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Retention of Liquid by Solids in Suspensions and Sediments

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Abstract

The effects of associated immobile liquid on the settling behavior of particles in solid-liquid suspensions are investigated. The effects are studied with respect to such sedimentation parameters as suspension porosity ε, final sediment volume fraction v_s, internal void fraction of the flow unit v_i, Steinour's parameter A, and McKay's packing factor p which corrects for the effective volume and density of the flow units in a sedimenting suspension. It is shown that the degree of liquid association with the settling particles is a function of their physicochemical characteristics. Suspensions containing clays and carbonates, owing to their ionic nature and loose packing behavior, absorb large amounts of liquids and result in larger settled sediments of a compressible nature. On the other hand, systems like glass beads-liquid suspensions absorb little associated liquid and virtually result in noncompressible settled sediments due to their rigid spherical shapes and close packing. The addition of hydrocolloids (such as natural gums) provides ionic stability to suspensions by giving them low cmpressibility and low values of v_s, v_i, A, and p.

INTRODUCTION

The sediment behavior of solids in liquids and the characteristics of the resulting sediments are of importance in the chemical processing and mineral extraction industries. The suspensions formed at a high solid concentration tend to exhibit hindered settling, characterized by the formation of a definite sediment-supernatant boundary, the rate of fall of which is linear initially (linear zone), then decreases gradually (compression zone) until the final sediment volume is reached (stationary zone). The rate of fall of the boundary in the linear zone is dependent upon the concentration of the dispersion; an increase in the proportion of the dispersed phase causing a decrease in the rate of settling.

The first general study of suspensions in the flocculated state was carried out by Coe and Clavenger (*1*), and they clearly recognized the various concentration zones which exist within the settling suspensions. They described the upward flow of displaced supernatant as a special case of pore flow. Smellie and La Mer (*2*) studied flocculated clay suspensions and proposed an extrapolation procedure to estimate the ultimate volume of the settled sediment.

Gaudin, Fuerstenau, and Mitchell (*3*) used x-ray adsorption techniques to measure the local solid concentration in settling suspensions as a function of time and position. The density profiles so obtained are shown in Fig. 1 from which it is noted that the suspension settles as a plug of a uniform solid concentration equal to that of the initial suspension concentration. As the sedimentation proceeds, the concentration in the lower zones (presumably the compression zone and the stationary zone) increases, but the concentration in the linear zone remains constant until the whole suspension settles down to a final stationary sediment.

These observations were reinvestigated here for concentrated suspension for different solids in water. The solids used were clay, ground limestone, and calcium fluoride of Analar grade. Deionized water was used as the liquid medium. Suspensions of 200 cm^3 volume were prepared in 250 mL graduated open top cylinders. Samples of fixed volumes were withdrawn with a pipette (shown in Fig. 2) from varying heights of these suspensions after different intervals of time. The samples were then dried and weighed, and the concentration of solid calculated. The density profiles of the suspensions thus obtained are shown in Figs. 3a–3c, which show good agreement with the data put forward by Gaudin et al. (*3*).

Gaudin et al. assumed from their observations that during the early stages of sedimentation the displaced supernatant fluid leaves the bed through pores of comparatively large diameters, whereas in the later stages of sedimentation, such as in the compressive phase of sediment consolidations (*3*), the fluid is expelled via much smaller tubules.

The studies cited above represent an important contribution to the knowledge of relative movement of solids in liquids during settling and consolidation, but the part played by the liquid associated with sedimenting solid species in these phenomena is still not clear.

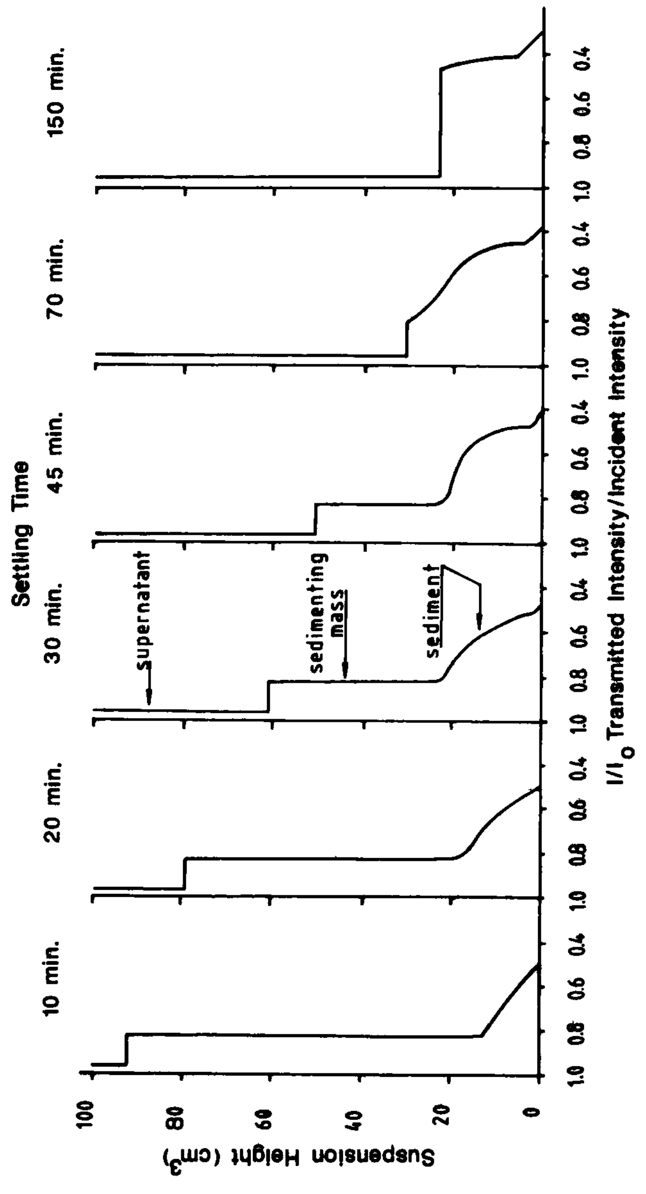

FIG. 1. Transmission of x-rays through sedimentary clay suspensions.

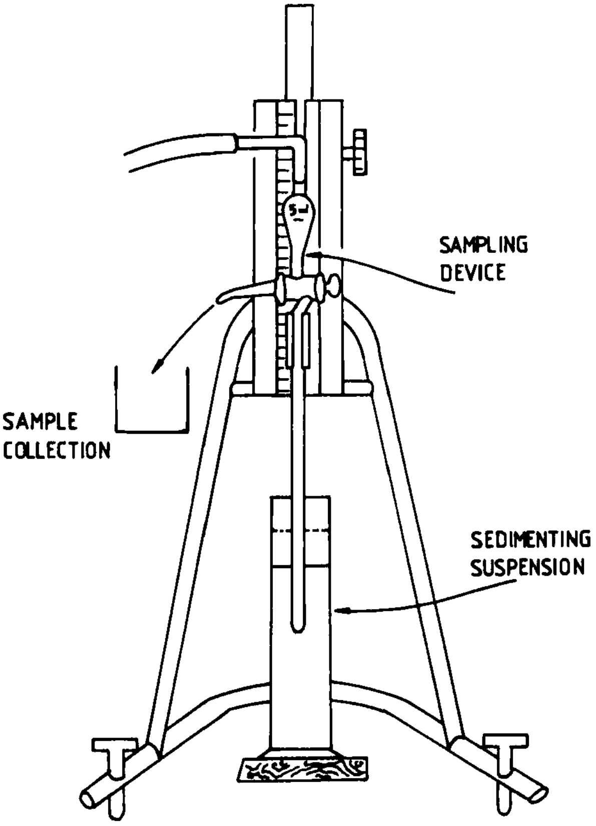

FIG. 2. Apparatus used for collecting samples of sedimenting suspensions at different intervals.

McKay (*4*) has termed the sedimenting solid species as "flow units" capable of carrying a large proportion of associated immobile liquid. Their effective volumes will be larger and their densities less than those of the solid itself, which would result in a final sediment volume greater than that predicted from the solid weight and density.

The association of immobile liquid with the settling flow units and the retention of liquid in the final sediment was estimated by McKay by using a modified empirical Steinour equation (*5*) which corrects for the effective volume and density of the flow units. He applied an appropriate packing factor p in Steinour's equation to account for the proportion of liquid in the sediment that is not associated with and immobilized by the flow units in the linear settling zone, e.g., liquids in voids between flow units packed in the sediment. The calculation of the effective density of the flow units was then made by allowing for the amount of liquid present within the flow units and between the flow units in the sediment. McKay derived the final form of equation to describe the movement of the interface as

$$Q = \frac{2gr^2(\rho_s - \rho_l)C(1 - pv_s)^2 r^2 \times 10^{-1.82pv_s}}{9\eta pv_s} \tag{1}$$

where Q = interface settling rate (cm/s)
g = acceleration due to gravity (cm/s^2)
r = radius of the particle (cm)
ρ_s = density of the particle (g/cm^3)
ρ_l = density of the liquid (g/cm^3)
C = solid volume fraction by volume
p = McKay's packing factor
v_s = final sediment volume fraction
η = viscosity of the liquid in poise (g · cm^{-1} · s^{-1})

McKay has used this equation in a series of experiments carried out on the sedimentation of organic pigments.

The calculations of p, however, are complex for the experiments in the present investigation. The main difficulty is the evaluation of ω_s which, according to McKay (*4*), is calculated from the following expressions:

$$v_i = \frac{pv_s - C}{pv_s} \tag{2}$$

and

$$pv_s = (1 - \varepsilon^*) \tag{3}$$

where ω_i = internal void volume fraction of the flow unit
ε^* = corrected value of liquid volume fraction allowing for ω_i

The evaluation of the correct values for ω_i and ε^* may be based on a wrong assumption however, and the present investigations have been carried out with model systems of glass beads–water, clay–water, and clay–water–gum suspensions. Evaluations of the parameters p, v_i, and v_s

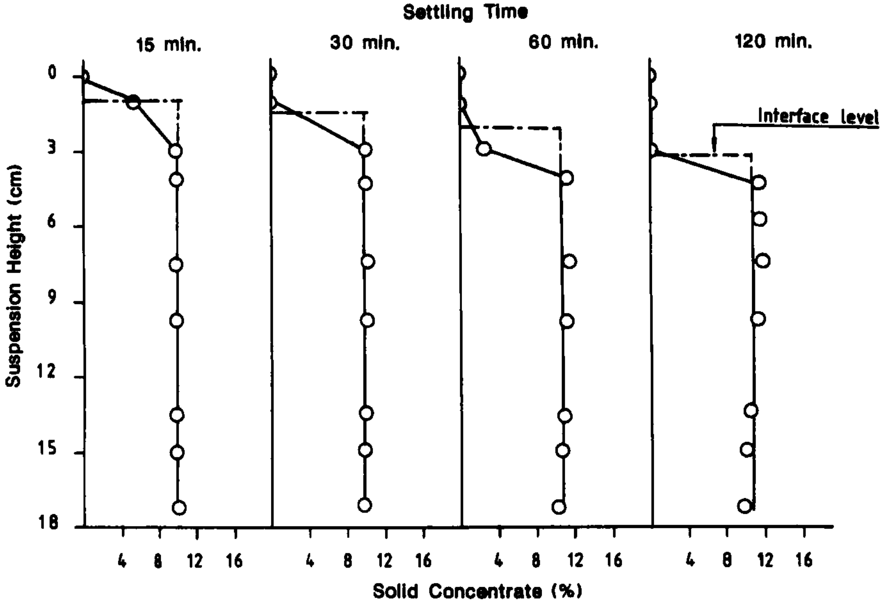

FIG. 3a. Concentration/suspension height plots at different time intervals for clay-water suspensions.

for these systems have been made, and their effects on the sedimentation behavior have been discussed.

EXPERIMENTAL

In the model system of glass beads and deionized water, the glass beads used were of the Grade I soda type with an approximate diameter range of 53-65 μm and a density of 2.50 g/cm^3. The sedimentation experiments were carried out in 225 mL graduated cylinders, and the suspension volume was fixed at 150 cm^3. A fixed amount of glass beads was added each time, and the suspension cylinder was inverted 20 times before being set down in front of a fluorescent light box. The corresponding interface settling measurements were made at appropriate time intervals, taking zero time as the instant when any initial turbulence had ceased. The final sediment volume was recorded wehn the settling ceased altogether.

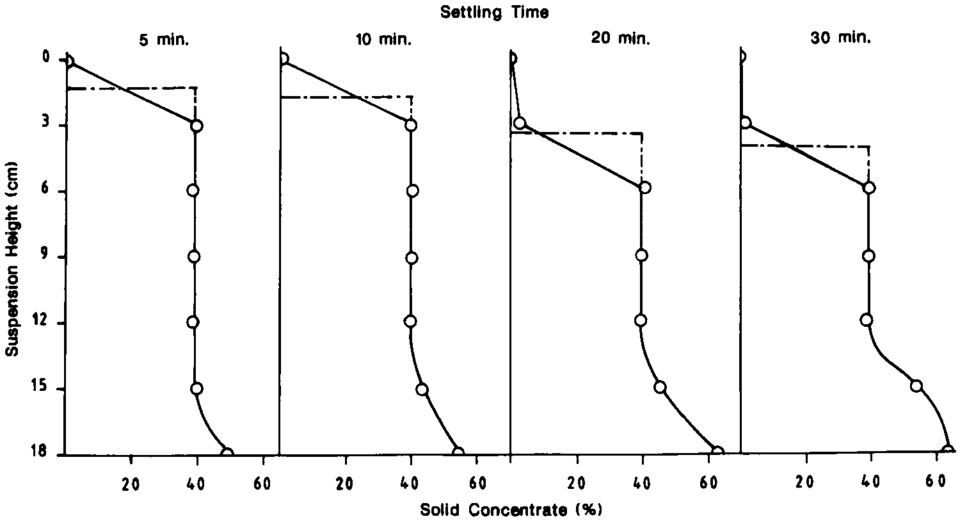

FIG. 3b. Concentration/suspension height plots at different time intervals for ground limestone-water suspensions.

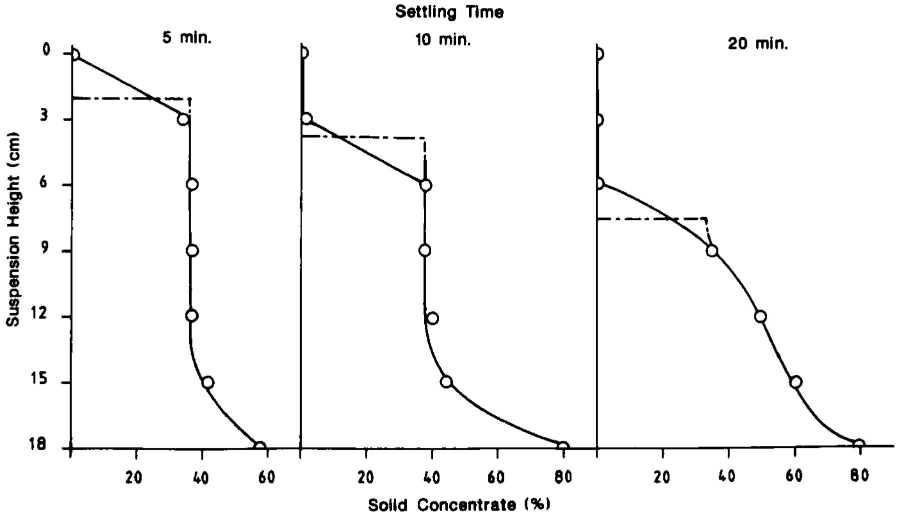

FIG. 3c. Concentration/suspension height plots at different time intervals for calcium fluoride-water suspensions.

TABLE 1
Chemical Analysis and Particle Size Distribution of Clay

Chemical analysis		Particle size distribution	
Compound	wt%	Size (μm)	wt%
SiO_2	46.20	+30	5.26
Al_2O_3	38.70	−30 + 20	8.40
Fe_2O_3	0.56	−20 + 10	6.26
TiO_2	0.09	−10 + 5	14.46
CaO	0.20	−5 + 2	14.87
MgO	0.20	−2	50.75
K_2O	1.01		
Na_2O	0.07		
Loss on ignition	13.14		

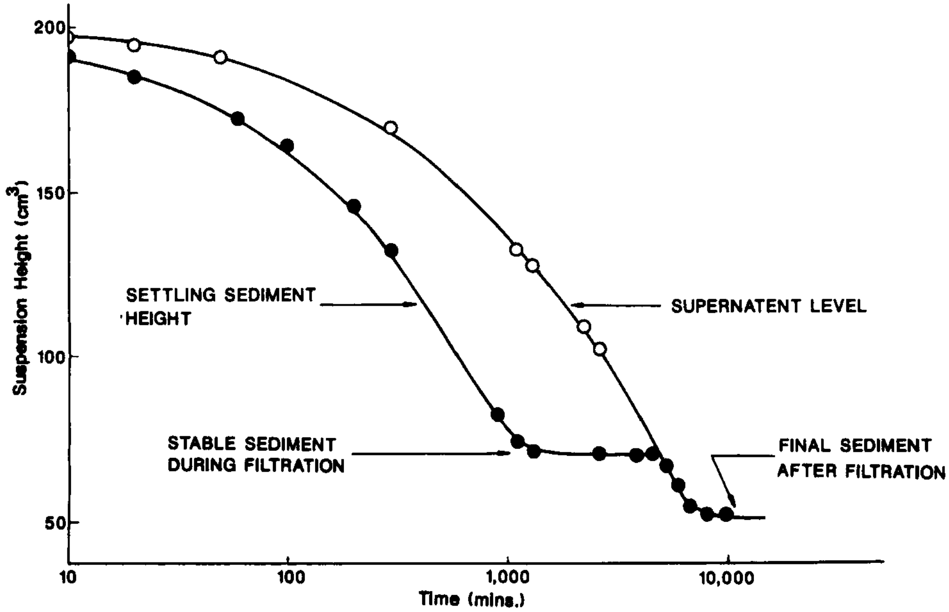

FIG. 4a. Simultaneous sedimentation and filtration plots for clay-water suspensions. (Bed is same as sediment.)

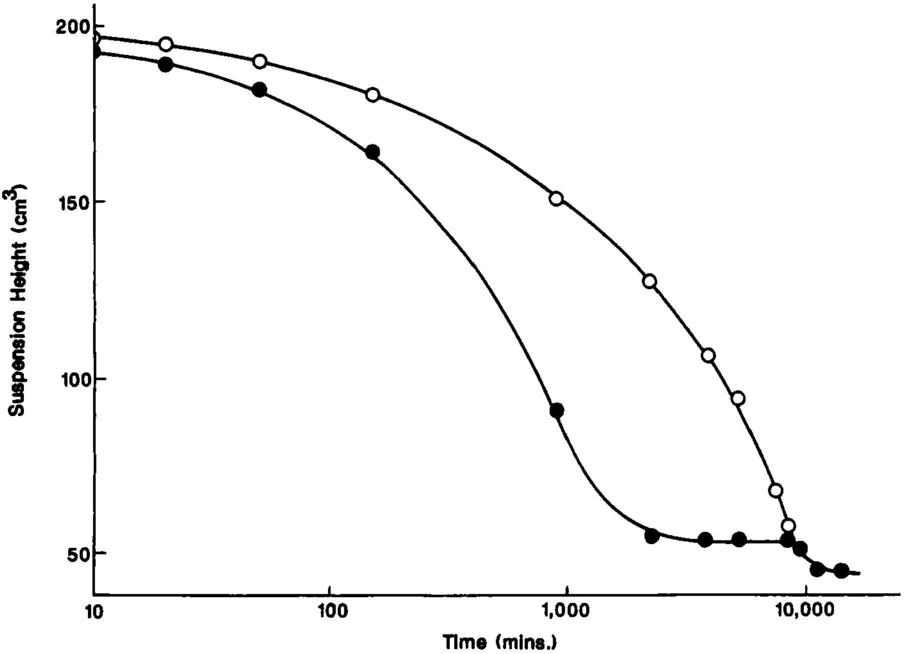

FIG. 4b. Simultaneous sedimentation and filtration plots for clay-water-gum tragacanth suspensions.

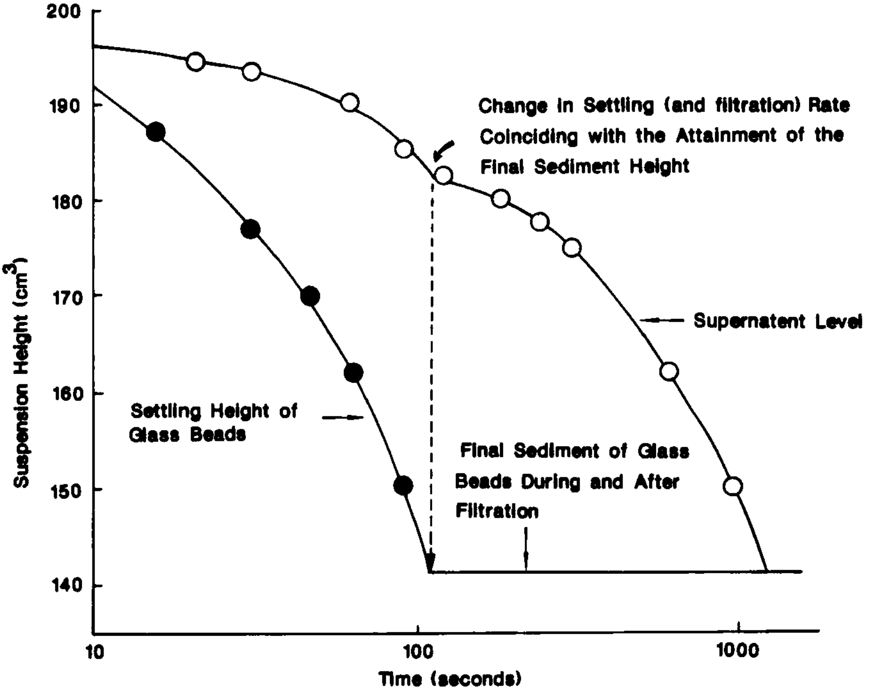

FIG. 4c. Simultaneous sedimentation and filtration plots for glass beads-water suspensions.

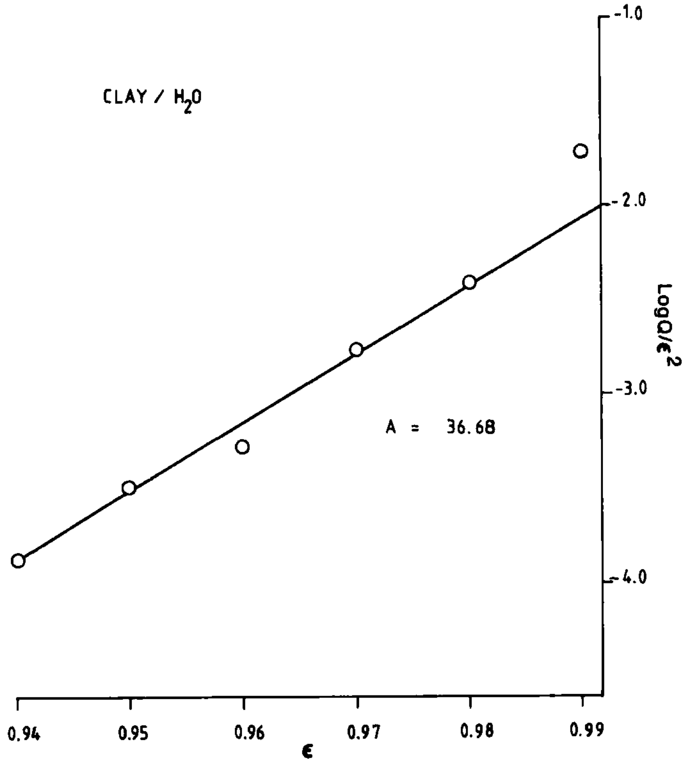

FIG. 5a. Measurement of parameter *A* for clay-water suspensions.

Experiments with clay-water and clay-water-gum suspensions were carried out similarly with the exception that the experiments were performed in 250 mL graduated cylinders and the suspension volumes were fixed at 200 cm^3. The gum was of persian gum tragacanth type R2 (PGTR2) and was provided by the Department of Chemistry, University of Isfahan, Iran. The chemical and particle size analysis of the clay is given in Table 1. In clay-water-gum tragacanth suspensions the concentration of the gum was kept at an optimum flocculation concentration of 80 ppm (by weight) of clay (*6*).

Filtration experiments on these suspensions were conducted in graduated cylindrical columns with perforated bottoms. Filter papers were placed at the bottoms of the columns, and thoroughly premixed suspensions were gently poured into the columns. The sedimentation and

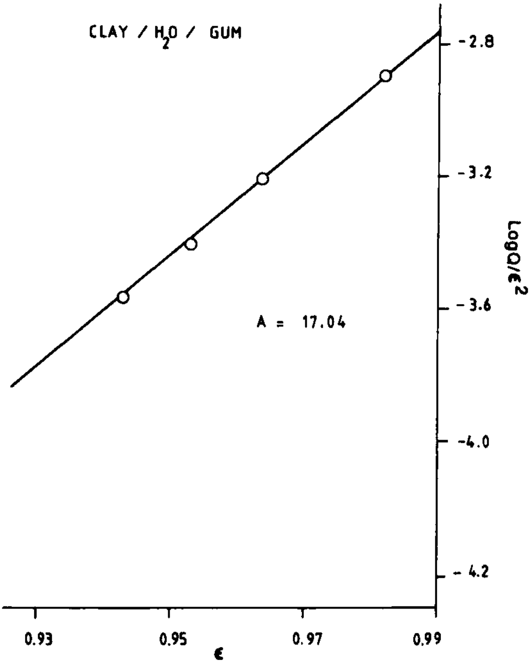

FIG. 5b. Measurement of parameter A for clay-water-gum tragacanth suspensions.

filtration observations were conducted immediately afterward. The settling of supernatant and sediment heights were recorded simultaneously; their plots against time are given in Figs. 4a–4c.

The solid concentrations in suspensions were converted to the solid volume fractions in C, which is also equivalent to $(1 - \varepsilon)$. The interface settling rate was designated as Q (cm/s). Table 2 gives the values of C, Q, and ω_s (the final sediment volume fraction) for the suspensions investigated in this report.

Steinour's A was calculated from the slopes of plots of log Q/ε^2 and ε for each suspension. The plots are given in Figs. 5a–5c.

McKay's p was calculated by solving pairs of McKay equations (typically as in Eq. 1) corresponding to different runs of the same set of experiments. For instance, with the first run of the experiment the equation is

$$Q_1 = \frac{V_s[1 - p(v_{s1})]^2 C_1 \times 10^{-1.82p(v_{s1})}}{pv_{s1}} \tag{4}$$

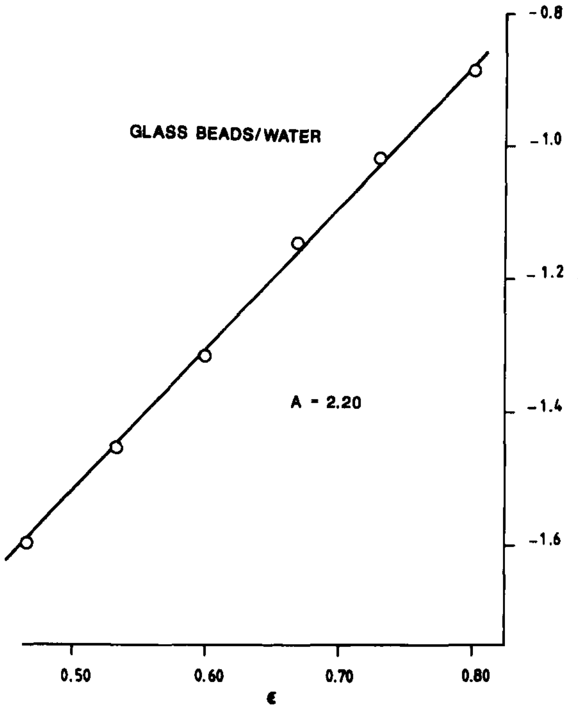

FIG. 5c. Measurement of parameter *A* for glass beads-water suspensions.

[where V_s (the Stokes terminal velocity) is $2gr^2(\rho_s - \rho_l)/9\eta)$ in Eq. 1], and for the second run is

$$Q_2 = \frac{V_s[1 - p(v_{s2})]^2 C_2 \times 10^{-1.82p(v_{s2})}}{p v_{s2}} \tag{5}$$

Therefore:

$$\frac{Q_1}{Q_2} = \frac{[1 - p(v_{s1})]^2}{[1 - p(v_{s2})]^2}\left(\frac{C_1}{C_2}\right)\left(\frac{v_{s2}}{v_{s1}}\right) \times 10^{1.82p(v_{s2} - v_{s1})} \tag{6}$$

which can be solved for *p*, assuming this is a constant. The two sides of Eq. (6) can be balanced by assigning various values to *p*. The value of *p* that gives the best fit to the equation should be the best value of *p*.

Values of *p* and ω_i calculated for varying concentrations of the suspensions under investigation are given in Table 3.

TABLE 2
Variation of Sediment Volume Fraction (υ_s) with Solid Concentration for Different Solid–Liquid Suspensions

Weight of glass beads (g)	Solid volume fraction of suspension $(1 - \varepsilon)$	Settling rate of suspension, Q (cm/s)	Sediment volume fraction (υ_s)	Weight of clay (g)	Solid volume fraction of suspension $(1 - \varepsilon)$	Setting rates Q (cm/s)		Sediment volume fraction, υ_s	
						Clay–H_2O suspension	Clay–H_2O–gum suspension	For clay–H_2O suspension	For clay–H_2O–gum suspension
75	0.20	0.084	0.34						
100	0.27	0.052	0.45	10	0.02	0.003	0.0012	0.25	0.24
125	0.33	0.032	0.56	15	0.03	0.001		0.35	
150	0.40	0.017	0.68	20	0.04	0.0004	0.0006	0.44	0.40
175	0.47	0.010	0.79	25	0.05	0.0002	0.0004	0.51	0.44
200	0.53	0.006	0.90	30	0.06	0.0001	0.0002	0.59	0.48

TABLE 3
Calculated Values of p and v_i for Different Solid–Liquid Suspensions (p calculated for pairs of suspensions as indicated)

Glass beads–water, $A = 2.2$				Clay–water, $A = 38.68$				Clay–water–gum tragacanth, $A = 17.04$			
$1 - \varepsilon$	v_i	p		$1 - \varepsilon$	v_i	p		$1 - \varepsilon$	v_i	p	
0.20	0.10			0.02	0.94			0.02	0.92		
		0.65				1.24					
0.27	0.09			0.03	0.94			0.03		0.83	
						1.52					
0.33	0.09			0.04	0.93		1.27	0.04	0.90		1.00
		0.67	0.64			1.08					
0.40	0.10			0.05	0.93			0.05	0.89		
						1.25				1.05	
0.47	0.09			0.06	0.92			0.06	0.87		
		0.63									
0.53	0.09										
Average p values:		0.65				1.27				0.96	

DISCUSSION

Large flow units which form due to flocculation settle quickly provided their size with respect to the container diameter is small. For a very large flow unit the settling rate may fall due to the tortuous nature of the upward fluid flow. For smaller flow units, however, the settling behavior would be less tortuous and would probably be controlled by the liquid associated with the flow units. The amount of liquid content associated with flow units could be estimated from the values of parameters, like Steinour's A, McKay's p, and v_i, mentioned previously. It seems that McKay's p in his modified Eq. (1) corrects for deviation of Steinour's A (5) from the value of 1.82 for different suspension systems. Therefore, a large p corresponds to a large A in Steinour's equation:

$$Q = V_s \varepsilon^2 \times 10^{-A(1-\varepsilon)} \tag{7}$$

and would correspond to a relatively large amount of associated liquid. This could lead to greater hindrance because:

(a) Particles would be effectively made larger (by associated liquid) with less free liquid to flow upward

(b) The effective particle would be correspondingly less dense and would thus settle more slowly under gravity

This can be verified from the values of p calculated for different suspensions shown in Table 3. It should be noted that the value of p for glass beads–water suspensions is smaller than that for clay–water suspensions. This clearly indicates that the amount of liquid associated with the flow units in the clay–water suspensions is much larger than that for the glass beads–water suspensions, which would correspond to an expected larger value of v_i for the clay–water suspensions than for the glass beads–water suspensions. Large amounts of associated liquid result in low floc densities, and the suspensions would exhibit low settling rates, and vice versa. This seems obvious for the suspensions in the present investigation, as shown in Table 2. The effect of gum tragacanth on the settling behavior of the flow units in the system has been shown by its tendency to adsorb onto the ionic surface of clay platelets which could cause reduction in the amount of liquid associated with the flow units. This results in a reduced hindrance to settling, i.e., increased settling rates of the flow units as shown in the Table 2 data.

For a given suspension the sediment volume fraction, v_s, and the solid volume fraction, $(1 - \varepsilon)$, are also correlated and influenced by the association of immobile liquid. The values shown in Table 2 for different suspensions and the plots of v_s against $(1 - \varepsilon)$ shown in Fig. 6 are verified to this point. Values of v_s in the case of glass beads–water suspensions vary uniformly as compared to the clay–water and clay–water–gum tragacanth suspensions. The straight-line plot for the glass beads–water suspension also exhibits the incompressibility of the sediment and the relatively larger values of the solid volume fractions $(1 - \varepsilon)$ in the settled sediments.

The values of parameter A for glass beads–aqueous electrolyte were calculated by Steinour to be 1.82 (*5*), whereas in the present investigations on glass beads–water suspensions the value of A has been calculated to be nearly 2.2 (almost 20% higher). This deviation can be explained by the nature of the suspension medium, which now is deionized water. In suspensions with $A = 1.82$, less hindrance is expected because the suspension medium is an aqueous electrolyte which causes compression of the double layer over the solid particle and results in a smaller amount of associated liquid, therefore giving higher settling rates to the suspensions. On the other hand, for glass beads–water suspensions with $A = 2.2$, the associated liquid is relatively larger, causing the flow units to settle slower.

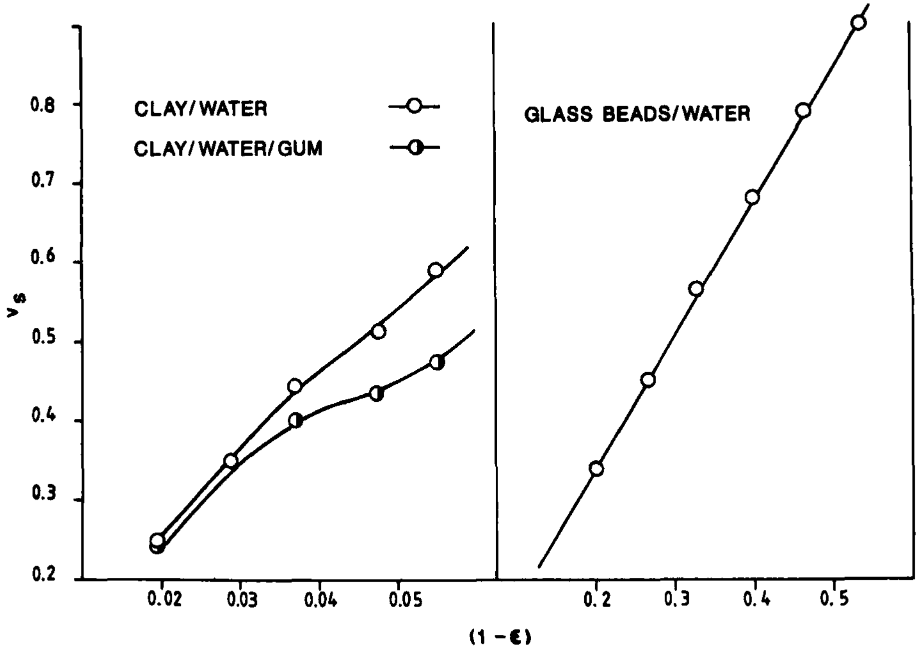

FIG. 6. Variation of v_s with $(1 - \varepsilon)$ for clay-water, clay-water-gum tragacanth, and glass beads-water suspensions.

The nature of the suspension medium and the temperature of the system could also affect the behavior of a suspension, as proposed by Hansen (*7*). Higher temperature generally results in smaller amounts of associated liquid and therefore smaller values of p and v_i. This may result in higher settling rates of the suspension and low sediment volumes. This aspect is not investigated in this report.

It is generally concluded (*8*) that a suspension with $p > 1$ results in a sediment of a less compressible nature. In other words, the sedimenting volume, i.e., the total volume of sedimenting material, solid, and associated liquid, is greater than the final sediment volume, which means that the liquid is forced off the sedimenting material as it settles into a sediment. This behavior is generally expected with a relatively less flocculated suspension.

For suspensions with $p < 1$ a sediment of a compressive nature is formed, in that the sedimenting volume is less than the sediment volume (i.e., the settled sediment volume). This means that a large amount of free liquid stays between particles (flow units) in the sediment. This kind of behavior is expected with highly flocculated suspensions (*8*) which give a sediment with an increased number of interstices.

For a suspension with $p = 1$ the indication is that the sedimenting volume of suspension equals the settled sediment volume. The amount of associated liquid stays with the flow units even after they have settled. These sediments have been termed "incompressible sediments." Aqueous suspensions of glass beads are expected to result in incompressible sediments (*8*). Preliminary observations on sedimentation and simultaneous filtration by recording the settling levels of the suspension and sediment reveal that the volume of the sediment may also change even after all the supernatent has been filtered off, due mainly to

(a) The release of unassociated liquid in the sediment (which may account for the amount of loose liquid staying in the interstices)
(b) The forced release of part of the associated liquid due to squeezing of the sediment caused by the downward force of filtration

Some of the preliminary experiments conducted on glass beads–water, clay–water, and clay–water–gum suspensions show evidence of this behavior. See, for instance, Figs. 4a–4c. The sediments do not undergo further squeezing after all the loose and partly associated liquids have been filtered off. In the case of the glass beads–water suspension, the sediment volume remains unchanged once it has attained its final volume "during" filtration. See Fig. 4c for an explanation. The reason is doubtless that there is virtually no immobile liquid associated with the glass beads. Moreover, their uniform size and spherical shape lead to close-packing of the sediment. It may be noted, however, that the filtration rate of the glass bead–water suspension is reduced soon after the sediment has reached its final volume. A kink in the sedimentation level corresponding to the attainment of the final sediment volume, as shown in Fig. 4c, provides evidence to this behavior. In the cases of clay–water and clay–water–gum tragacanth suspensions, the filtration times are extremely large (span days) as is obvious from Figs. 4a and 4b. Therefore, a critical filtration behavior like that observed with the glass beads–water suspensions is difficult to monitor.

Filtration becomes increasingly tortuous with increasing amounts of immobile liquid associated with the particles. This tortuous fluid flow reduces both the sedimentation and filtration rates, as noted with suspensions containing clays. A schematic diagram of particles with associated liquids in a settled sediment is given in Fig. 7. The length L_e is

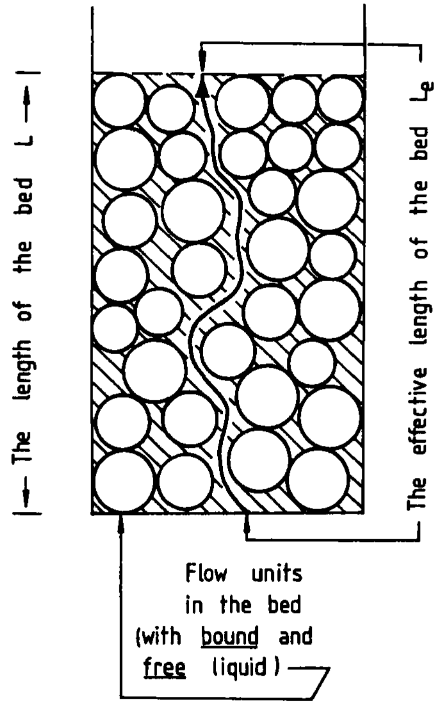

FIG. 7. Effective path length L_e of an upward liquid flow through a tortuous bed (schematic).

the effective liquid path during filtration, which is longer than the apparent length L of the sediment itself. After complete filtration, only the associated immobile liquid stays in the sediment. Amounts of this liquid for each system under investigation are shown by the values of ω_i given in Table 3.

CONCLUSIONS

The association of immobile liquid to the flow units in solid-liquid suspensions results in large sediment volumes. The degree of liquid association depends primarily upon the nature of the solid-liquid suspension. Larger volumes of associated liquids slow down the settling rates of suspensions and result in compressible sediments of reduced porosity. They also exhibit slow filtration due to increased tortuosity.

Acknowledgments

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