.10 % saline = 1.001 g/mL, and

1.0 % saline = 1.010 g/mL.

Only the stock Percoll had to be concentrated to provide necessary working densities above its stock density of 1.13 g/mL; media concentration was accomplished using dialysis. Dialysis tubing was boiled for ten minutes in distilled water with a small amount of sodium carbonate. Percoll was poured into the tubing, and both ends of the filled tube were tied with plastic clips. The tube was then laid in a pan and covered with polyethylene glycol flakes which extracted water from the Percoll through the dialysis tubing. The concentrated Percoll was removed after approximately two hours.

The densities of the concentrated Percoll and the stock sucrose solution were determined by first measuring each medium's refractive index. Standard curves for each medium which relate refractive index and density (Figures 8 and 9) were then used to obtain the density. The maximum density of Percoll attained through dialysis was 1.285 g/mL.

Step gradients were formed in 10 mL glass centrifuge tubes by layering, in order of decreasing density, 1 mL of each "step" solution. Each layer was added from a 5 mL disposable polyethylene pipette gradu-

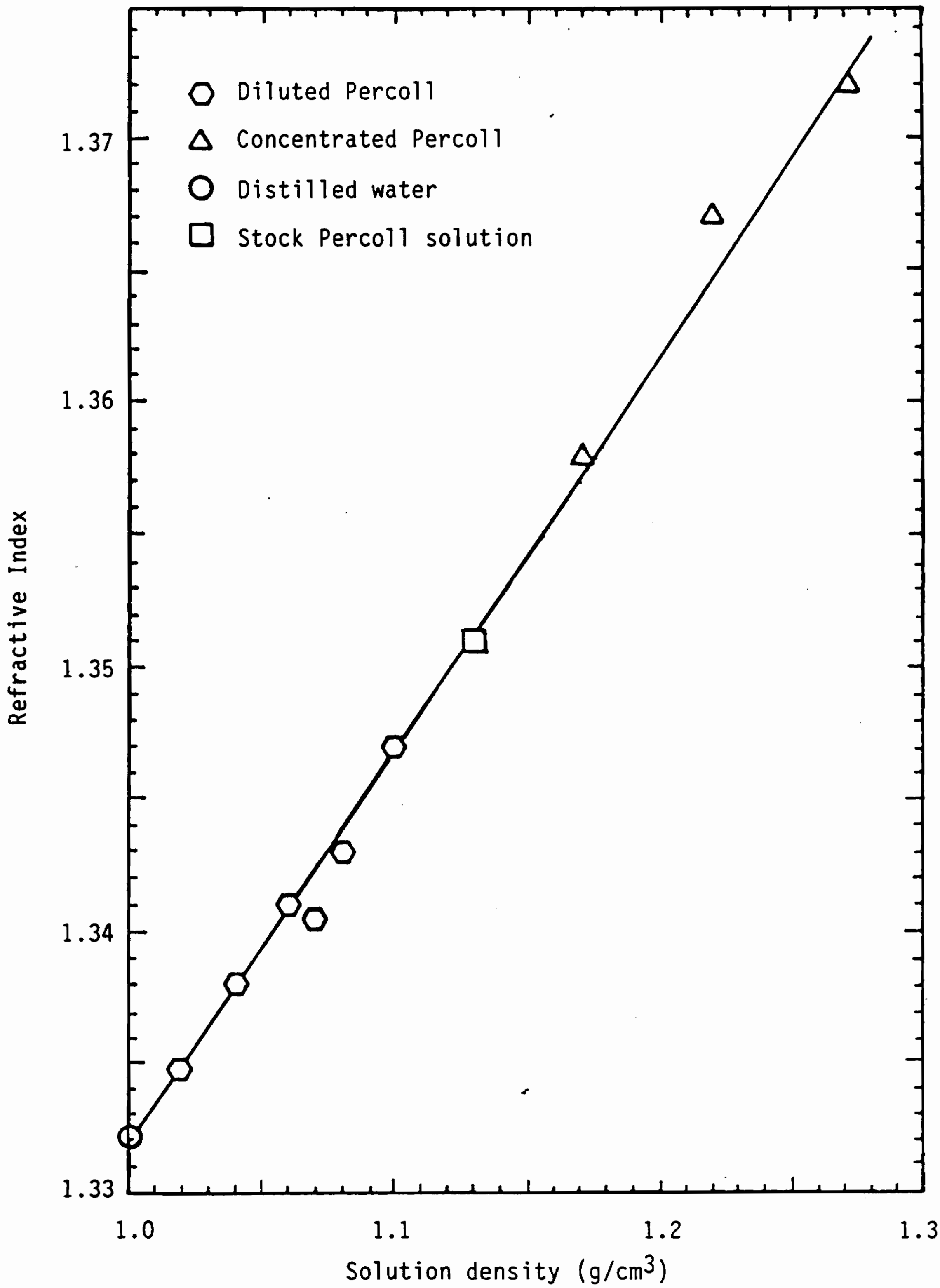

Figure 8. Calibration curve relating the density of Percoll solutions of varying density to the measured refractive index (temp. = 24.5°C) (4).

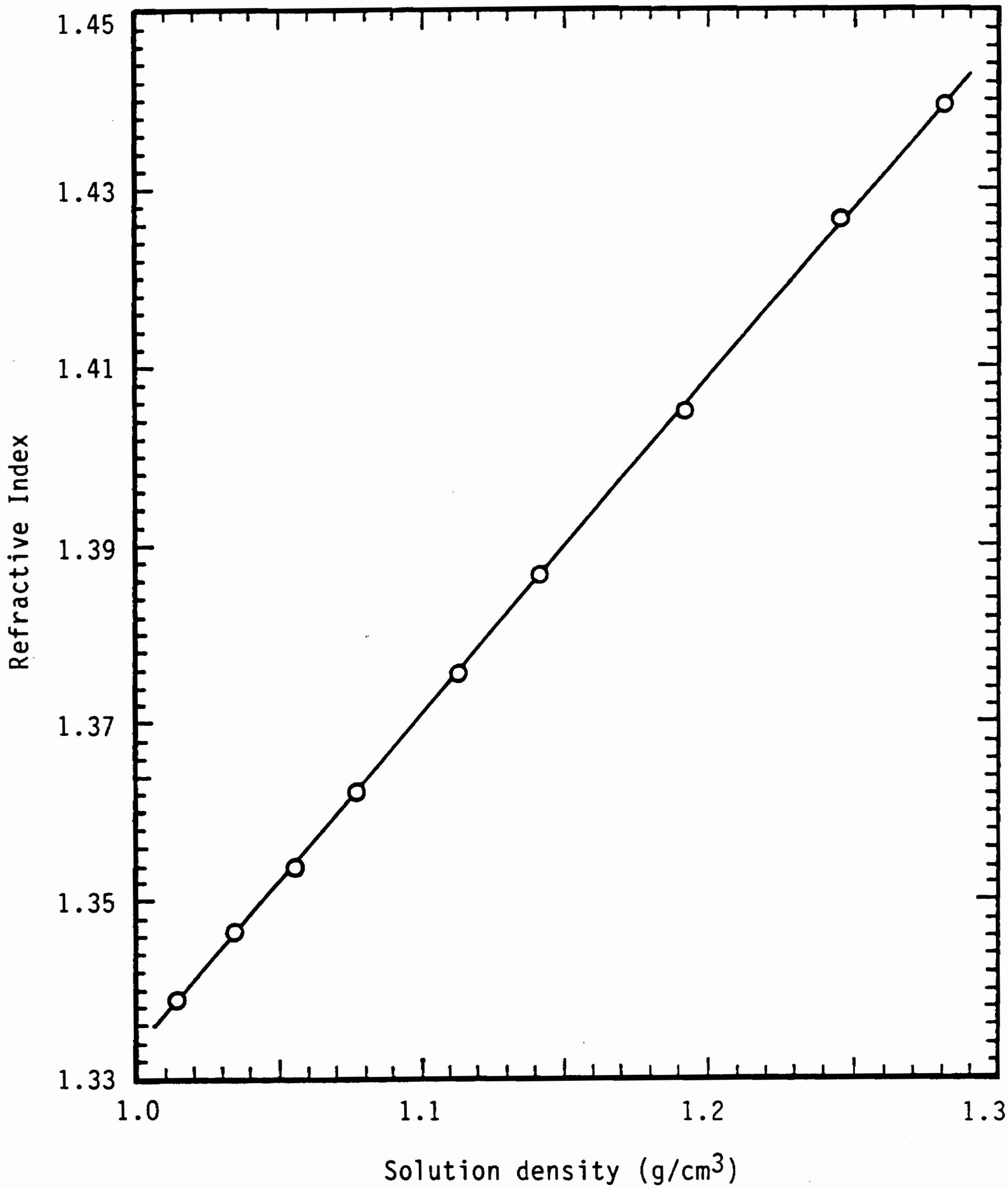

Figure 9. Calibration curve relating the density of sucrose solutions of varying density to the measured refractive index (temp. = 20°C) (19).

ated to 1 mL in 0.25 mL units while keeping the tip of the pipette just above the liquid surface. A mark was made on the outside of the tube at each interface to serve as reference points. Normally, a step gradient consisted of three to six layers with steps of 0.01 or 0.02 g/mL.

Continuous gradients were formed using only the Percoll medium. When a solution of Percoll is centrifuged at more than 10,000 g (g refers to relative centrifugal force or the average g-force exerted at the midpoint of the tube) in a fixed angle rotor, a gradient forms isometrically around the starting density and becomes steeper with time. The shape of this gradient will be approximately linearly related to the g-force and time of centrifugation. Density marker beads can be used to define the shape of the gradient as demonstrated in Figure 10 (18).

Density marker beads are supplied by Pharmacia and are derivatives of Sephadex^R (cross-linked dextran). There are ten color-coded types, each modified to have a specific density in gradients of Percoll diluted with sucrose or saline. The beads' densities must be recalibrated for use with Percoll diluted by another medium, such as water. This is accomplished by forming a gradient in the desired medium with the marker beads present and then fractionating the gradient and measuring the refractive index of the fractions of the fractions containing the marker beads. Table 2 summarizes the densities of the ten marker beads as exhibited in Percoll diluted with water (aqueous Percoll). These beads are useful only in the density range of 1.03 to 1.093 g/mL. Therefore, only sludges with relatively low floc densities were evaluated in continuous gradients. Continuous gradients were not utilized at higher densities due to the necessity of extracting medium from the gradient

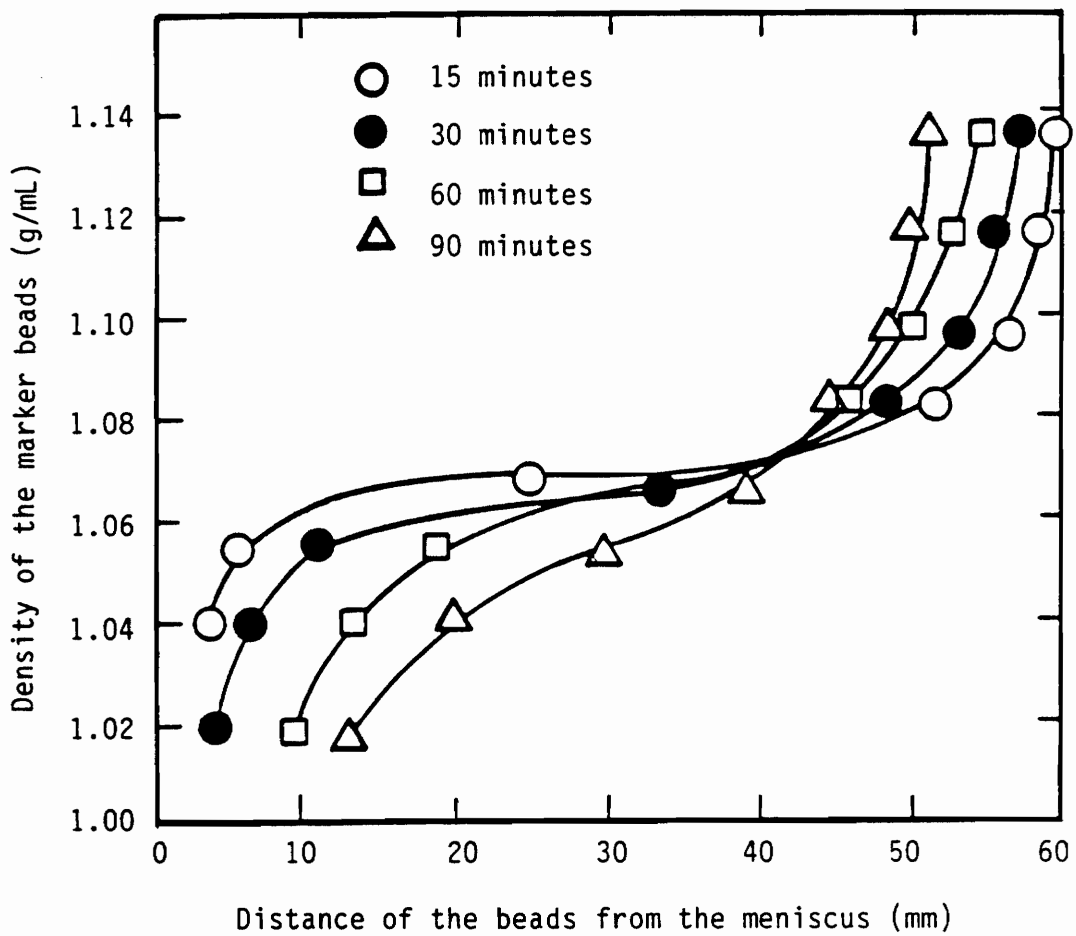

Figure 10. Location of calibrated density marker beads defining the shape of a self-generated density gradient in Percoll. The curves were formed by centrifuging 1.07 g/mL Percoll at 20,000 gav in an angle head rotor for increasing lengths of time (14).

Table 2. Density of calibrated density marker beads in gradients of Percoll diluted with water (14).

Bead no.	Bead color	Bead density in aqueous Percoll (g/mL)
1	blue	1.030
2	yellow	*
3	green	1.046
4	red	1.051
5	blue	1.060
6	orange	1.077
7	green	1.085
8	red	1.091
9	violet	1.093
10	blue	1.053

* not calibrated

and measuring its refractive index in order to determine the density at any one level within the gradient. This methodology was deemed to be too time-consuming.

To form a continuous gradient, a dilution of Percoll (having a density in the middle of the expected range of floc density to be measured) was added to a 10 mL plastic centrifuge tube. Density marker beads were then added on top of the solution. The tube was centrifuged for approximately 30 minutes at 15,000 rpm (17,600 g) in a high-speed Beckman centrifuge (Beckman Instruments, Inc., Fullerton, California, Model J-21C, rotor type JA-20). This provided an adequate gradient exhibiting ample bead separation. A continous gradient could be stored under refrigeration for several days without any effect on the shape of the gradient.

Gradient/Sample Centrifugation. The method of centrifugation of the sludge sample in the gradient was evaluated in terms of location of sample addition, centrifugation speed, centrifugation time, and temperature of the centrifuge and gradient environment.

Sample Addition. In both step and continuous gradient tests, sludge samples were added either on top or at the bottom of the gradient. This variation was made to establish whether the floc density test result would be affected by the location of sample addition in the tube.

A disposable polyethylene pipette was used to deposit the sample on the surface of both step and continuous gradients. Samples were deposited in the bottom of step gradients with a disposable pipette prior to layering the gradient. Bottom sample addition to continuous gradients was done after the gradient was formed using a 1 mL syringe, being careful not to disturb the pre-formed gradient and marker beads. Each sample consisted of one to three drops of sludge.

A different approach to sample addition was taken for testing dewatered sludge cake samples due to their solid consistency compared to the liquid nature of unthickened or gravity thickened sludges. For these samples, a small quantity (equivalent approximately to half of one liquid drop in volume) of sludge cake was gently scraped onto the tip of one of the plastic pipettes. The sample was then placed just below the surface of the gradient medium and simultaneously deposited in and mixed with the medium by repeatedly touching and rubbing the tip of pipette against the side of the centrifuge tube.

Relative Centrifugal Force. The centrifuge speed was varied to determine what effect centrifugal acceleration (g) had on the measured floc density. Samples in step gradients were centrifuged in a low-speed table top unit with a swinging-bucket rotor at speeds of 1300 and 2700 rpm (200 and 920 g, respectively) and at 5000 rpm (1960 g) in the Beckman centrifuge. Samples were centrifuged in continuous gradients at speeds of 2000, 5000, 10,000, and 15,000 rpm (310, 1960, 7830, 17,600 g, respectively) in the Beckman centrifuge. The floc density determined at each speed was plotted versus the relative centrifugal force to appraise the effect of the applied g-force.

Centrifugation Time. At each centrifuge speed, the rotor was stopped at one- to two-minute intervals to record the floc density at that time. Plots were made of floc density versus centrifuge time to determine whether the test's result is impacted by the length of centrifugation.

Temperature. Step gradient centrifugation analyses were conducted under two different room temperatures to evaluate this factor's effect on the test. One set of tests was performed in a room with uncontrolled temperature. During the floc density measurements, temperature was approximately 27^{o}C. Another set of tests was carried out in a controlled-temperature laboratory having a temperature of 20^{o}C. The effect of the temperature in the test environment was evaluated from the behavior of the gradients in terms of convection and/or diffusion. The continuous gradients were all centrifuged in the same environment (i.e., a cooled rotor); therefore, the effect of temperature effect on this type of gradient was not determined experimentally.

Evaluating the Floc Density. Sludge floc particles exhibited the following reactions to density gradient centrifugation:

1. Floated. Sludge floc particles floating on the surface of the gradient were too light for the selected density range.
2. Formed one distinct band. The floc reached its isopycnic point; all floc were of similar density.
3. Formed multiple bands or remained in a scattered suspension. The sludge particles were not of uniform density. The points where flocs came to rest revealed a distribution of floc densities within that sample.
4. Pelleted. Sludge that pelleted to the bottom of the tube or gradient was too dense for the chosen density range.

A density measurement was recorded as a single value for a distinct band or as a range of values for multiple bands or a scattered suspension.

Density results from step gradients were read using the demarcations made on the tube which represented the location of the interface between the gradient's layers. Values from continuous gradients were evaluated by measuring the distance in millimeters from the top and bottom of scattered sludge bands to the marker beads immediately above and below the sample particles. These distances were then used to interpolate between the beads' densities to calculate the range of the sludge's floc density.

Sludge Dewatering Methods

One method used for sludge dewatering has already been described, i.e., gravity thickening. Three mechanical dewatering processes were also used: pressure filtration, centrifugation, and vacuum filtration. Mechanically dewatered samples were analyzed for dewatered cake solids concentration, floc density, and bulk density.

Pressure Filtration. Only the alum II + polymer sludge was dewatered by pressure filtration. A laboratory-scale plate-and-frame filter press (JWI Filter Press, Model 250.15, JWI, Inc., Holland, Michigan) was used to accomplish this type of dewatering. The press was filled with approximately 20 L of sludge for each test. Compressed air then gradually pressurized the filter press chambers to 100 psi. The pressure was then released and the dewatered sludge cake from several areas of the various filter cloths was manually removed for analysis.

Centrifugation. Four of the laboratory-generated sludges and the two unconditioned alum sludges were dewatered by centrifugal means. A sample of each sludge was poured into six centrifuge tubes. The six tubes were then inserted into the high-sped Beckman centrifuge and subjected to a speed of 10,000 rpm for five minutes. The water which was separated from the solids was poured off and the dewatered solid phase was analyzed.

Vacuum Filtration. Vacuum dewatering was performed on all the sludges except the bentonite + alum, WAS I and II, and the lime sludge. This method utilized a Buchner funnel apparatus (Figure 11). Sample sludge volumes of 50 to 100 mL were poured into the funnel and allowed to stand for 30 seconds. A pressure of 15 inches of mercury was applied across the sample as water passed through the #40 ashless filter paper. Dewatering was discontinued when the sludge cake failed, i.e., when large cracks appeared across the cake and the pressure differential across the cake began to drop.

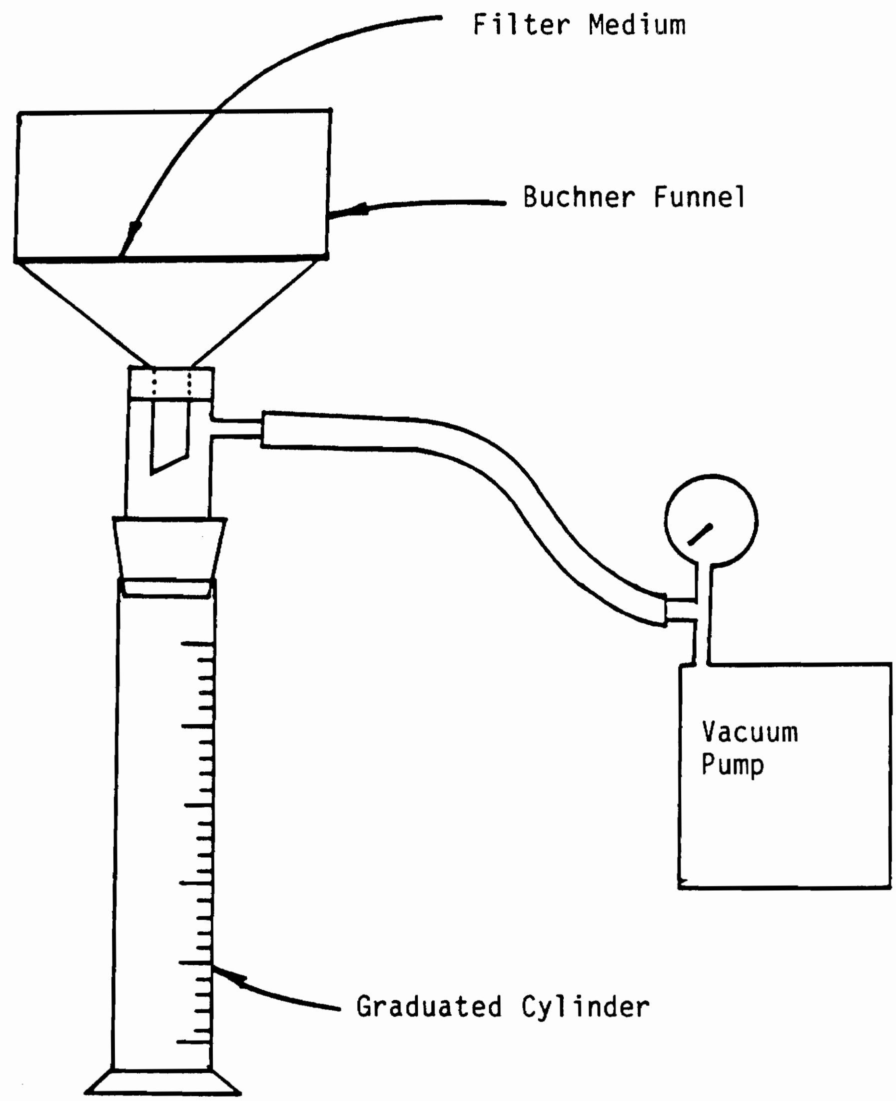

Figure 11. Schematic diagram of Buchner Funnel apparatus used for vacuum dewatering tests.

CHAPTER IV. RESULTS AND DISCUSSION

The laboratory data that resulted from this research will now be presented and evaluated. The results are divided into two main sections, beginning with the density gradient centrifugation method for determining sludge floc density followed by a review of the results of the sludge dewatering tests.

Floc Density Measurement

An evaluation of density gradient centrifugation as a method for determining sludge floc density was made on the basis of the type of gradient media, method of gradient formation, and method of gradient/sample centrifugation. Data for this area of the research were generated using twelve sludges, including data obtained from another researcher's work with an iron hydroxide sludge.

Gradient Media. The usefulness of three density gradient media (CsTFA, Percoll, and sucrose) was examined in terms of their osmotic pressure, viscosity, and cost. Osmotic pressure was investigated for its effect on sludge samples, while medium viscosity was evaluated to determine what impact, if any, it had on the centrifugation time required for sample particles to reach their isopycnic point and on medium handling.

Floc density values of four sludges as measured in different gradient media are presented in Table 3. It is apparent from these data that the measured floc density of a given sludge varied depending on the medium. For instance, the floc density of alum II sludge was in the range 1.22 - 1.25 g/mL when measured in aqueous Percoll but was determined to be 1.9 g/mL in CsTFA. It may be concluded that these floc

Table 3. Floc densities of several sludges as measured in media exhibiting different osmotic pressures.

Sludge	Media	Floc density (g/mL)	Osmotic pressure (psi)
Alum II	Aqueous Percoll	1.24	4
	CsTFA	1.9	2400
Iron *	Aqueous Percoll	1.11	3
	Sucrose	1.3	1000
Lime	Aqueous Percoll	1.5	60
	CsTFA	1.9	2400

* data from Dove (16)

density variations were a result of the osmotic pressures of the media (also displayed in Table 3) by noting, for example, that aqueous Percoll of density 1.22 - 1.25 g/mL has an osmotic pressure of approximately 4 psi while that of CsTFA at a density of 1.9 g/mL is 2400 psi. In view of these results, high osmotic pressures evidently force water out of the floc structure and thereby increase the floc's dry solid fraction and corresponding apparent density.

Table 4 shows more floc density results obtained using various media. Samples of a waste activated sludge (WAS II) were analyzed in Percoll diluted with low ionic strength water (aqueous Percoll), Percoll diluted with saline to provide three different levels of ionic strength as shown, and in CsTFA. These data reveal that as the osmotic pressure exhibited by the density medium increased, the apparent floc density also increased, thereby demonstrating further the critical nature of density medium osmotic pressure. In view of these data, the point must be stressed that dilutions of Percoll for use in density gradients need to be made in low ionic strength water.

Viscosity proved to be an important parameter in terms of both centrifugation time and media handling. Figures 12 through 16 demonstrate the apparent effect that viscosity had on the required centrifugation time. In dilutions of Percoll with densities greater than about 1.20 g/mL, four to six minutes of centrifugation were required before the sludge floc particles reached their isopycnic point. After this time the movement of particles in the gradient was negligible. This scenario differs from that at lower densities (e.g., 1.02 g/mL) which was typified by required centrifugation times of only three minutes or less.

Table 4. Floc density of a waste activated sludge as measured in media exhibiting different osmotic pressures.

Media	Floc density (g/mL)	Osmotic pressure (psi)
Aqueous Percoll	1.02	0.5
Percoll in 0.01% saline	1.02	1.2
Percoll in 0.10% saline	1.03	7
Percoll in 1.00% saline	1.04	62
CsTFA	1.40	1100

Percoll at a density of 1.02 g/mL has a viscosity of 1.3 centipoise (cp) which increases to approximately 18 cp at a density of 1.2 g/mL (see Figure 7 in Chapter III). Therefore, on the basis that aqueous Percoll grows more viscous with increasing density, the time needed to complete an isopycnic centrifugation increases with greater medium viscosity.

In qualitatively comparing the media on the basis of ease of handling, it was found that lower viscosity media was much easier to work with. In general, the more viscous the medium the more difficult and time-consuming it was to prepare dilutions and gradients. Percoll was the only medium that caused handling problems due to its sharply increasing viscosity at densities above about 1.07 g/mL.

Gradient Formation. The two types of density gradients utilized in this study were step and continuous gradients. This section will compare these types of gradients in terms of their formation time, the accuracy of results using each, and their useful lives. These comparisons will be made only on the basis of tests using Percoll.

Forming a step gradient first required the creation of several dilutions (normally four or five) of the stock medium (Percoll) and then slowly and carefully layering these dilutions into a centrifuge tube. Forming a continuous gradient involved placing only one dilution or density level of the stock medium in a centrifuge tube along with density marker beads and centrifuging the mixture for approximately 30 minutes to provide adequate marker bead separation. Although the use of both types of gradients necessitated the dilution of the stock media, many more dilutions were needed to form a step gradient that covered the same range of densities provided by one dilution in a continuous gradient. Furthermore, diluting dialyzed Percoll to create different density levels for use in step gradients was very time-consuming due to the viscous nature of the dialyzed medium. However, continuous gradients were not useful with dialyzed Percoll due to the absence of marker beads calibrated at densities above that of the stock Percoll (i.e., 1.13 g/mL). When considering the time spent both making the medium dilutions and actually forming the gradient at densities less than 1.13 g/mL, continuous gradients were formed in less time than were step gradients.

Continuous gradients also have an advantage over step gradients in the accuracy of the test results. The floc density value is measured in continuous gradients by linearly interpolating between the densities of the marker beads (values known to an accuracy of 0.001 g/mL) immediately above and below the sample particles. In this manner, the precise density of a particular sludge floc particle may be determined or the density range of the entire sample may be established. On the other hand, sample particles either come to rest within a layer (step) or layers of a step gradient, indicating the floc density is equal to that of the particular step(s), or they settle at the interface between steps, signifying the floc density is somewhere in the range between the densities of the gradient layers immediately above and below the particles. The latter case was by far the more prevalent in this study because the minimum difference in density between steps was set at 0.01 g/mL. This was practiced in order to maintain individual density layers within the gradient and to provide a visible plane at the layer interfaces. Therefore, floc density results from step gradients were normally read as ranges with the minimum range being 0.01 g/mL, while results from continuous gradients could be reported more accurately.

Another comparison that may be made between the two types of gradients is that of their useful lives after formation. According to its supplier (14) and verified during this research, continuous gradients of Percoll are good for several days after they are formed, provided they are covered and refrigerated. On the other hand, step gradients were not useful after approximately 30 minutes. The major advantage of the longer life of continuous gradients is that several may be prepared simultaneously and then used as needed.

Maintaining step gradients was discovered to be particularly more difficult at higher room temperatures. The higher temperatures evidently promoted convection and/or diffusion in the medium which in turn caused the gradient layers to mix. This negative effect caused by the test environment was controlled by performing step gradient centrifugations in an air-conditioned room.

Gradient/Sample Centrifugation. Different methods of centrifuging sludge samples in the density gradients were investigated. The parameters varied were the point of sample addition, centrifuge speed, centrifugation time, and the type of centrifuge rotor used.

Sludge samples were added either at the bottom or the surface of gradients. This variation was found to have a very slight effect, if any, in the measured floc density. Figure 12 demonstrates this point with plots of floc density versus time under centrifugation in continuous gradients. The only instance when the sample addition point

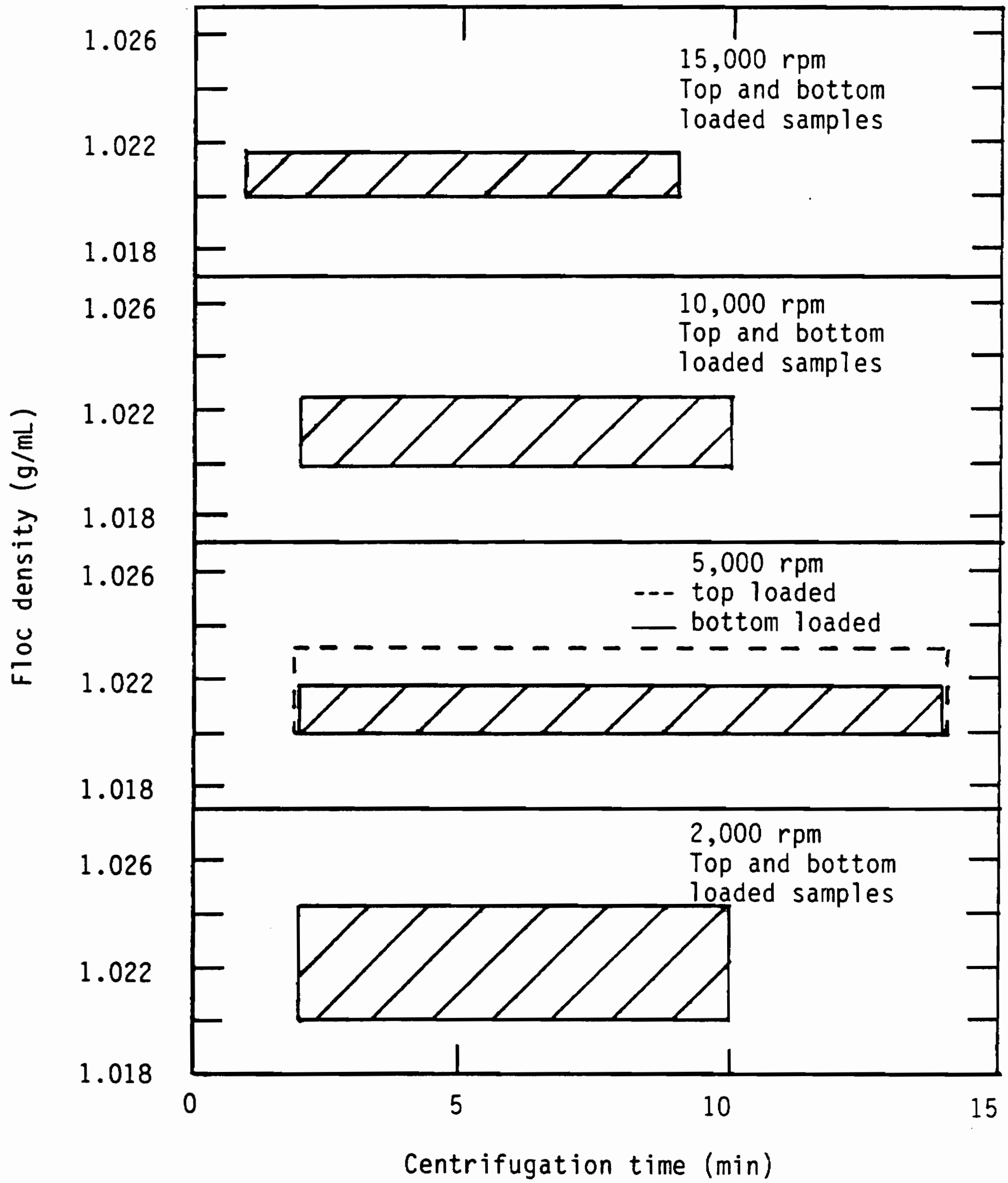

Figure 12. Variation of WAS II sludge floc density with time, centrifuge speed, and location of sample addition point when measured in continuous gradients of aqueous Percoll.

affected the results was at 5000 rpm; but, the midpoint of the two floc density ranges differed by only 0.001 g/mL. Bottom versus top-loading of sample also made no significant difference when step gradients were used.

The centrifuge speed, or relative centrifugal force (g), was found to be significant only when conducting tests in the more viscous medium in step gradients. A few examples of this are evident in Figures 13-16. The results in Figure 13 for the alum I sludge show that the sample particles migrated downward to their isopycnic point more quickly under the higher centrifugal force and that after 8 minutes the sludge flocs ranged over higher densities at this force than at the lower g. The reader will also note that, under the greater g force, a portion of the particles eventually moved back upward to lower density levels. This was an isolated occurrence in these experiments that is, nevertheless, noteworthy because it indicates the potential for floc aggregation (i.e., formation of lower-density particles) and/or mixing of the gradient steps during centrifugation.

The plots for the alum II sludge shown in Figure 14 are typical for this study. In this case, the higher centrifuge speed served to bring the sludge flocs to the top of their density range sooner, while the floc particles at the lower part of the range took longer to reach their isopycnic point than they did during centrifugation at the lower g force. After ten minutes, the results at the two different speeds are practically equal.

The alum I and II plots discussed above involved floc densities greater than 1.20 g/mL which were, therefore, measured in rather viscous

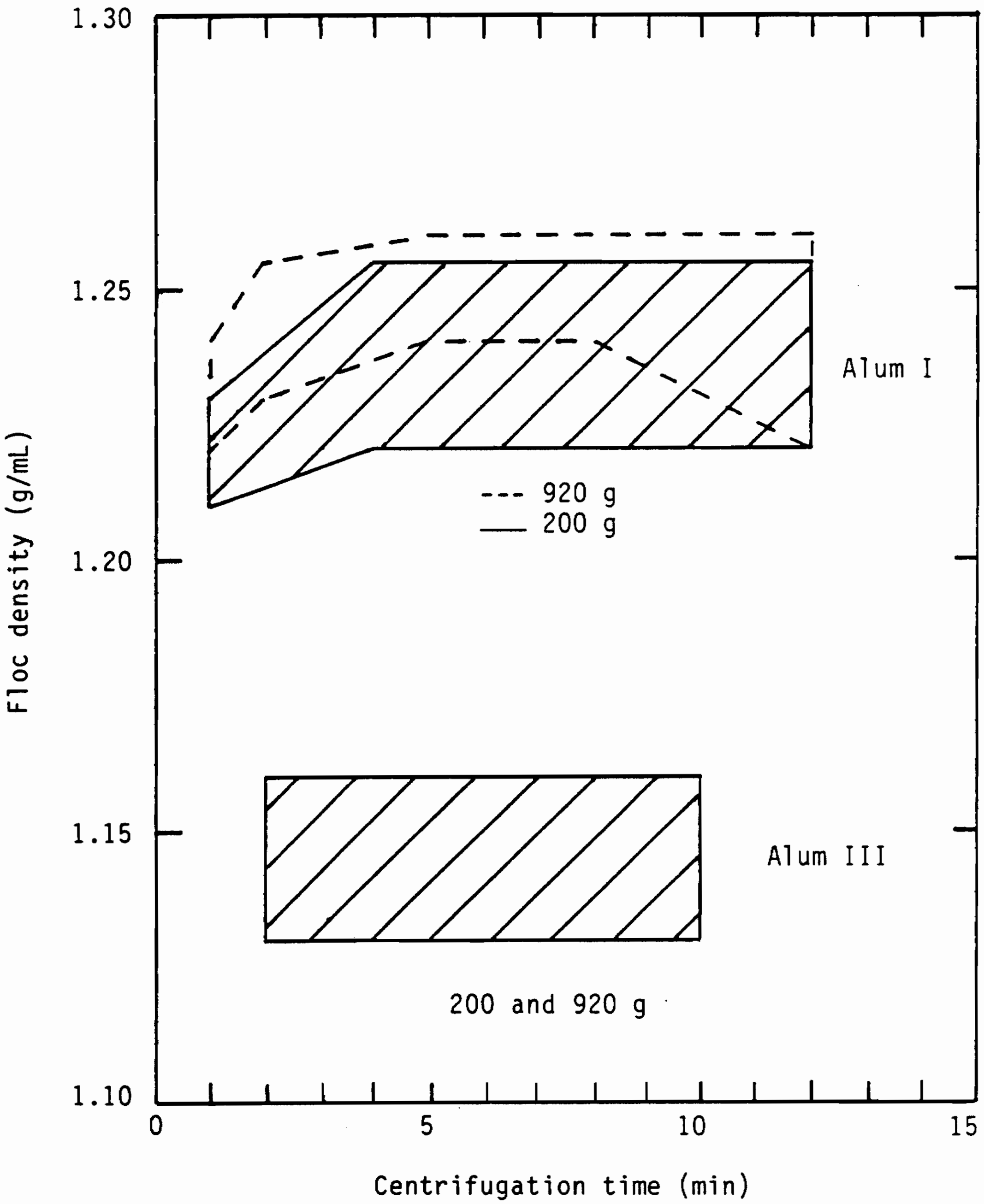

Figure 13. Variation of Alum I and Alum III sludge floc density with time and centrifuge speed when measured in step gradients of aqueous Percoll.

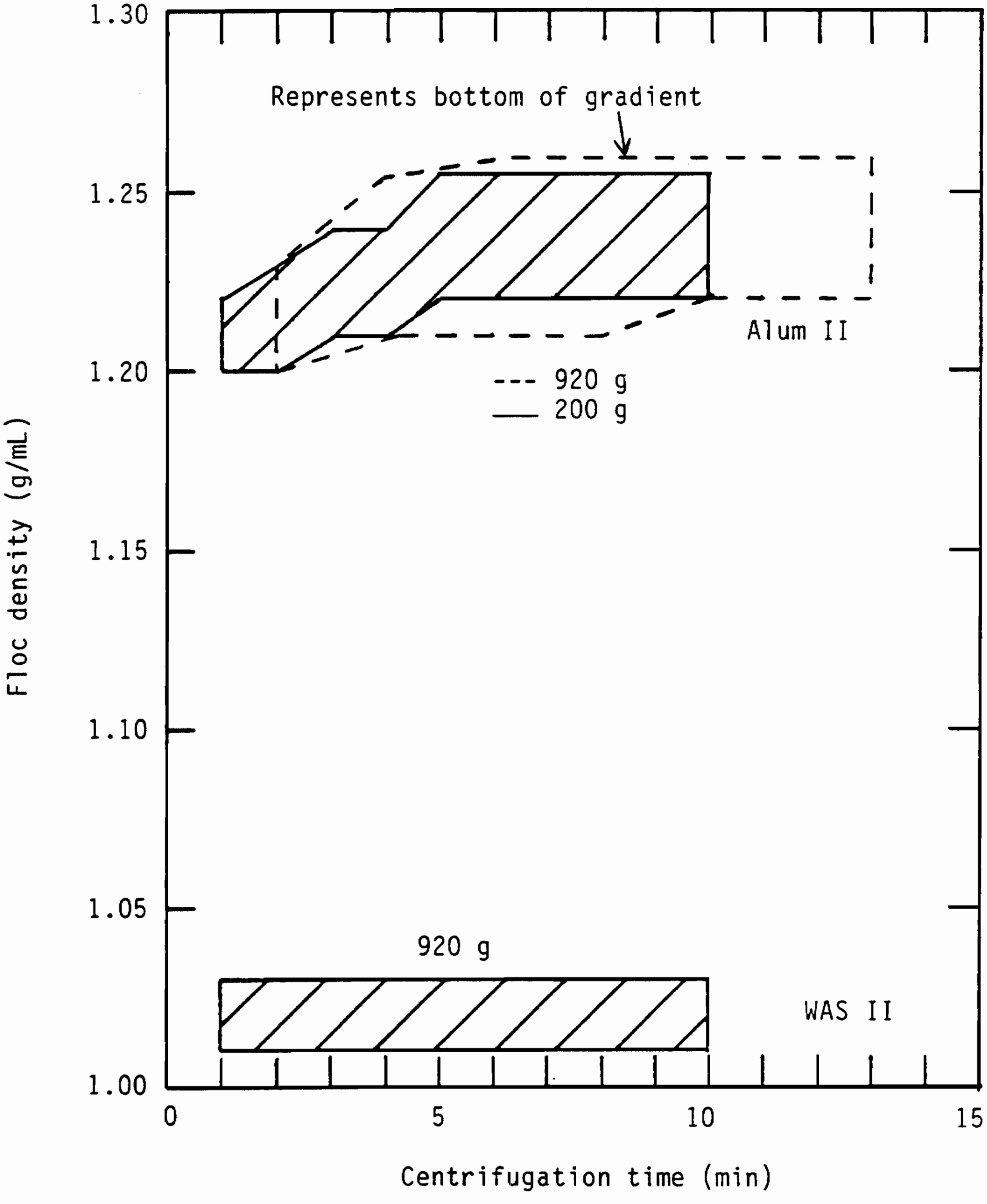

Figure 14. Variation of Alum II and WAS II sludge floc density with time and centrifuge speed when measured in step gradients of aqueous Percoll.

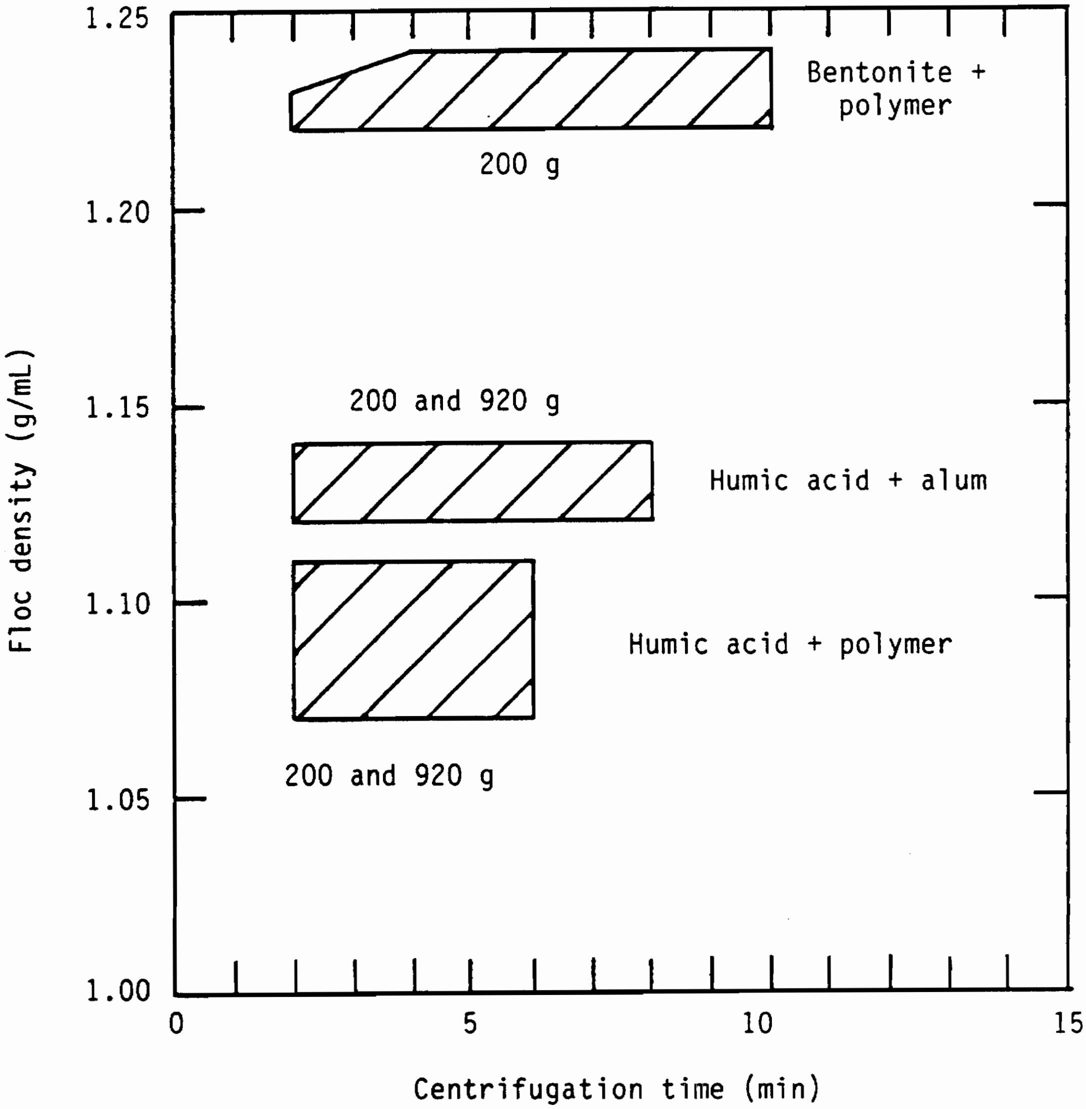

Figure 15. Variation of bentonite + polymer, humic acid + alum, and humic acid + polymer sludge floc density with time and centrifuge speed when measured in step gradients of aqueous Percoll.

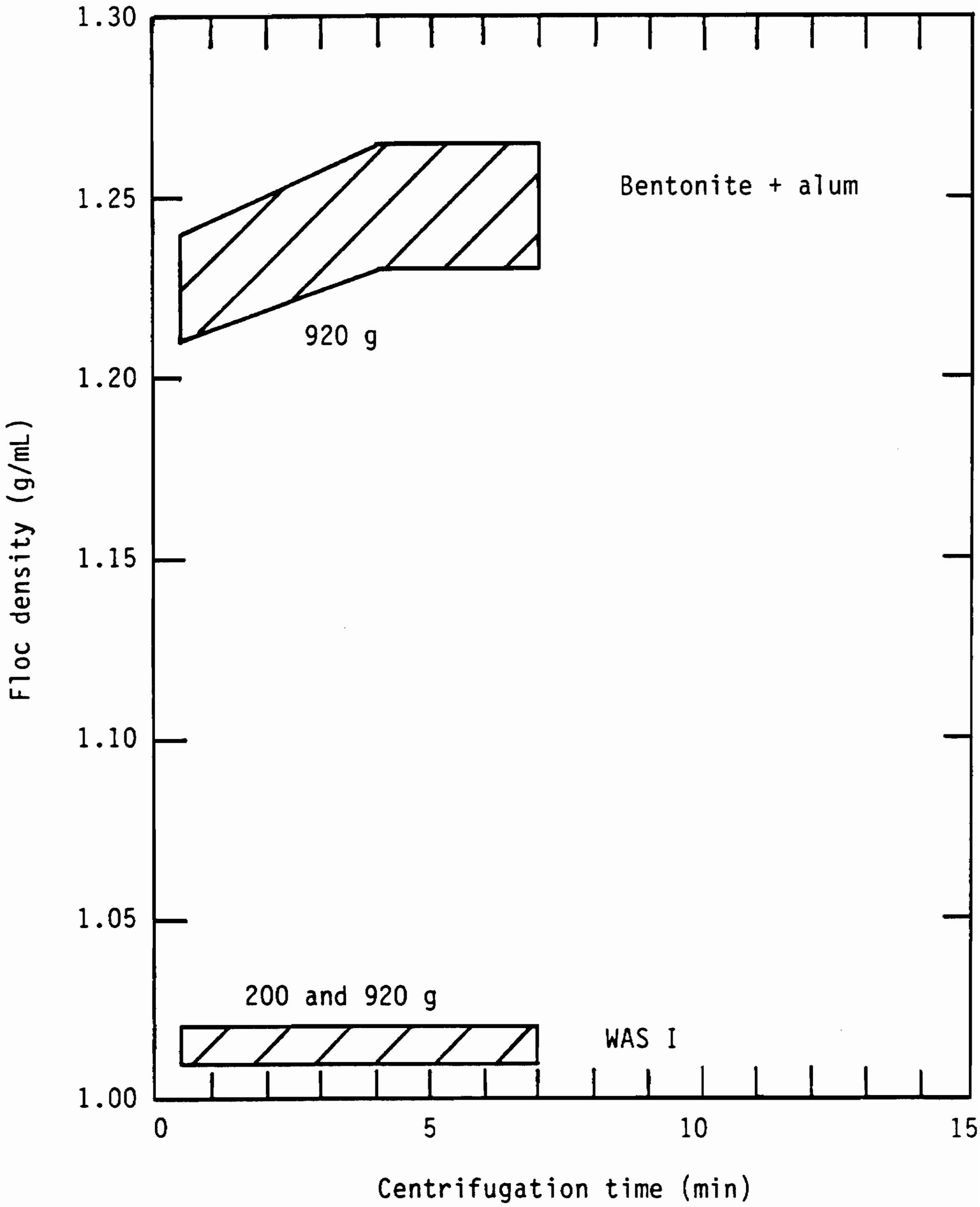

Figure 16. Variation of bentonite + alum and WAS I sludge floc density with time and centrifuge speed when measured in step gradients of aqueous Percoll.

Percoll media. The plots for alum III sludge and WAS I, as shown in Figures 13 and 16, respectively, demonstrate that the viscosity of the gradient medium evidently determines whether the g force will affect the floc density results. These two plots reveal that the resulting floc densities, which were lower and, therefore, measured in less viscous media than were the alum I and II samples, were not affected by centrifuge speed.

The plots for bentonite + 1195 and bentonite + alum in Figures 15 and 16, respectively, along with the alum I and alum II data discussed above, demonstrate that the measured sludge floc density can vary with centrifugation time. For each of these sludges, their floc density increased with time for a while and then stabilized. As explained previously, the value for the alum I sludge decreased after a period of remaining constant. These four samples exhibited floc densities that required them to be analyzed in high viscosity gradients. It therefore appears that the viscosity of the Percoll media caused the measured floc density to increase with time in the first few minutes of the test by slowing the downward movement of the sample particles to their isopycnic points through increased friction.

A fixed-angle rotor was used to centrifuge both step and continous density gradients, while a swinging-bucket rotor was used only with step gradients due to the unit's inability to reach the high speeds necessary to utilize continous gradients. It was found that the fixed-angle rotor was suitable only for use with continous gradients. In the fixed-angle rotor, step gradients mixed and their samples smeared against the inside of the outer wall of the tube.

Recommended Floc Density Test Methodology. Consideration of the results of this study's density gradient centrifugation tests allows the proposal of a sludge floc density test methodology. The following methodology is recommended:

1. Gradient media. A low osmotic pressure, low viscosity medium should be used. In this study, Percoll was found to be the most favorable medium even though it was very viscous at the higher densities. Osmotic pressure was more critical to the test results than viscosity in that it caused a significant increase in the floc particles' density, whereas viscosity only created handling problems and altered the time required to reach the isopycnic point. If a medium becomes available that is less viscous than Percoll yet has the same low osmotic pressures then, naturally, it should be used.

2. Type of gradient. Continuous gradients are preferred over step gradients. However, the use of continuous gradients of Percoll at densities above 1.13 g/mL is not practical at the present time due to the lack of the necessary density marker beads.

3. Gradient formation.

Continuous gradients. A dilution of the stock Percoll having a density near the middle of the expected range of the sample's floc density is added to a 10 mL plastic centrifuge tube and then centrifuged for 30 minutes at approximately 18,000 g in a fixed-angle rotor. One to two drops of three to four different density marker beads are added to the Percoll prior to centrifugation. The goal here is to provide ample marker bead separation to later allow the precise measurement of the distance between the beads and the sample.

Step gradients. Step gradients should be formed in 10 mL glass or plastic centrifuge tubes by layering, in order of decreasing density, 1 mL of each "step" dilution. It is recommended that the medium be added using disposable polyethylene pipettes graduated to 1 mL while keeping the tip of the pipette just above the liquid surface. A different pipette must be used for each layer. The interface between each step should be marked on the outside of the tube. The number of steps and the difference in density between adjacent steps will vary depending on the desired accuracy of the floc density results. However, when dealing with densities below 1.13 g/mL, the minimum density step should be 0.02 g/mL; above 1.13 g/mL the minimum step should be 0.01 g/mL. The temperature of the environment used for working with step gradients should maintained at 20-22^{o}C to minimize mixing of the gradient due to convection and/or diffusion.

4. Media and gradient storage. The various dilutions of Percoll must be covered and stored under refrigeration with as small an air space as possible above the liquid. This will minimize evaporation of the water in the dilutions and the resulting increase in density. Continuous gradients, if refrigerated, will be useful for several days. On the other hand, step gradients must be used within approximately 30 minutes after formation; therefore, storage is not an issue.

5. Sample addition. No more than three drops of a sludge sample should be added to a gradient to keep the volume of the sample sufficiently small in comparison to the volume of each density step of a step gradient and to help maintain the linearity of continuous gradients. Samples may be added either on the top or at the bottom of

density gradients. However, adding the sample to the top of continuous gradients prevented the disturbance of the gradient.

6. Gradient/sample centrifugation.

Continuous gradients. Varying centrifuge speed (or centrifugal force) and centrifugation time had no appreciable effect on the floc density results. It is suggested that samples be centrifuged in continuous gradients (for densities less than 1.13 g/mL) at approximately 2000 g for 5 minutes.

Step gradients. Sludge samples analyzed in step gradients should be centrifuged only in swinging-bucket rotors. In gradients of dialyzed Percoll, the samples should be centrifuged at a g-force of at least 200 for 8 minutes. For gradients below a density of 1.13 g/mL, samples may be centrifuged for a shorter time, approximately 3 minutes, at at least a 200 g-force.

7. Floc density evaluation.

Continuous gradients. The floc density value obtained from a continuous gradient is calculated by measuring the distance in millimeters from the top and bottom of the band of sludge floc particles to the marker beads immediately above and below the particles. These distances are then used to interpolate between the beads' densities to calculate the sludge's floc density range.

Step gradients. Floc densities are read in step gradients using the demarcations made on the tube which represented the interface between the gradient steps. The value is reported as a single density if the total sample comes to rest in one layer or as a density range if the sample bands over more than one gradient layer. This proposed floc density methodology was used for all subsequent tests, the results of which are presented in the next section.

Sludge Dewatering

The purpose of this section is to present and interpret the data collected during the laboratory gravity thickening and mechanical dewatering of different sludges. An attempt will be made to gain insight into the behavior of sludges during these dewatering processes. Additionally, the data will be used to investigate the effect sludge microproperties have on this behavior and on the extent of sludge dewatering.

Gravity Thickening. Gravity thickening of sludges was simulated using batch settling tests as described in Chapter III. Using values of the sludge bulk density, dry solids concentration, and dry particle density, which were measured directly or calculated, and graphically analyzing settling data, parameters related to the aggregate fraction (Aggregate Volume Index, aggregate particle density, and aggregate volume fraction) of the sludges were derived. All of these sludge properties were then incorporated into an investigation of sludge behavior during thickening. Table 5 contains values of several microproperties of the seven sludges utilized in the dewatering studies and will be referred to throughout the remainder of this chapter.

The traditional plot of log (V_s) versus initial dry solids concentration was made to compare settling characteristics among the six sludges (Figures 17 and 18). A sludge exhibiting the least variation in V_s with respect to mass solids concentration is most desirable, as this helps to maximize solids flux through a gravity thickener. The alum I

Table 5. Values of various fundamental sludge microproperties.

Sludge type	ρ_a (g/mL)	ρ_f		ρ_k (g/mL)	V_o (m/hr)	AVI
		range	average			
Alum I	1.007	1.22-1.255	1.24	2.45	13	205
Alum III	1.008	1.13-1.16	1.14	2.55	4.5	220
Alum III + polymer	*	1.17-1.18	1.17	2.30	*	*
Humic acid + polymer	1.003	1.08-1.11	1.10	2.51	2.1	560
Humic acid + alum	1.001	1.12-1.14	1.13	2.50	4.6	980
Bentonite + polymer	1.006	1.22-1.24	1.23	2.27	9.6	195
Bentonite + alum	1.007	1.24-1.265	1.25	2.15	10	170

* Settling tests were not performed on this sludge to evaluate these properties.

** These values were obtained prior to mechanical dewatering.

*** average of three or four values

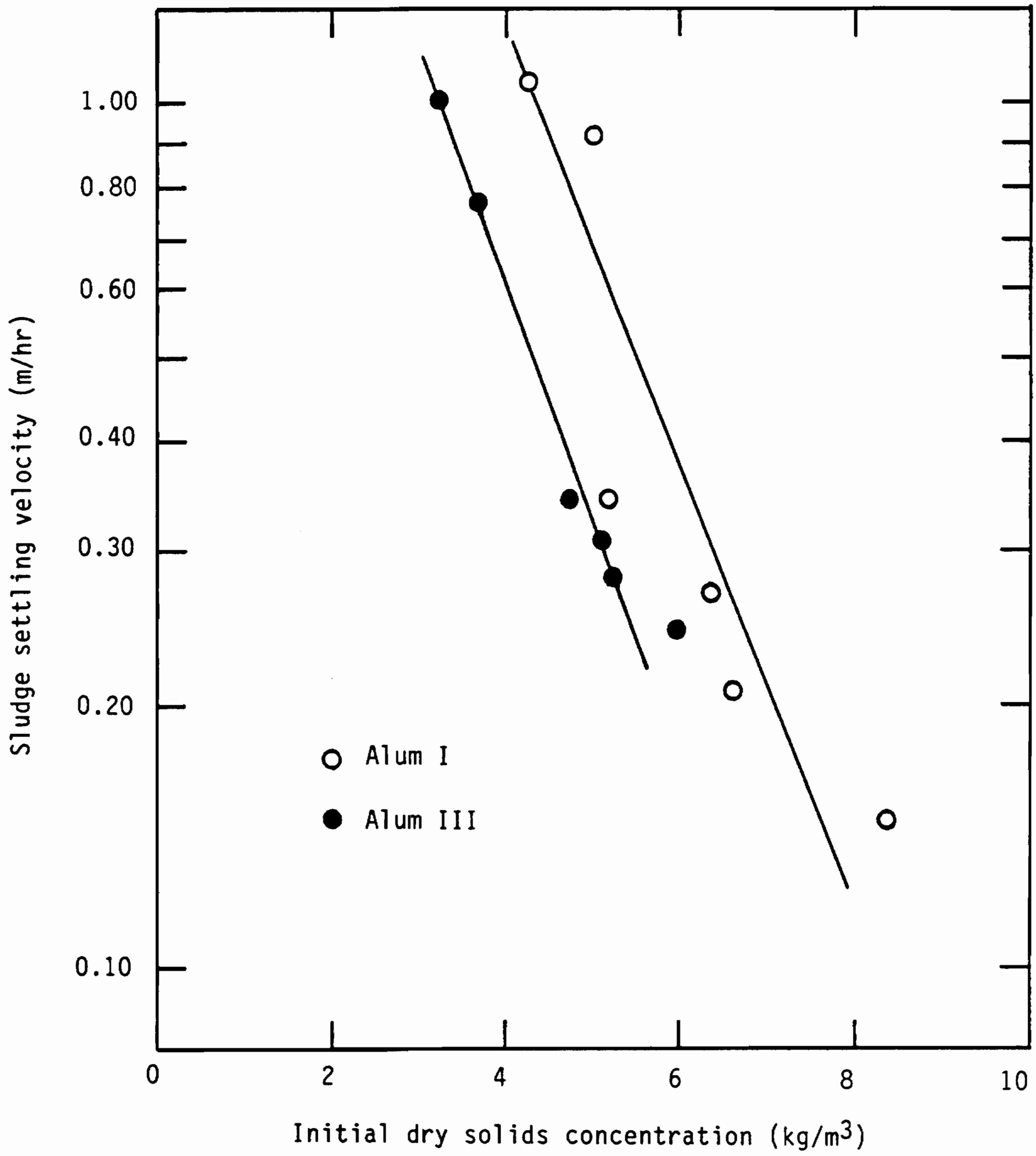

Figure 17. Relationship of sludge settling velocity (V_s) to initial dry mass solids concentration (C_o).

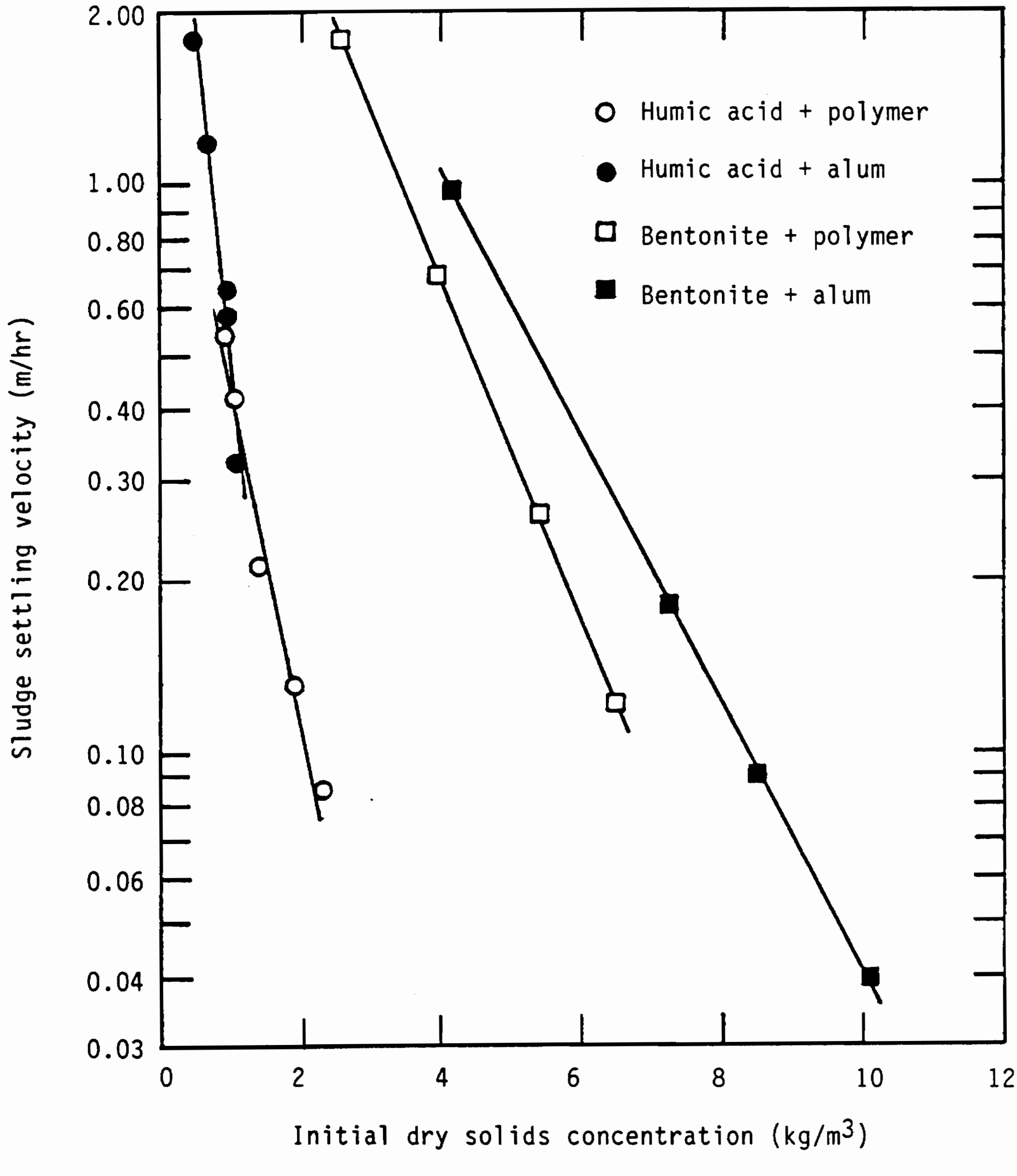

Figure 18. Relationship of sludge settling velocity (V_s) to initial dry mass solids concentration (C_o).

and III and the bentonite sludges showed nearly the same relationship of V_s to initial dry solids concentration. The settling of the humic acid sludges was most sensitive to changes in dry solids concentration, with the humic acid + alum sludge showing the greatest sensitivity.

It can also be seen from Figures 17 and 18 that, at a given dry solids concentration, the humic acid sludges settled at a considerably lower rate than did the other sludges. Hence, although these plots do not reveal a definite relationship between sludge gravity thickening rates and dry solids concentration, they do suggest that there may be basic differences between different types of sludges which affect their thickening behavior. In search of parameters that influence sludge thickening rates, the data in Table 5 will be discussed and the batch settling data will be analyzed further.

Interpretation of the parameters listed in Table 5 allows one to compare the sludges with respect to aggregate density, size, and relative water content. The two humic acid sludges had the highest aggregate water content (by virtue of their greater values of AVI). The water treatment plant alum sludges, alum I and III, had a much lower aggregate water content and heavier aggregate particles. The bentonite sludges had slightly less aggregate water content than the alum sludges. Their aggregates appear to be slightly lighter than those of the two alum sludges; this could be due to the more dense dry solids present in the alum sludges. Since V_o is considered to be an indication of particle size, the humic acid sludges contained the smallest aggregates, while the alum I sludge and the bentonite sludges had the largest apparent aggregate sizes.

In consideration of the above discussion, the source or content of a sludge can effect the characteristics of its aggregates. It appears that sludges with higher organic content, such as the humic acid sludges, have aggregates with greater water content than do inorganic sludges, which are represented in this study by the bentonite sludges. The alum I and III sludges are, in view of their sources, generally inorganic in content. However, based on the fact that both sludges are from treatment facilities which treat water containing some measure of organic matter, with some portion of this organic material being incorporated into the sludge during treatment, they are assumed to contain more organic material than the laboratory-prepared bentonite sludges. Research by Dulin (20) yielded the plots shown in Figure 19. These data support the idea that higher organic content in sludge results in more intra-aggregate water (i.e., higher AVI). Furthermore, from Table 5, the sludges with less organic material also contained larger aggregate particles.

In this study, two different coagulants were used: a low molecular weight cationic polymer and alum. The variation in sludge properties caused by the use of a different coagulant can be seen by evaluating data presented in Table 5. Both the humic acid and the bentonite sludges made with polymer exhibited lower floc densities and smaller size than the sludges formed by alum coagulation. In regard to the humic acid sludges, the polymer had a distinct positive effect when comparing the AVI values of the two sludges. The humic acid + alum sludge had an AVI nearly twice that of the humic acid + polymer, thereby indicating a greater water content.

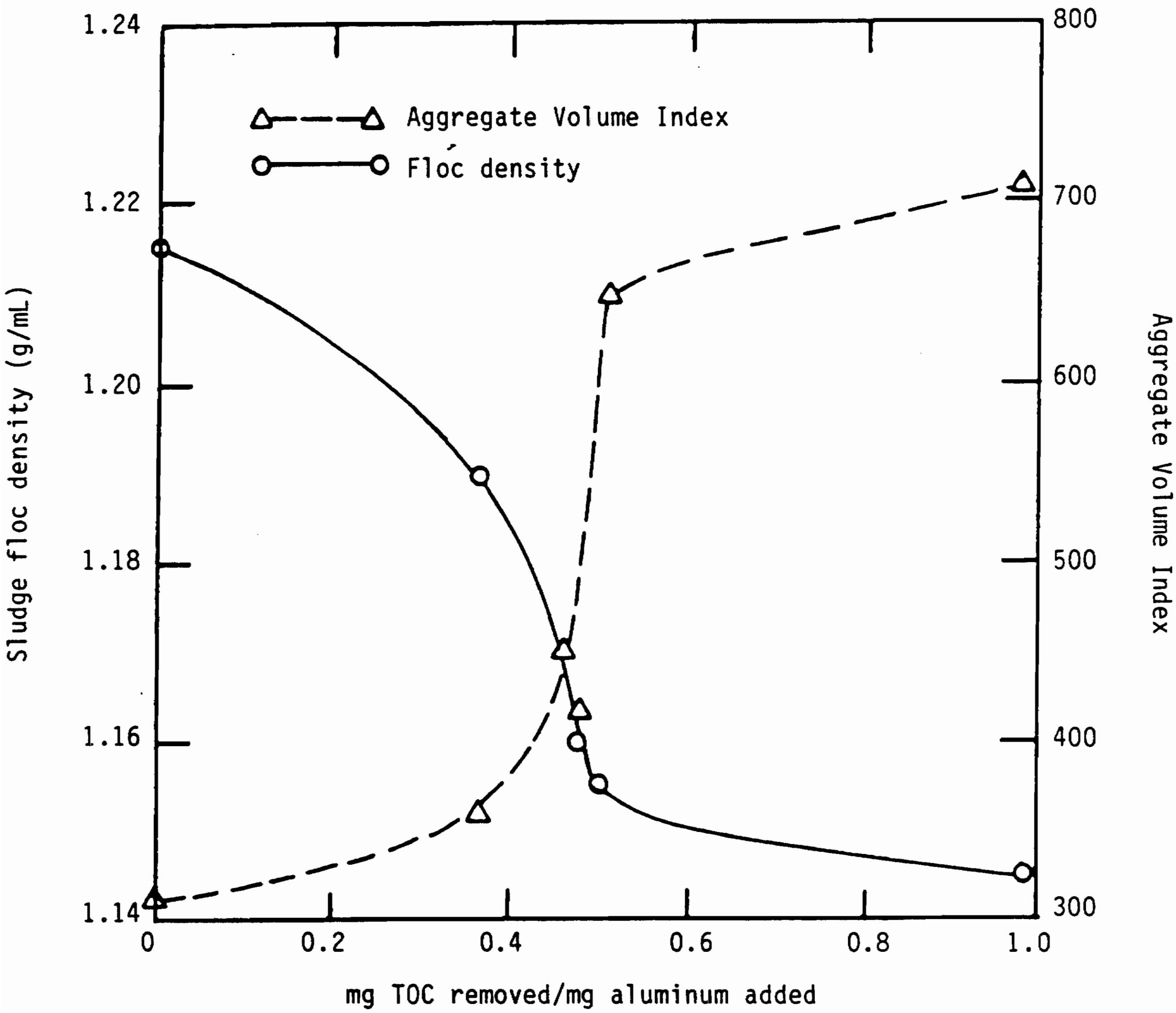

Figure 19. Effect of organics removed on floc density and Aggregate Volume Index of sludge (20).

It has been shown previously that comparison of dry solids concentration alone is not adequate for analyses of sludge gravity thickening rates. Therefore, in search of a parameter bearing a more direct influence on sludge settling velocity, plots were made of V_s versus the aggregate volume fraction or concentration, Φ_a. By referring to equations [5] and [11], one will notice the parameters that have a bearing on the value of Φ_a. Although a comparison of sludge settling velocity in terms of Φ_a will still be influenced by the value of C, it also serves to account for the density of the aggregate particles and dry sludge particles. By accounting for these densities, it was revealed that a significant relationship exists between sludge settling velocity and the aggregate volume fraction. Figures 20, 21, and 22 show a remarkable similarity in settling characteristics among all six sludges. The lines are all of nearly equal slope, indicating that sludges of unequal aggregate, floc, and dry particle densities and dry solids concentrations respond similarly to changes in the volumetric aggregate concentration (Φ_a). Other researchers (4,9) have also reported this phenomena.

Mathematical expressions for the lines in Figures 17 and 18 were generated of the form:

$$V_s = V_o e^{-k_1 C} \qquad [24]$$

using the values of V_o obtained previously in the plots of $V_s^{1/4.65}$ versus Φ_k. The values of k_1, which represent the slope of each line, are recorded in Table 6. The plot of k_1 versus AVI for each sludge

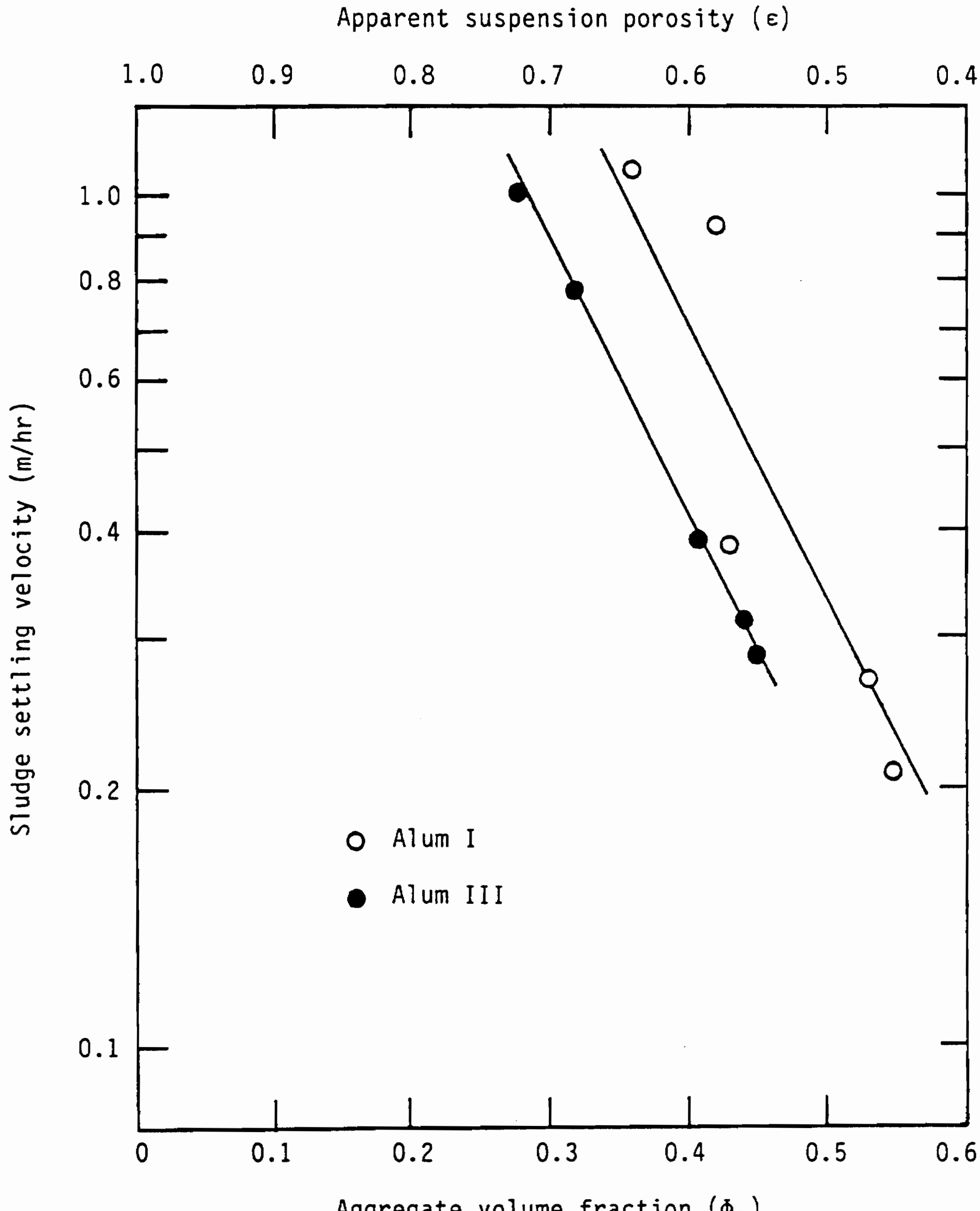

Figure 20. Relationship of sludge settling velocity to sludge aggregate volume fraction for the alum I and alum III sludges.

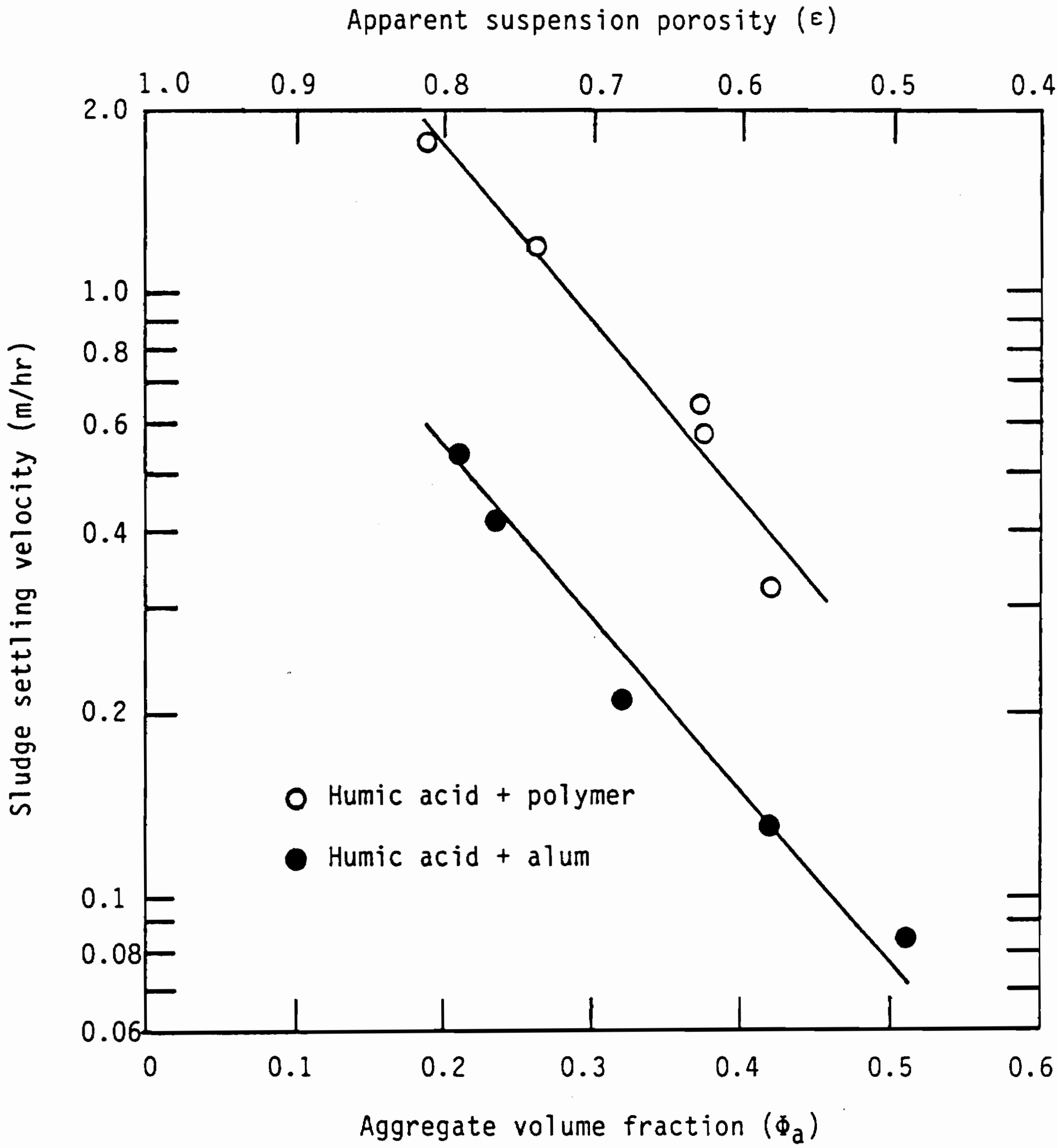

Figure 21. Relationship of sludge settling velocity to sludge aggregate volume fraction for the two humic acid sludges.

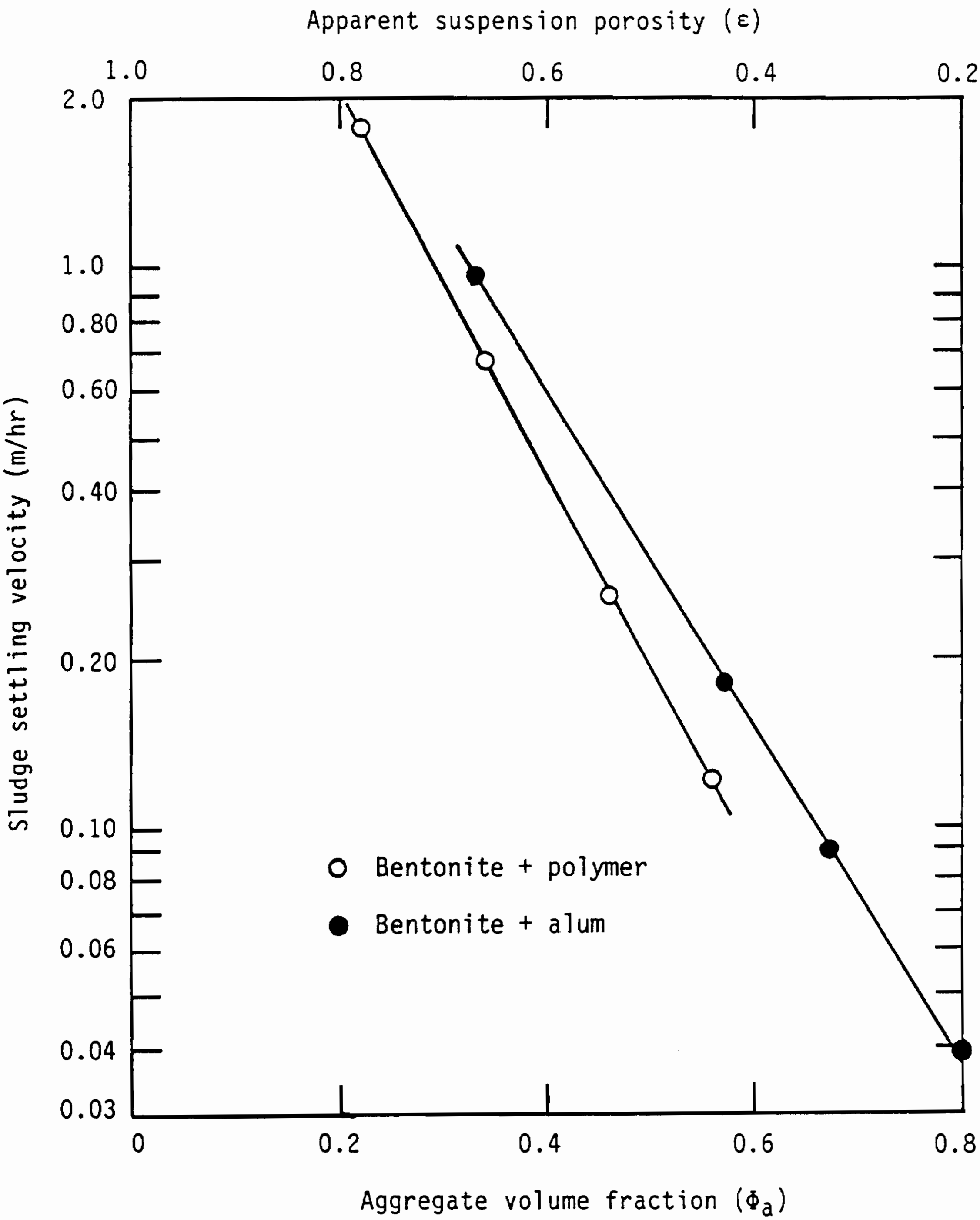

Figure 22. Relationship of sludge settling velocity to sludge aggregate volume fraction for the bentonite sludges.

Table 6. Values of thickening constants for Equations [24] and [25] as derived from Figures 17 and 18 and 20-22.

Sludge	Thickening constants	
	k_1 (m^3/kg)	k_2
Alum I	0.57	7.37
Alum III	0.60	7.37
Humic acid + polymer	1.45	6.59
Humic acid + alum	2.09	7.37
Bentonite + polymer	0.67	7.74
Bentonite + alum	0.55	7.02

(Figure 23) highlights the influence aggregate water content had on sludge settling velocity.

Similarly, equations were derived for the lines in Figures 20, 21, and 22 of the form:

$$V_s = V_o e^{-k_2 \Phi_a} \qquad [25]$$

The values of k_2 are also presented in Table 6. It is noted that the k_2 values all equal approximately 7.0. Although no explanation has been developed for this, other investigators have also found the thickening constant k_2 to be near 7.0 (4, 9).

In each case in Figures 20, 21, and 22, the sludge with the highest V_o value is on top in the plot. That is, at each aggregate volume fraction value (or suspension porosity, which will be discussed later), the sludge with the larger particles (as estimated by a larger V_o value) settled faster, a result predicted by Stoke's Law. Note also that, in the case of the two bentonite sludges, one might predict that the sludges contain similar size aggregates given the fact that their V_o values are almost identical.

The significance of sludge porosity in gravity thickening will now be discussed. The aggregate volume fraction, established as an influential parameter on sludge settling velocity, is related to sludge porosity. According to Javaheri and Dick (6), the porosity of a sludge (ε) is comprised of the interstices between the aggregates. In mathematical terms:

$$\varepsilon = 1 - \Phi_a \qquad [26]$$

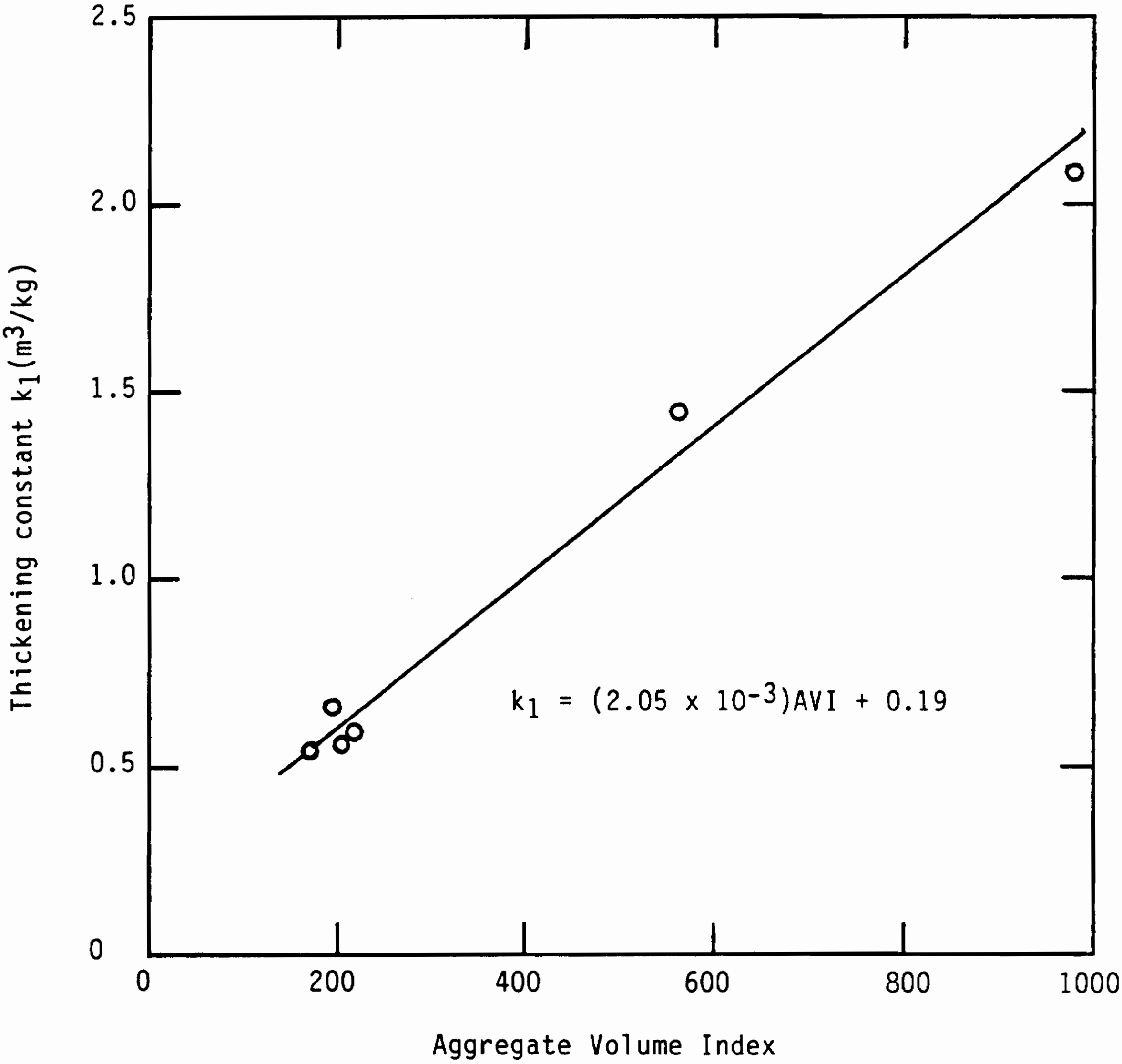

Figure 23. Observed experimental data relating thickening constant k_1 to AVI.

According to equation [26], Φ_a and ε are inversely related, i.e., in a sludge of high porosity the volume occupied by aggregates will be relatively small. The data in Figures 20, 21, and 22 indicate that when porosity was high, settling velocity was also high. Likewise, lower velocities were associated with lower suspension porosities. This may be explained by the resistance to fluid flow caused by drag forces on the surface of the aggregates. Higher porosities and thus, lower Φ_a, translate to lower aggregate specific surface area. With less surface area to create drag forces, the sludge liquid is displaced more easily around the aggregates, leading to faster sludge settling. An example is found in comparing the porosities and settling velocities of the humic acid + polymer and the bentonite + polymer sludges at a dry solids concentration of approximately 0.25%. Using Equation [26]:

humic acid + polymer: $\varepsilon = 1 - 0.51 = 0.49$; $V_s = 0.085$ m/hr

bentonite + polymer: $\varepsilon = 1 - 0.22 = 0.78$; $V_s = 1.80$ m/hr

The lower thickening rate of the humic acid sludge at 0.25 % dry solids concentration could be explained by its substantially lower porosity.

By analyzing the plots of $V_s^{1/4.65}$ versus Φ_k for a sludge, certain comments can be made about aggregate behavior during settling. According to Javaheri and Dick (6), a linear plot indicates unchanging V_o and AVI values. In their study of waste activated sludge settling, they discovered the plot of ($V_s^{1/4.65}$) versus Φ_k to be curved, indicating a significant change in aggregate size and a release in water content during thickening. This behavior was evident in all of the gravity thickened sludges in this study. Figure 24 demonstrates this for the alum I, alum III, and the humic acid sludges. Values of AVI and V_o were deter-

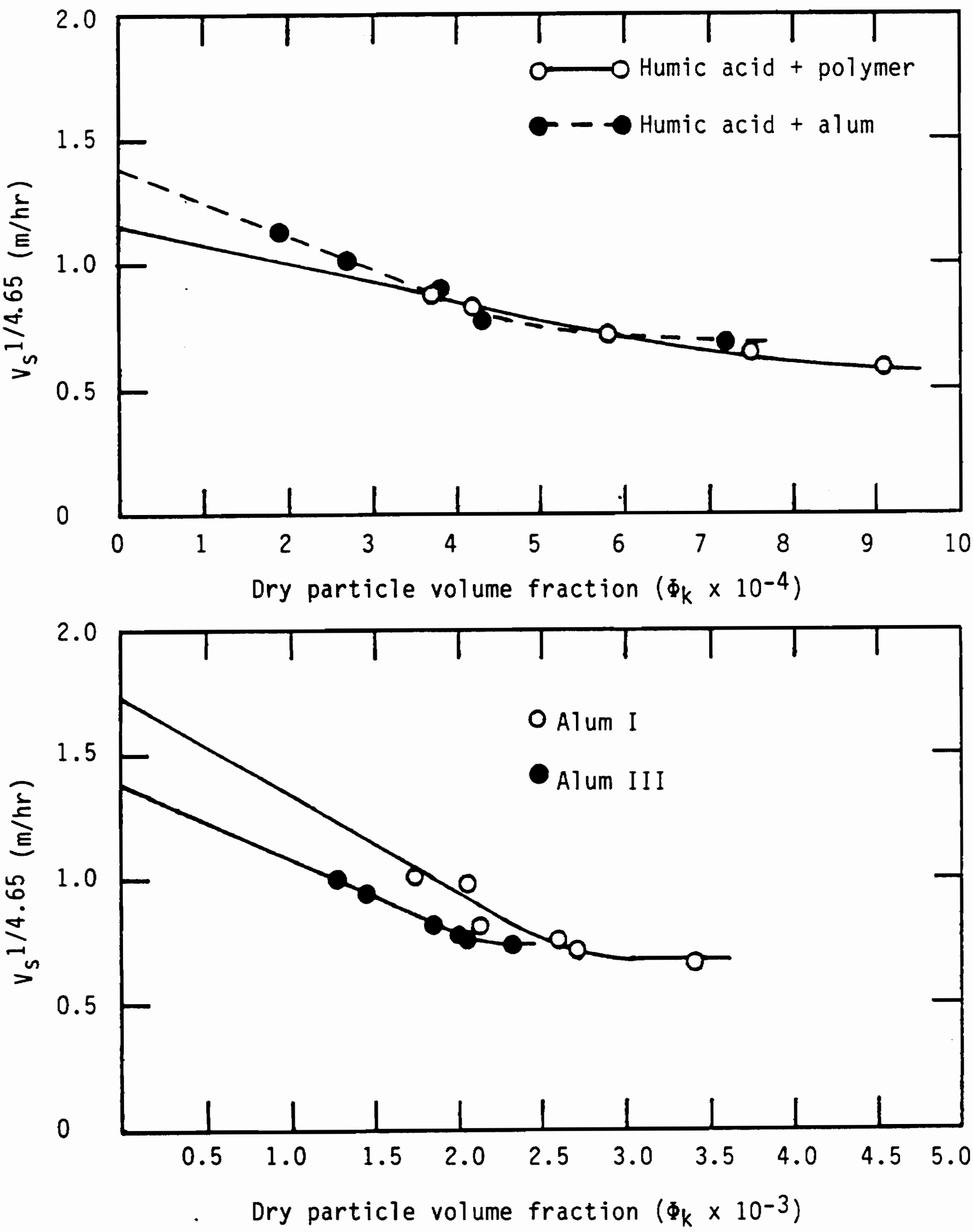

Figure 24. Relationship of sludge settling velocity to volume fraction of dry solids for the alum and humic acid sludges.

mined for these four sludges at various Φ_k by taking the tangent to the curved lines in Figure 24. Figures 25 and 26, which show the AVI and V_o increasing sharply at a certain range of dry solids concentration particular to the sludge, supports the idea that aggregate particles breakup during a compression phase of thickening. Therefore, in view of these data and by applying ideas from the literature (6), one may conclude that the aggregates in the thickened sludge contained less water and were of smaller diameter than those in the unthickened sludge. This result is typical of a sludge being compressed by the weight of overlying solids and supernatant.

The AVI and dry solids concentration data in Figure 25 was translated into a normalized plot of AVI (i.e., AVI/AVI_o, where AVI_o is the AVI at the lower solids concentrations where the plot of $V_s^{1/4.65}$ versus Φ_k is linear) versus the aggregate volume fraction for each sludge (Figure 27). The data in this plot reasonably overlap indicating that these sludges move into the compression phase of thickening at similar values of aggregate volume fraction. This idea correlates well with the conclusions drawn previously from data presented in Figure 2.

The maximum solids concentration achieved during gravity thickening of the six sludges was plotted versus both Aggregate Volume Index and floc density (Figure 28). It was found that this thickened solids concentration generally increased with decreasing values of AVI and increasing ρ_f. This relationship with AVI could be expected in view of the inverse relationship of V_s with the AVI. A higher Aggregate Volume Index indicates a greater fraction of the volume of the aggregate particle itself is made up of water. In order to achieve optimum dry

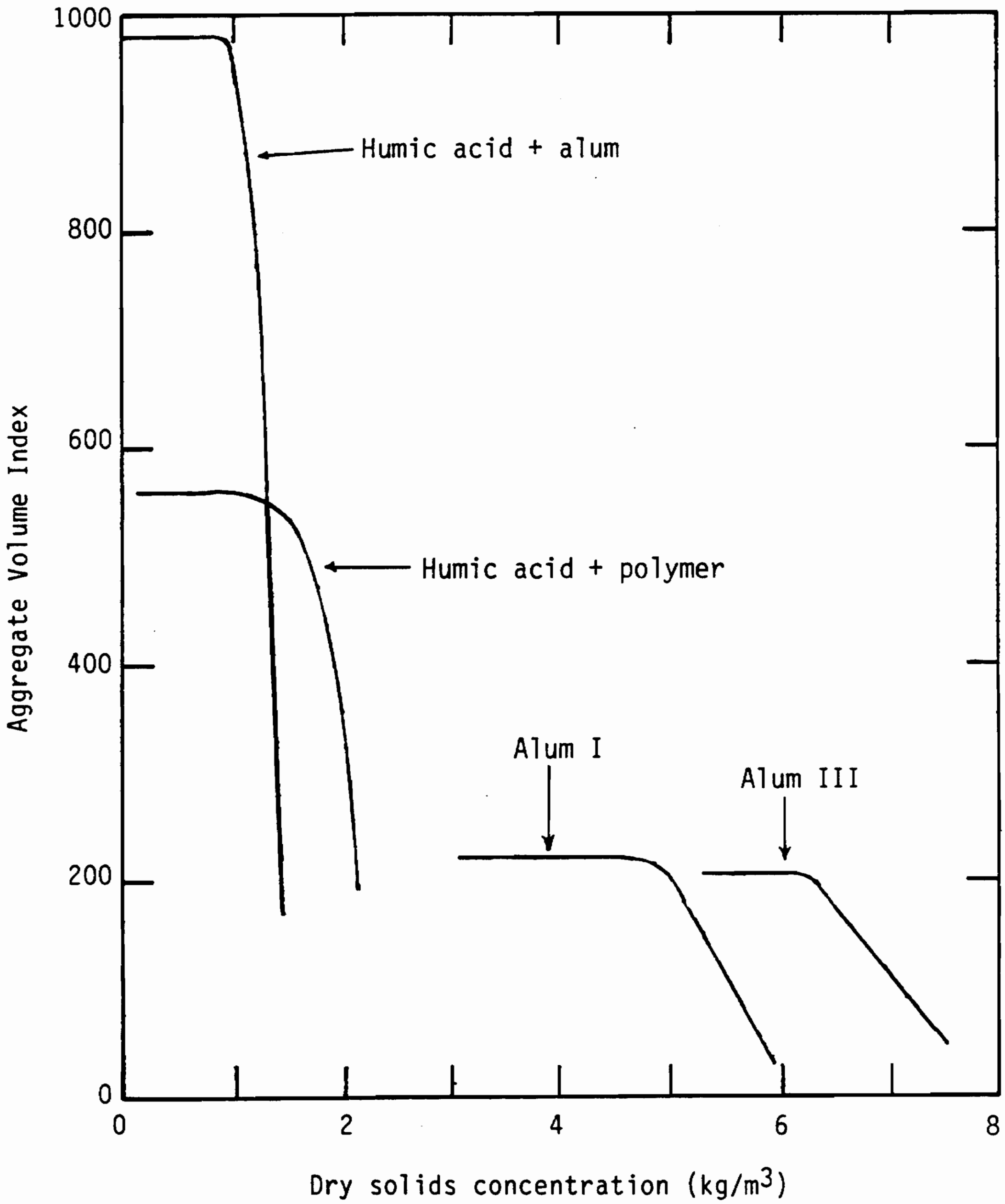

Figure 25. Effect of increasing dry solids concentration on the AVI.

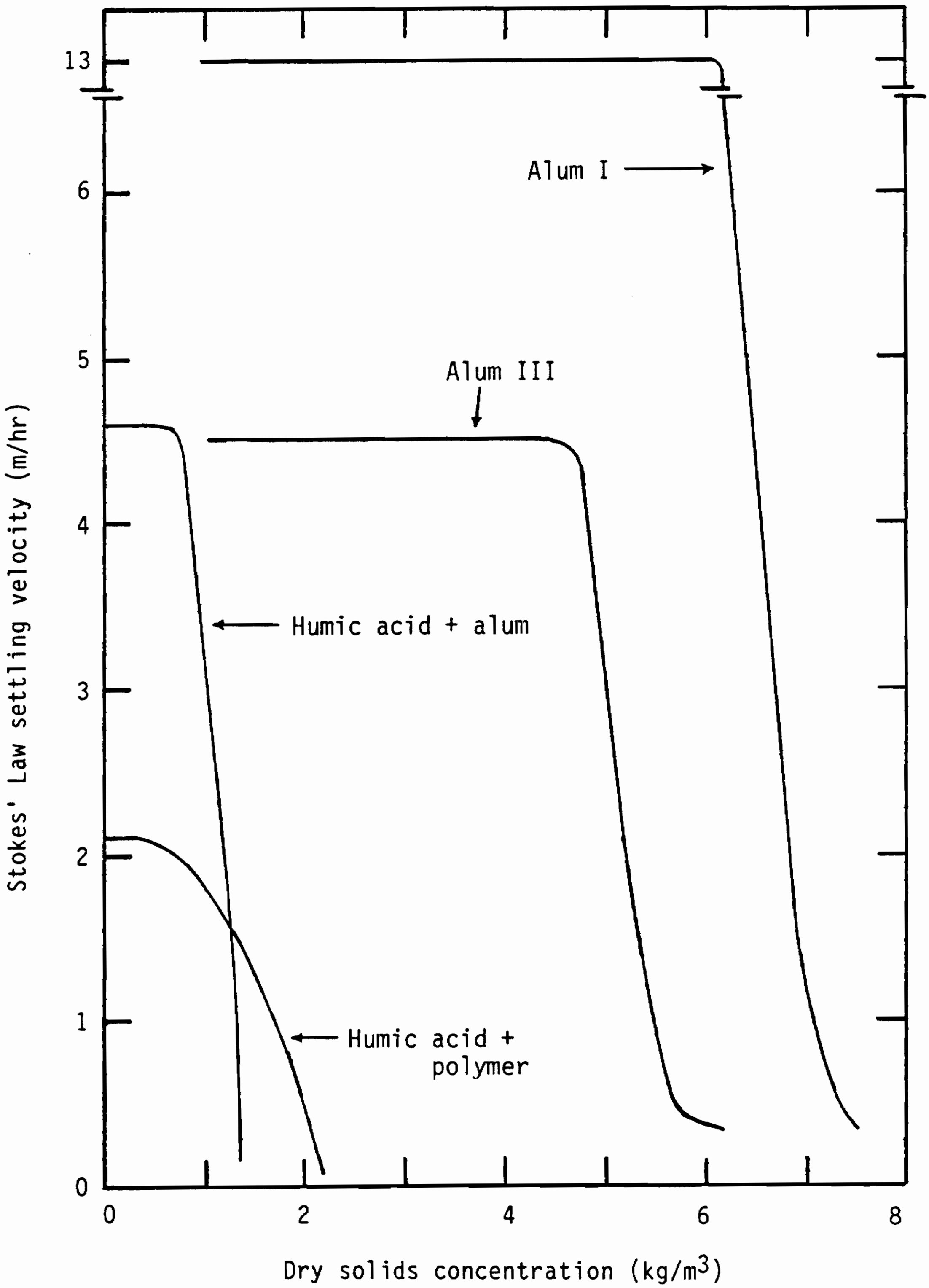

Figure 26. Effect of increasing dry solids concentration on the Stokes' Law settling velocity.

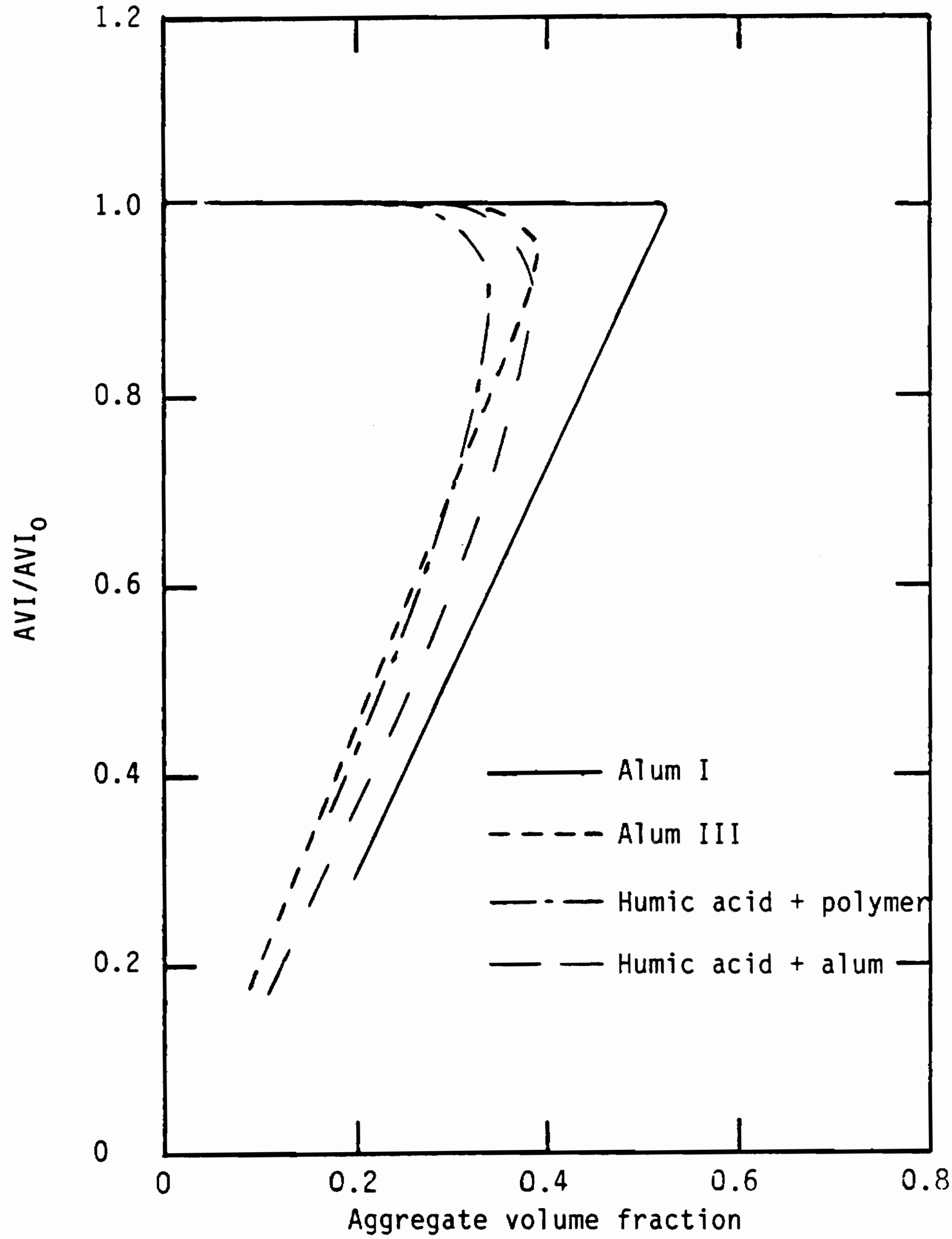

Figure 27. Normalized Aggregate Volume Index versus Aggregate volume fraction.

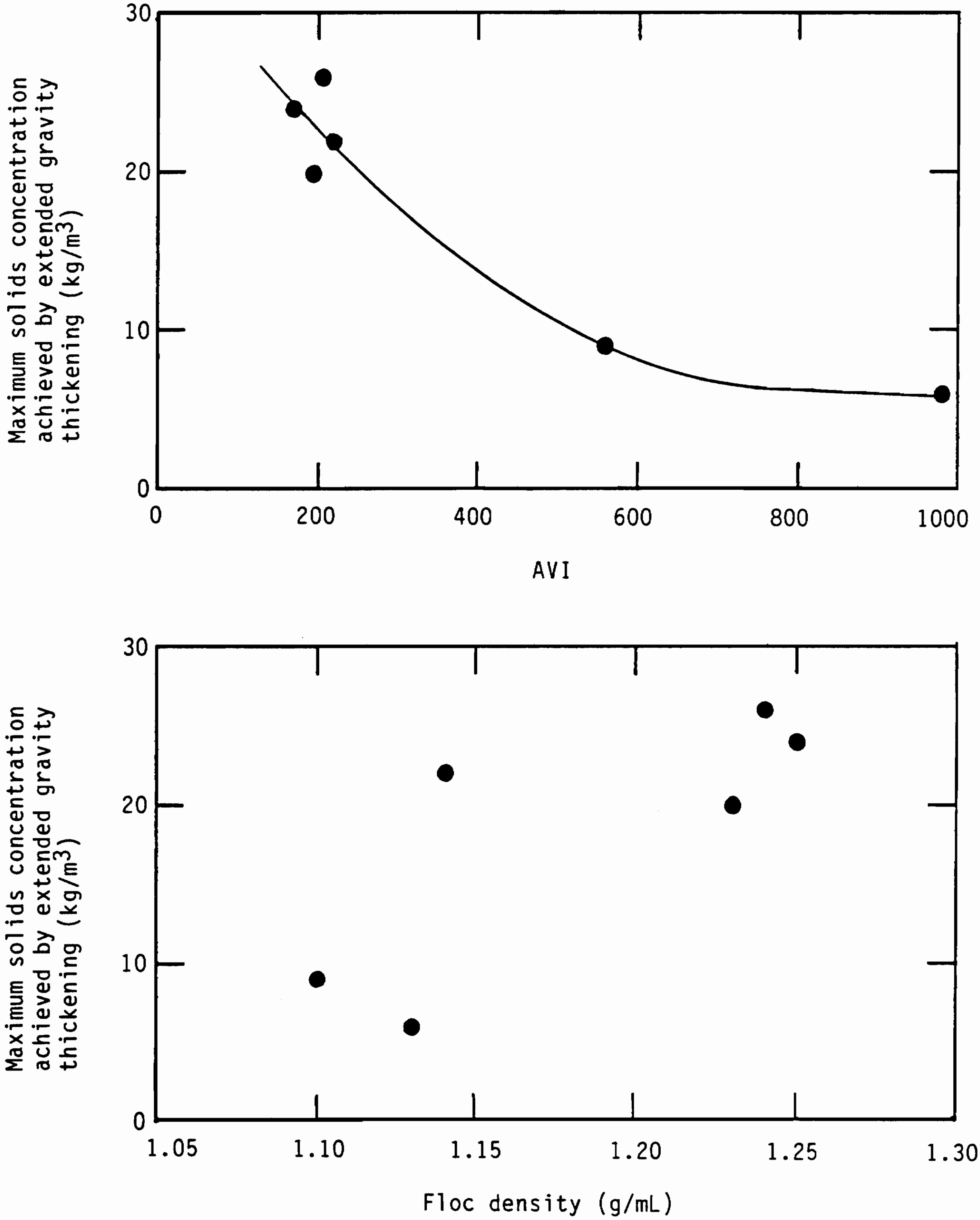

Figure 28. Variation of maximum solids concentration of gravity thickened sludges with their Aggregate Volume Index and undeformed floc density.

solids concentrations by extended gravity thickening, this intra-aggregate water must be removed. However, if there is an excessive amount of intra-aggregate water present in a sludge, the weight of the overlying solids and supernatant may not be great enough to force a sufficient amount of this water out of the aggregates.

It can be concluded from Figure 28 that the floc particle density has a relationship to the dry solids concentration achieved by gravity thickening generally opposite that of the AVI. This might well be expected when one considers the fate of aggregates during gravity thickening of sludges to high solids concentrations, as explained in literature cited previously (1,5,6). Overlying solids and water cause the gradual collapse and split of sludge aggregates into their constituent flocs. As this process continues, an increasingly greater volume fraction of the sludge will consist of floc particles. This leads to greater sludge dry solids concentrations because the floc particle density is higher than the aggregate density. Therefore, sludges with higher floc densities will tend to settle to greater solids concentrations under extended gravity thickening.

For each sludge, sludge bulk density was plotted versus dry solids concentration through various types of dewatering: from gravity thickening to mechanical dewatering. These data are presented in Figures 29 through 35. A discussion of these figures as they relate to mechanical dewatering will be provided in the next section. As far as gravity thickening is concerned, the plots illustrate the potential for water release from sludge aggregates during the compression phase of

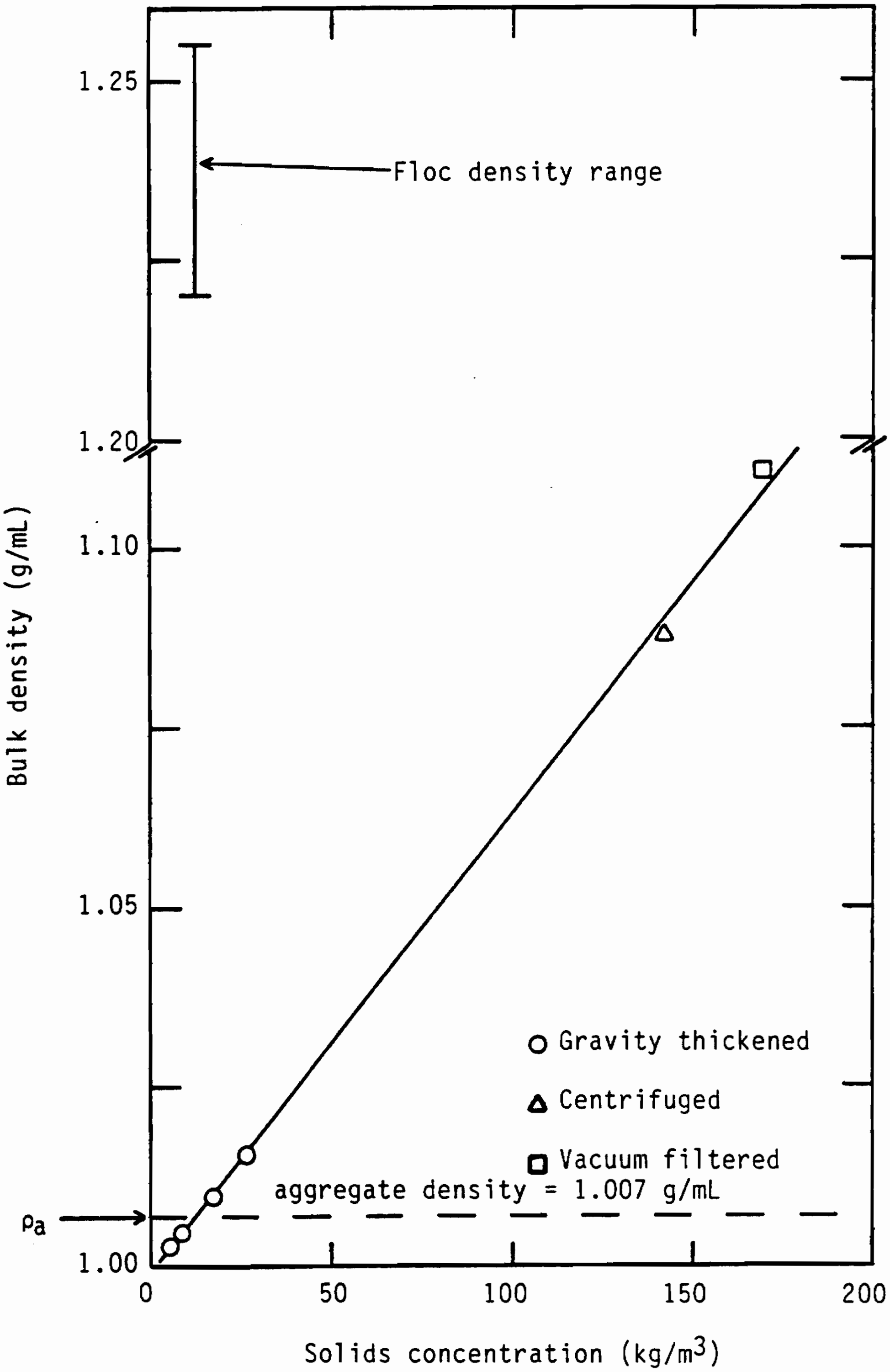

Figure 29. Relationship between bulk density and solids concentration during the dewatering of alum I sludge.

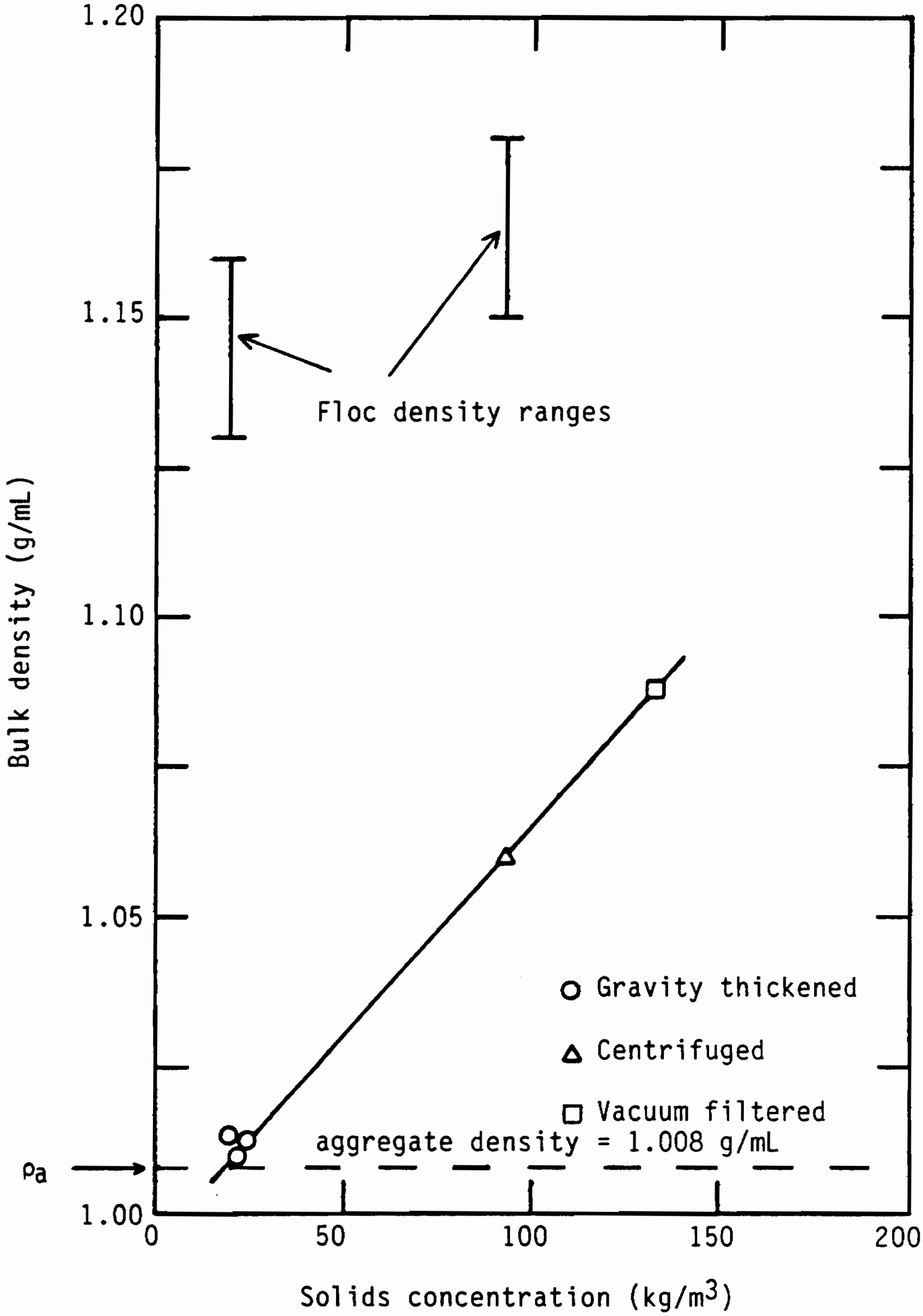

Figure 30. Relationship between bulk density and solids concentration during the dewatering of alum III sludge.

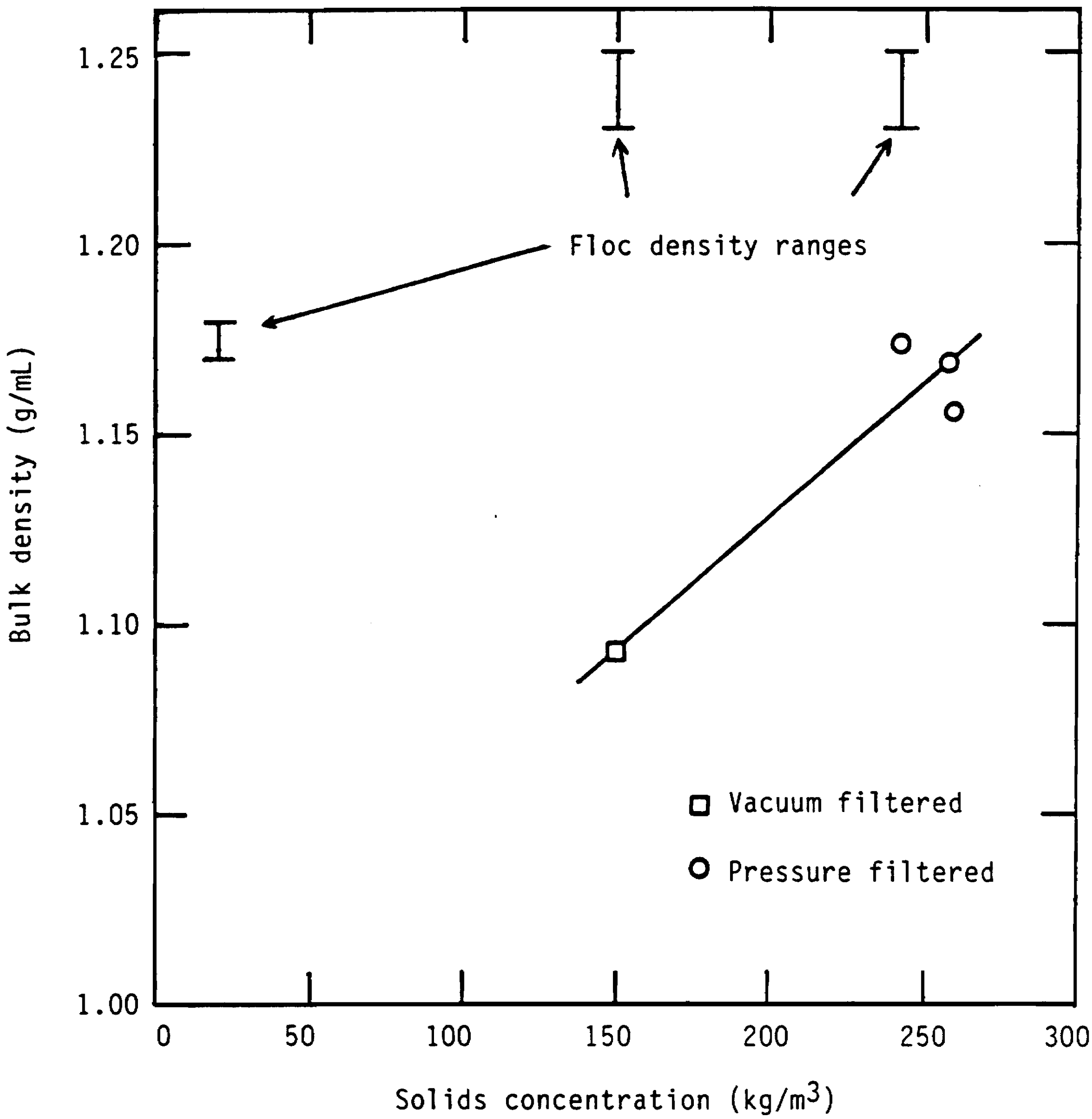

Figure 31. Relationship between bulk density and solids concentration during the dewatering of alum III + polymer sludge.

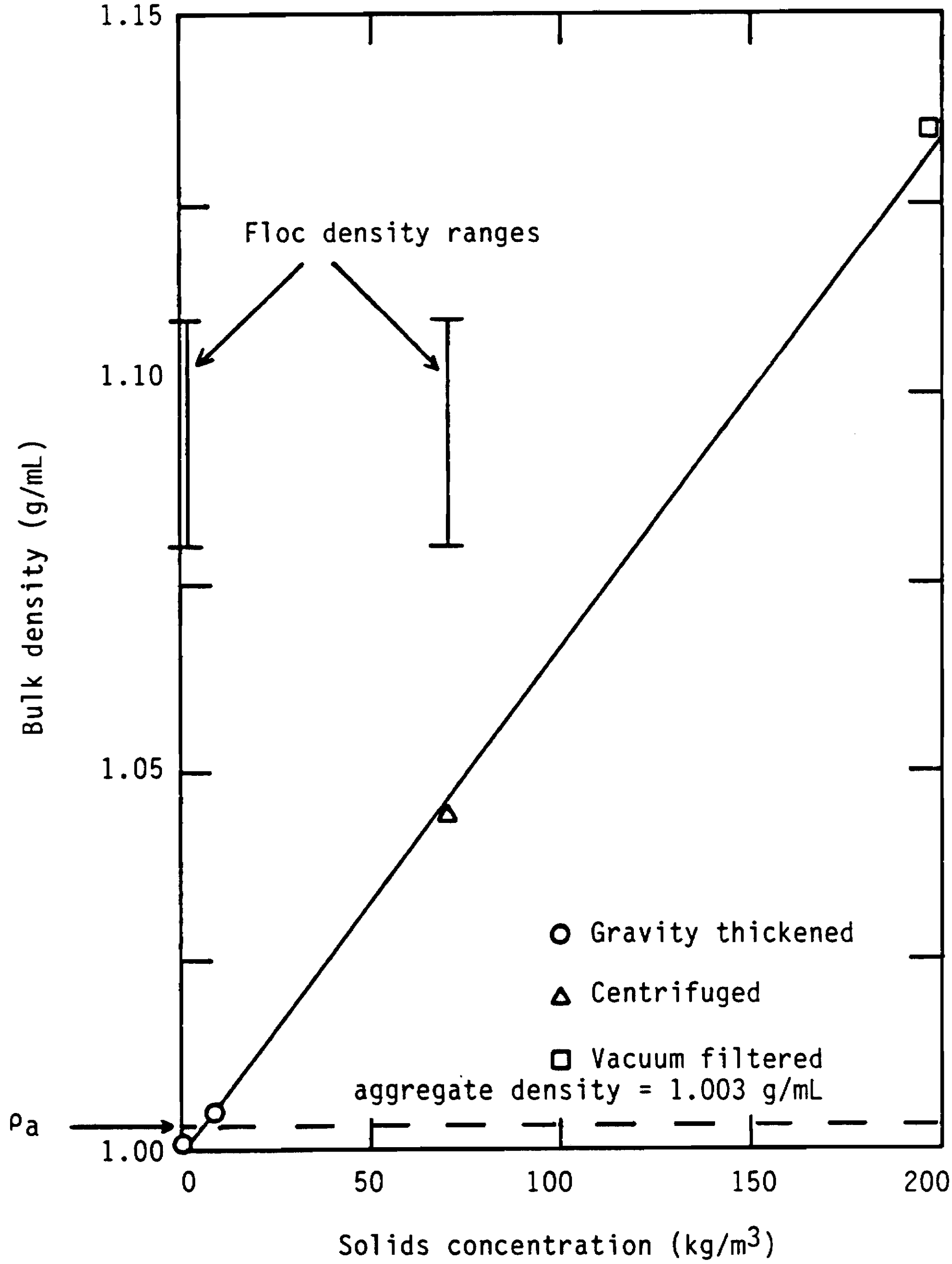

Figure 32. Relationship between bulk density and solids concentration during the dewatering of humic acid + polymer sludge.

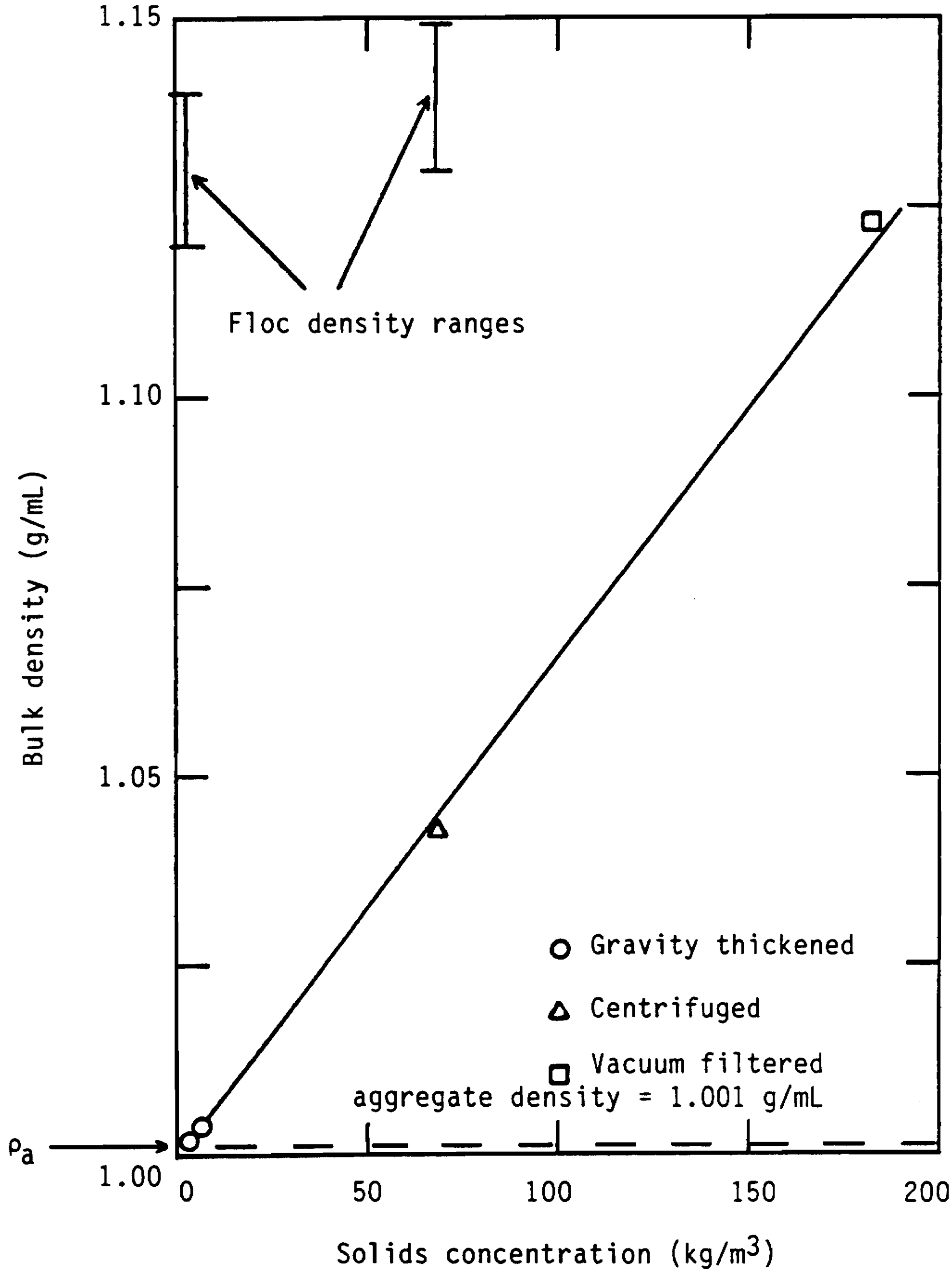

Figure 33. Relationship between bulk density and solids concentration during the dewatering of humic acid + alum sludge.

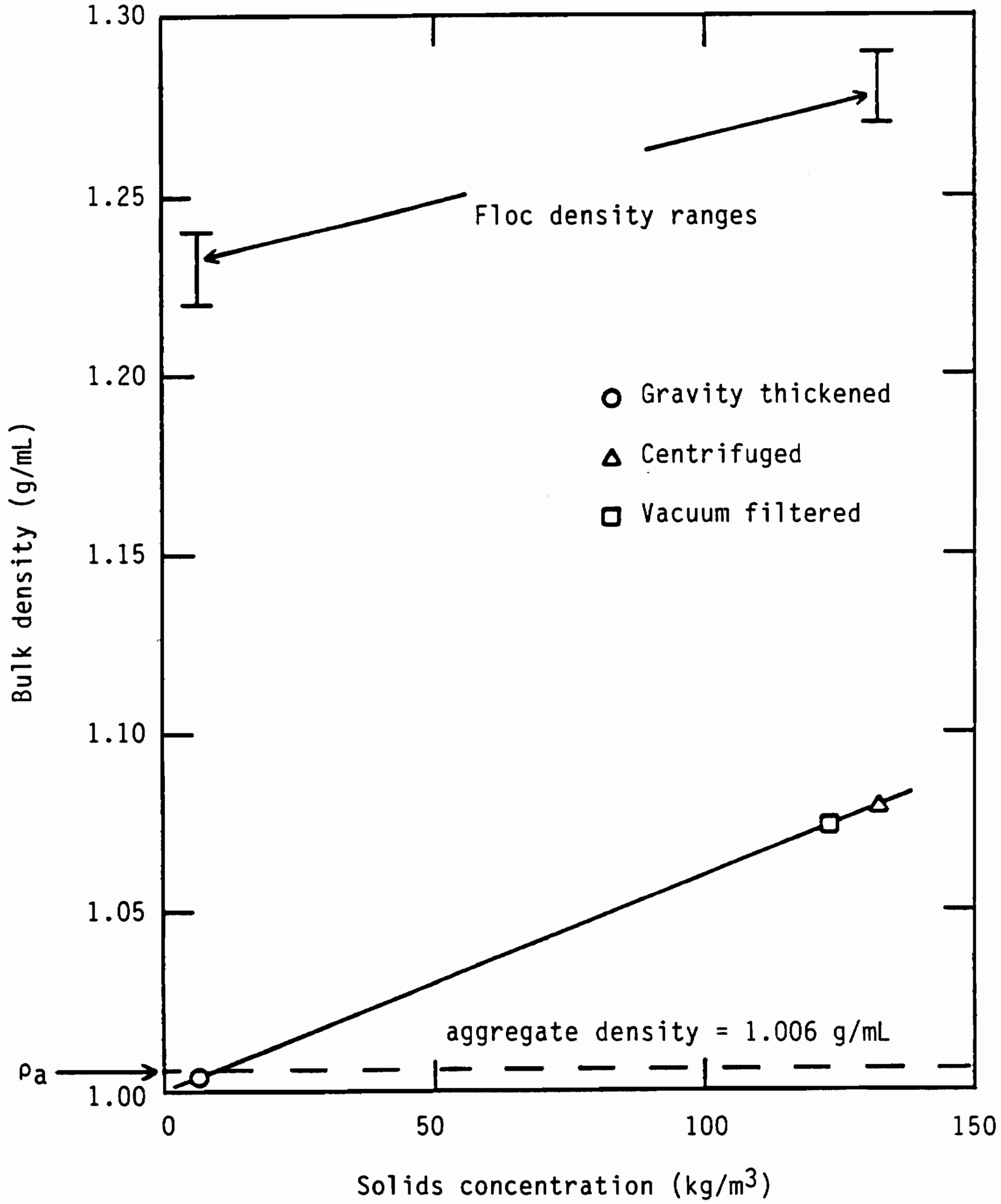

Figure 34. Relationship between bulk density and solids concentration during the dewatering of bentonite + polymer sludge.

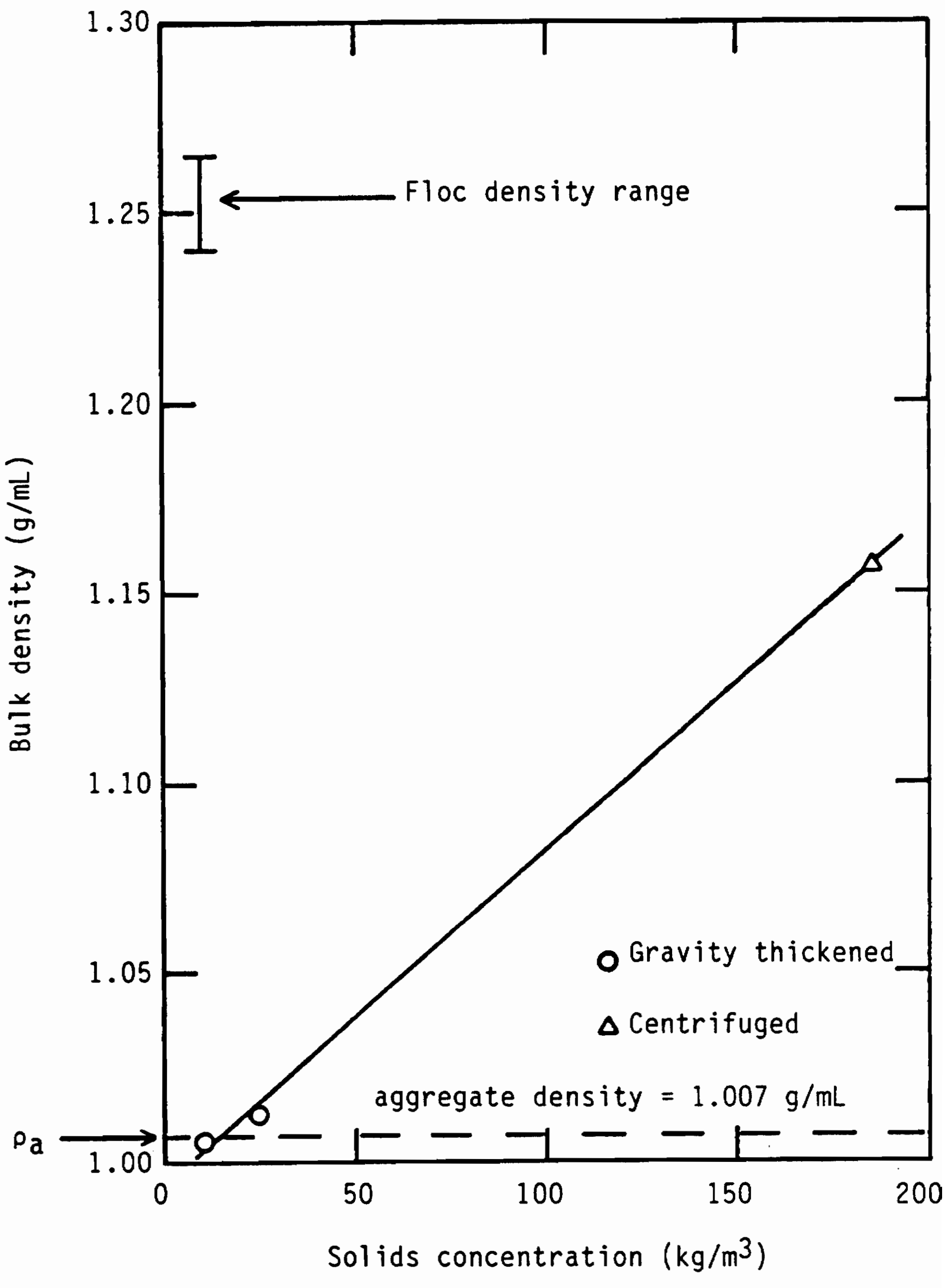

Figure 35. Relationship between bulk density and solids concentration during the dewatering of bentonite + alum sludge.

sludge settling (i.e., the third phase of gravity thickening as defined by Michaels and Bolger (1)). This is indicated by the instances where thickened bulk densities exceeded calculated aggregate densities.

Mechanical Dewatering. Pressure filtration, centrifugation, and vacuum filtration were used to study sludge behavior during mechanical dewatering. First, the relationships between the extent of dewatering and certain sludge properties were analyzed. Secondly, parameters were calculated to evaluate their influence on the extent of dewatering.

An analysis of the plots of bulk density versus dry solids concentration in Figures 29 through 35 yields some noteworthy observations:

1. In every case, the bulk densities achieved by all three of the mechanical dewatering methods exceeded the aggregate density. This signified that the aggregate structure had been altered. Furthermore, in three cases (alum III + polymer and the two humic acid sludges), the bulk density obtained using pressure filtration and vacuum filtration, respectively, surpassed the measured floc density of the undewatered sludge. One interpretation of these results is that certain mechanical dewatering methods will actually fracture the floc structure of some types of sludges. According to the following mass balance (given previously in Chapter II):

$$\rho_b = \Phi_f \rho_f + (1 - \Phi_f) \rho_w \qquad [6]$$

bulk density is the sum of the proportional fractions of floc density and the density of the interfloc water. Thus, bulk density should be less than or equal to the actual floc density. If the original floc structure was deformed, causing a release of intrafloc water, then the bulk density could exceed the measured, undeformed floc density.

2. Further inspection of Figures 29 through 35 reveals that, for all samples except the humic acid + polymer sludge, the floc density appears to have increased during mechanical dewatering. (The floc density of the alum I and the bentonite + alum sludges was not measured following mechanical dewatering.) Two explanations are offered for these results. First, as was the interpretation offered in item 1 above, the floc particles could have been deformed and collapsed to a certain extent. If this was the case, then their density would increase due to the release of intrafloc water. A second explanation is related to the technique used for measuring the density of flocs. In developing the floc density test, it was assumed that the centrifugal force applied to the sludge samples during centrifugation was sufficient to completely break the aggregate particles into their constituent flocs. This assumption may or may not have been accurate in light of the higher apparent floc densities measured following mechanical dewatering. It is possible that the samples used in the floc density tests after mechanical dewatering were comprised of the actual undeformed floc particles and the samples analyzed prior to dewatering consisted of mainly deformed aggregates with perhaps some individual floc particles. On the other hand, waste activated sludges were analyzed in continuous gradients at increasing centrifugal force and their apparent floc densities did not vary with centrifugal force, as was pointed out in the first part of this chapter.

3. For each sludge, the maximum dewatered bulk density measurement remained below the calculated value for the dry particle density. This reinforces the concept that even higher pressure dewatering cannot remove all traces of moisture from sludges. The water remaining might be classified as representing, to a significant extent, the chemically bound water fraction that Vesilind (2) and Moller (3) referred to in their respective models of sludge water distribution.

4. The effect of polymer conditioning on the alum III sludge can be seen by comparing Figures 30 and 31. Polymer conditioning increased the apparent floc density both before and during mechanical dewatering. Another effect of the polymer is that it provided a higher solids concentration from vacuum filtration.

The remainder of this section will deal with comparing volume fraction ratios and floc density to dry solids concentration achieved in mechanical dewatering. The volume fractions and floc densities used in these comparisons were calculated for each sludge based on its dry solids concentration just prior to mechanical dewatering.

Figures 36 and 37 show the ratio of undeformed aggregate volume fraction to undeformed floc volume fraction versus the dry solids concentration achieved by centrifugation and vacuum filtration, respectively. Analysis of these data reveal that the volume fraction ratio had no effect on the solids concentration from mechanical dewatering. The ratio Φ_a/Φ_f was first introduced by Michaels and Bolger (1) to give a relative indication of the volume of water present when comparing the aggregate structure to the floc structure. Therefore, the lack of fit between final dewatered solids concentration and the aggregate:floc

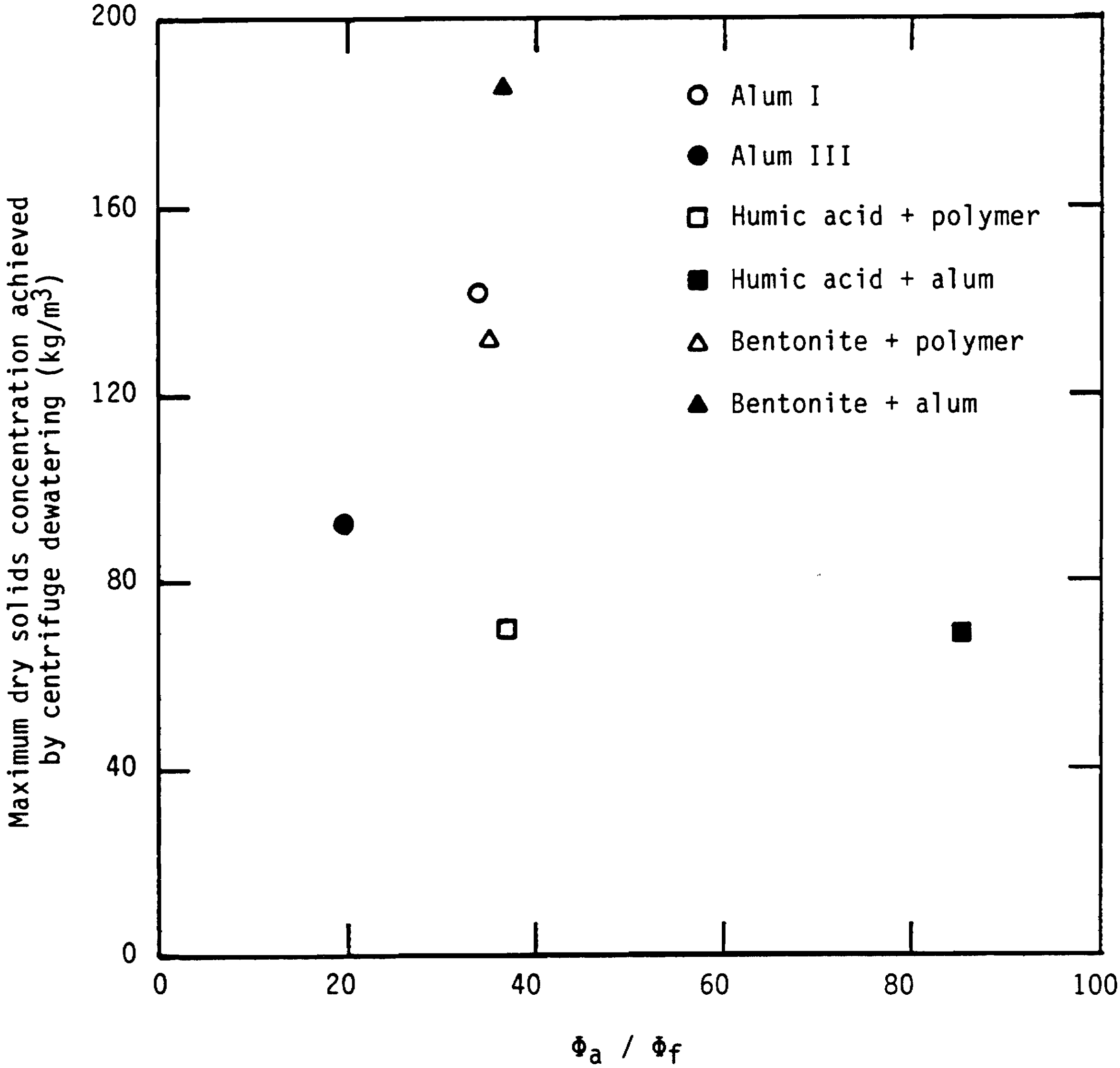

Figure 36. The maximum solids concentration of centrifuged sludges versus the undeformed aggregate:floc volume fraction ratio.

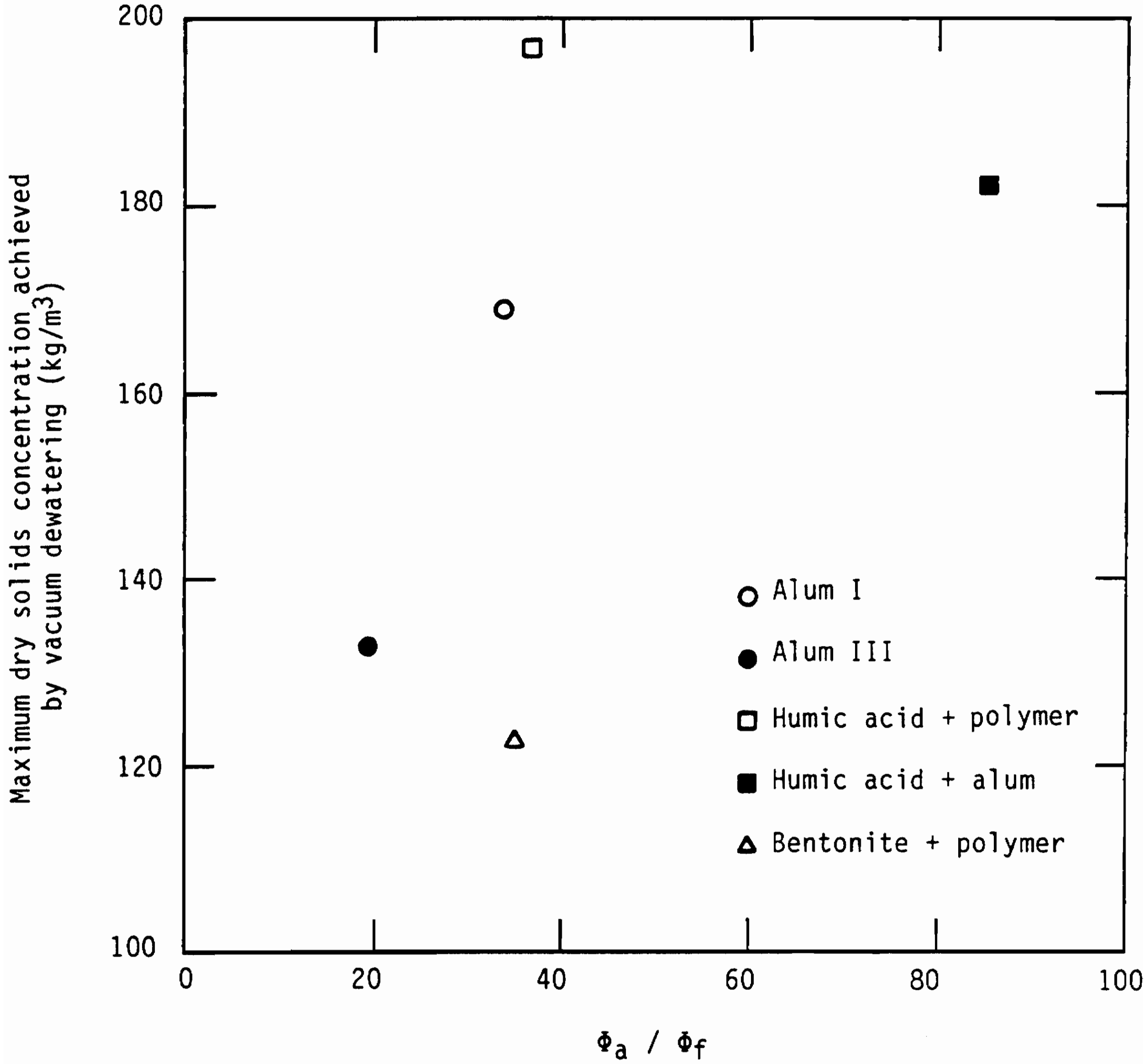

Figure 37. The maximum solids concentration of vacuum filtered sludges versus the undeformed aggregate:floc volume fraction ratio.

volume ratio is not surprising since this ratio is related to water that is theoretically outside the floc structure and, thus, available for removal during gravity thickening and mechanical dewatering.

The undewatered floc density was plotted versus the dry solids concentration achieved by centrifugation and vacuum filtration, as shown in Figures 38 and 39, respectively. Figure 38 reveals a rather well-defined relationship between these two parameters, with higher floc densities resulting in more extensive dewatering by centrifugation. However, the data in Figure 39 indicate no impact of floc density on the extent of vacuum filtration dewatering. One explanation for these results may be the subjective and variable nature of the vacuum dewatering test in comparison to the centrifugal test. All the samples dewatered by centrifugation were done so simultaneously under identical conditions. On the other hand, a significant amount of variability can enter into vacuum filtration test regarding when the test is stopped. At some point, the judgement must be made on when the cake has failed; the length of the filtration run is based on this judgement. Furthermore, the amount of vacuum filtered sludge taken for the dewatered solids sample will impact on the final cake solids concentration. There is a significant variation in cake solids concentration on the surface of a Buchner funnel, with much poorer degrees of dewatering occuring along the outer edges. This makes it more difficult to be very reproducible from one test to the next.

The dry solids concentrations attained by centrifugation and vacuum filtration of each sludge was plotted versus the ratio of the undeformed floc volume fraction to the volume fraction of dry solids, Φ_f/Φ_k

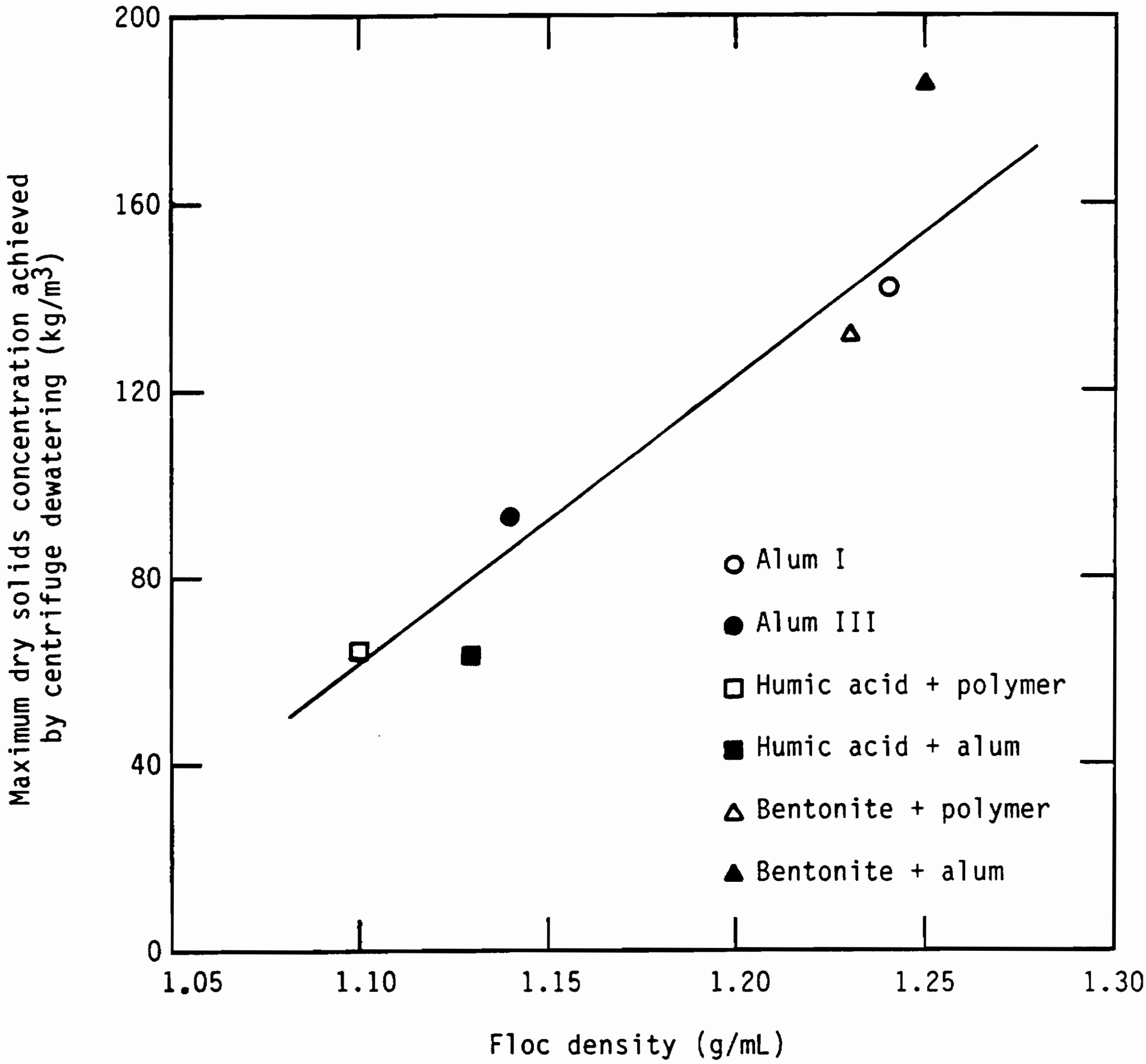

Figure 38. The maximum solids concentration of centrifuged sludges versus the undeformed floc density.

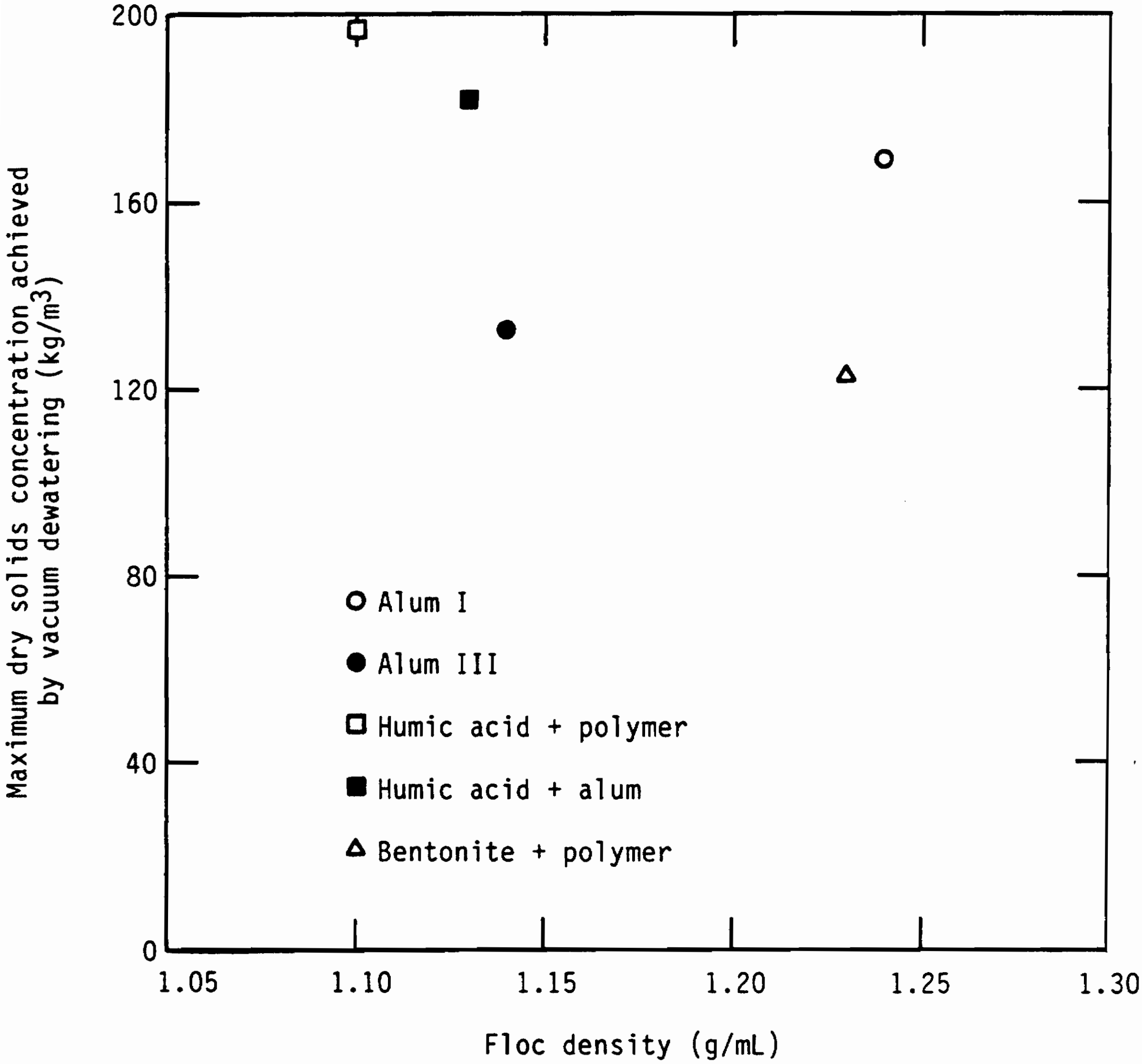

Figure 39. The maximum solids concentration of vacuum filtered sludges versus the undeformed floc density.

(Figures 40 and 41). The ratio Φ_f/Φ_k, which is similar to the AVI, helps to quantify the volume of intrafloc water and may be termed the floc volume index. The intrafloc water is the most difficult sludge water to remove (except, of course, for the water bound to the primary particles). Therefore, the inverse relationship between C and Φ_f/Φ_k that is indicated by the data in Figure 40 should be expected. The scattered vacuum filtration data shown in Figure 41 contradicts that of Figure 40. The explanation offered previously for the vacuum filtration data in Figure 39 could be repeated at this point to explain this difference in results.

Figures 42 and 43 contain plots of the ratio of undeformed aggregate volume fraction to undeformed floc volume fraction (Φ_a/Φ_f) and undeformed floc volume fraction to undeformed dry particle volume fraction (Φ_f/Φ_k), respectively, versus the ratio of dry solids concentration achieved by centrifugation to maximum possible dry solids concentration. The maximum solids concentration achievable by normal dewatering methods, C_{max}, is calculated using the following formula:

$$C_{max} = \frac{\rho_k}{\rho_f} \frac{(\rho_f - 1)}{(\rho_k - 1)} \times 1000 \qquad [27]$$

where C_{max} is in units of kg/m^3 and ρ_f represents the value of floc density measured prior to dewatering. This equation represents a mass balance for a sludge from which all the water outside its floc particles has been removed. Therefore, assuming that intrafloc water is not removed by normal methods but that all water outside the floc structure

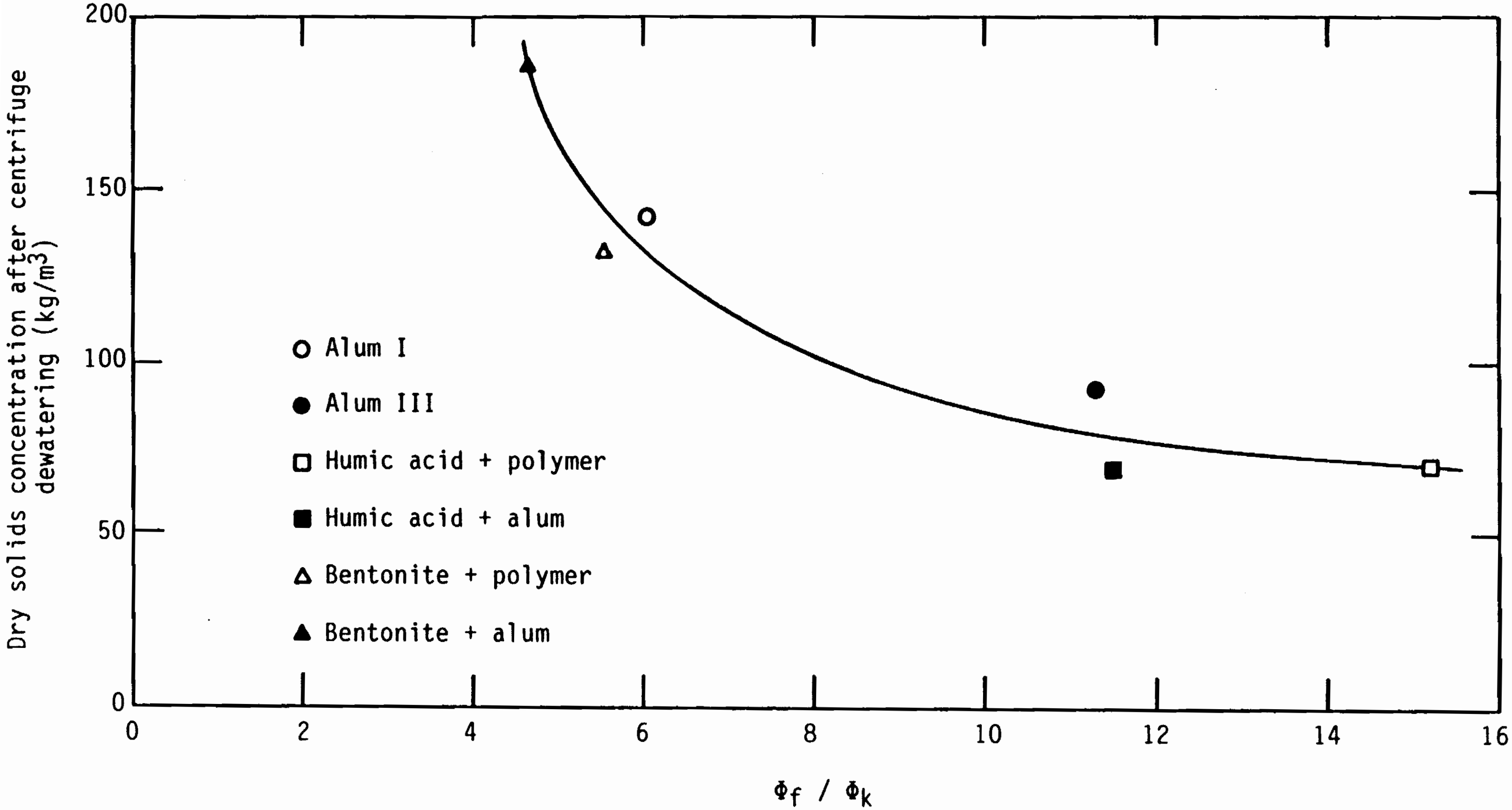

Figure 40. Comparison of the undeformed floc volume fraction to dry solids volume fraction ratio to solids concentration attained by centrifugation.

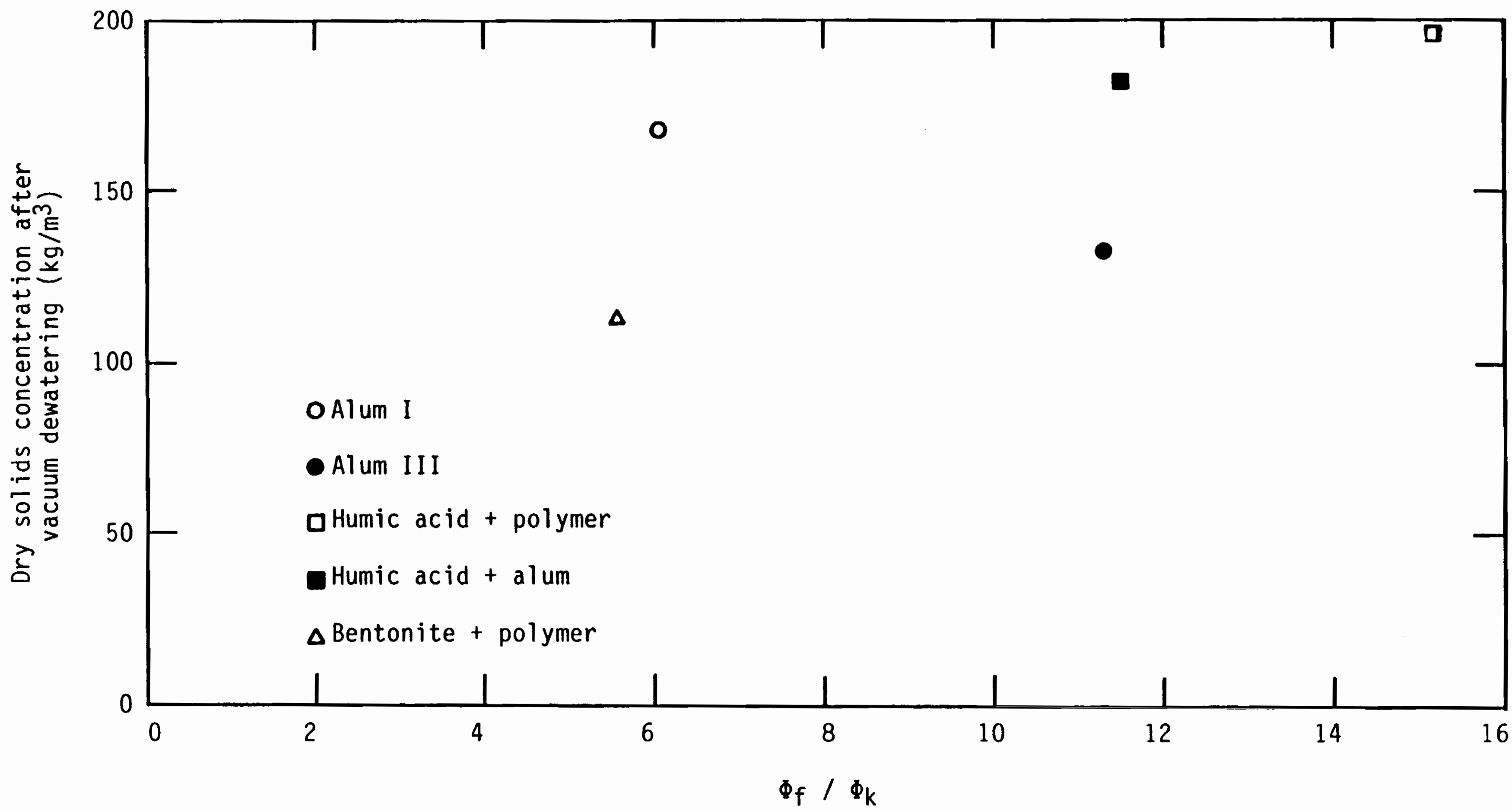

Figure 41. Comparison of the undeformed floc volume fraction to dry solids volume fraction ratio to solids concentration attained by vacuum filtration.

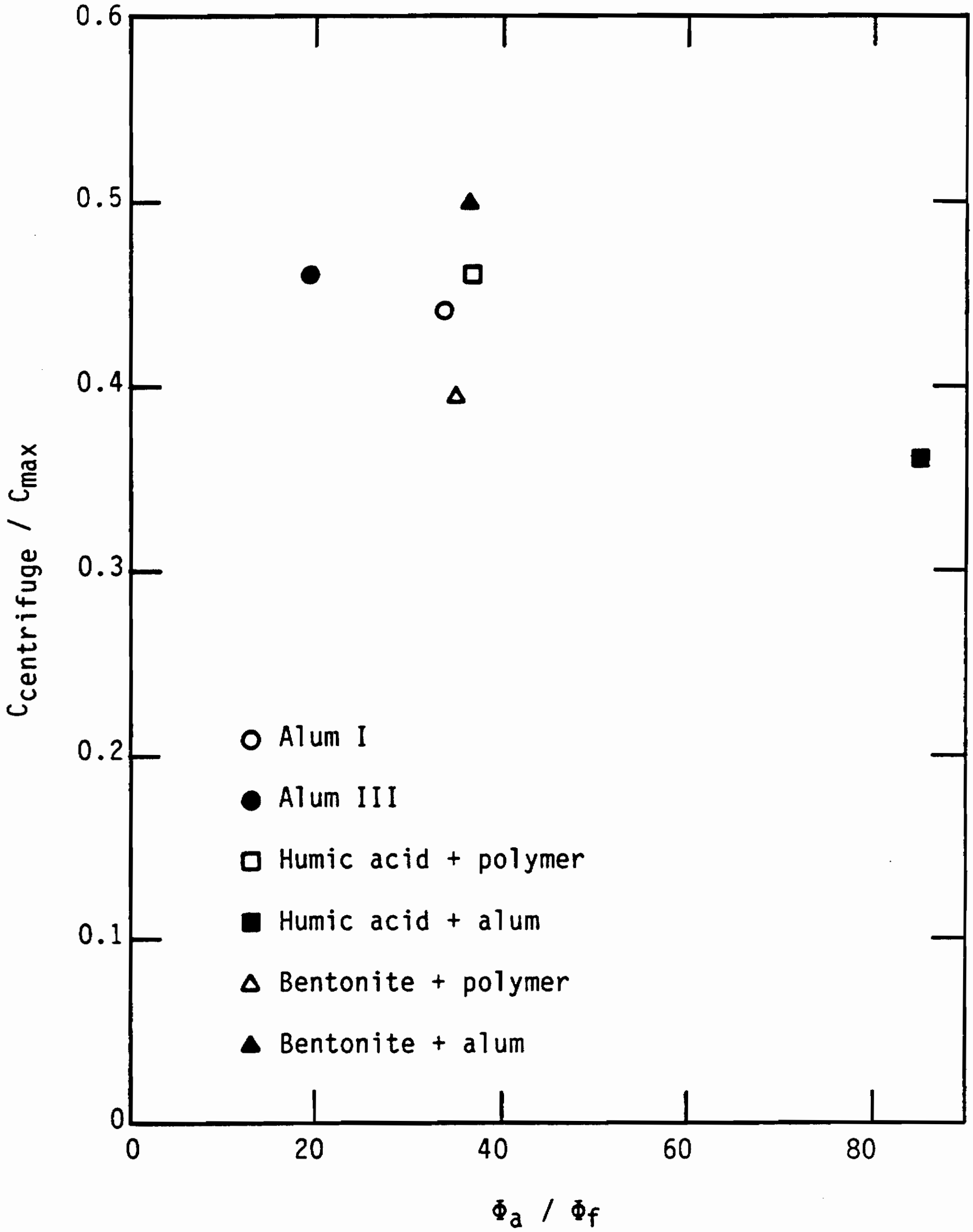

Figure 42. Effect of the ratio of aggregate volume fraction to floc volume fraction of undewatered sludges on the ratio of solids concentration attained by centrifugation to the maximum possible solids concentration.

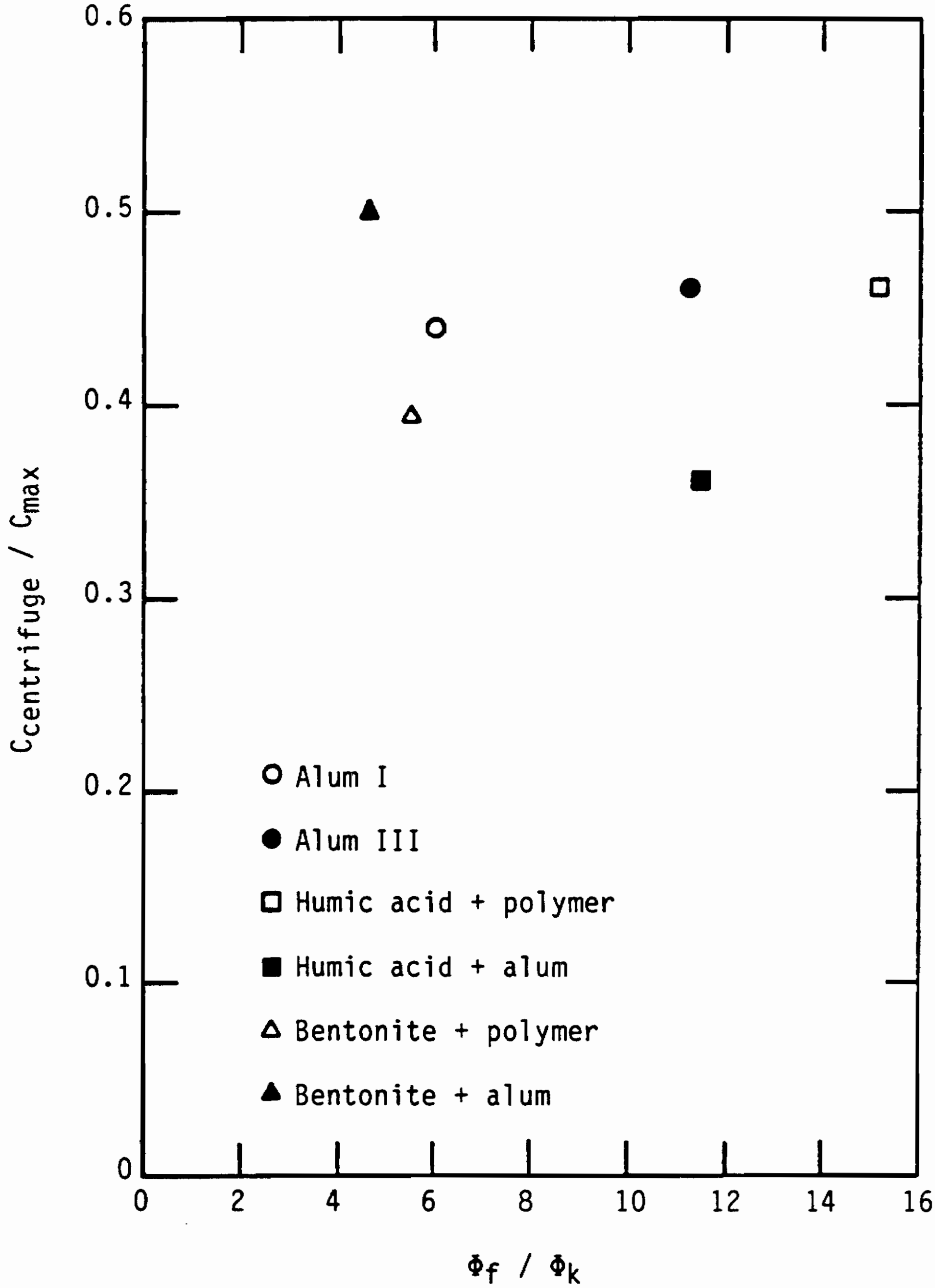

Figure 43. Effect of the ratio of floc volume fraction to dry solids volume fraction of undewatered sludges on the ratio of solids concentration attained by centrifugation to the maximum possible solids concentration.

is removed, C_{max} will be the ultimate dry solids concentration for a particular sludge.

The data in Figures 42 and 43 suggest similar conclusions concerning the relationship between the two plotted ratios. One can see from both of these plots that, regardless of the aggregate:floc or floc:dry particle volume ratio, the cake solids ratio averaged approximately 45%. That there is no relationship in these plots, other than a relatively horizontal line, may not be surprising when one remembers that the C_{max} calculation already takes into account the water content of the floc structure. The data indicate that it may be possible, given a measured floc density and dry solids density, to predict centrifuge performance in reference to cake solids concentration.

Another point to be made is that all the Φ_a/Φ_f ratios are well above that reported by Michaels and Bolger (1) of 3.8. One explanation for this difference is that this research dealt with highly flocculant materials which should have more of a tendency to come together and form large aggregate structures composed of many smaller floc particles; whereas, Michaels and Bolger were working with salt flocculated kaolin which would tend to result in smaller aggregates. Secondly, it is not clear from their work the method and/or formula Michaels and Bolger used to arrive at the values for floc density and the floc volume fraction. Therefore, it may be pointless to compare the Φ_a/Φ_f ratios obtained in this study to those of Michaels and Bolger.

CHAPTER V. SUMMARY AND CONCLUSIONS

Sludge microproperties are established by the characteristics of the different types of particles making up water and wastewater treatment sludges and the interaction between these particles. This research sought to investigate the effect variations in microproperties (and macroproperties) have on sludge dewatering. To this end, a method was to be established for measuring one of these microproperties, the floc particle density.

It is evident from the data gathered during this study that sludge microproperties do indeed affect dewatering performance. Furthermore, the importance was seen of considering microproperties in dewatering investigations rather than looking only at macroproperties. Variations in microproperties such as the AVI and floc density are dependent on the type of sludge; for instance, whether it is organic or inorganic in nature. The batch settling tests supported and built upon the ideas of past researchers concerning the different phases of gravity thickening and the fate of aggregates during these phases; namely, that the sludge settling velocity is dependent on the relative water content of the aggregates and the porosity of the slurry and that aggregate particles can be deformed and ultimately destroyed in the compression phase of thickening.

A method was recommended for measuring the floc density. The author raised questions about this method when bulk densities measured after mechanical dewatering exceeded the floc density measured prior to dewatering. These dewatered bulk densities, however, never approached the dry particle density, indicating that normal dewatering methods cannot completely remove the water phase of sludge.

The following are important specific conclusions drawn from this research:

1. The floc density test methodology presented in Chapter IV is recommended. However, it was noted that the sample particles analyzed by this method may consist of a mixture of aggregate and floc particles. Therefore, the resulting density may not be that of the actual floc particle. In view of this, a recommendation is made in the latter part of this chapter to further investigate the floc particle.

2. An increase in Aggregate Volume Index, or relative water content, results in decreasing gravity thickening rates. Inasmuch as it is related mathematically to the AVI, the aggregate volume fraction, Φ_a, has a consistent effect on sludge settling velocity from one sludge to another; namely, as Φ_a increases, settling velocity decreases. This effect of Φ_a on settling rates is due to its relationship to porosity, which is the prime factor in determining thickening rates. Higher values of porosity translate to lower values of Φ_a and provide greater thickening rates.

3. The maximum dry solids concentration achieved by gravity thickening is greater with decreasing AVI and with increasing floc density. This indicates that at higher AVI there is more water that must be removed to attain a given dry solids concentration and that this extra volume of water is not removed. Higher floc densities provide greater sludge dry solids concentrations because the aggregates break down partially or completely to floc particles during extended thickening.

4. The extent of mechanical dewatering is impacted by the floc density, the ratio of aggregate to floc volume fractions, and the ratio of floc to dry particle volume fractions.

The results of this research suggest a number of possible additional studies. First, the relationships between aggregate, floc, and dry sludge particles should be further considered with the goal of better differentiating between aggregate and floc particles. The floc density test methodology could be used in such studies by analyzing sludges having low apparent floc densities in continuous gradients of Percoll and over a broad range of centrifugal force. The same sludges could be gravity thickened and mechanically dewatered and then tested for floc density again to check for changes in the floc that may have been caused by dewatering. Finally, attempts should be made to develop a single test that will relate several of the microproperties mentioned in this study (i.e., AVI, floc density, Φ_a/Φ_f, and Φ_f/Φ_k) and that will thereby closely aproximate sludge thickening and dewatering characteristics without having to perform several batch settling and/or lab-scale dewatering tests. The floc density methodology recommended herein could be a starting point for developing such a test.

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