Measurement of chemical sludge floc density and implications related to sludge dewatering

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ABSTRACT: An isopycnic centrifugation procedure was developed for estimating the characteristic floc density present in several chemical sludges. Issues addressed included selection of a low osmotic pressure gradient medium; selection of appropriate centrifugal acceleration and time values for testing; viscosity effects; and need for temperature control. Increases in sludge floc density were predictive of an increase in cake solids concentration following mechanical dewatering. Most sludge water removal during dewatering was hypothesized to be external to the floc structure. *Water Environ. Res.*, **65,** 735 (1993).

KEYWORDS: centrifugation, floc density measurement, sludge dewatering, sludge testing.

The primary objective of sludge dewatering is to increase the cake solids concentration by removing as much liquid as possible in an efficient manner. Numerous research studies have shown that sludge microproperties such as floc size and density have a role in defining both the rate and extent of water removal observed for waste sludges. Various methods have been used for estimating sludge floc density (Li and Ganczarczyk, 1986); most involve the evaluation of settling rates or movement through a medium of known characteristics.

Analytical centrifugation has been used successfully in the biological and medical sciences for isolating and characterizing various biological particles. One method of analytical centrifugation is called density gradient or isopycnic centrifugation. The premise of this method is that particles migrate through a varying density liquid until their density equals that of the specific liquid layer, defined as the isopycnic point. Knowledge of the gradient density allows the particle density to be estimated. Separation of particles by isopycnic sedimentation is independent of particle settling rates or particle size and is a function of particle density only.

Isopycnic centrifugation has been used to quantify the apparent particle density of several bacteria prevalent in environmental treatment systems (Scherer, 1983; Vincent and Nadeau, 1984; Woldringh *et al.,* 1981). These studies involved the use of solutions of high ionic strength for maintenance of osmotic balance during the test procedure. Dammel and Schroeder (1991) used a low osmotic pressure medium for estimating the density of activated sludge solids, reporting such solids to vary in density from 1.03 to 1.06 g/mL.

Floc density values have been reported for various chemical sludges. Tambo and Watanabe (1979) used settling velocity measurements coupled with floc diameter data to estimate floc density based upon a modified Stokes equation. Lagvankar and Gemmell (1968) estimated $Fe(OH)_3$ floc density based on settling behavior in sucrose solutions; no centrifugation was used. An inverse relationship between floc size and density was observed. The sucrose solutions were characterized by extremely high osmotic pressure values. This resulted in a need for immediate estimation of floc density because exposure to the sucrose medium for any length of time resulted in an increase in the measured floc density, most probably due to floc water release initiated by the osmotic pressure of the sucrose solutions.

Dulin and Knocke (1989) used a low osmotic pressure medium (Percoll) to characterize the density of alum sludge flocs as a function of solution pH and incorporated organic matter content. Flocs produced by alum coagulation of turbidity alone had an average density near 1.20–1.21 g/mL. Incorporation of dissolved organic carbon (DOC) into the floc structure resulted in a significant decrease in measured floc density (1.14–1.16 g/mL). This decrease in floc density corresponded to a sharp decrease in vacuum dewatered cake solids concentration.

This paper reports on the use of low osmotic pressure gradient media for measuring the density of chemical sludge flocs. The first portion addresses issues associated with the development of a floc density protocol, including such items as centrifugal speed, time, and medium osmotic pressure. A second emphasis is on using such data in describing the response of various chemical sludges to conditioning and/or mechanical dewatering.

Particle Structure Notation

This paper utilizes the three-tiered particle structure notation proposed by Michaels and Bolger (1962) in describing the floc density measurement technique and its application to sludge characterization. Michaels and Bolger defined a primary particle as being composed of dry solids and associated bound water. Flocs are produced by the clustering of such primary particles, along with internal water that is retained between clusters as part of the floc structure. The floc structure is characterized by significant shear strength. Under lower turbulence (mixing) or quiescent conditions, these flocs form large-size aggregates through loose associations. These aggregates have low shear strength and are easily ruptured by external forces.

Methods and Materials

Sludge sources. Sludges selected for use in this study included a range of anticipated floc density values. Specifics about the sludges investigated are below. (1) Three alum sludges (called Alum I, Alum II, and Alum III) were obtained from treatment facilities that coagulated low turbidity, low organic content surface water. (2) A polymer coagulation sludge was obtained from a treatment facility that used a cationic polymer (Nalcolyte 8101) to coagulate a low turbidity, high organic content surface water. (3) An iron coagulation sludge (called Iron I) was obtained from a water treatment facility that used ferric chloride coagulation of an iron-containing groundwater. (4) A second iron coagulation sludge (called Iron II) was collected from a surface water treatment facility where ferric chloride was used to coagulate a low turbidity, low organic content surface water. (5) Lime sludge was collected from a groundwater facility that practiced selective calcium removal for softening; minimal amounts of magnesium were incorporated into the sludge. (6) A pyrite (iron sulfide) sludge was collected from a pyrite mining operation.

Each sludge sample was collected onsite and gravity thickened for a minimum of 24 hours (with subsequent supernatant removal) prior to testing.

Sludge dewatering procedures. Sludge samples were dewatered by various mechanical systems. Centrifugation was performed with a high-speed centrifuge (samples dewatered at 2 000–17 000 rpm). Following centrifugation, the supernatant was decanted and the resulting dewatered sludge cake analyzed. Vacuum filtration was accomplished according to methods described by Vesilind (1979). A 9-cm diameter Buchner funnel was used with Whatman 40 filter paper used as the filter medium. A pressure differential from 6 to 28 inches mercury (0.2 to 0.9 atm) was employed.

High-pressure filtration was accomplished using a stainless steel cylinder pressurized at 30–160 psig with a nitrogen tank. A 30-mL volume of sludge was poured into the cylinder on Whatman 40 filter paper. Samples were removed following dewatering for subsequent solids analysis.

Certain sludge samples were conditioned with various cationic polymers prior to mechanical dewatering. A jar test apparatus (Phipps & Bird; Richmond, Va.) was used with rapid mixing (200 rpm, 1 minute) followed by slow mixing (30 rpm, 5 minutes) to assess appropriate polymer dosing requirements. A 500 mL sludge sample volume was used in these studies. No attempt was made to find the optimum sludge-polymer-dosing-mixing configuration; instead, the intent was to find a polymer conditioning scheme that improved sludge dewatering rates so that the effects of polymer conditioning on floc and sludge density values could be evaluated.

Samples of the polymer water treatment plant sludge and Iron I sludge were conditioned using freeze-thaw methods. Sludge (2-L volume) was placed in open vessels and stored in a freezer (−5–−8°C) for up to 72 hours. After freezing, the sludge was allowed to warm to room temperature and free fluid decanted. Samples were dewatered and analyzed immediately upon reaching 20°C.

Sludge analyses. Both undewatered and dewatered sludge samples were analyzed for several parameters. Sludge total solids concentration was determined by oven drying at 103°C according to *Standard Methods* (1985). Initial testing of each sludge indicated that dissolved solids concentrations were less than 2% of the total solids concentration present; thus, suspended solids (SS) concentrations were assumed to be essentially equal to total solids concentration values.

Sludge bulk density (p_b) was evaluated for both thickened and dewatered sludge samples using calibrated glass pycnometers. Using several paired values of sludge bulk density and dry solids concentration, it was possible to estimate the dry solids density (p_{dry}) of each sludge

$$\frac{100}{p_b} = \frac{(100\text{-}C)}{p_w} + \frac{C}{p_{\text{dry}}} \qquad (1)$$

Where

C = sludge SS concentration, %;
p_b = sludge bulk density, g/mL;
p_w = density of water at the temperature of interest, g/mL; and
p_{dry} = sludge dry particle density, g/mL.

Development of a floc density protocol. Important variables in the development of the protocol included gradient medium, method of gradient formation, method of sample addition, osmotic pressure effects, centrifugation speed and time, and temperature.

Gradient media. Media considered were cesium trifluoroacetate (CsTFA™), Percoll®, and sucrose solutions. CsTFA is marketed by Pharmacia Fine Chemicals AB of Sweden. Percoll, also marketed by Pharmacia, consists of colloidal silica particles coated with polyvinylpyrrolidine (PVP). Several properties of both products (as reported by the manufacturer) 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.

Osmotic pressure. Osmotic pressure was investigated for its effect on the apparent floc density by comparing the density values obtained in gradients prepared with each of the three media. Osmotic pressure was calculated according to the following equation:

$$P_t = 14.7 \cdot S \cdot \mathrm{R} \cdot \mathrm{K} \qquad (2)$$

Where

P_t = solution osmotic pressure, psi;
S = solution osmosity, mole/L;
R = gas constant, 0.0821 L·atm/mol·K; and
K = temperature, K.

Solution osmosity information was obtained either from the manufacturer or from the literature (Chemical, 1979). Values were calculated for CsTFA and sucrose solutions as well as Percoll media suspended in solutions of various percent sodium chloride concentration. Results of these calculations are presented in Figure 1 as a function of media density. Note the impact of saline concentration on osmotic pressure for Percoll media and the high values of osmotic pressure for CsTFA and sucrose.

Table 1—Physical properties of CsTFA and Percoll.

Property	Specifications[a] CsTFA	Percoll
Composition	134 g cesium trifluoroacetate/ 100 mL of solution	Silica sol with nondialysable PVP[b] coating
Density	2.0 ± 0.05 g/mL	1.13 ± 0.005 g/mL
Conductivity	Not available	1.0 mS/cm
Osmotic pressure[c]	2 400 psi	3 psi
Viscosity	1.4 centipoise at 20°C	10 ± 5 centipoise 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

[a] As reported by manufacturers.
[b] PVP—polyvinylpyrrolidine.
[c] Calculated from osmolality information provided by manufacturers.

Preliminary tests were performed to assess the impact of osmotic pressure on observed floc density; representative values for three sludges are shown in Table 2. The aqueous Percoll gradients were prepared with distilled water. Clearly, osmotic pressure had an effect on the measured floc density value. It was hypothesized that osmotic pressure was causing intrafloc water to be removed from the floc structure, resulting in artificially high floc density values. As a result, CsTFA and sucrose were eliminated from consideration as a gradient medium, and further testing centered on the use of the Percoll media.

Media viscosity. Viscosity was considered for its impact on ease of handling of the media and on the rate at which a sludge floc reached its isopycnic point. Values of viscosity for Percoll (reported by the manufacturer) are shown in Figure 2; comparison is made to reported values for sucrose and CsTFA (Chemical, 1979). Under higher media density situations, the viscosity values indicate that longer centrifugation times may be necessary to reach a true isopycnic point in the gradient.

Media density and gradient preparation. Percoll was supplied by the manufacturer at a density of 1.13 g/mL. Gradients comprised of lower density values were generated by dilution of the media. Percoll could also be concentrated using dialysis to achieve density values as large as 1.30 g/mL. Dialysis tubing was boiled for 10 minutes in distilled water containing approximately 200 mg/L Na_2CO_3 as per manufacturer's recommendations. Percoll was then poured into the tubing, laid into a pan, and covered with polyethylene glycol flakes (depth of coverage approximately 2 cm) which extracted water through the dialysis tubing. Dialysis was typically complete after 2 hours.

Density of the Percoll media was quantified by measuring its refractive index at 25°C using a refractometer. This required the initial development of a standard curve relating the two parameters. To do this, solutions containing varying amounts of

Table 2—Floc densities of different sludges as measured in media exhibiting different osmotic pressures.

Sludge	Gradient median	Floc density, g/mL	Osmotic pressure, psi
Alum II	Aqueous Percoll	1.24	4
	CsTFA	1.9	2 400
Iron II	Aqueous Percoll	1.11	3
	Sucrose	1.3	1 000
Lime	Aqueous Percoll	>1.3	60
	CsTFA	1.9	2 400

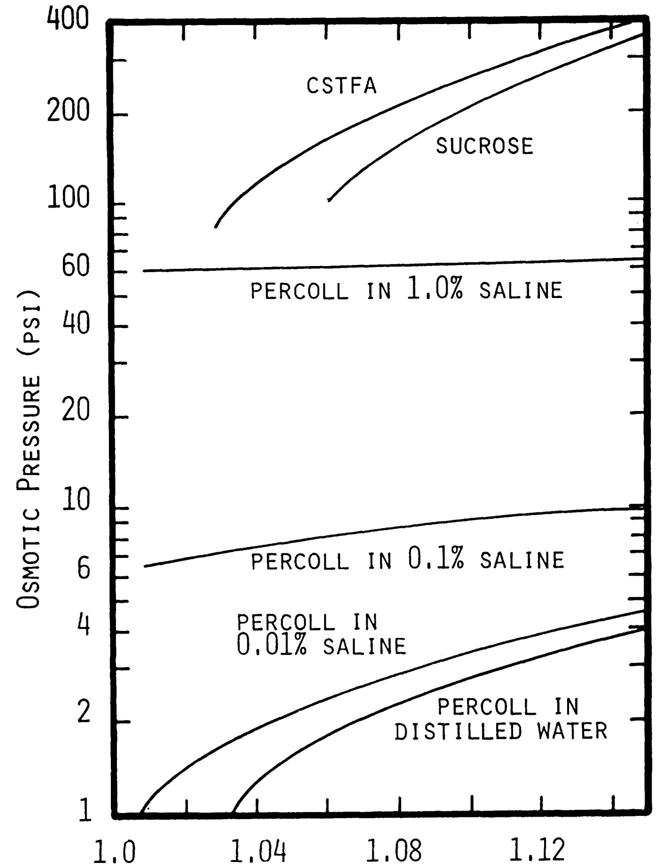

Figure 1—Relationship between medium density and osmotic pressure for various centrifugation media.

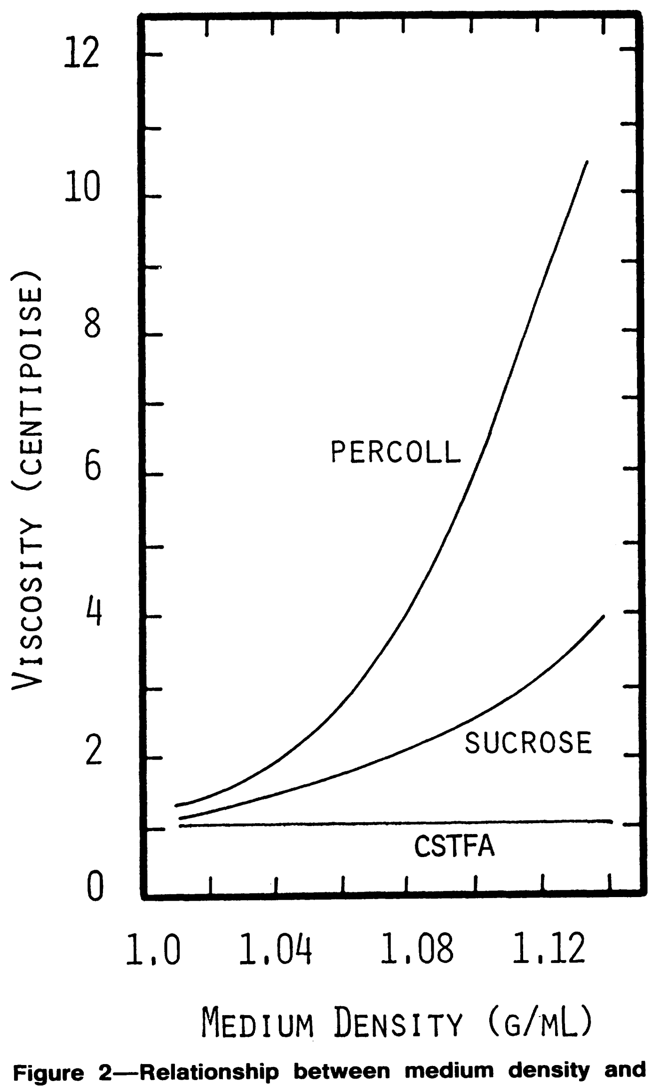

Figure 2—Relationship between medium density and viscosity for various centrifugation media (Chemical, 1979, and Sheeler, 1981).

Percoll and distilled water were prepared and analyzed for density (using a pycnometer) and refractive index. The resulting paired values were plotted (Figure 3) and used throughout the study as a rapid means for determining media density.

Two types of gradients were prepared for testing: continuous and step gradients. A continuous Percoll gradient was produced in a 10-mL centrifuge tube with diluted media at a density equal to the desired midpoint density of the gradient. The tube was then centrifuged for approximately 30 minutes at 13 000–15 000 rpm in a high-speed centrifuge (Beckman Model J-21C, rotor type JA-20; Fullerton, Calif.). Density marker beads (supplied by Pharmacia) were added prior to centrifugation. These beads (density determined in distilled water; range from 1.02 to 1.09 g/mL) provided a means for estimating density at various gradient locations.

Step gradients were formed in 10-mL centrifuge tubes by layering, in order of decreasing density, 1 mL of each "step" solution. Normally, a step gradient consisted of up to six layers of media with density increments between layers of 0.01 to 0.025 g/mL. Layering involved the use of a 1-mL pipette for solution transfer. The transfer was done carefully to minimize intermixing of the adjacent density layers. Media refractive index is directly related to density; thus, each of the step layers could be identified by a visual interface that developed between adjacent steps. Concentration gradient effects between layers produced diffusion over time near the layer boundaries. As a result, step gradients were typically usable only for 30–40 minutes after creation. Step gradients were used most frequently for analyzing flocs with density values between 1.13 to 1.30 g/mL. Density values were reported as either the density of the layer in which flocs were observed (after centrifugation) or as a range of values if flocs banded at the interface between two layers.

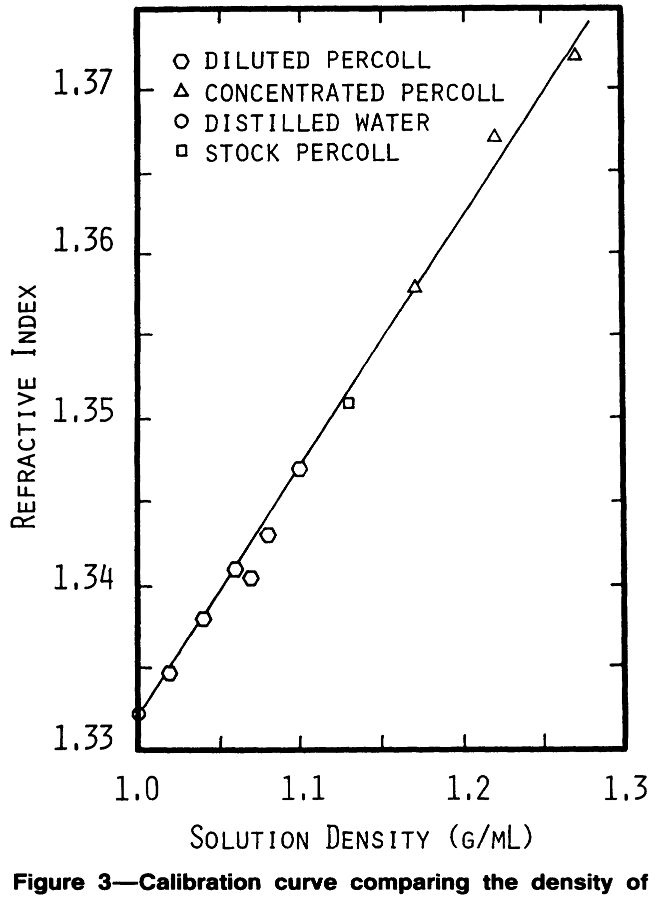

Figure 3—Calibration curve comparing the density of Percoll solutions' measured refractive indexes (temperature = 24.5°C).

Centrifugation variables. Speed and time of centrifugation were varied to determine what effect (if any) such changes 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 up to 2 700 rpm (up to 900 g). Higher speeds were found to produce greater intermixing of the step layers. Continuous gradient samples were centrifuged at speeds of 2 000–15 000 rpm in the high-speed Beckman centrifuge.

At each centrifuge speed, the rotor was stopped at 1–2-minute intervals to record the floc location in the gradient at that time. Centrifuge time effects were evaluated for total centrifugation times of up to 15 minutes.

Sample addition. In both step and continuous gradient tests, sludge samples (approximately one drop) were added either on the top or bottom of the gradient. This variation was investigated to determine whether the procedure yielded results that were truly isopycnic. A disposable polyethylene pipette was used to deposit the sample on the surface of the gradient in "top-loaded" tests. "Bottom-loading" to step gradients involved depositing the sludge sample in the bottom of the centrifuge tube prior to layering the gradient. Loading beneath continuous gradients was performed after the gradient was formed using a 1-mL, wide-bore syringe, being careful not to disturb the preformed gradient and marker beads.

Typically, the source of sludge flocs for analysis was from the thickened sludge sample (prior to dewatering). However, in a few tests, an attempt was made to analyze for changes in floc density that may occur during dewatering. Following dewatering, extremely small (<1 mm diameter) pieces of sludge were removed from the cake and placed into a density gradient for analysis. While this procedure did not ensure the transfer of individual flocs or aggregates, it did allow for at least a semi-quantitative estimate of density to be undertaken.

Temperature effects. The impact of variations in temperature were not investigated as part of this study. Instead, it was recognized that temperature variations during the course of an analysis would be a source of experimental error. Temperature changes could affect media properties such as osmotic pressure, viscosity, and/or density. Thus, all analyses were conducted under controlled (20–22°C) conditions.

Controlled particle formation and testing studies. A limited number of experiments were conducted that involved the formation of flocculated aggregates and the subsequent analysis of these aggregates via isopycnic gradient centrifugation. Latex particles (Accubeads; Fastek Co., Liverpool, N.Y.) of known size (0.5 μm or 5.0 μm; ρ_{dry} = 1.05 g/mL as reported by Fastek) were flocculated with a low molecular weight cationic polymer, producing large-size, visible aggregates which were separated by sedimentation. These aggregates were placed into a Percoll gradient and allowed to settle until they reached an apparent isopycnic point (noted as a lack of further observed settling). Following the notation of this gradient location, the Percoll media was then centrifuged at 15 000 rpm for approximately 1 hour, at which time the particle location was again noted. This procedure permitted estimation of the degree of aggregate rupture during centrifugation.

Results and Discussion

Controlled aggregation studies. Results from the polymer flocculation of Latex particles are shown in Table 3. The aggregates formed by polymer addition had measured density values markedly lower (<1.03 g/mL) than the manufacturer's (Fastek) reported dry density of 1.05 g/mL; likewise, this value was well below the density range of 1.043–1.046 g/mL observed when unflocculated Latex particles were centrifuged in a Percoll gradient. However, on application of centrifugal forces, these aggregates were compressed or ruptured in some form, yielding floc that had a measured density much closer to the original unflocculated Latex particle density. Thus, the density gradient method does apparently produce forces sufficient to promote the rupture of large-size aggregates. These results indicate that the centrifugation method has the *potential* for measuring floc density values for sludges that originally contain large-size aggregates.

Effects of various parameters on measured floc density. The impact of variations in centrifugation speed and time on measured floc density can be evaluated by reviewing data summarized in Table 4 for the three alum sludges. Because of high floc density values, the three sludges listed in Table 4 were all analyzed using step gradients in the low-speed, table-top centrifuge. In general, the use of centrifugal acceleration values of 200 g or greater resulted in no change in measured floc density. Likewise, centrifugation time values of 5 minutes and longer produced essentially the same range of floc density values. Tests involving lower density floc showed no difference when using centrifugation times as short as 2 minutes. The greater viscosity of the Percoll media at higher density values was hypothesized to be the reason slightly longer centrifugation times were required to reach an isopycnic point.

These results are similar to those obtained by Dammel and Schroeder (1991) who observed no measurable change in activated sludge particle density when using centrifugal acceleration values of 200 g or greater. Using the same methods as described in this paper, Dishman (1988) reported that activated sludge solids density did not vary when measured over a centrifugal acceleration range from 200 g to 16 000 g.

Comparison of the results obtained from parallel studies involving top versus bottom loading for sample addition indicated no discernible difference in measured floc density. Thus, it was concluded that the results obtained in this procedure represented a true isopycnic condition.

Table 3—Comparison of centrifugal gradient density measurements for unflocculated and polymer flocculated latex particles.

	Particle density, g/mL	
Particle size and type	No flocculation	Polymer flocculated
5.0 μm particles	1.046[a]	<1.030[b] 1.043[a]
0.5 μm particles	1.043[a]	<1.030[b] 1.042[a]

[a] Centrifuged in Percoll gradient at 16 500 rpm for 1 hour.

[b] Density measured in Percoll gradient following sedimentation only; no centrifugation provided.

Table 4—Effect of centrifugation acceleration and time on measured sludge floc density.

Sludge type	Centrifugal acceleration, g	Centrifugation time, minutes	Floc density, g/mL
Alum I	200	1	1.21–1.23
	200	4	1.22–1.25
	200	12	1.22–1.25
	900	1	1.22–1.24
	900	2	1.23–1.255
	900	8	1.23–1.255
	900	12	1.22–1.255
Alum II	200	2	1.20–1.22
	200	3	1.21–1.23
	200	5	1.22–1.25
	200	10	1.22–1.25
	900	2	1.20–1.23
	900	5	1.21–1.25
	900	8	1.22–1.25
	900	12	1.22–1.25
Alum III	200	2	1.13–1.16
	200	10	1.13–1.16
	900	2	1.13–1.16
	900	10	1.13–1.16

Effects of sludge conditioning on floc density and sludge dewatering characteristics. Sludge conditioning via polymer addition resulted in an increase in measured floc density; representative data are contained in Table 5. Knowing the dry density of solids contained in the floc as well as the measured floc density, it was possible through mass balance considerations to calculate the degree of fluid release from the floc obtained following polymer conditioning. Release was typically in the range of 25–40% of the water originally present in the floc structure. This observation compares favorably with the work of Robinson and Knocke (1992) who used isothermal drying techniques and dilatometric procedures to quantify the release of fluid from flocs during polymer conditioning. The authors reported up to 50% release of bound water from flocs following polymer application. Likewise, Katsiris and Kouzeli-Katsiri (1987) found that polymer application to waste biological sludges produced a significant (up to 50%) reduction in floc-bound water content.

Polymer conditioning also increased the rate of water removal from each sludge as characterized by a decrease in the specific resistance value obtained during vacuum dewatering. However, polymer application did not improve the extent of water removal

Table 5—Effects of polymer conditioning on floc density and sludge dewatering characteristics.

Sludge type	Polymer conditioned ?	Floc density, g/mL	Specific resistance, m/kg × 10^{11}	Cake solids concentration, %
Polymer WTP	No	1.06–1.08	250	15–16
	Yes	1.08–1.11	20	14–15
Alum III	No	1.14–1.16	—	13–14
	Yes	1.16–1.18	—	13–14
Iron I	No	1.26–1.28	120	25–26
	Yes	>1.28	70	23–25

Table 6—Effect of freeze-thaw conditioning on floc density and dewatered cake solids concentration.

Sludge type	Floc density, g/mL	Dewatered solids concentration, % dry wt			
		Gravity thickening	Vacuum filter	Centrifuge	Pressure filter
Polymer WTP (unconditioned)	1.06–1.08	1.2–2.7	15–17	5–11	15–21
Polymer WTP (freeze-thaw)	>1.30	20–25	45	28–33	37–40
Iron I (unconditioned)	1.26–1.28	8.1–8.9	21–27	17–31	18–30
Iron I (freeze-thaw)	>1.30	7–16	47–54	38–44	48–53

achieved as indicated by comparable cake solids concentrations for both unconditioned and conditioned sludge samples.

The impact of freeze-thaw conditioning on floc density and cake solids concentration achieved following gravity thickening and mechanical dewatering can be seen in Table 6. Floc density values increased substantially following freezing and subsequent thawing of the sludge samples. For example, the floc density for the polymer water treatment plant sludge increased from approximately 1.08 g/mL to a value exceeding 1.3 g/mL, the maximum value that could be measured in the Percoll gradient. Knowing the dry density of solids present in this sludge (ρ_{dry} = 1.58–1.60 g/mL), mass balance calculations determined that freeze-thaw conditioning resulted in at least an 80% reduction in internal floc water content. This release of floc water resulted in an ability to remove water from this sludge to a much greater extent, regardless of the dewatering method used. For example, gravity thickened solids concentration increased from the 1–1.5% dry solids range to values in excess of 20% dry solids after freeze-thaw treatment. Likewise, cake solids concentrations increased by as much as 300%.

This increase in floc density following freeze-thaw treatment is well supported by the literature. Robinson and Knocke (1992) reported that freeze-thaw treatment of chemical and biological sludges resulted in floc bound water release of up to 75% by weight. Likewise, others (Katsiris and Kouzeli-Katsiri, 1987, and Tsang and Vesilind, 1990) have reported significant removal of floc water in association with freeze-thaw conditioning of sludges.

Interpretation of floc density data and implications for sludge dewatering performance. Sludge samples were mechanically dewatered and analyzed for the resulting cake solids concentration and corresponding cake bulk density. Representative results for two of the sludges considered are shown in Figures 4 and 5. As expected, an increase in cake solids concentration correlated with sludge cake density because cake volume was occupied by sludge solids with densities much greater than 1.0 g/mL. Also, application of high-pressure differentials (for example, high-pressure filtration) yielded increased cake solids concentrations.

Floc density values were also measured for flocs collected from the thickened sludges (prior to mechanical dewatering) and are included in the two Figures. Cake bulk density values were, in almost every instance, well below the measured floc density value. This result suggests that the application of mechanical dewatering forces promoted the removal of fluid which was, for the most part, external to the floc structure. Tsang and Vesilind (1990) as well as Robinson and Knocke (1992) reached a similar conclusion when assessing the removal of water from waste biological sludges using mechanical dewatering.

This issue can be further evaluated by considering bulk density data as well as density data measured on flocs collected both before and after mechanical dewatering. As stated previously, a limited number of attempts were made to analyze floc density using samples obtained from dewatered sludge cakes. Data for the Alum III sludge (both unconditioned and polymer conditioned) are contained in Table 7, both before and after vacuum filtration. The results show that measured floc density increased following vacuum filtration, indicating the loss of some amount of internal floc water.

Knowing values for bulk density, floc density, and dry particle density, mass balance considerations allowed for the calculation of the amount of internal and external floc water present in each sludge, both before and after vacuum filtration. While the loss of internal floc water was significant on a percentage basis, the

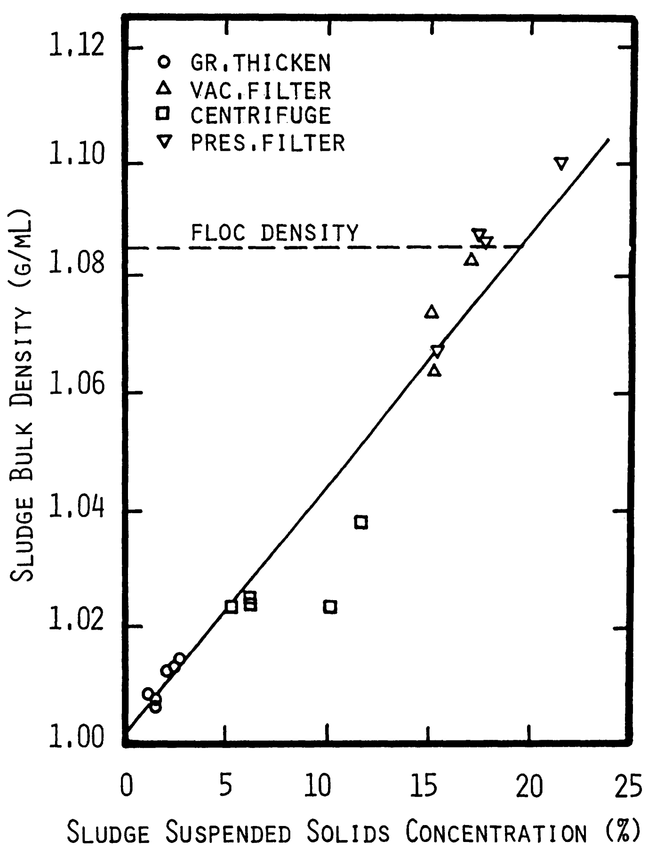

Figure 4—Sludge bulk density-suspended solids concentration relationship for the polymer water treatment sludge.

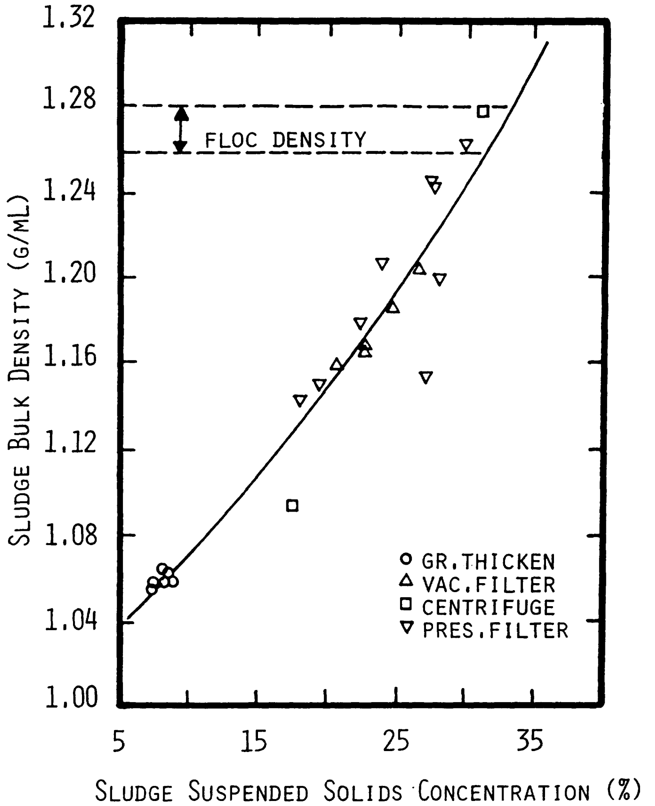

Figure 5—Sludge bulk density-suspended solids concentration relationship for the iron coagulation water treatment (ferric II) sludge.

amount of water within the floc structure was a relatively small fraction of the total water present in the sludge. As a result, the calculated contribution of internal floc water content loss to overall sludge water loss was small (<4% by weight). These results support the conclusion that the majority of the water removed from a sludge during dewatering was external to the floc structure.

Values of floc density, dry particle density, and cake solids concentration achieved following the thickening and mechanical dewatering of various chemical sludges are summarized in Table 8. In general, higher values for sludge floc density were predictive of higher cake solids concentrations being achieved after dewatering, regardless of the dewatering technique used. Special note should be made of the lime and pyrite sludges. Both had floc densities in excess of 1.3 g/mL, the maximum that could be measured in a concentrated Percoll gradient. These sludges produced the highest cake solids concentrations observed.

The value of greater than 1.9 g/mL listed for the pyrite sludge was included because the thickened sludge bulk density of this sludge was 1.9 g/mL. With all other chemical sludges, the thickened sludge bulk density was well below the measured floc density. This relationship was assumed to be equally true for the pyrite sludge; thus, this value was provided simply for reference to the other chemical sludges listed.

Relationship of these findings to the published literature. An important issue to be addressed is the interpretation of the floc density results obtained from isopycnic procedures in relation to the work of others. Tambo and Watanabe (1979) calculated floc density values based upon the analysis of quiescent settling rates for flocculated solids. They reported that floc density decreased as floc size increased. Lagvankar and Gemmell (1968) reached a similar conclusion based upon sucrose gradient measurements of flocculated particles produced in iron coagulation systems.

An analysis of the results of these two studies was undertaken. A key issue in relation to the current study was the relative size and density of flocs being measured in each. Most of the flocs reported by the two studies had diameters greater than 500 μm. Table 9 summarizes floc density information for a variety of chemical sludges based on the smallest floc diameters reported in each study. The floc density values observed were all well below that measured in the current study by isopycnic centrifugation for aluminum and iron based sludges.

The differences observed can be explained by consideration of the three-tiered particle structure (Michaels and Bolger, 1962) that was central to the formulation of the current study. The two literature studies both reported information based on the settling characteristics of what would be interpreted as aggregates in the Michaels and Bolger framework. Tambo and Watanabe (1979) rightly concluded that this size range would be important in sizing quiescent sedimentation basins. However, it is doubtful that these aggregates would retain their physical size during thickening and mechanical dewatering because of the shear and/or compressive forces exerted on the aggregates. For example, Javaheri and Dick (1969) reported that the size of activated sludge aggregates decreased significantly as the sludge went through the transitional and compression periods of gravity thickening. The decrease in size was felt to be because of the low shear strength of the sludge aggregates.

Tambo and Hozumi (1979) showed that floc diameter was inversely proportional to the effective rate of energy dissipation

Table 7—Changes in sludge density values and corresponding water loss following vacuum filtration.

	Density value, g/mL				Water loss during vacuum filtration		
	Before dewatering		After dewatering				
Sludge type	Bulk	Floc[a]	Bulk	Floc[a]	Floc water loss, % by wt	Overall sludge water loss, % by wt	Percent of overall water loss due to floc water loss
Alum III	1.02	1.145	1.062	1.165	14	69	2.5
Alum III plus polymer	1.02	1.175	1.092	1.24	30	80	3.8

[a] Average floc density value (for range of density values observed in gradient).

Table 8—Summary of floc density and dewatered solids concentration data for several chemical sludges.

Sludge type	Floc density, g/mL	Dry solids density, g/mL	Maximum dewatered solids concentration, % by wt			
			Gravity thickening	Vacuum filter	Centrifuge	Pressure filter
Polymer WTP	1.06–1.08	1.58	2.7	17	11	21
Polymer WTP[a]	>1.30	1.60	25	45	33	40
Iron I	1.26–1.28	2.86	8.9	27	31	30
Iron I[a]	>1.30	2.86	16	54	44	53
Alum I	1.22–1.25	2.55	3.0	17	15	25
Alum II	1.14–1.16	2.45	3.0	14	10	—
Lime	>1.30	2.47	13	41	42	—
Pyrite (iron sulfide)	>1.9	3.86	61	83	—	92

[a] Freeze-thaw conditioning provided.

the suspension was exposed to. Again, the floc sizes reported ranged from 200 μm to 2 000 μm (consistent with the aggregate definition of the current study). Stated differently, larger size flocs were highly susceptible to shear input, resulting in floc rupture and a decrease in floc size (with a corresponding increase in floc density).

Significant amounts of shear are present in many mechanical dewatering systems, making aggregate rupture and fragmentation into smaller size flocs likely. For example, in the current study, polymer generated aggregates exposed to high speed centrifugation were ruptured back to smaller size flocs with densities near that of the uncoagulated Latex solids. Also, Novak and Lynch (1990) showed that significant shear can develop within sludge cakes because of the movement of liquid through the cake. The authors calculated mean velocity gradient (G) values as large as 450–500 seconds^{-1} within sludge cakes exposed to vacuum filtration.

Finally, one additional point to be noted is the relationship between bulk density and measured floc density values for various chemical sludges. Table 9 contains maximum floc density data reported by Tambo and Watanabe (1979) as well as Lagvankar and Gemmell (1968) for various water plant sludges. Comparing these numbers to the values reported for bulk density for dewatered sludges (Figures 4 and 5 are representative for two sludges), it can be seen that the bulk density values measured in the current study often exceeded the maximum floc density values of Table 9, a condition that would not be theoretically possible in a sludge cake. Rather, the maximum bulk density would be the floc density (assuming that all external floc water had been removed from the cake).

The summation of this information leads to the conclusion that aggregate rupture occurs during thickening and mechanical dewatering. As such, it is proposed that the centrifugal isopycnic procedure leads to the measurement of a floc density that may be more representative of the characteristic size particles that predominate in these mechanical dewatering systems.

Summary and Conclusions

The purpose of this study was to define a useful protocol for estimating sludge floc density. A second objective was to use these data in interpreting the response of several chemical sludges to mechanical dewatering.

Various recommendations can be made regarding an appropriate protocol for floc density analysis using isopycnic centrifugation. First, it is important that gradient medium osmotic pressure conditions be comparable to that present in the sludge sample. For most chemical sludges, this would typically indicate the need for a low osmotic pressure medium. Percoll gradient media suspended in either distilled water or low salt concentration (for example, 0.01% sodium chloride by weight) would satisfy this requirement. Reported values for floc density testing using high osmotic pressure gradient media should be interpreted with caution.

Recommended test parameters include use of a centrifugal acceleration of at least 200–500 g and a centrifugation time of 5–10 minutes to ensure that the flocs reach an isopycnic point in the gradient. Isothermal conditions should be maintained throughout the analysis period to minimize temperature effects on viscosity and measured density.

Floc density information can provide helpful insights into the

Table 9—Density—size information reported by various literature sources.

Source	Sludge type	Smallest floc sizes reported, μm	Maximum floc density values reported, g/mL
Tambo and Watanabe (1979)	Alum (low turbidity, low DOC)[a]	200–300	1.04–1.05
	Alum (high turbidity, low DOC)	200–300	1.06–1.07
	Iron (low turbidity, low DOC)	200–400	1.02–1.04
	Alum (low turbidity, high DOC)	600–800	1.004–1.006
	Iron (low turbidity, high DOC)	400–600	1.004–1.01
Lagvankar and Gemmell (1968)	Iron (low turbidity, low DOC)	500–600	1.005–1.006
	Iron (high turbidity, low DOC)	400–500	1.005–1.006

[a] DOC—dissolved organic carbon.

dewatering characteristics of chemical sludges. The data indicate that most of the water removal during mechanical dewatering is external to the floc structure.

Polymer conditioning did result in the release of floc water as evidenced by an increase in measured floc density. Likewise, freeze-thaw conditioning produced a large increase in floc density and a corresponding increase in the extent of sludge dewatering achieved by various methods.

Further work is warranted to determine whether this isopycnic centrifugation technique for measuring floc density can provide insights into the dewatering characteristics of waste biological sludges. It is realistic to assume that the floc and aggregate formation characteristics of chemical sludges may be different than those of activated sludge. The role of exocellular materials in promoting aggregate stability and the likelihood of centrifugal test procedures disrupting such stability must be evaluated before the method can be effectively used to characterize biological sludges.

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