

COLLOID SCIENCE IN SOLID/LIQUID SEPARATION TECHNOLOGY: IS IT IMPORTANT?

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ABSTRACT

Of the various fine particle/liquid separation techniques available to the process engineer the most commonly used are variants of filtration, expression and sedimentation. Their simple concept and apparent ease of operation enable the efficient separation of many solid/liquid systems. However, the separation of finer, near colloidal sized, particle suspensions using these techniques frequently poses problems which have undesirable consequences. For example, on occasions the wetness of filter cakes can be inexplicably high, with the result that thermal drying is the only viable method by which the required moisture removal can be achieved. With cost increases of energy forever pending, and demands for improved separation technologies, there is a growing interest in developing alternative and more cost effective methods of separation. To meet this goal it is necessary for both engineers and scientists to work together and to have a basic understanding of how the technology of one affects the science of the other (and *vice-versa*). This paper considers some of the implications of the work of colloid scientists for the separations technologist, with particular reference to sedimentation and filtration processes.

INTRODUCTION

Process selection and design of solid/liquid separation equipment generally starts once a representative sample of suspension is available, with preliminary evaluations from jar sedimentation and leaf filter tests. At this stage consideration may be given to modifying the suspension by chemical or physical treatments to improve its settling or filtration characteristics. Results from these are in the form of a sedimentation rate and sediment volume from jar tests, and filtration rate and cake moisture content from leaf tests. The filtration rate is sometimes expressed via a specific cake resistance or cake form time. So that the process engineer is better able to manipulate the suspension it is becoming increasingly important that he or she has a 'feel' for what is behind these measurements - that is, a basic understanding of the phenomena which dictate whether or not a suspension is easily separable.

THE PRIMARY FORCES

Colloidal particles (less than one micron diameter) in suspension do not settle readily, and the suspension is said to be stable. The particles and/or macromolecules in these dispersions maintain their resistance to aggregation (i.e. their stability) through interactions (forces) at their surfaces. A consequence of the small particle sizes in a particular solution environment can make these surface forces appreciably large; these in turn determine the properties of the dispersion and the resultant force magnitude can govern separation efficiency. For example, it may be necessary to apply expression pressures greater than several hundred atmospheres in order to squeeze the liquid from very stable dispersions¹. When particles initially in a suspension are forced to approach one another, as when a sediment or cake is formed, the role of the forces of interaction become more dominant.

Origins of the Surface Charge

Most particles acquire a surface electric charge when brought into contact with a polar (e.g. aqueous) medium. The various charging mechanisms may include ionisation (e.g. proteins

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acquire their charge mainly through the ionisation of carboxyl and amino groups to give COO^- and NH^+ ions), the unequal adsorption of oppositely charged ions at the particle surface, and the unequal dissolution of ions from the surface. These phenomena give rise to the electric double layer which surrounds colloidal particles, the thickness of which is approximated by:

$$\kappa^{-1} = \left(\frac{1000e^2 N_A \cdot 2I}{\epsilon \epsilon_0 k_B T} \right)^{0.5} \quad (1)$$

where l is the ionic strength (i.e. the thickness varies inversely with the ionic strength of the solution). The double layer is composed of the Stern layer and a diffuse layer, as shown in Figure 1. The Stern layer is that region between the surface of the particle and the plane of closest approach to it (of the order of 0.3 nm). The diffuse layer contains counter-ions and the charge density varies with distance from the interface.

The main types of interaction are several and include:

- van der Waals attractions
- Electrical repulsions
- Solvation (hydration) forces
- Steric interactions
- Polymer bridging.

The first two are of primary, although not of exclusive, concern in this paper. The relative magnitude of the repulsive and attractive components determine the stability of the suspension.

van der Waals Attractions

Attractive forces between atoms and molecules were first postulated by van der Waals. Those postulates relating to intermolecular attractions have been generally scaled-up to apply to fine particles. Such attractions originate in three ways:

- Two molecules with permanent dipoles mutually orientate each other such that attraction results
- Dipolar molecules induce dipoles in other molecules such that attraction results
- Fluctuations in the charge distribution in a molecule causes polarisation of neighbouring molecules, leading to the so-called universal attractive forces (also known as dispersive forces).

The first two only arise if both interacting molecules are polar, but the third occurs even between non-polar molecules and accounts for almost all the van der Waals attractions which are operative. Similar effects are thought to happen at the surfaces of colloidal particles. The relationship for the net attractive interaction force between two spheres is:

$$F_A = \frac{4A}{3a} \left(\frac{2}{s^3 - 4s} - \frac{s}{(s^2 - 4)^2} - \frac{1}{s^3} \right) \quad (2)$$

where $s = 2+x'/a$ and A is the Hamaker function reflecting the polarisability of the particle in the fluid. The range of values for the Hamaker function for substances immersed in water can be summarised as follows

30	10	3	1	0.3	$\times 10^{-20}$ J
metals		oxides and halides		hydrocarbons	

Interparticle Repulsion

Origins of the repulsive force lie in the distribution of solution ions around the charged particle, and the resultant electrical charge is dependent on the chemical species present at the particle surface. A potential energy of repulsion may extend appreciable distances from the particle surfaces, but its range may be compressed by increasing the electrolyte content of the solution environment - this implies that under some conditions double layer repulsion competes with van der Waals attractions in both magnitude and range to govern particle interactions.

The conditions under which approaching particles first influence one another are at large separation distances, and the relationship for the repulsive interaction force is:

$$F_R = \frac{64\pi n_s z k_B T (a + \delta)^2 (\kappa (x' + 2a) - 1)}{\kappa^2 (x' + 2a)^2} \tanh^2 \left(\frac{ze_e \psi}{4k_B T} \right) \exp(-\kappa (x' - 2\delta)) \quad (3)$$

For practical purposes the potential is taken to be the zeta-potential (ζ). This expression is complex, but several features important to the process engineer should be highlighted:

- The interaction increases with the solids volume fraction in the solid/liquid mixture
- The ionic strength of the solution plays an important role (through the double layer thickness κ^{-1})
- The interaction increases with increasing zeta-potential of the particles
- Reducing the magnitude of this repulsive force causes the dispersion to become unstable and generally more easily separated
- Repulsive forces can be reduced either by (a) adding an indifferent electrolyte to the solution to change the distribution of solution ions around the particle, or (b) altering the electrical charge on the surface of the particle by the specific adsorption of certain ions.

For systems where only electrical and van der Waals interactions need to be considered, these can be added to give the total interaction force (F_T) between the particles as a function of the solids volume fraction. Typical interaction energy curves are shown in Figure 2 and some example values for the resultant interaction forces are given in Table 1, where it is seen that as the particles come closer together so the repulsive force increases substantially. This makes it very difficult to bring the particles into true contact with each other, and hence reach a filter cake with a minimum moisture content.

SEPARATION PROCESSES

Terminology tends to differ between the colloid scientist and the process engineer, and although in general terms both are quite readily understood by each other it can be difficult to interconvert rapidly. The engineer requires numerical information about materials in bulk, whereas the colloid scientist frequently talks of isolated particles. For example, the above equations are written to involve particle separation distances, but solids concentrations or volume fractions are required by the engineer. Figure 3 shows an approximate conversion from separation distances to volume fractions and indicates where different separation technologies may be considered in relation to

these terms. Repulsive forces can be operable at volume fractions well below 0.1 in the case of stable suspensions at one end of the scale, and control deliquoring of filter cakes by expression at the other end. Not only can such forces be important to different separation processes, but they can also affect the performance of the process at various states in its cycle, i.e. during cake formation, deliquoring, washing and discharge.

A typical separation process begins with a treated suspension in which the particulates might be dispersed or aggregated. A cake or sediment is formed which has a structure capable of sustaining stress. These structures vary considerably in their resistance to compressive forces, and their compressibility is a key factor in the behaviour of thickeners, filters, centrifuges and expression equipment³. In thickeners the characteristics of the compressing sediment is of greater importance than sedimentation of the particles, although the performance of gravity clarifiers is controlled by free settling of the particles. In filters, centrifuges and expression equipment, porosity and permeability of the cakes are major factors affecting their operational behaviour.

The extent of particle aggregation in suspensions determines the degree of compressibility. Flocs or aggregates from the pretreatment process are deposited as a cake or sediment surface under a zero stress. As increasing deposits cover the surfaces, developing stresses continually compress the bed. Compressibility depends on particle size distribution and shape, and degree of aggregation (Figure 4). The degree of aggregation is dependent on the surface charges on the particles and their solution environment. Beds composed of particles larger than 20 microns are essentially incompressible, and their final solids volume fraction is mainly dependent on particle shape. Beds composed of finer particles have solids volume fractions which depend not only on particle geometries but also on the primary interparticle forces.

The following are a few examples of how colloidal phenomena affect some of the more common separation processes.

Suspension Pretreatment

Suspension pretreatment is generally carried out either to increase the rate of separation or to enable formation of a more tightly packed cake or sediment. At the primary level this is done by reducing the interparticle repulsive forces. The most common methods used are either to add an indifferent electrolyte to the solution or to add certain ions which specifically adsorb on to the particles. In many instances attempts to achieve repulsive force reductions to effect coagulation/flocculation and subsequent aggregation rely on trial-and-error methods. For example, the 'sweep-floc' mechanism widely used in the wastewater treatment industry achieves separation through a precipitation process which is not strictly coagulation. Here, large enough amounts of an ammonium or ferric salt and alkali produce a woolly hydroxide precipitate which engulfs the fine particles as it settles. This has been particularly successful when a low initial solids concentration is involved. However, once the suspension has settled it can sometimes be difficult to separate the fine particles from the precipitate. Filtration is then only needed if a further reduction in the moisture content of the sediment is required.

Thus, many separation processes are often carried out without due regard to the surface chemical properties of the particles which affect their stability against aggregation⁴. There is a need for a more systematic approach to the filtering of stable suspensions where cakes and compacts with the required packing and texture can be produced.

Laboratory Testing

Engineering processes are inherently less ideal than the carefully controlled experiments used in studies of fundamental phenomena. The former commonly involves the use of materials that are not pure and materials that are not scrupulously clean. In contrast, colloid science experiments demand the highest degree of cleanliness, due to the unpredictable effects of impurities on the

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primary process. For example, water forms droplets instead of spreading evenly on exposed machine surfaces - in this case the presence of adsorbed organic matter renders the surface hydrophobic.

Figure 5 illustrates how the cleanliness of equipment can affect results from the ubiquitous jar settling test. In one test the jar was thoroughly cleaned, and in the other the inner walls of the jar were covered by a thin film of grease. In this instance settling was more rapid in the clean jar.

In a similar manner, impurities adsorbed onto the surface of the particles can have serious consequences for separation rates. Figure 6 shows differences in expression rates for two samples of anatase (titanium dioxide). The 'uncleaned' anatase was powder as supplied, whilst the cleaned material had undergone the following cleaning procedure:

- Removal of impurities from the particle surfaces by acid leaching⁵
- Washing the particles with twice distilled water, where the second distillate was from a solution of alkaline potassium permanganate which destroyed residues of organic matter
- Cleaning the apparatus with a mild detergent solution and thereafter rinsing with copious amounts of twice distilled water⁶⁻⁸.

Expression of the cleaned anatase was in this instance slower than for the impure, and the cake contained a significantly lower volume fraction of solids.

Apart from the needs for good housekeeping in the laboratory and for using a truly representative sample of suspension in testwork, the consistency of experimental methods (e.g. mixing and shaking of suspensions) is also important. Fresh suspensions of the mixture which is to be separated should be used for testwork to mitigate the so-called ageing effects. In the case of Figure 6 the 'uncleaned' anatase is actually being affected by an ageing process – in this case by the development of inorganic chemical species on the surface of the particles. In the case of titanium dioxide the zeta-potential changes with time as a result of the acidic dissociation of surface hydroxyl groups. This is because sparingly soluble metal oxides in contact with water usually undergo hydrolysis and dissolution at the surface. The latter effect occurs over a longer period. The expression results were probably affected in accordance with the development of inorganic species on the surface which contribute to the zeta-potential.

The potential dangers behind inadequate simulation of fine particle suspensions for use in tests are demonstrated in Figure 7 which shows results from the expression of an anatase suspension. In both cases the anatase was mixed into water and a solution with a pH of about 7.5 resulted, but in one case distilled water was used in the preparation and in the other case tapwater was used.

From a pH measurement alone it would be expected that these two suspensions would behave in similar fashion, but the additional zeta-potential against pH curves would have yielded the vital information that a higher repulsive force existed in the distilled water suspension. This would have been shown up by a high zeta-potential. But, due to the high repulsive forces, at this high zeta-potential the suspension is well dispersed. On compression of the suspension the particles are therefore reluctant to approach each other too closely unless a high enough external force is used, as in filtering or expressing the suspension, whence the repulsive force acts to 'lubricate' the compressing structure and a high packing density of the particles is achieved.

Settling Test

The importance of interparticle forces in sedimentation is demonstrated in many fine particle suspensions. As an example, Figure 8 shows tile settling characteristics 1 micron calcite particles suspended in 0.00001M CaCl₂ solution. At the isoelectric point (IEP) the zeta-potential is zero and

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there is no interparticle repulsion. Aggregation of particles occurs here without hindrance, resulting in the formation of larger aggregates which settle more rapidly. That is, the IEP represents the pH at which most rapid settling occurs. In the presence of calcium ions the IEP of calcite is shifted from its natural pH of about 9.3 to pH's of about 6 to 7 and 12 to 13. Hence, in Figure 8 more rapid settling is observed at pH's of 6 and 12.

Filtration Test

Laboratory filter tests usually yield cake specific resistance (or cake form rate) and moisture content data, which are then usable in process design and scale-up calculations. The effect of the primary forces can be seen in these types of data. Figure 9 shows the effect of pH on the filtration of an aqueous china clay suspension. The results were obtained from a pressure driven leaf filter and show how reducing the pH causes the formation of a lower resistance (i.e. higher permeability) cake with a higher moisture content.

For this china clay suspension the zeta-potential versus pH plot is shown in Figure 10 where it may be seen that an IEP occurs at a pH of about 2.9. Without doing any filtration testing it could be predicted that the fastest rate of filtration and the wettest cakes would occur at this pH: such results are confirmed in Figure 9 and in Table 2 which relates to the experiments reported in Figure 9.

The further data in Figure 10 relate to calcite and anatase suspensions, and are illustrative that a variety of such curve forms exist. The calcite has a low surface potential and so is relatively easy to filter; furthermore, calcite filter cakes are expected to be quite incompressible. Anatase (titanium dioxide) suspensions show a high zeta-potential ranging from about +80 mV to about -100 mV, passing through an IEP at a pH of about 5.9. Anatase cakes and sediments will therefore show a wide range of compressibility effects and moisture contents, and indeed this is what is experienced. Combined with these surface potential effects is also the possible influence of particle size, size distribution and shape. These factors are believed to have a role in determining the compressibility of the sediment or cake, and the bulk concentration of the suspension may also need to be considered. However, attempts have not yet been made to elucidate the separate effects of these parameters whilst controlling the surface charges on the particles.

Expression Tests

A series of suspension compression/expression tests have been carried out in a laboratory piston press for a wide range of different suspensions. The results from one sequence of such tests is shown in Figure 11 (the anatase suspensions here are different from those used in the construction of Figure 10). The IEP is between 4 and 5 for this suspension, at which the expression rate is seen to be the fastest. At $\text{pH} < 4$ and $\text{pH} > 6$ the value of the zeta-potential (positive and negative respectively) is in excess of about $|80|$ mV and expression rates are correspondingly slow.

On an operational expression filter it is generally not possible to measure filtration rates, but cake moisture contents can be determined. Some such data for a different suspension/cake taken from a horizontal tube press are shown in Figure 12. The minimum cake solids volume fraction occurs close to the IEP of the suspension.

Higher volume fractions occur when the zeta-potential is greater, that is, when the repulsive forces between the particles are greater and there is lesser hindrance to the formation of a tight particle packing structure in the cake. However, the solids volume fraction is seen to decrease at the extreme pH values. This is also expected because at these extremes the ionic strength of the solution is high, which causes a compression of the thickness of the electrical double layer around each particle, and consequently a lowering of the zeta-potential (and the repulsive force). A similar effect can be observed in Figure 11 between pHs of 8.15 and 11.22.

Field Assisted Separations

Field assisted and crossflow separations are attracting considerable attention as being suitable for the filtration of colloidal and near colloidal sized particles. Crossflow techniques include ultrafiltration and microfiltration, and field assisted techniques include electrofiltration and electrothickening (applicable to both deadend and crossflow modes of filtration), dielectrophoretic filtration, and electro-acoustic filtration. In these operations the interparticle interaction forces are of general importance in so far as they control the state of aggregation in the suspension, but the surface charge on the particles is utilised in some way to effect the electrokinetic motion of the particle (e.g. by electro- or dielectro- phoresis) or of the fluid (e.g. by electroosmosis). As it is beyond the scope of this paper to discuss the role of primary forces in all of these separation processes, let it suffice to concentrate attention on crossflow electrofiltration.

In crossflow filtration a fouling layer deposits on the membrane surface, however high the crossflow velocity. This results in a decreasing rate of filtration. When an electrical field is superimposed over the suspension flow volume above the membrane, an appropriate polarity of field will cause an electrophoretic velocity to be imparted to the particle which counteracts the drag induced velocity of the particle towards the membrane surface. The effect is to reduce, or in some instances eliminate, deposition at the membrane surface and thus facilitate a high average rate of filtration. Figure 13 shows the effect that an electric field gradient has on increasing the filtrate rate obtained from the filtration of a china clay suspension by a polypropylene membrane. Similar effects can be demonstrated with most sub-5 micron suspensions when the zeta-potential is greater than about $|20| \text{ mV}^{9-11}$.

A lesser potential is required when the particle size is smaller, which probably is indicative that the suspension needs to be reasonably well dispersed in order that the technique can be effected.

CONCLUSIONS

There is overwhelming evidence pointing to the importance of colloid science in solid/liquid separation technology. The dominant phenomenon affecting the separation behaviour of finer (smaller than about 10 micron) particles is the repulsive interparticle force. This is affected by the ionic strength of the solution and increases with the solids volume fraction in the solid/liquid mixture and with the zeta-potential. An important plot, and one which is often measurable for process suspensions, is the zeta-potential versus pH diagram. Figure 14 links the effects of the repulsive forces at the scale of the particle to what is observed at the process level through such a plot. This plot has to be measured for each suspension being scrutinised as the precise curve is dependent on impurities at the particle surfaces, particularly for metal oxides. For example, the IEP of anatase (titanium dioxide) has been measured to vary over the range:

pH = 6 for washed particles
 = 5.9 for 'dirty' particles
 = 5.7 for washed and heated particles
 = 4.5 for particles aged 6 months in distilled water

Around the IEP the process engineer can expect:

- Faster settling rates
- Rapid filtration/expression
- Higher moisture content cakes/sediments.

due to the aggregation of particles in a suspension where interparticle repulsive forces are very small.

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At maximum or minimum zeta-potentials the process engineer can expect:

- Slower settling rates
- Slower filtration/expression rates
- Lower moisture content cakes/sediments.

due to the existence of greater repulsive forces maintaining the particles better dispersed in the suspension.

At pHs beyond those at which maximum or minimum zeta-potentials first occur the process engineer can expect intermediate settling, filtration and expression rates and intermediate cake/sediment moisture contents. This is due to compression of the electrical double layer around the particle, caused by high ionic strengths in the solution, whilst a relatively high zeta-potential exists.

ACKNOWLEDGEMENTS

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NOMENCLATURE

a	particle radius (m)
A	Hamaker function (J)
C	volume fraction of solids in solid/liquid mixture (-)
e_e	elementary charge (C)
F_A	attractive force between two particles (N)
F_R	repulsive force between two particles (N)
F_T	net force acting between two particles (N)
I	ionic strength of the solution (mol l^{-1})
K_B	Boltzmann constant (J K^{-1})
n_s	number of ion pairs in the solution (m^{-3})
N_A	Avogadro's constant (mol^{-1})
T	absolute temperature (K)
x'	shortest interaction distance between two particles (m)
z	valence of an ion (-)

Greek symbols

δ	thickness of the Stern layer (m)
ϵ	dielectric constant (-)
ϵ_0	permittivity of free space (F m^{-1})
κ	reciprocal thickness of the electrical double layer (m)
ψ	potential at the plane where the diffuse double layer begins (V)
ζ	zeta-potential (V)

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TABLES AND FIGURES

C (-)	>0.7	>0.7	0.7	0.55	0.1
x' (nm)	0.1	1	10	100	1000
F_A (N)	-4.43×10^{-8}	-4.14×10^{-10}	-3.99×10^{-12}	-2.83×10^{-14}	-2.35×10^{-17}
F_R (N)	4.53×10^3	4.52×10	3.76×10^{-1}	2.31×10^{-5}	2.24×10^{-35}
F_T (N)	4.53×10^3	4.52×10	3.76×10^{-1}	2.31×10^{-5}	-2.35×10^{-17}

Table 1: Approximate interaction forces for 1 micron particles with a 20 mV zeta-potential suspended in water with monovalent ions in solution.

pH	Specific resistance of cake (m/kg)	Porosity of cake	Moisture content of cake (%)
2.9	2.76×10^{11}	0.79	59.4
5.65	5.07×10^{11}	0.8	60.9
8.0	21.5×10^{11}	0.79	59.4
9.45	40.0×10^{11}	0.76	55.2
11.6	96.1×10^{11}	0.73	51.3

Table 2: Cake resistance and moisture content variations with suspension pH.

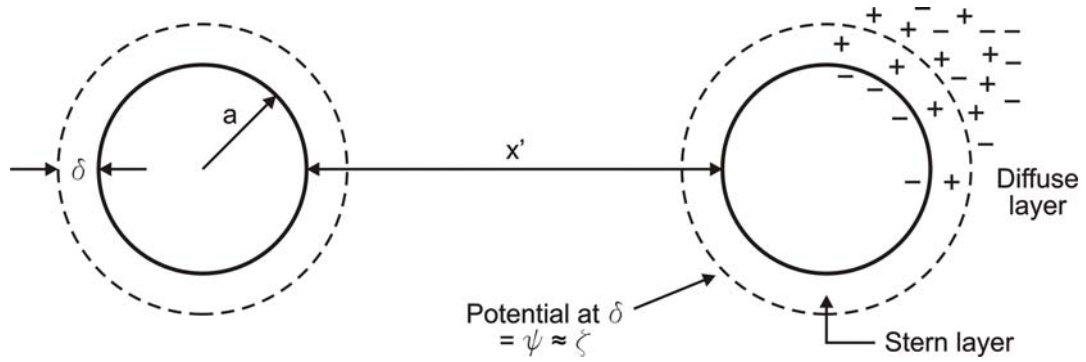


Figure 1: Single particles in solution.

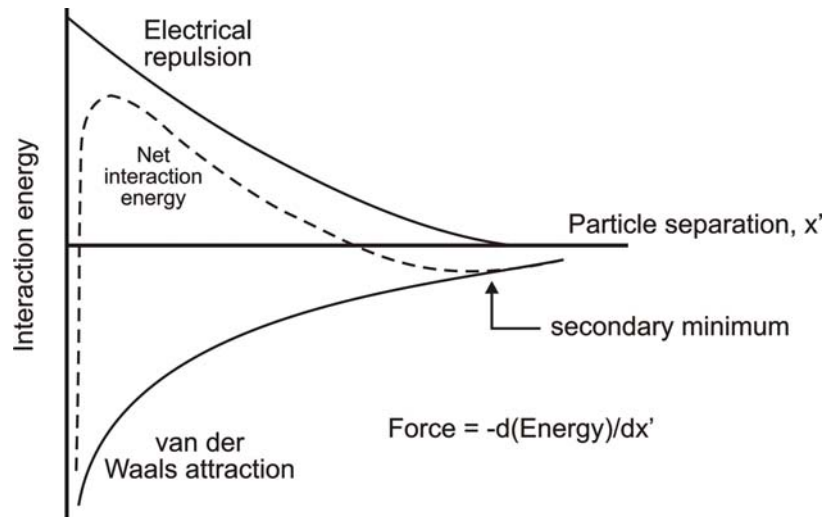


Figure 2: Interaction energies between particles.

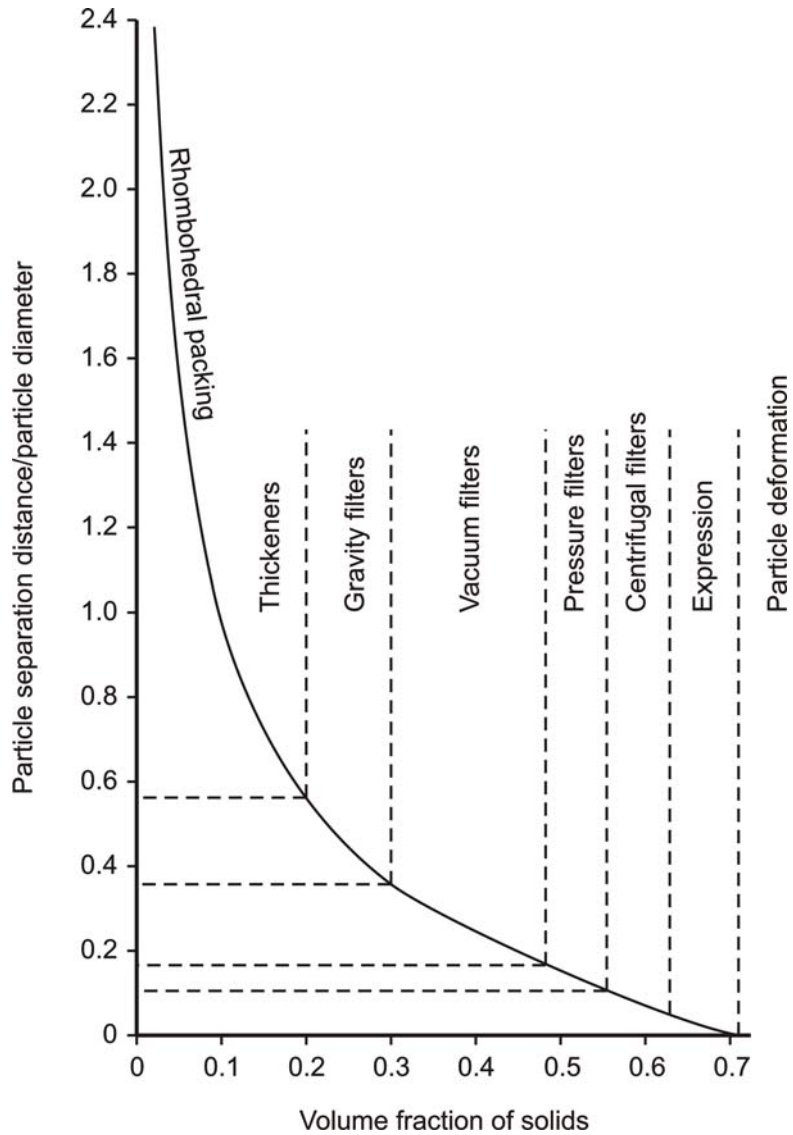


Figure 3: Separation processes in relation to an ideal particle packing and particle separation distance.

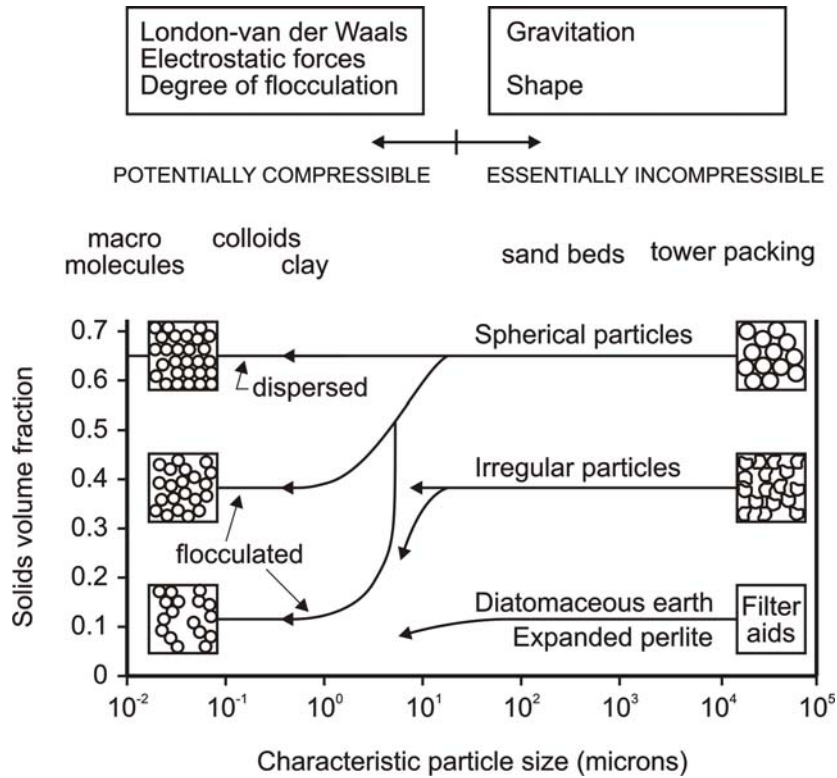


Figure 4: Relation of sediment/cake solids volume fractions to particle size, shape and degree of aggregation.

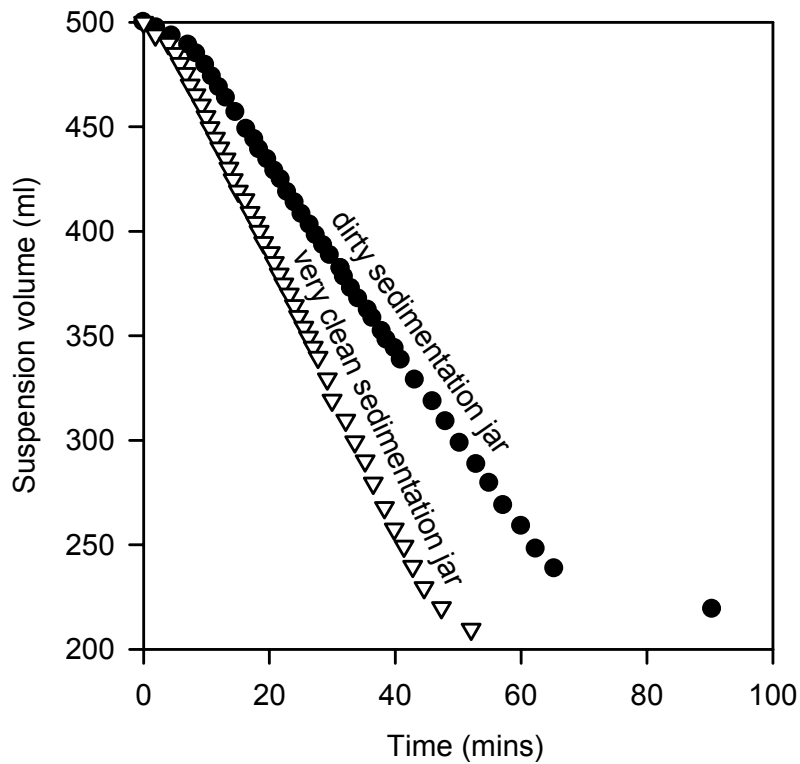


Figure 5: Sedimentation of ground calcite. Initial volume fraction 0.025; pH 10.0; Average particle size = 3 microns; Sedimentation jar diameter = 40 mm.

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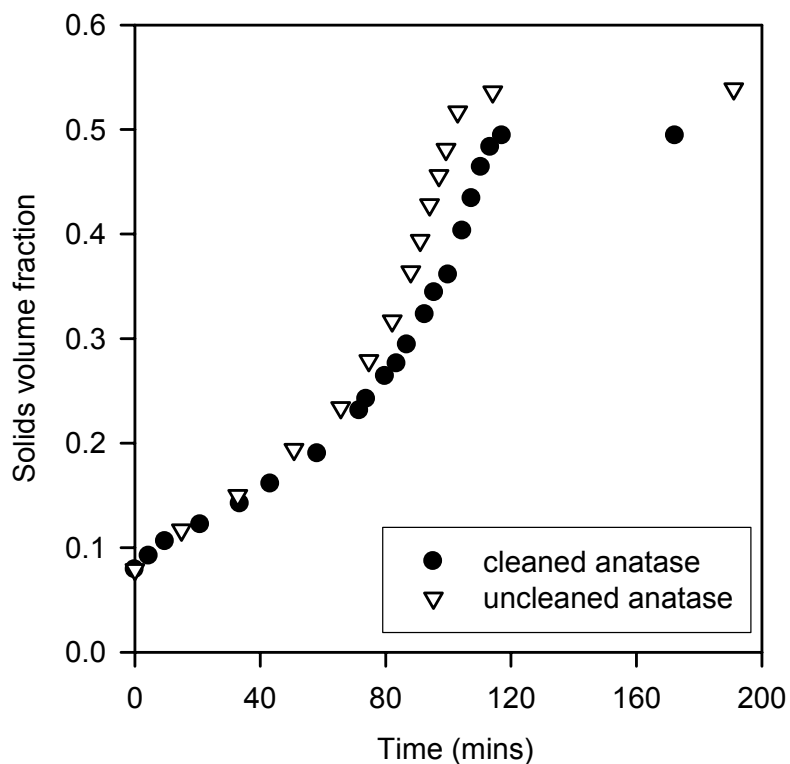


Figure 6: Effect of particle surface impurities on expression of anatase.

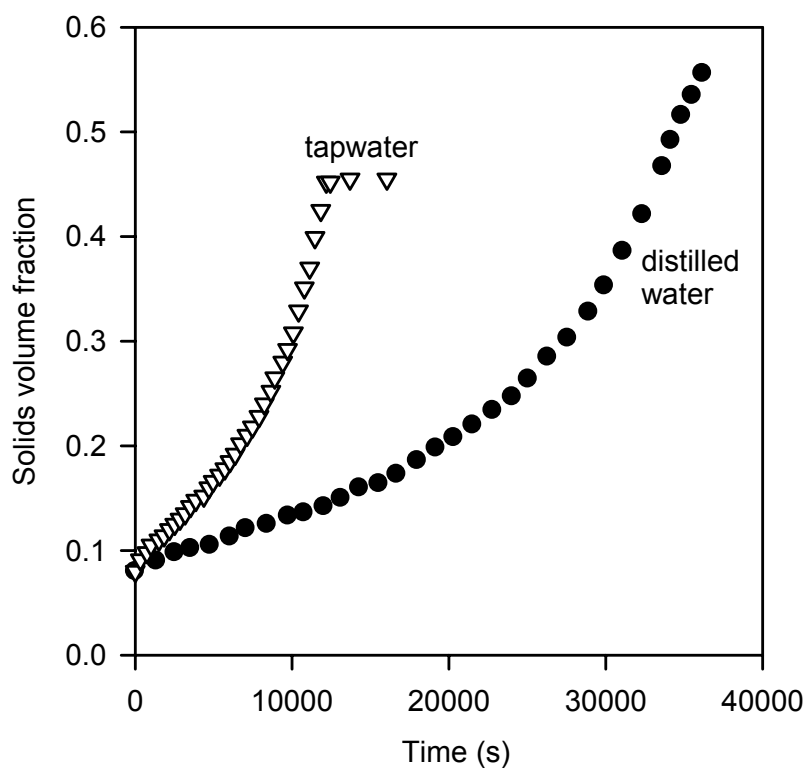


Figure 7: Effect of solution environment (pH = 7.46 and 7.49) on expression of anatase.

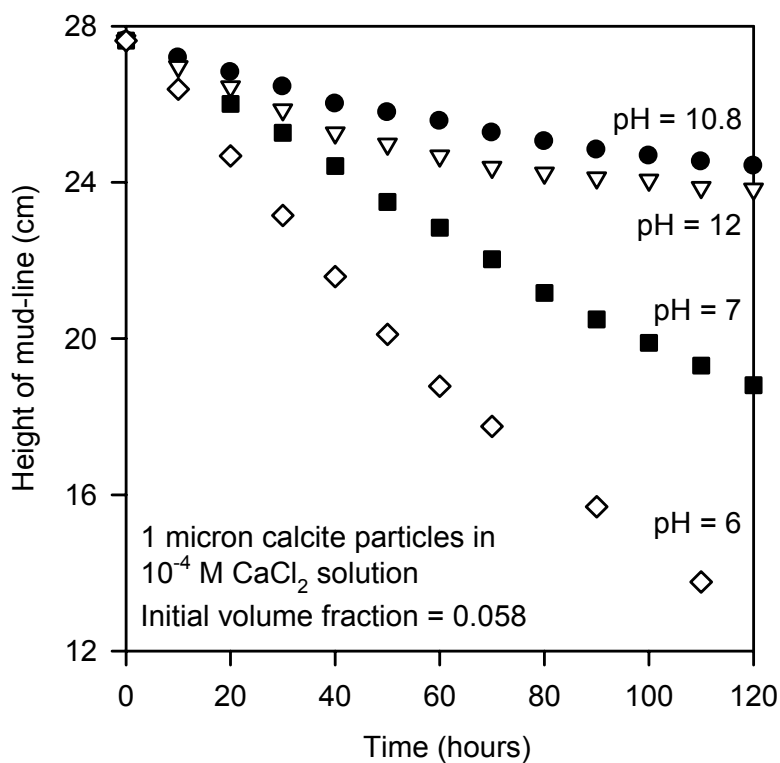


Figure 8: Effect of pH on sedimentation of calcite.

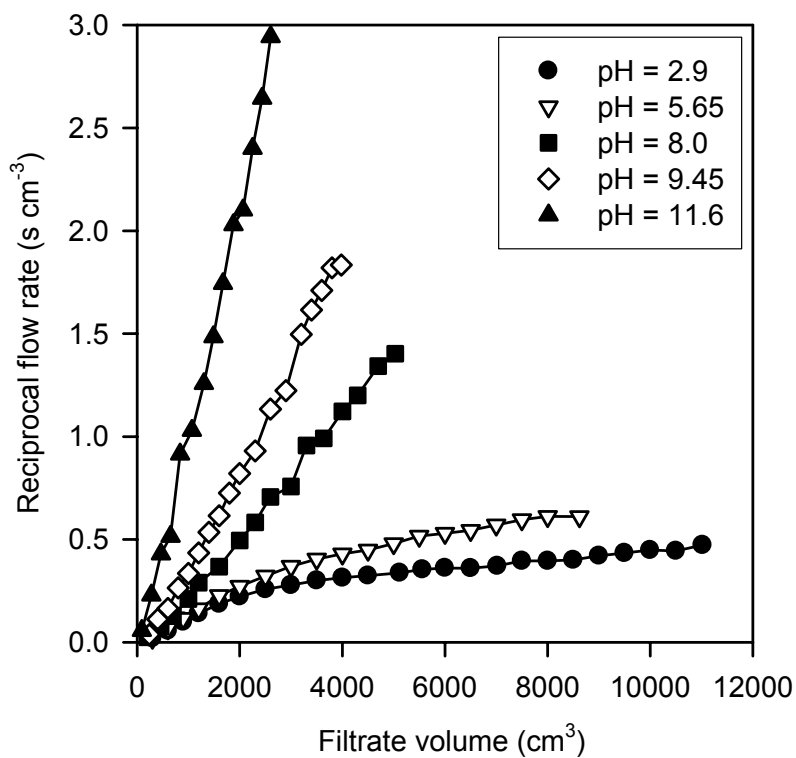


Figure 9: Effect of pH on filtration of china clay. Suspension concentration = 0.5% by volume; Average particle diameter = 0.5 microns; Filtration pressure = 20 psi.

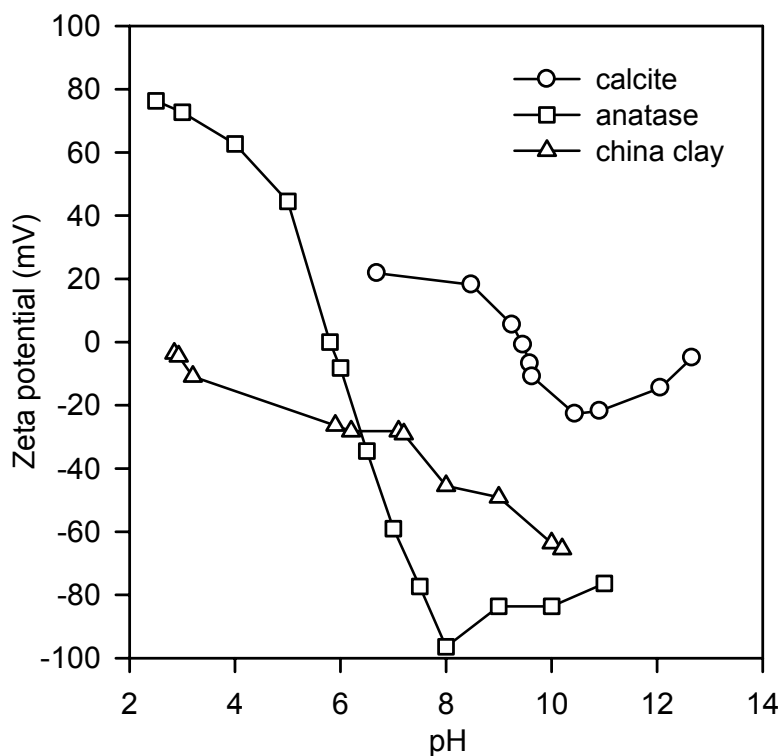


Figure 10: Zeta-potential vs. pH diagrams for example suspensions of calcite, anatase and china clay.

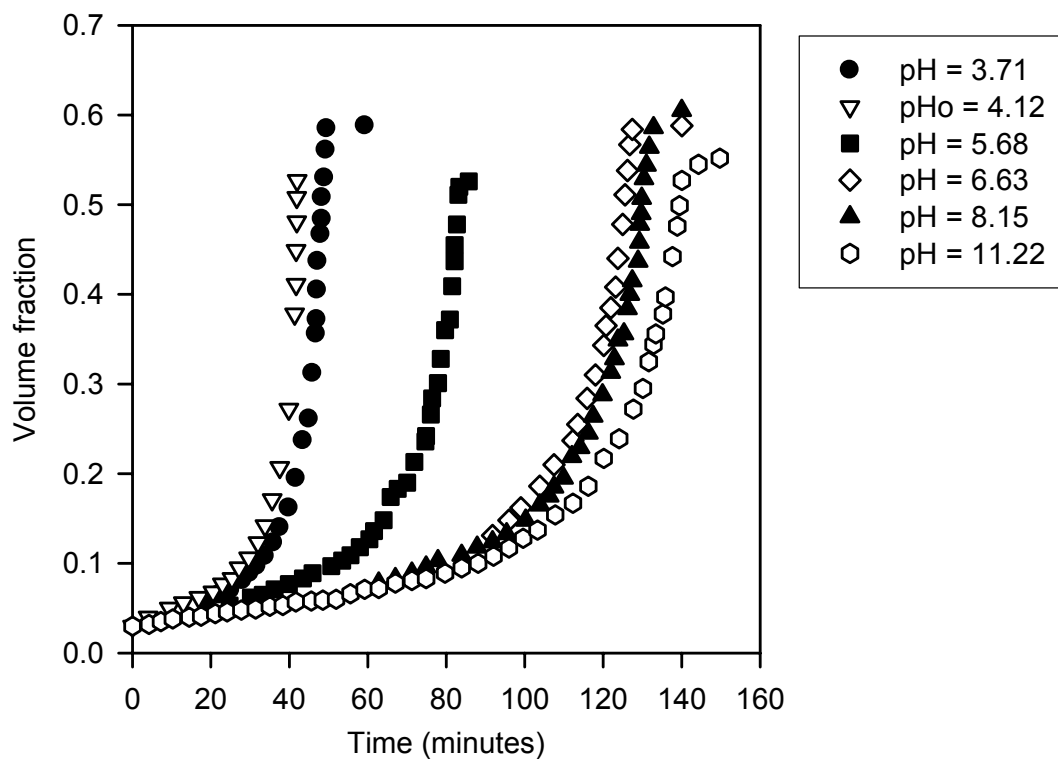


Figure 11: Effect of pH on expression of anatase (at constant pressure).

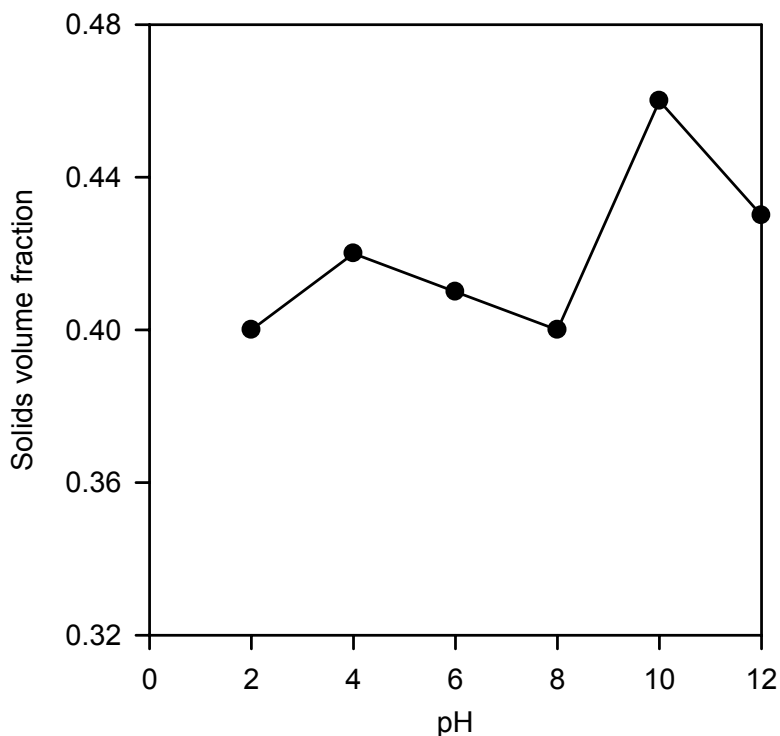


Figure 12: Effect of pH on anatase cake moisture contents in a tube press filter.

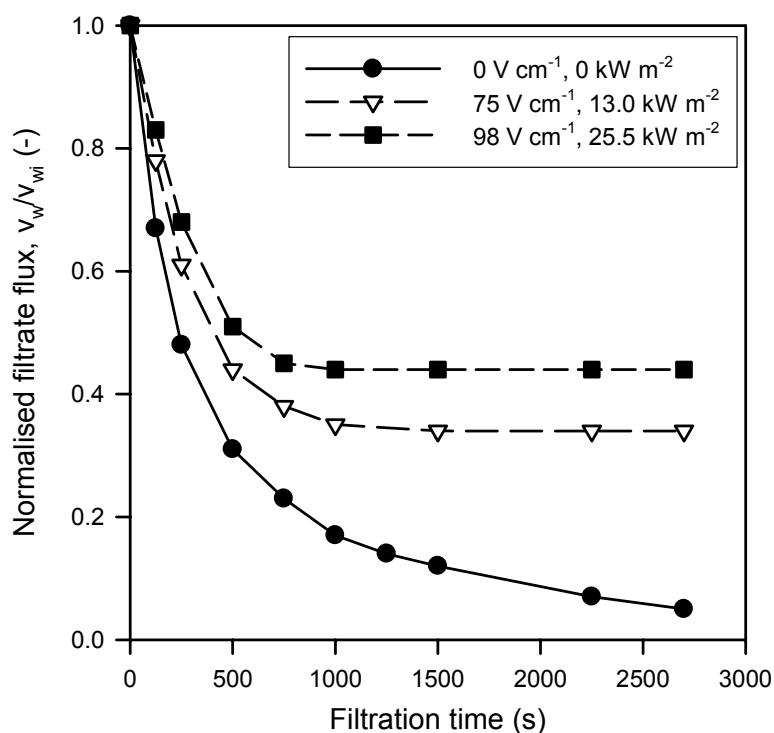


Figure 13: Effect of electrophoretic velocity on permeate rates from a polypropylene crossflow filter. Suspension concentration = 0.39% v/v china clay in water; Crossflow velocity = 0.9 m s⁻¹; Initial filtration rate = 0.97 m³ m⁻² h⁻¹; Suspension conductivity = 1.439 × 10⁻⁴ ohm⁻¹ cm⁻¹; Membrane type = pleated polypropylene; Electrode separation = 4 cm.

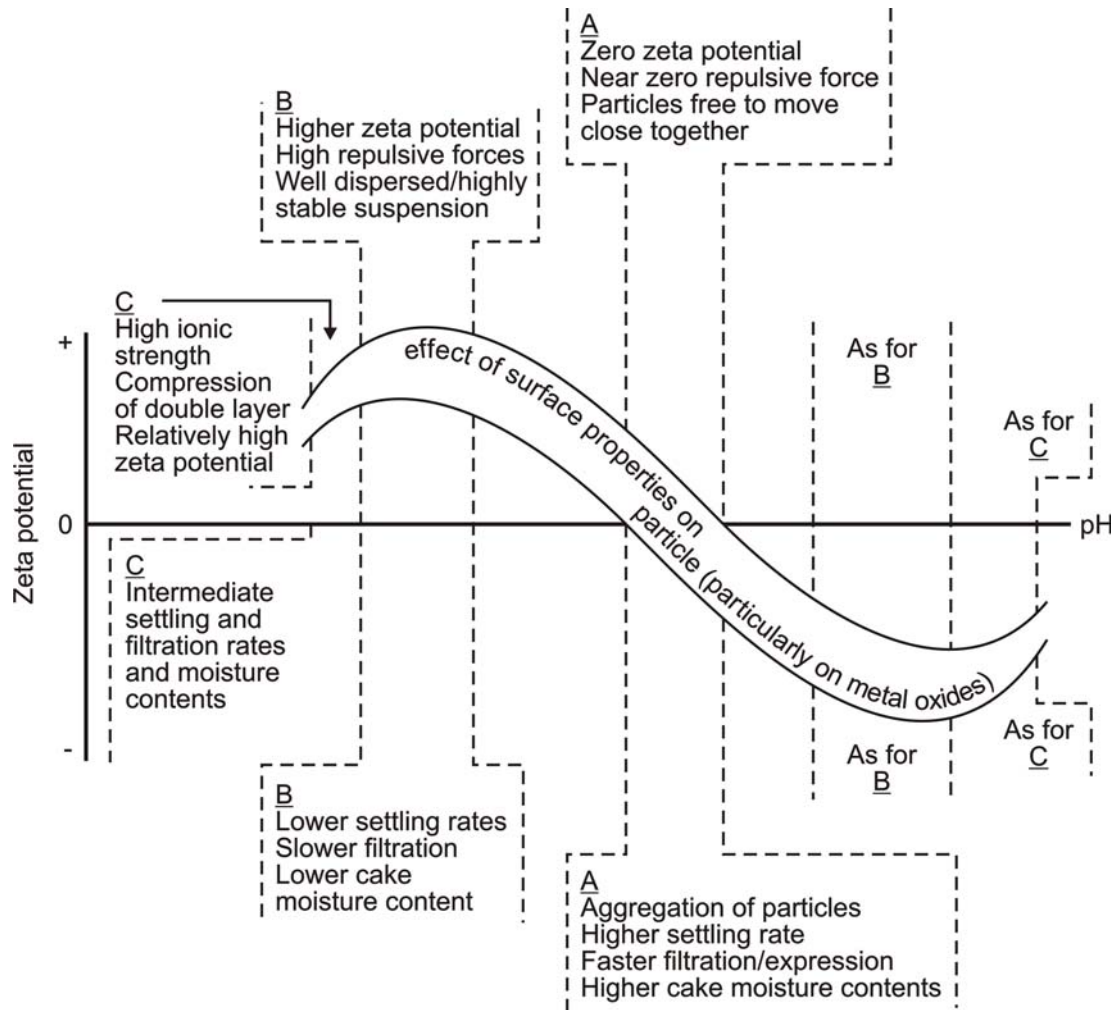


Figure 14: The link between particle phenomena and process separations.