Aggregate Structures and Solid-Liquid Separation Processes†

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Abstract
Solid-liquid separation processes for dispersions of fine particles continue to present significant challenges in many areas. As an example, in filtration it is usually possible to achieve either fast filtration or a dry cake, but rarely both. To achieve both a compact dry cake and rapid filtration will rely upon an increased ability to control the particle network structure of the filter cake throughout the whole process, with a probable requirement being a controlled structural change from the beginning to the end of the filtration. An obvious pre-requisite is a detailed knowledge of how to control the structure of sediment beds during flocculation processes. Recent advances in measurement technology have provided unique insights of the relationship between the mechanism (bridging, depletion, charge neutralisation) and conditions of flocculation and resultant aggregate structures. As a consequence, the link between aggregate structures and sediment bed properties is also being investigated with renewed interest. Clearly, the link between flocculation, aggregate structure and sediment bed is vital in the understanding and control of solid-liquid separation processes. This paper will present a review of recent research linking aggregate formation and characterisation of solid-liquid dispersions to subsequent physical properties of the sediments and filter cakes that they produce.

Keywords: Colloid, Filtration, Sedimentation, Yield stress, Filter cake, Aggregate structure

1. Introduction

The control of solid-liquid separation processes, continues to present major challenges in a number of significant industrial sectors including water and waste-water treatment, minerals processing, paper manufacture, pharmaceuticals, and fine chemicals†. It is now widely recognised that accurate control over the macroscopic properties of any particulate dispersion originates from a detailed understanding of the microscopic particle-particle interaction forces‡. For example, a stable dispersion can be created if a net repulsive interaction exists between the particulate objects. On the other hand, if the particles can be made attractive to one another they will aggregate forming large multi-particle units that can sediment rapidly facilitating separation of the phases. Thus, to gain good control over a particulate dispersion it is necessary to accurately characterise the strength of any inter-particle forces that may be operating.

The flocculation or coagulation of a stable dispersion to form larger aggregates is of central importance to the efficient operation of most solid-liquid separation processes, including filtration, thickening, and clarification§. The relationship between aggregate properties, such as size, structure, and strength, and the efficiency of these unit operations has been recognised for many years∥. However, measurement and analysis limitations significantly hindered any systematic approach to understanding how aggregate properties effect these operations until comparatively recently. Whilst measurements of aggregate sizes have been available for many decades, reliable measurements of aggregate densities have only been
reported during the last decade or so. The reliable measurement and analysis of sediment bed properties, such as shear and compressive yield stresses, are also a comparatively recent development.

When considering the formation of aggregates, the inter-particle interaction forces are of central importance since these define the kinetics of aggregation and the strength of particle-particle bonds both of which ultimately influence aggregate structures. When considering the properties of sediment beds, the strength of particle bonds and the number of bonds that have to be broken, which is determined by the network structure which is in turn related to the aggregate structures, are of critical concern. Hence, an understanding of what controls aggregate properties and how these properties impact on sediment bed properties is fundamental to optimising or improving basic solid-liquid separation processes.

In the sections that follow, a brief review of surface forces is given and this is followed by a discussion of how these forces impact on aggregate and sediment structures. The impact of these structural features on standard process operations such as filtration and thickening are also briefly considered.

2. Particle-Particle Surface Forces in Aqueous Systems

The stability of any aqueous particle dispersion is usually described through the so-called DLVO theory. This simple theory, developed in the 1940’s, defines the potential energy of interaction between two particles as the summation of two independent components, one attractive and the other repulsive, according to;

\[ V_{\text{at}} = V_A + V_R = \frac{aA_H}{12H^2} + \frac{2\pi e^2 \varepsilon_0 \varepsilon \zeta^2 e^{-e/\kappa H}}{1 + e^{-e/\kappa H}} \]  

(1)

Where \( V_A \) is the attractive component and \( V_R \) is the repulsive component. In these equations, \( a \) is the particle radius, \( A_H \) is the effective Hamaker constant, \( \varepsilon \) the dielectric constant of the medium, \( \varepsilon_0 \) is the permittivity of a vacuum, \( \zeta \) is the particle zeta potential, \( H \) the surface-surface separation, and \( \kappa \) the inverse Debye length.

The attractive component arises from the bulk material properties of the particles and is caused by dipolar fluctuations of the atoms. The detail is not required here: It is sufficient to note that the strength of the attraction is essentially independent of the medium for an aqueous dispersion (ie. electrolyte type and concentration); it is always active; and, it decays very rapidly with surface-surface separation distance.

The magnitude of the interaction is controlled by the Hamaker constant (\( A_H \)) which includes the dielectric information for the particles and medium.

The repulsive component to the interaction arises as a result of the attraction of counter-ions by a charged surface. It should be noted here that in an aqueous environment most surfaces can develop a charge, either by the dissociation of inherent surface charges or by specific ion adsorption. Entropy considerations mean that the counter-ions to these charges are not restricted to a surface adsorbed layer but can be found anywhere within a defined volume which extends from the surface. This volume, known as the electrical double layer, ensures electrical neutrality between the particle and its counter-ions. However, when two particles approach each other, the two double layers must overlap. This results in a local non-equilibrium concentration of the counter-ions which is osmotically unfavourable and hence leads to a repulsion. The magnitude of the repulsive interaction is defined by the size of the surface charge (determines the counter-ion concentration in the double layer) and the extension of the double layer away from the surface. The double layer extension is given by the Debye length, \( 1/\kappa \), where;

\[ \kappa \propto (\zeta^2 \varepsilon_0)^{1/2} \]  

(2)

and depends upon the electrolyte concentration (\( c_0 \)) and counter-ion charge (\( z \)).

Thus, the concentration and the type of electrolyte play a significant role in determining the overall energy of interaction between two particles. As a function of particle-particle separation, the DLVO theory gives the following types of interaction curves;

At sufficiently high electrolyte, the primary bar-
ribe coagulation is suppressed and the particles can enter the thermodynamically favoured primary minimum; the depth of this minimum determines the strength of the bonds formed. Factors such as the exact type of counter-ion and the presence of surface roughness have been shown to influence the strength of the attractive minimum although the exact mechanisms for quantifying these effects are still undecided. It is also worth noting that aggregation can be induced even when there is a small barrier of a few kT (curve B). The presence of any repulsive barrier will be sufficient to slow the aggregation rate and here the process is considered as reaction-limited. The rate of the aggregation process is maximised when all repulsive interactions are suppressed, under these conditions the rate is purely diffusion limited (curve C).

Since the development of the DLVO theory, a large number of systems that exhibit apparently anomalous stability behaviour have been reported. This has led to a number of other interaction forces being described. Amongst these, and relevant here, are steric polymer forces, ionic hydration forces, and hydrophobic interactions.

The adsorption of any large polymeric molecules at a particle surface can significantly affect the observed interactions. At the most basic level, an uncharged polymer can adsorb to a particle surface creating a hydrated molecular sheath. If two particles approach one another, overlap of these polymer layers leads to a loss of molecular freedom which is unfavourable and hence, a strongly repulsive interaction.

The distance over which this repulsion operates is dependent upon the “thickness” of the adsorbed film. This is a complex function of many parameters such as polymer molecular weight, adsorption energy, solution concentration etc.; it will suffice here to note that when the layer is sufficiently thick, the attractive dispersion forces can be permanently suppressed and a fully stable dispersion can result. Obviously, the use of charged polymers introduces a further electro-static component into the repulsion that is strongly affected by the added electrolyte level. However, the broad analysis of the origins of stabilisation is the same as given above.

The presence of different electrolyte types can also have a significant effect on particulate stability. For example, it has been known for a long time that systems such as clays and silica can exhibit anomalous stability. Direct measurements using force balance techniques were used to demonstrate that this stability was caused either by the strong hydration of the surface or by the specific adsorption of strongly hydrated ions. Close approach of the surfaces is prevented by the need to either de-hydrate the surface or cause ion desorption, respectively. Once again, this prevents the particle contacts from achieving close enough contact to reach the energy minimum caused by the attractive dispersion forces.

The effect of concentrated electrolyte solutions on particle-particle interactions have also been the subject of much interest recently. It has frequently been observed that simple 1-1 electrolytes at concentrations of around 1M and higher can lead to significant short-range interaction forces not described by DLVO theory. These interactions are driven by ion adsorption at the solid-liquid interface caused by the high salt concentration; the effects are seen in anomalous colloid dispersion behaviours. Despite the obvious importance of high electrolyte systems across many technologies, a unified understanding of these effects is not yet available. A number of models have been proposed to explain the observed ion adsorption data. These are largely based around an analysis of the hydration state of the surface and the ionic species in solution. It is assumed that a “like-adsorbs-like” concept can be used to explain the adsorption of ions at a surface. Thus, a weakly hydrated surface would favour the adsorption of weakly hydrated counter-ions and vice versa. This approach satisfactorily explains the observed adsorption of ions on surfaces such as silica and alumina. Unfortunately, these hydration based approaches are unable to explain all of the data observed across all of the available particle types. It is also worth noting that in almost all cases, data were collected for a ho-
mological series of counter-ions (whether cations or anions) adsorbing on oppositely charged substrates will little or no analysis of the role of co-ions. An exception to this is the work of Kosmulski and co-workers who have published a series of papers over the last decade in this field\(^3\). They make extensive use of the hard ion-soft ion model to explain their data since this model does allow for co-ion influences.

Finally, it is worth noting that hydrophobic particles in an aqueous medium will attract one another very strongly. Obviously, by definition they are in an unfavourable solvent environment. The exact origins of this interaction and its full quantification theoretically have yet to be resolved. However, it is accepted that a hydrophobic interaction will be attractive and will in general result in a significantly stronger and longer range interaction than is seen for the attractive dispersion forces\(^{13, 22, 23, 26}\).

### 3. Measurements and Analysis of Aggregates, Particle networks, Sludges and Slurries.

As noted above, stable particle dispersions can be destabilised in a variety of ways including through the addition of electrolyte. When an attractive particle-particle interaction force is established, any collision between two particles can result in a `sticking event’. In this way, aggregates within dispersions can form and grow\(^{39}\). A fundamental parameter that is often used to characterise this process is the initial aggregation rate. As well as the interaction potential between the particles, the rate of aggregation is dependent upon factors such as particle volume fraction, medium viscosity, and whether the system is quiescent or mixed. Under quiescent conditions and in the absence of any repulsive interaction potential, the limiting rate of aggregation is given by the Smoluchowski theory as\(^{37}\);

\[
K_0 = \frac{4kT}{3\eta}
\]

Where \(\eta\) is the viscosity of the solvent.

Full characterisation of an aggregation event usually involves determination of the rate of aggregation, the aggregate size distribution (as a function of time), the sedimentation rate, and the aggregate structure\(^{36}\). Aggregation rate, aggregate sizes, size distributions, and densities are most frequently determined using static light scattering\(^5\) although there is a significant body of research that utilises less complex turbidity measurements\(^{39}\). The main drawback of light scattering approaches arises from the requirement to maintain a dilute dispersion that prevents multiple scattering events. It should also be noted that determination of the aggregate densities relies upon the calculation of a so-called fractal dimension for the aggregates\(^{40}\). Practically, this can be obtained from measurements of the scattered intensity as a function of the scattering angle for a system of aggregates. According to relevant theories, it is critical that no multiple scattering events occur within any given aggregate: This is usually achieved for small open aggregates.

For a mass fractal aggregate consisting of monodisperse primary particles, the fractal character of an aggregate can be revealed if the primary particles satisfy the criteria of the Rayleigh-Gans-Debye (RGD) regime; the particles should behave as point scatterers and their should be no multiple scattering events. Assuming these conditions are met, the scattered intensity from the aggregate, \(I(Q)\), shows a power-law behaviour, according to,

\[
I(Q) \propto Q^{-D}
\]

provided that \(1/R < Q < 1/r_o\). Here \(D\) is the aggregate mass fractal dimension, \(R\) and \(r_o\) are the respective radii of the aggregate and the primary particles, and \(Q\) is the magnitude of the scattering wavevector.

\[
Q = \frac{4\pi n_o \lambda_0}{\lambda_o \sin \left( \frac{\theta}{2} \right)}
\]

where \(n_o\) is the refractive index of the dispersion medium, \(\theta\) is the scattering angle and \(\lambda_o\) is the in vacuo wavelength of the incident light. Since \(1/Q\) is the length scale probed in a scattering experiment, the low and high regions of \(Q\) reveal the overall structure of the aggregate and the structure of the primary particles, respectively. The spatial arrangement of the primary particles inside an aggregate can only be probed when \(1/R < Q < 1/r_o\). Typical scattering data from a fractal aggregate are shown below in Fig. 3.

This approach has been used to show that both the size of aggregates that form, and their density (structural compactness) can be altered by changing one or all of the particle interaction forces\(^{41}\), particle volume fraction\(^{39}\) and/or the mixing conditions\(^{40}\). More recently, alternative techniques that utilise ultrasoneics or back scattered light have begun to be utilised to measure aggregation in situ for more concentrated dispersions\(^{44, 46}\).

There are two main limitations on the growth of aggregates, the presence of shear\(^{43}\) is one and the second is sedimentation. When a shear field exists, aggregate growth is limited since large tenuous ag-
gregates can be easily disrupted leaving a system of smaller more compact aggregates. In the case of a quiescent system where there is no barrier to aggregation, if there were no sedimentation then growth of the aggregates would continue until there was only one single aggregate. This has been seen for polystyrene latex in water which is an almost neutrally buoyant system. However, more typically the particles are more dense than the medium and begin to settle rapidly as the aggregates grow. This prevents them growing too large, and may also influence the average size and shape.

As we noted above, aggregate densities are characterised using the mass fractal dimension, $D$. $D$ corresponds to the space-filling capacity of the aggregate and hence is a good measure of the structural compactness. In 3-dimensional Euclidean space, it can have values of $1 < D < 3$ where $1$ corresponds to a relatively linear tenuous aggregate and $3$ to a totally compact object. Fractal behaviour has been reported for a wide range of colloidal materials including latex, gold, silica, alumina, hematite, bentonite clay and carbon black. Such structural characterisation has been reported for aggregates formed using a variety of aggregation mechanisms including the addition of electrolyte, polymer bridging and polymer depletion. The aggregation caused by addition of salt is by far the most understood both in experiment and theory. The theory predicts two regimes of behaviour. In the absence of any potential energy barrier (Fig. 1), all collisions lead to a sticking event which results in the rapid formation of large open clusters, this is known as diffusion limited cluster-cluster aggregation (DLCA); when there is a small potential energy barrier, not all collisions lead to a sticking event and individual particles have longer to find a pathway into the core of an aggregate, this leads to smaller more compact aggregates and is known as reaction-limited cluster-cluster aggregation (RLCA). Theory and experiment have shown that for DLCA, $D = 1.7 - 1.8$, and for RLCA, $D = 2.1 - 2.2$. Examples of aggregates corresponding to these values are shown in Fig. 4.

In general, it has been shown that the aggregate density is a sensitive function of the ratio of the particle-particle sticking rate to the structural rearrangement rate for any aggregated particle. Essentially, if the sticking rate is high, rearrangements are unlikely before further collisions (and sticking events) and an open aggregate structure becomes locked-in. Reductions in the collision rate by decreasing the particle volume fraction or increasing the medium viscosity have been shown to cause more compact aggregates. In general, for systems which show limited sedimentation it has been observed that aggregates which form under RLCA conditions can undergo rearrangements over long timescales which result in more compact aggregates than are initially observed. Such rearrangements, for aggregates with little or no settling are relevant to natural
aquatic colloids or living species such as algae that may undergo aggregation.\textsuperscript{54,56} It has also been shown recently that there is a gradual transition between the limiting RLCA and DLCA cases across a narrow range of added electrolyte\textsuperscript{57}; the limiting cases and this intermediate regime can be defined using the colloid stability ratio, $W$, which defines stability on the basis of a ratio of the observed aggregation rate to the maximum possible aggregation rate (diffusion limited). So, when $W = 1$, the system is at its maximum instability and a large value of $W$ defines a stable system. The intermediate behaviour regime is defined as $5 < W < 100$; in this regime D was seen to gradually rise from a typical DLCA value to the RLCA limiting value.

The introduction of shear\textsuperscript{43, 58, 59} is also an important factor and it usually results in smaller more compact aggregates. The impact of shear is affected by the primary particle size. Smaller particle sizes generate more compact aggregates through an increased level of aggregate rearrangement in the shear field. By contrast, larger particles exhibit a fragmentation and re-aggregation mechanism as a result of the breakage of tenuous features from the aggregates in the shear field.

Sedimentation rates are obviously strongly dependent upon both the aggregate sizes and their net density (a function of the primary particle density and the aggregate compactness). Typically, sedimentation rates have been investigated by recording changes in turbidity as a function of both time and height in a sedimentation column. More recently, the sedimentation rates of individual aggregates have been reported as a function of the aggregate size. Such data can be used to calculate the apparent density of an aggregate, and if the data are collected over a sufficiently broad size range they may also be used to determine the fractal dimension of the aggregates\textsuperscript{48}. The key difficulties with this approach are associated with the porosity of the aggregates and their generally non-spherical shapes. Shape effects can be managed through the use of appropriate empirical corrections to the drag coefficient.\textsuperscript{6} The porosity of the aggregates, and the associated flow of liquid around or through these aggregates are more difficult to understand and account for. The implications of a fractal structure are that as an aggregate gets larger, at a fixed fractal dimension, its net density must decrease and the effective porosity must increase. Furthermore, the porosity of an aggregate must increase with its radius away from the centre\textsuperscript{69}; it seems improbable therefore that large aggregates do not show some level of liquid flow through their structure during sedimentation.\textsuperscript{63} However, correct modelling of this flow through is difficult as a result of the complex relationship between aggregate size, fractal dimension, and porosity as a function of aggregate radius. Nonetheless, the sedimentation approach has been used to characterise fractal aggregates.\textsuperscript{4, 48, 62} It is especially useful when the size of the aggregates leads to problems with the more common light scattering approach as a result of violations of the available RGD theory. The use of polymer flocculants usually results in such large aggregates, and it also introduces a much wider range of possible fractal dimensions\textsuperscript{50} as a result of the increased complexity of the aggregation process and its dependence on a greater number of parameters such as polymer molecular weight, type, method of addition, and concentration.\textsuperscript{62}

The result of particles or aggregates which sediment from a dispersion is the formation of a sediment bed (sludge). The properties of this sediment are strongly dependent on the aggregate feed properties, especially the aggregate sizes and densities.\textsuperscript{53, 64} It is also important to understand how these aggregates interact within the sediment; for example, aggregates formed by salt coagulation will probably bind at any contact points since the salt can still be expected to have an influence continuously whereas aggregates formed by polymer bridging may not actually bind to each other and can remain as discrete objects. The size of the aggregates is important since, for any given fractal dimension, aggregates are more ‘open’ at the outer edges than they are at the core.\textsuperscript{64} Thus, in a sediment larger aggregates may generate a more porous network; it is worth noting however that the edges may be inherently weak and more compressible resulting in greater compaction of the sediment under gravity. The strength of a sediment bed is also important since this defines its resistance to compaction and ultimately has an influence on the porosity. The total strength of any sediment will depend on the strength of the particle-particle bonds and the network structure within the bed.\textsuperscript{63} As noted above, this overall network structure will be influenced both by the structures of the individual aggregates as well as by how these aggregates interact with each other. The strength and structure of sediment beds is therefore clearly important to consolidation, filtration, and re-suspension.

The energy required for re-suspension is clearly related to the product of the bond number and average bond strength\textsuperscript{4, 12, 66}, although complications in-
In the case of aggregates formed by suppression of the double layer through the addition of electrolyte, all the aggregates in the sediment bed can aggregate together forming a continuous gel network structure. The resistance of this network to an external shear field is characterised by its shear yield stress, \( \tau_y \). The shear yield stress is defined as the applied stress beyond which material will flow and is indicative of a transition between solid-like and liquid-like behaviour\(^{67, 68}\). Although the existence of a yield stress has traditionally been the subject of contention, it is now generally accepted that for a cohesive particle network \( \tau_y \) has real meaning\(^{69}\). Boger and co-workers\(^{70-79}\) have pioneered the use of a vane technique for the characterisation of particle networks through the measurement of \( \tau_y \). The advantage of the vane for these measurements is the absence of any wall slip when performing the rheological test\(^{69}\). More commonly, it is reported that the yield stress can be obtained from an extrapolation of shear stress-shear rate data to zero shear rate\(^{80-87}\). This is often performed using a rheology model such as the Bingham, Herschel-Bulkley or Casson. This approach relies upon accurate data at low shear rates. Unfortunately, wall slip is a big problem for conventional measuring geometries (cup and bob or cone and plate) when the system has a significant yield condition. This can lead to significant errors in the flow data and hence the yield stress.

In addition to its effect on the shear yield stress, a significant inter-particle interaction can impact strongly on the compressive properties of any particle network\(^{6, 88-96}\). Compression is usually characterised through the compressive yield stress \( P_y \). As before, \( P_y \) is defined as the compressive pressure beyond which the network consolidates inelastically. Measurement and calculation of \( P_y \) is considerably more complex\(^{86-102}\) than \( \tau_y \) and, as a result, there is much less data available in the literature.

In general, a stronger particle-particle attraction will result in a network that has a higher yield stress (shear and compression), a more elastic character (larger storage modulus) and a lower overall particle packing density\(^{70}\). Recently, controlled rheological investigations using model mineral particles such as clean and pure alumina or silica have been used to show that there is a direct relationship between the rheological properties of a sediment bed and the net inter-particle force\(^{2, 69, 71, 72}\). For example, Scales et al\(^{70}\) have proposed that for an aqueous particle dispersion where only DLVO forces are operable, the shear yield stress can be given as:

\[
\tau_y = K_e \left[ \frac{A_H}{12\eta^2} - \frac{2\pi \varepsilon_0 \varepsilon K e^{-\varepsilon_0}}{1 + e^{-\varepsilon_0}} \right] \tag{6}
\]

where the terms in brackets account for the DLVO forces of interaction and \( K_e \) is a term that depends upon the detailed network structure, primarily the coordination number. Using the above relationship, good correlation between factors such as the zeta potential (\( \zeta \)) and the shear yield stress have been demonstrated\(^{85, 104}\). The influence of other inter-particle forces on \( \tau_y \) such as polymer bridging forces\(^{105}\), hydrophobic forces\(^{106}\), and polymer steric forces\(^{104}\) has also been investigated and good correlation with the inter-particle forces has been demonstrated. Preliminary investigations of the influence of other factors such as particle size\(^{66}\), particle size distribution\(^{66}\), particle volume fraction\(^{78}\), and specific ion effects\(^{12, 30}\) have also been described. However, detailed understanding of their influence on the yield stress is currently not as well developed. Franks et al\(^{66}\) have examined the effect of aggregate size on the network strength using a careful series of experiments where all other features of the aggregate samples were held constant, such as bond strength, fractal dimension, and primary particle size. It was found that larger aggregates produce sediments which have a lower gel point and higher shear & compressive yield stresses when all other factors remain constant. Given that larger aggregates become more tenuous, at a fixed fractal dimension, this result seems somewhat counter-intuitive and is, as yet, not fully explained.

There is considerably less literature evidence for
the relationships between inter-particle forces and compressive yield stresses\textsuperscript{6, 90-92, 94}. Despite this, $P_y$ is a crucial factor in understanding the properties of highly concentrated dispersions since it can indicate the maximum concentration of solids that will flow\textsuperscript{6}. In general, knowledge of the maximum concentration for a dispersion that can still flow is highly sought after. This is particularly true in industries such as mining where the disposal of semi-dry solids is increasingly used to minimise the volume of waste. Recent theory and experiment developments have resulted in relatively rapid determinations of the compressive yield stress for settled solids using a pressure filtration approach\textsuperscript{6, 90, 92, 95, 96, 98, 107}. This technique also offers important complementary information about the hindered settling factor (and hence filtration efficiency). Aziz et al\textsuperscript{94} have used such an approach to investigate the influence of inter-particle forces on the compression and filtration of a sediment. They show that a strongly repulsive inter-particle force results in a very compact dense sediment. However, there is a corresponding reduction in the filtration efficiency of this bed and a decrease in the dewaterability of the bed. Green and Boger\textsuperscript{95} note that careful control of the compressive properties for a particle network can be used to control the final solids concentration under a given applied pressure. They also show that a highly concentrated particle system can still be pumped and, in fact, will require a lower pumping energy due to operation in a laminar regime within the pipe. Finally, they suggest that problems of start-up, as well as erosion and settling problems will be reduced if a system of more concentrated solids is used. Zukoski and co-workers have also investigated how inter-particle forces can affect the development of inhomogeneities in the sediment bed and how these flaws influence the compressibility\textsuperscript{90, 91}.

The importance of rheology for the characterisation of a sediment bed is that it can offer a direct measurement of the energies needed to re-suspend the solids under process relevant conditions. Indeed, in principle the measurement of yield stress, elastic modulus and bed structure should give direct access to the inter-particle interactions. In practice this is not easily done and more direct force balance techniques can give a much better indication of the operable forces. This detailed information is crucial for the control and manipulation of forces between particles\textsuperscript{15-19, 29, 108}.

As yet, there are few studies which combine the macroscopic rheology measurements of inter-particle forces with the microscopic data available from a technique such as AFM. One report, by Yan et al\textsuperscript{109} shows that the two techniques are definitely complementary and in a broad sense give the same results. Burns et al\textsuperscript{111} have also shown that rheology and AFM data can be used in combination to more completely explain the flow behaviour of a concentrated suspension. The influence of polymer steric layers on the flow properties of dispersions have also been examined using both rheology and direct force measurements\textsuperscript{82, 97, 110, 111}. There is definitely value in exploring these links further.

4. Aggregate Properties and Solid-Liquid Separation

In considering the effect of aggregate structures on different solid-liquid processes or treatment strategies, it is important to define the range of conditions that are experienced by the particles when treated. For example, the impact of shear on aggregation and on aggregate structure is known to be important\textsuperscript{43, 58, 59}. So, the use of mixing when adding a coagulant or flocculant can have a significant effect on the final operational performance of a given process unit\textsuperscript{4}. The application of shear causes the formation of smaller more compact aggregates; this is seen most dramatically under DLCA conditions where larger more pendulous aggregates would be formed in a quiescent state. The diffuse arms of these large aggregates are relatively weak and easily sheared off resulting in the formation of smaller aggregates. The sheared fragments are expected to penetrate into the aggregates before re-attaching resulting in the formation of smaller more dense aggregates. It has also been noted that both the intensity of shear and the duration of its application are important control parameters for producing different size and density aggregates\textsuperscript{50, 112, 113}.

It has also been noted previously, that the performance requirements of different solid-liquid separation processes, such as filtration or thickening, are often very different\textsuperscript{3, 4} As a result, the optimum aggregate properties for each are most likely very different. Furthermore, it is likely that different modes of operation within a given unit will also affect any decision about optimal aggregate properties. As noted earlier, significant progress on the analysis of aggregates and sediment bed properties has been made during the last decade. However, understanding of the impact of different aggregate properties on water treatment operations remains limited.

One of the most well developed areas is sedimentation; here researchers have demonstrated clear links
between aggregate sizes and size distributions, as well as aggregate densities and sedimentation. It remains unclear about how to best model an open fractal aggregate and how to define the correct drag coefficient for such an object. For example, will fluid flow through or around such an aggregate; given that the effective porosity of a fractal aggregate increases with aggregate size it seems likely that the opportunity for flow through the aggregate will be greatest towards its edges and decrease towards the centre. Glover et al. showed that for small porous aggregates of alumina it is important to allow for some flow through the aggregates if we are to correctly interpret sedimentation data.

The links between the consolidation behaviour of sediments and aggregate properties also remain relatively poorly understood with only a limited number of reports available. Waite and co-workers have, in a series of papers, investigated the effect of aggregate sizes and structures on the performance of ultrafiltration for colloidal solids. The relationship between fractal aggregate structures and filter cake porosity is explored in this work through application of a modified Carmen-Kozeny relationship, which is given as:

\[ \alpha = \frac{180(1 - \varepsilon)}{\rho g d^2 \varepsilon^2} \]

where \( \alpha \) is the specific resistance of the filter cake, \( \rho \) the density of the particles, \( d \) the mean diameter of the particles, and \( \varepsilon \) is the void volume of the cake. This relationship is known to be valid for cakes formed from discrete particles having porosities of less than 0.5. It is clear from this expression, that the porosity (through the void volume, \( \varepsilon \)) of the cake has a strong influence on the performance of any filter cake. Hence, filter cakes that are formed from fractal aggregates of colloids will be influenced by aggregate sizes and fractal dimensions. It has been noted many times that as aggregates get larger, at a fixed fractal dimension, that they must get more porous. Hence, when they form a filter cake we might expect that the porosity of that cake would be higher. On the other hand, if the aggregates are weak they may be susceptible to collapse and the bed may consolidate under gravity resulting in a loss of porosity and hence permeability. From the work of Waite and co-workers, it seems that, in general, larger aggregates tend to form more porous filter cakes and these filter cakes are more resistant to collapse. Smaller aggregates, with a higher fractal dimension, form filter cakes that are initially of lower porosity; in addition, these cakes exhibit higher levels of consolidation with the associated poor filtration performance. Interestingly, these results appear to agree with the shear and compressive yield stress data of Franks et al., which showed that larger aggregates were more resistant to an applied force. These results from filtration tests appear quite general and have been confirmed on a variety of systems including biosolids and apple juice colloids.

5. Conclusion

Whilst it is clear that there must exist links between the feed of aggregates and the performance of a given solid-liquid separation process, relationships between the two are still not fully developed. The last decade has seen significant advances in measurement capability and this has led to an unprecedented degree of insight into aggregation and the properties of aggregates formed. Parallel advances in the characterisation of sediments and filter cakes have also occurred using rheology and pressure filtration techniques. Despite this, links between the properties of the aggregate feed and the subsequent behaviour of a sediment bed are still poorly developed; this is primarily related to the complexity of the system and the number of inter-dependent variables that have to be controlled for a systematic study to be undertaken.

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**Author’s short biography**

**Simon Biggs**

Simon Biggs was appointed to the RAEng/BNFL Chair at the University of Leeds in September 2002. He joined the University of Leeds from The University of Newcastle (New South Wales, Australia) where he had been a staff member for 8 years. Prior to this, he developed his research career through post-doctoral positions at the Institut Charles Sadron (Strasbourg, France) and the University of Melbourne (Australia).

The main research interests of Prof. Biggs’ group are in the field of colloid and interface engineering. He has a strong interest in the measurement, control, and manipulation of inter-particle forces to allow more efficient process engineering of particulate systems. In related research, his group have a growing interest in the full 3-dimensional characterisation of adsorbed surfactant or polymer layers at the solid liquid interface. This research is being used to better understand adsorption mechanisms and adsorbed layer properties for these systems. In particular, we are interested in combining in-situ imaging with direct measurements of both adsorbed amounts and adsorption kinetics. His research involves both fundamental investigations, using precision instrumentation such as light scattering and atomic force microscopy, and applied problem solving related to industrial scale issues.

Simon Biggs has been the chief investigator on numerous research projects and he is an author of over 150 publications in refereed journals.