

# Measurement of bound water in sludges: A comparative study

D. J. Lee, Y. H. Hsu

**ABSTRACT:** The centrifugal settling, expression, differential scanning calorimetry (DSC), and drying tests are employed for measuring the bound water content in a sludge made of  $\text{CaCO}_3$ , PVC, or glass powders, an inorganic  $\text{Cu}(\text{OH})_2$  sludge, and an activated sludge. The bound water is found to be an operationally defined value that differs greatly when different measurement technique is applied. A classification scheme for dividing the moisture content in a sludge based on the bound water data is proposed. *Water Environ. Res.*, 67, 310 (1995).

**KEYWORDS:** bound water, centrifuge, drying, settling.

Dewatering is one of the most difficult process in wastewater sludge treatment. Because the moisture distribution within a sludge shows strong correlation between the bound water content and the performance of many processes (Drost-Hansen, 1977; Sato *et al.*, 1982; Katsiris and Kouzeli-Katsiris, 1987; Robinson and Knocke, 1992), people have tried for years to differentiate the water content which is in different states (Herwijn *et al.*, 1992).

The simplest way of differentiation is to divide the water content into two parts: the "bound water," which is held chemically, physically, or both onto the flocs, and the "free water," which is the remaining water content that behaves the same as the bulk water and is easily removed via mechanical means.

Vesilind and Martel (1990) (see also Tsang and Vesilind, 1990; Vesilind *et al.*, 1991) classified the water content further into four catalogues: free water, interstitial water, surface water, and bound water. Herwijn *et al.* (1992) divided the water content in a similar matter as made by Vesilind and Martel (1990); however, the physical interpretation is very different. Smollen (1990) also made detailed classifications of moisture in a sludge.

When water is "bound," the chemical potential should be different from that in the bulk water (Herwijn *et al.*, 1992). Heulekeian and Weinberg (1956) defined the bound water as the water content that does not freeze at temperatures below the freezing point. Various methods for measuring the bound water content have been proposed following the same principle. Some among others are as follows: differential thermal analysis (DTA) (Katsiris and Kouzeli-Katsiri, 1987), differential scanning calorimetry (DSC) (Lee *et al.*, 1975), water vapor sorption isotherms (Herwijn *et al.*, 1992), nuclear magnetic resonance (NMR) (Haschemeyer *et al.*, 1977; Sato *et al.*, 1980, 1982) and suction pressure (Lewicki *et al.*, 1978). Drying test has been used to measure bound water content because it is believed that the resistance to evaporation should be larger for bound water (Sato *et al.*, 1982; Smollen, 1990; Tsang and Vesilind, 1990; Matsuda *et al.*, 1992; Robinson and Knocke, 1992). Herwijn *et al.* (1992) proposed that the water content with bond enthalpy  $>1$  kJ/kg is referred as the bound water.

Matsuda *et al.* (1992) defined the "bound water" of an activated sludge as the internal water and the adhered surface water which are difficult to remove. Therefore, centrifugal settling method had been proposed to find the "wet solid," which contains only the solid phase and the bound water (Kawasaki *et al.*, 1990b; Matsuda *et al.*, 1992). Expression tests are frequently used in characterizing sludge dewaterability (Kawasaki *et al.*, 1990a). Following the same reasoning, the moisture content that cannot be removed by increasing applied pressure in an expression test should also in some sense be the bound water.

Although all these techniques had been successfully for estimating the bound water content, it was argued that the bound water is merely an operationally defined value depending on the measurement methods (Robinson and Knocke, 1992). In this work, drying, DSC, centrifugal settling, and expression tests are employed for measuring the bound water content in sludges made of  $\text{CaCO}_3$ , polyvinyl chloride (PVC), glass powders of various diameters, an activated sludge, and an inorganic  $\text{Cu}(\text{OH})_2$  sludge. The bound water data obtained are compared and a way to differentiate moisture distribution in a sludge is proposed.

## Measurement of bound water in a sludge

The tests used in this work can only divide the water content into two parts. The term "bound water," therefore, is used solely to represent the portion of water different from the bulk water phase in the sense of that measurement method.

**Drying Test.** A typical drying rate versus moisture content curve following Vesilind and Martel's interpretation is illustrated schematically in Figure 1. When a porous media is dried in a constant temperature/humidity environment, the drying curve can be distinguished into the constant rate period (curve AB), the first falling rate period (curve BC), the second falling rate period (curve CD), and the equilibrium stage (curve DE) (McCabe *et al.*, 1985).

In Tsang and Vesilind (1990), the points B, C, and D in Figure 1 are taken as the transition points between free, interstitial, surface, and bound water, that is, the moisture content in equilibrium with the surroundings is defined as the "bound water" ( $W_b$  in Figure 1). Smollen (1990) classified the water content in sludges into free, immobilized, (physically) bound, and chemically bound water. From his paper the so-called physically bound water is the sum of interstitial and surface water, and the chemically bound water is the bound water in Vesilind and Martel (1990). It is also noted that the bound water defined in Sato *et al.* (1982), Robinson and Knocke (1992), and Matsuda *et al.* (1992) is actually the sum of the interstitial, surface, and bound water in Tsang and Vesilind's interpretation. The definition of bound water between works employing drying technique varies from case to case.

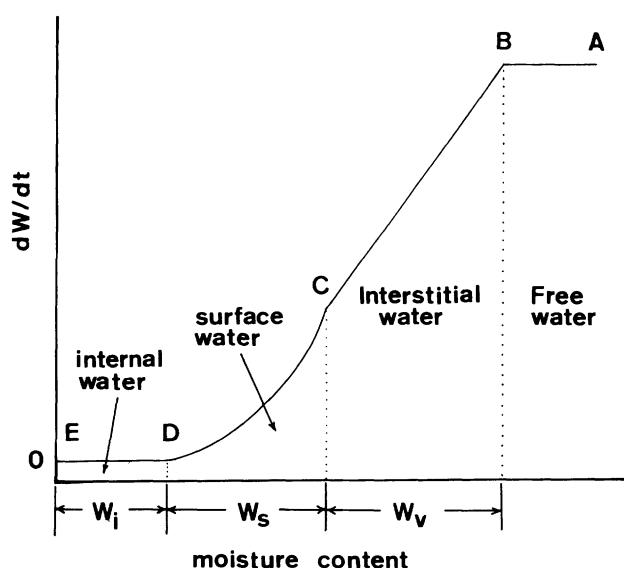


Figure 1—Drying rate vs moisture content plot when treating a porous media.

The drying process for a packed bed composed of porous particles can be simply interpreted as follows if the moisture diffusion resistance within the cake can be neglected. In the constant rate period, the evaporation rate for the free and interstitial water, whose properties are the same as those in bulk water, is a constant when the surrounding temperature/humidity is fixed. When drying continues and when the water on the particle surface cannot form a continuous film (which reduces the effective mass transfer area) and/or the capillary suction mechanism cannot provide enough water from the interior of the particle to the surface (which reduces the water supply rate), the evaporation rate decreases and the process enters the first falling rate period. If the material is dried further, surface water exhaust and the evaporation rate is controlled by moisture diffusion within the particles. The process then enters the second falling rate period. Finally, the moisture becomes in equilibrium with the surroundings, which makes evaporation rate zero (McCabe *et al.*, 1985). Note that the surface water content in this sense is equal to  $W_s + W_v$  in Figure 1.

**DSC test.** The assumption behind a DSC test is the bound water will not freeze at temperatures below, for example,  $-20^\circ\text{C}$  (Katsiris and Kouzeli-Katsiri, 1987). The enthalpy absorbed during freezing is therefore proportional to the free water amount and the bound water content can be calculated via simple mass and energy balance (Lee *et al.*, 1975; Katsiris and Kouzeli-Katsiris, 1987).

However, because the sampling amount in a DSC test is usually small ( $<10$  mg), the potential difficulty is the sample uniformity. If the sludge contains large particles or flocs, which is usually the case in many industrial/biological sludges, the data might deviate greatly.

**Centrifugal settling test.** The centrifugal settling method is originally utilized for measuring the “packed cell volume” by extrapolating the sludge sediment height to  $N \rightarrow \infty$  limit (Mitani *et al.*, 1983). The equilibrium sediment height for an activated sludge is found to vary linearly with  $N^{-1}$  when rotational speed ranges from 500 to 3500 rpm (Mitani *et al.*, 1983; Kawasaki *et al.*, 1990b).

The porosity-compressive pressure relationship for a compressible cake is proposed as follows (Tiller, 1955, 1962; Murase *et al.*, 1989):

$$1 - \epsilon = EP_s^\beta \quad P_s \geq P_{si} \quad (1a)$$

$$1 - \epsilon = EP_{si}^\beta \quad P_s \leq P_{si} \quad (1b)$$

or

$$e = e_0 - C_c \ln P_s \quad P_s \geq P_{si} \quad (2a)$$

$$e = e_0 - C_c \ln P_{si} \quad P_s \leq P_{si} \quad (2a)$$

where  $P_{si}$  is a small value. By assuming the arm length is much larger than the sediment height, the following equation can be obtained by combining the force balance equation and Equation 1 as (Murase *et al.*, 1989):

$$h = C_1 \Omega^{-2\beta} \quad (3a)$$

where

$$C_1 = \frac{\omega_0^{1-\beta}}{E(1-\beta)} [(\rho_s - \rho_L)R]^{-\beta}$$

When the force balance equation is combined with Equation 2, the result reads:

$$h = C_2 + C_3 \ln \Omega \quad (3b)$$

where

$$C_2 = (1 + e_0 + C_c)\omega_0 - C_c\omega_0 \ln [(\rho_s - \rho_L)\omega_0 R]$$

$$C_3 = -2C_c\omega_0$$

$\omega_0$  is the equilibrium sediment height at infinite rotational speed searched for (Kawasaki *et al.*, 1990b) and can be found from the linear relationship between sediment height and  $N^{-1}$ . Murase *et al.* (1989) found that Equations 3a and 3b work well with two inorganic sludges whose solid-phase volume data are known.

Because the centrifugal force at  $N \rightarrow \infty$  limit should be infinitely large and because bound water is recognized as the portion of moisture hard to remove via mechanical means, it is proposed that the sediment in equilibrium with infinite rotational speed should contain only the solid phase and the bound water (Mitani *et al.*, 1983; Kawasaki *et al.*, 1990b). By simple mass balance, the bound water content can be estimated with help of the bulk sludge density and the solid-phase density (Matsuda *et al.*, 1992). However, as discussed in detail later, linear equilibrium sediment height versus  $N^{-1}$  relation does not exist in tests with highly compressible sludges, such as activated sludge or  $\text{Cu}(\text{OH})_2$  sludge. If  $\omega_0$  cannot be found *a priori*, the three unknowns in Equations 3a ( $\beta$ ,  $E$ , and  $\omega_0$ ) or 3b ( $e_0$ ,  $C_c$ , and  $\omega_0$ ) can only be possibly obtained from nonlinear regression analysis.

**Expression test.** In an expression test, the residual water in equilibrium with various applied pressures can be found. Kawasaki *et al.* (1991) had combined the porosity-compressive pressure data from gravity settling and centrifugal settling tests to estimate the undetermined coefficients in Equation 2(a). If Equations 1 or 2 are universally valid, the combined expression/centrifugal settling test might also be able to estimate the undetermined coefficients in these equations. This point will be checked experimentally.

## Experimental sections

**The samples.** Sludge with nonporous particles was prepared by adding fixed amount of  $\text{CaCO}_3$ , PVC powders, or glass spheres

**Table 1—The characteristics of nonporous materials tested.**

Sludge	$D_p$ , $\mu\text{m}$	$\rho_s$ , $\text{kg/m}^3$
$\text{CaCO}_3$	21	2 746
PVC	350	1 429
Gla I	500	2 481
Gla II	230	2 481
Gla III	33	2 481
Gla IV	10	2 481
Gla V	7.1	1 097

For  $\text{CaCO}_3$  and glass III to V powders,  $D_p$  is Stoke's diameters from Sedigraph test; for PVC and glass I and II powders,  $D_p$  is the mean diameter from microscopic observations. Gla: glass powders.

into a beaker stirred vigorously with a magnetic stirrer. The particle size distribution was measured by Sedigraph 5100C (Micromeritics) or an optical microscope (Projectina). The true density of the powders was measured by a pycnometer (AccuPyc). The characteristics of these sludges are listed in Table 1. Microscopic observation and the mercury penetration test showed that these powders are basically nonporous, and no internal water existed in these sludges by definition.

An inorganic  $\text{Cu}(\text{OH})_2$  sludge was prepared by adding 50 g  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  and 18 g NaOH into 950 g deionized water. The weight percentage of the solid phase of the sludge was estimated as 1.83% and used within 1 hour to prevent quality change.

The activated sludge sample was taken from the wastewater treatment plant in Hsinpu Fiber Plant, Far Eastern Textile Ltd., Hsinchu, Taiwan, and tested within 2 hours after sampling. The biochemical oxygen demand (BOD), chemical oxygen demand (COD), and suspended solids (SS) of the sludge were, respectively 15, 84.4, and 17 mg/L, and the weight percent was measured as 0.558%. Density measurements gave  $\rho_s = 1450 \text{ kg/m}^3$  and  $\rho_b = 1005 \text{ kg/m}^3$ .

The sludge samples for drying and DSC tests were first vacuumed filtrated and the resulting cake was pressed firmly to remove most of the free water. Samples were taken from the resulting cake. At least three runs for each experimental condition were conducted to check the reproducibility. The total water content for each sample was measured by drying under  $102^\circ\text{C}$ .

**Bound water measurements.** A digitally controlled centrifuge (Kubota 2100) with arm length of 13.5 cm served for the centrifugal settling tests. The rotational speed ranges from 0 to 4000 rpm, and time of the test was set to 1 hour because the sediment reached its equilibrium height within about 0.5 hour. Four tubes of a sludge are measured simultaneously and the equilibrium heights are recorded under various rotational speeds. A digital density meter is used to measure the bulk sludge density with accuracy up to 0.0001 g/mL.

A differential thermal analyzer (DuPont model 2000) equipped with a scanning calorimeter cell is employed for recording the thermograms of samples. The temperature is first decreased at a rate  $-10^\circ\text{C}/\text{min}$  to  $-60^\circ\text{C}$ , and then increased back to the room temperature at the same rate. Sample freezing mainly occurs during  $-5$  to  $-15^\circ\text{C}$ , and the amount of heat absorbed from 0 to  $-20^\circ\text{C}$  is used to measure free water content. The bound water can then be subsequently obtained.

A constant temperature/humidity drying apparatus is em-

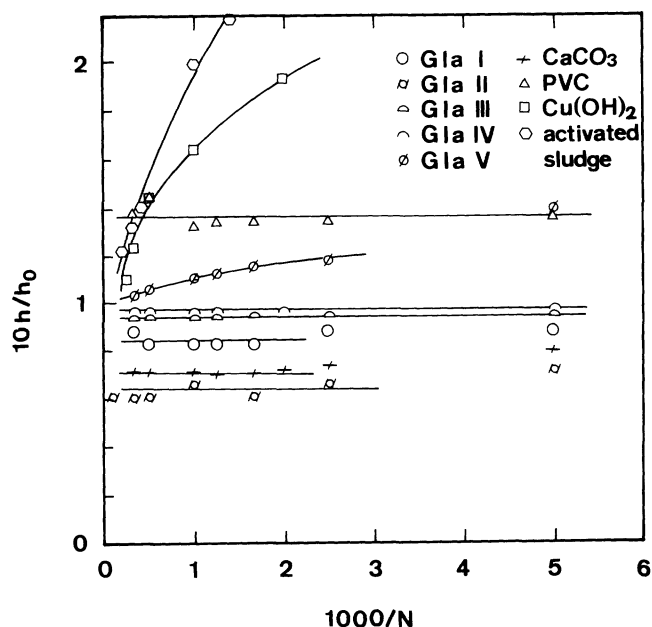
ployed for the drying test. The drying temperature is kept at  $40^\circ\text{C}$  and the humidity is kept at about 60%. An electronic balance connecting to a personal computer is employed for automatically recording the sample weight during an experiment. A typical test takes about 24 hours for the system to attain to the equilibrium state. The residue water content remained in the cake is determined by further drying the cake at  $102^\circ\text{C}$ . It is found, however, that the amount of the residue water content is usually negligible. The sludge thickness is fixed as 1 mm for the sake of comparison and to minimize the moisture diffusion resistance within the sludge cake.

A constant head piston press (Triton Electronics Ltd., type 147) is employed for finding the moisture content in sludge that is in equilibrium with the applied pressure. The applied pressure ranges from 500 to 4500 psi.

## Results and discussions

**Centrifugal settling test.** The results of centrifugal settling tests for  $\text{CaCO}_3$ , PVC, glass powders,  $\text{Cu}(\text{OH})_2$  sludge, and the activated sludge are summarized in Figure 2. The equilibrium height for  $\text{CaCO}_3$  and glass powders attain to a constant when the rotational speed is larger than about 300 rpm. Because the sediment height does not decrease when rotational speed increases up to 4000 rpm, this constant height is  $h_0$  by definition. The case with PVC powders shows gradual decrease of equilibrium height with increasing rotational speed; however, the  $h/h_0$  versus  $1/N$  curve is linear and the equilibrium height at  $N \rightarrow \infty$  limit can be easily determined. The bound water content can then be calculated and are listed in Table 2.

The  $h/h_0$  versus  $1/N$  data for  $\text{Cu}(\text{OH})_2$  sludge and activated sludge are quite different. Nonlinear relation exists especially



**Figure 2—Experimental results for centrifugal settling tests.  $\text{CaCO}_3$  sludge, 5% w/w; PVC sludge, 6.75% w/w; Gla I: glass powder, 500  $\mu\text{m}$ , 8.6% w/w; Gla II: glass powder, 233  $\mu\text{m}$ , 4.4% w/w; Gla III: glass powder, 33  $\mu\text{m}$ , 7.8% w/w; Gla IV: glass powder, 10  $\mu\text{m}$ , 9.2% w/w; Gla V: glass powder, 7  $\mu\text{m}$ , 10% w/w;  $\text{Cu}(\text{OH})_2$  sludge, 1.83% w/w; activated sludge, 0.558% w/w.**

Table 2—Bound water contents measured via centrifugal settling (CS), expression (EX), drying test (Drying), and DSC tests (DSC).

Sludge	CS, kg/kg	EX, kg/kg	Drying, kg/kg	DSC, kg/kg	W <sub>v</sub> , kg/kg	W <sub>s</sub> , kg/kg	W <sub>i</sub> , kg/kg
CaCO <sub>3</sub>	1.03	0.02	0.31	0.28	0.72	0.29	0.02
PVC	1.15	0.03	0.09	0.08	1.06	0.06	0.03
Gla I	0.56	0.001	0.013	×	0.55	0.013	0.01
Gla II	0.58	×	0.018	×	0.56	0.018	0.0
Gla III	0.54	×	0.027	×	0.51	0.027	0.0
Gla IV	0.61	×	0.036	0.041	0.57	0.036	0.0
Gla V	0.60	×	0.047	0.056	0.55	0.047	0.0
Cu(OH) <sub>2</sub>	×	0.44	1.86	×	×	1.42	0.44
Activated sludge	×	3.85	6.70	×	×	2.85	3.85

The bound water contents are in kg-bound water/kg-dry solid. Gla: glass powders. The symbol × indicates that the data is unavailable or the deviation is too large. W<sub>v</sub>, W<sub>s</sub>, and W<sub>i</sub> are interstitial, surface, and internal water content, respectively.

when the rotational speed is high. In these cases, the method for estimating the bound water proposed by Matsuda *et al.* (1992) fails.

Figures 3a and 3b show the centrifugal settling data in a log-log scale and in a semi-log scale, respectively. It is clear that when rotational speed is high, linear relation exists in both scales. The correlation coefficients for linear regression analysis are all higher than 0.99 and the best-fitted β's from linear regression analysis (equation 2a) are listed in Table 3.

β is an index for the cake compressibility. For CaCO<sub>3</sub> and glass powders of large diameters, the β's are essentially zero. Those with PVC powders and glass spheres of 7 μm in diameter are small positive values, which indicates an incompressible or slightly compressible cake. On the other hand, the β values for Cu(OH)<sub>2</sub> sludge and the activated sludge are 0.119 and 0.175, respectively, indicating two highly compressible sludges, the ac-

tivated sludge being more compressible than the Cu(OH)<sub>2</sub> sludge. Other parameters in equations 3a or 3b, including the “solid phase” volume ω<sub>0</sub>, cannot be determined uniquely except further information can be obtained.

The use of nonlinear regression analysis for estimating these parameters from experimental data is also proved inadequately. For a specific sludge, because the linearity of the data shown in Figure 3a is quite well, the “best” ω<sub>0</sub><sup>1-β</sup>/E is a constant. The “best” β value is very close to the value from the slope of linear regression analysis and is also a constant. There exists therefore infinite sets of (ω<sub>0</sub>, E) pairs that minimize the sum of the squares of errors. A simplex analysis has confirmed this point.

Therefore, for highly compressible sludges, the information provided by centrifugal settling method is not enough to determine all the parameters in equations 3a or 3b, and the bound water content cannot be uniquely determined.

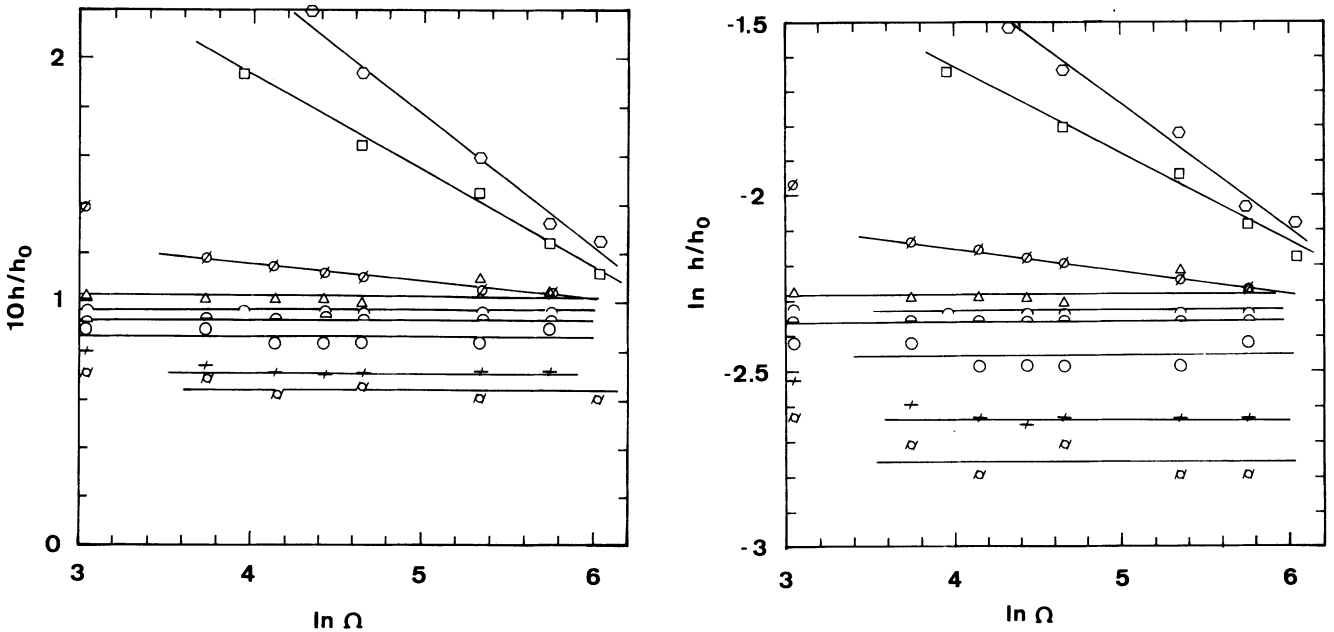


Figure 3—Experimental results for centrifugal settling tests in a log-log representation (left) and a semi-log representation (right). All symbols are defined as in Fig. 2.

Table 3— $\beta$ 's measured from centrifugal settling and expression test.

Sludge	CS	EX
CaCO <sub>3</sub>	0.0	0.073
PVC	0.015	0.091
Gal I	0.0	0.0
Cu(OH) <sub>2</sub>	0.119	0.185
Activated sludge	0.175	0.385

CS: centrifugal settling test; EX: expression test.

**Expression test.** A typical expression test for Cu(OH)<sub>2</sub> sludge is shown in Figure 4. The basic features for the expression tests with other sludges are similar and will not be illustrated here.

The experiment starts by applying 500 psi pressure onto a piston that presses the sludge to force the filtrate out. During this stage, the operation is a constant pressure filtration process. After the slurry has exhausted and the piston touches the cake, the operation enters the expression stage. The water is continuously removed until equilibrium is attained. Under 500 psi, the total removal amount of water is 523.6 g. Further increase the pressure to 1000 psi will force another 0.6 g water out of the cake. The pressure is then increased again. It is found that no water content can be removed when the applied pressure ranges from about 3000 to 4500 psi. The resulting cake is then dried at 102°C to find the final moisture content.

In several expression tests, some water still can be removed even when the applied pressure is as high as 4500 psi. The water removal amount decreases with increasing applied pressure, from which the final moisture content can be estimated by slight extrapolation.

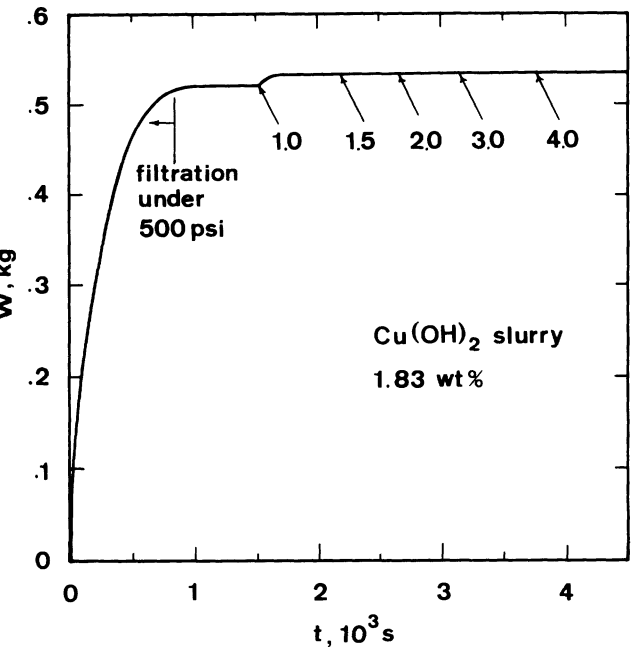


Figure 4—Expression test for Cu(OH)<sub>2</sub> sludge. Numerical values are the applied pressures in kpsi. The arrows indicate where the applied pressure changed. The initial applied pressure is 500 psi.

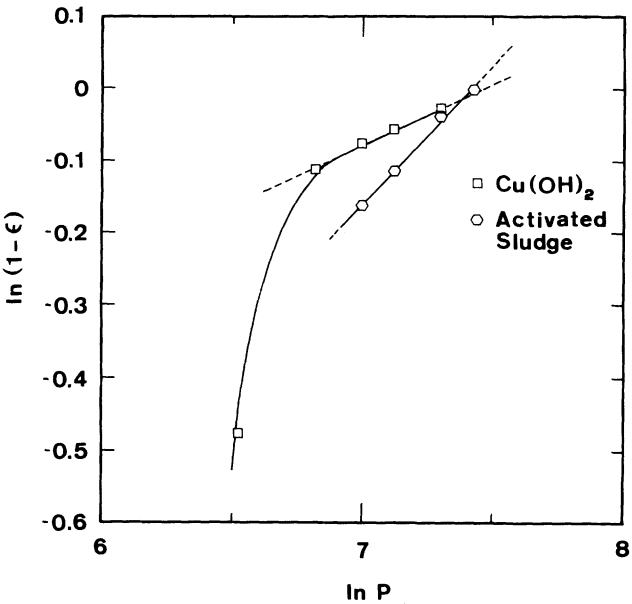


Figure 5—Porosity vs. compressive pressure. Cu(OH)<sub>2</sub> sludge and activated sludge. Cu(OH)<sub>2</sub>: 1.83% w/w; activated sludge: 0.558% w/w.

The final moisture content data (the bound water) are listed in Table 2. The tests with glass powders will cause the particles to crush; therefore, only the case with diameter 500  $\mu$ m is discussed further.

The porosity of sludge can be estimated from the water moisture in equilibrium with the applied pressure. Figure 5 shows the results for Cu(OH)<sub>2</sub> and for activated sludge. Linear relation exists and the coefficients in equation 1a can be estimated via linear regression analysis. The  $\beta$ 's are listed in Table 3 and are all larger than those from centrifugal settling test, indicating the  $\beta$  and  $E$ , or  $C_c$  and  $e_0$  data obtained from expression test cannot be used as the input data that are required in the centrifugal settling tests. The reason for the inconsistency might come from the different force field provided by these two tests, or simply that a unique empirical correlation can hardly describe the porosity variation under such a wide pressure range, or both.

**Drying test.** Typical drying curves are shown in Figure 6. Transitions from constant rate to falling rate period can be detected in all cases, with finite uncertainty (Smollen, 1990; Robinson and Knocke, 1992). Further classifying the falling rate period into the first and the second period as had been done in Tsang and Vesilind (1990), however, is impractical in this work. The moisture content at the transition point is the bound water as defined in Smollen (1990), Matsuda *et al.* (1992), and Robinson and Knocke (1992) and is listed in Table 2.

For the Cu(OH)<sub>2</sub> and the activated sludge, because there exists large amount of surface and internal water, the dimensionless weight at transition point  $W_{tr}$  is higher than those for nonporous materials. The data are also listed in Table 2.

**DSC test.** The reproducibility for the DSC tests with CaCO<sub>3</sub> and glass powders of small diameters (7 and 10  $\mu$ m) is fair. Nevertheless, the deviation for glass powders of large diameters, Cu(OH)<sub>2</sub> sludge, and activated sludge is often too large to make meaningful data analysis. Vigorous mixing the sludge can only gain slight improvement. The reason is believed as the sample

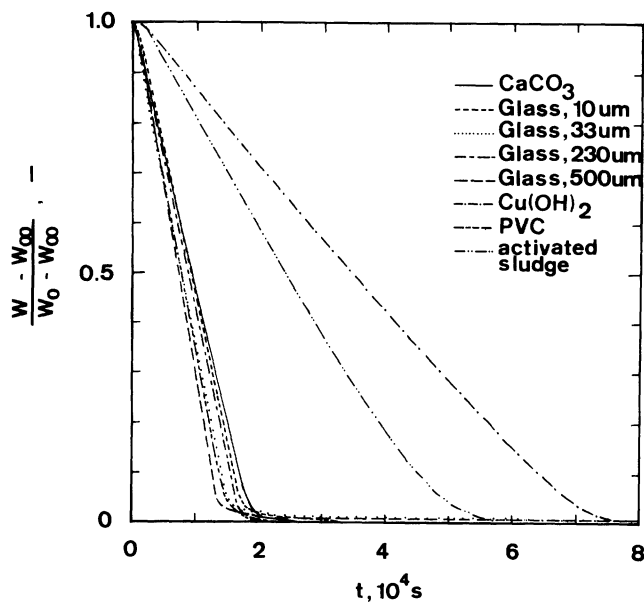


Figure 6—Drying tests.  $W_0$  and  $W_\infty$  are the initial weight and the equilibrium weight of sludge.

nonuniformity and the DSC test is therefore inadequate for measuring the bound water content of sludges except the sample uniformity can be guaranteed.

The available bound water data are listed in Table 2. Though the data are limited, it is noted that the bound water is close to the data from drying test, indicating the portion of water in a sludge exhibits a large resistance to evaporation also keeps unfrozen below  $-20^\circ\text{C}$ .

**Bound water contents.** Table 2 summarizes the bound water data measured via the four measurement methods. Clearly the bound water content is indeed an operationally defined value depending on the measurement techniques. Based on this information and some physical interpretations, a classification of the moisture among the sludge can be constructed accordingly. In the present study, just for the sake of convenience, the terms “free water,” “interstitial water,” “surface water,” and “internal water” as those proposed in Tsang and Vesilind (1990) are used, with different definitions. The moisture distribution in highly compressible sludges is much more complicated than the nonporous cases; however, certain characteristics can be highlighted from tests with nonporous materials.

Consider a sludge made of rigid particles. Finite void space always exists between these particles in centrifugal settling tests under high rotational speed. The water content within the cake (the bound water) is the sum of interstitial water, surface water, and internal water (zero for nonporous particles by definition). In an expression test, the bound water content for nonporous materials is close to zero, which implies that the bound water measured via expression test is practically the internal water. The bound water from drying tests and DSC tests (if available) are close to each other and are located between centrifugal settling and expression tests. From simple interpretation in previous section the transition between constant rate and first falling rate period signals the exhaust of thin water film on the particles, the bound water found from a drying test (and also DSC test) is interpreted as the sum of surface and internal water. With

such a classification, the moisture distribution among a sludge can be constructed with help of the bound water contents measured via various techniques. The calculated results are listed in Table 2.

From Table 2, the internal water for nonporous powders are near zero, which is consistent with the mercury penetration test. For  $\text{CaCO}_3$ , PVC, and glass powders, the void fraction calculated from the interstitial water content and the solid-phase density is 0.66, 0.58, and 0.60, respectively, which are reasonable values in cakes formed by rigid particles.

To get more insight into the surface water defined above, Figure 7 demonstrated the normalized weight at transition point found by drying glass powders of various diameters with the same solid volume and cake thickness. If the surface water is indeed a thin water film distributed uniformly all over the particle surfaces, the amount of surface water should be proportional to the specific area of the powders, or the inverse of the particle diameter if the solid volume is fixed. However, the data in Figure 7 give the slope  $-0.25$  rather than  $-1.0$ . This might occur from the curvature effect of the water film, the capillary suction action in porous bed, or the transition point indicates not only the surface water but also some interstitial water that is located near the contact region of the particles. Another possibility is that the so-called “surface water” is merely the portion of water within the sludge whose evaporation resistance is large and cannot be interpreted simply as a layer of water attached on particle surface as proposed in Tsang and Vesilind (1990).

A natural extension is to classify the water content in highly compressible sludges in a similar way. Because centrifugal settling method fails in treating such sludges and because the deviation in DSC data is often too large, the only information available are the surface and internal water provided by the drying and expression tests. The data are all listed in Table 2. These results should be considered as of a preliminary nature, because the validity of extrapolating the experimental findings from non-

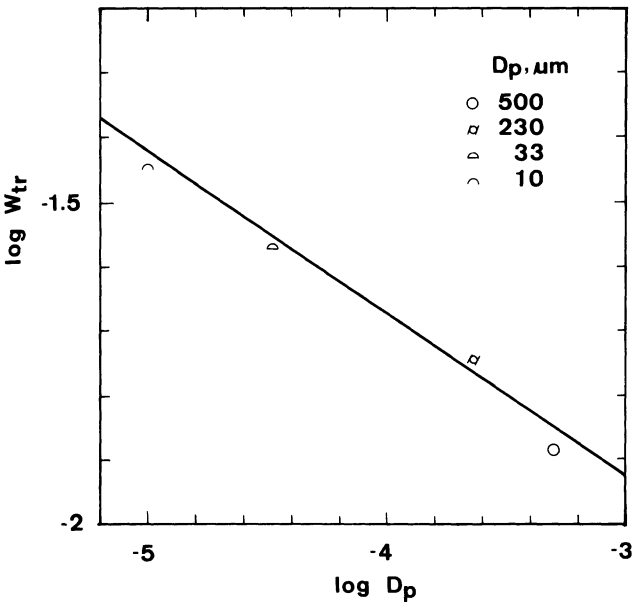


Figure 7— $\log W_{tr}$  vs.  $\log D_p$ . Drying tests. Glass powders.  $W_{tr}$  is the normalized water content at transition between constant rate and the falling rate period.

porous materials to highly compressible sludges is still in question.

Large amount of surface and internal water exist in  $\text{Cu}(\text{OH})_2$  and activated sludges. Note that both the internal and surface water content in activated sludge is the highest one among others, and the sum is close to the bound water content reported by Matsuda *et al.* (1992). Because the surface water can be forced out from sludge under very high pressure (for example, 4000 psi in expression test), it is the internal water that cannot be removed via any mechanical devices. Therefore, the upper limit for the solid concentration after a mechanical dewatering treatment is approximately 20 and 70% for the activated sludge and  $\text{Cu}(\text{OH})_2$  sludge employed in this work. The actual residual water in a dewatered cake is therefore mainly determined by the efficiency of surface water removal, which is affected by the design of dewatering device, cake compressibility, and others.

## Conclusions

The centrifugal settling, expression, DSC, and drying test are employed for measuring the bound water content in sludges made of  $\text{CaCO}_3$ , PVC, or glass powders, an inorganic  $\text{Cu}(\text{OH})_2$  sludge, and an activated sludge. The bound water evaluated from various method differs greatly and is an operationally defined value.

According to the bound water data for nonporous materials and some physical interpretations, the moisture in a sludge can be classified into free water, interstitial water, surface water, and internal water. The bound water from centrifugal settling test is interpreted as the sum of internal, surface, and interstitial water if the cake is incompressible or slightly compressible. Drying test gives the sum of surface and internal water. The DSC test are close to the drying test if sample uniformity can be guaranteed. Expression test gives the internal water content.

The results from nonporous materials had been extended to highly compressible sludges without proof. The DSC test fails for sample nonuniformity problem, and the centrifugal settling test can only provide the cake compressibility.

Large amount of internal and surface water exist in  $\text{Cu}(\text{OH})_2$  and activated sludge. The internal water is the upper limit for the performance of any mechanical dewatering device. The purpose of a dewatering process is therefore to remove as much surface water as possible.

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**Authors.** Dr. D. J. Lee is Associate Professor of Department of Chemical Engineering, National Taiwan University. Y. H. Hsu is currently working as an engineer for Hsinpu Fiber Plant, Far Eastern Textile Ltd., Hsinchu, Taiwan. At the time of this study, she was a Master student in the Department of Chemical Engineering, Yuan-Ze Institute of Technology, Taoyuan, Taiwan. Correspondence should be directed to Dr. D. J. Lee, Department of Chemical Engineering, National Taiwan University, Taipei, Taiwan, 10617, ROC.

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## Notations

$C_c$	coefficient defined in equation 1.
$C_1, C_2, C_3$	coefficients in equation 3.
$D_p$	diameter, m
$E$	coefficient defined in equation 1, $P\alpha^{-\beta}$
$e$	$= \epsilon/(1 - \epsilon)$
$e_0$	coefficient defined in equation 2
$h$	sediment height, m
$h_0$	initial sediment height, m
$N$	rotational speed, rpm
$P_s$	compressive pressure, Pa
$P_{si}$	reference pressure, Pa
$R$	arm length, cm
$W$	weight of drying cake, kg
$W_i$	internal water content, kg/kg solid
$W_s$	surface water content, kg/kg solid
$W_{tr}$	dimensionless weight of a drying cake at transition point between constant rate and first falling rate period,—
$W_v$	interstitial water content, kg/kg solid
$W_0$	initial weight of drying cake, kg
$W_\infty$	equilibrium weight of drying cake, kg

## Greek Letters

$\beta$	index defined in equation 1,—
$\epsilon$	porosity,—
$\rho_b$	bulk sludge density, $\text{kg/m}^3$
$\rho_L$	liquid density, $\text{kg/m}^3$
$\rho_s$	solid-phase density, $\text{kg/m}^3$
$\Omega$	rotational speed, rad/s
$\omega_0$	solid-phase volume per unit cross section, m

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