

## The Physical Characterization of Suspensions and Slurries: Why they have to be measured without dilution

A significant number of commercial products involve, either in the final state or at some stage of their production, suspensions of particulate materials dispersed into liquid vehicles often at high volume fraction. Quality dispersions are needed in applications as diverse as cosmetics, pharmaceuticals, and ceramics, but it is often under-appreciated that, in order to be properly formulated, these suspensions or slurries must be analysed as they are prepared and without dilution. The characteristics of the solid-liquid interface control dispersion behaviour and can influence, among other things, important processes such as adhesion, flotation and, in concentrated suspensions, rheological behaviour. This short Technical Note will explain why this is so and introduce a simple and powerful device for measuring key characteristics of virtually any dispersion.

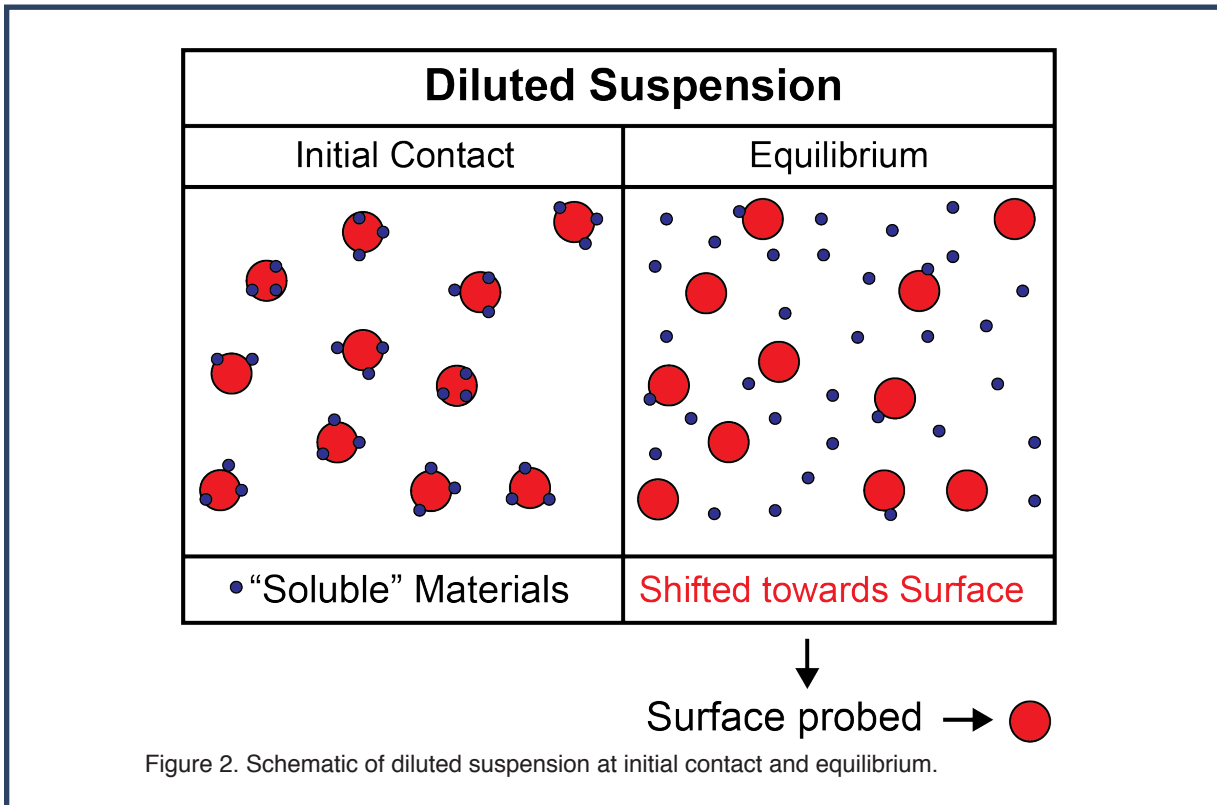
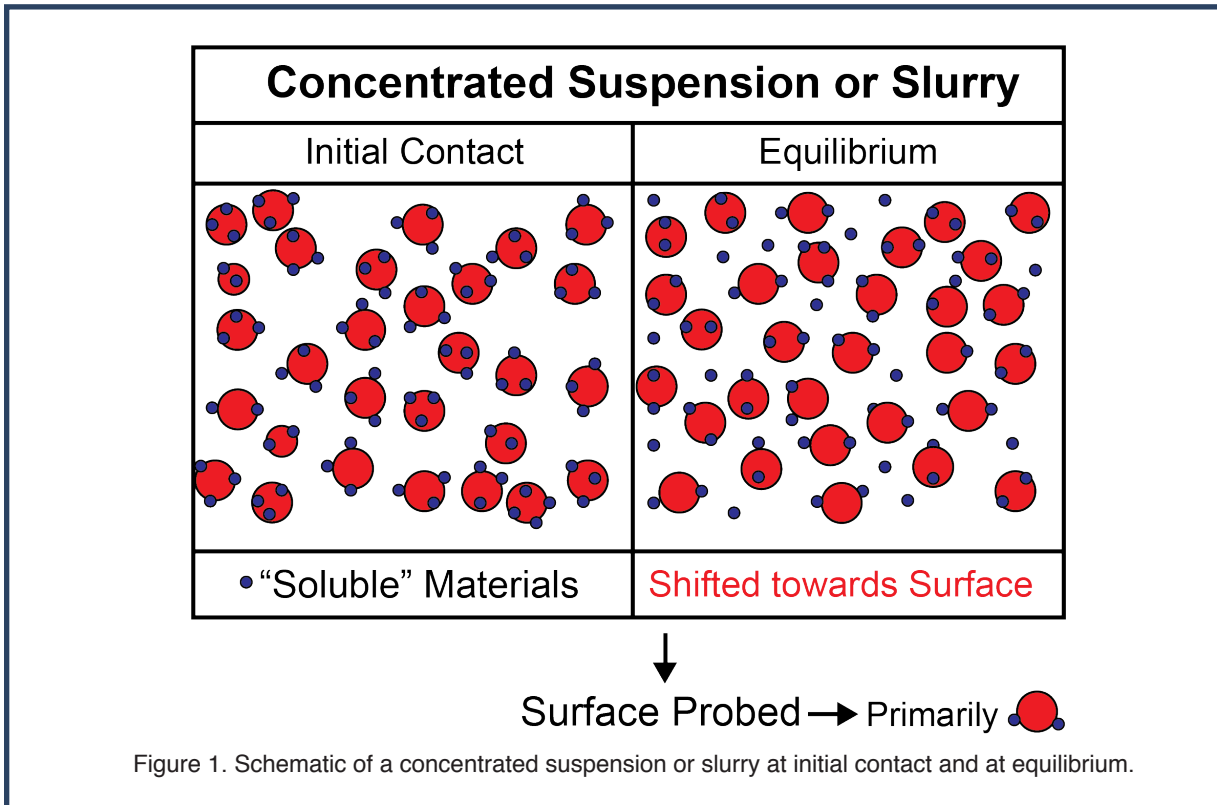
Let's look first at Figure 1. The illustration on the left depicts any concentrated suspension as it is made or formulated (called "initial contact") and provides a representation of particles of a material (red circles) within a liquid (white background). The "surface chemical nature" of the material is imagined as a series of blue dots. This could be the fundamental functional groups comprising the material, or something adsorbed on the material's surface. On the right-hand side is a depiction of that same suspension in which a defined chemical "equilibrium" has been established between the particles and the liquid. This means that any species that can potentially desorb, or dissolve, or dissociate to the limit or capacity of the available liquid phase. For a given volume of the suspension, the greater the particle concentration is, the less volume of free liquid

is available. This means that the solubility limit for any surface species can be exceeded, thus inhibiting any further desorption, dissolution, or dissociation. So, in concentrated suspensions, the chemical equilibrium is *always* shifted towards the surface, and the little icon at the bottom right-hand side of Figure 1 represents the surface that will be primarily "probed" (i.e., evaluated/measured/monitored) in such a situation.

Now let's look at Figure 2, which considers the same sample but after being diluted, as can happen when preparing samples for measurement/analysis. As we see on the left-hand side in this schematic, not surprisingly, the initial contact in a diluted suspension looks much like that seen in Figure 1 except that there are much fewer particles. However, once this system reaches its chemical equilibrium state, it is completely different because the total volume of liquid is now much larger, which allows more surface species to desorb, dissolve, or dissociate. The consequence is that the chemical equilibrium is now shifted away from the surface and towards the solution – and, the more dilute the suspension, the greater will be this shift.

Note that in the case of extreme dilution – as is necessary for particle characterization techniques that are based on, for example, light scattering – one can end up with a suspension where the surface probed can now be a surface that has a completely different surface chemical activity (i.e., very few or no blue dots), again represented by the small icon at the bottom of the Figure 2. This is now a totally altered situation from that which existed in the concentrated suspension (Fig. 1).

*“In order to be properly formulated, suspensions or slurries have to be analysed as they are prepared and without dilution.”*



This can be illustrated in the following example using mobility measurements of an aqueous suspension of barium titanate, which is used, for example, in the manufacture of ceramic capacitors. The mobility is related to the surface charge of a material; it is a good index of the magnitude of the electrostatic repulsive interaction between particles and can be used to predict and control dispersion stability. The dispersion characteristics of ceramics and refractories affect green body strength and the degree of shrinkage on

sintering. Thus, reliable mobility determinations are of practical concern in ceramics preparation, processing, and application. In these samples, the mobility was measured as a function of pH at a high solids concentration (20% w/w) using an electroacoustic device. The suspension was then extensively diluted using deionized water and the mobility measured, again as a function of pH, using an electrophoretic light scattering device and the results compared (Fig. 3).

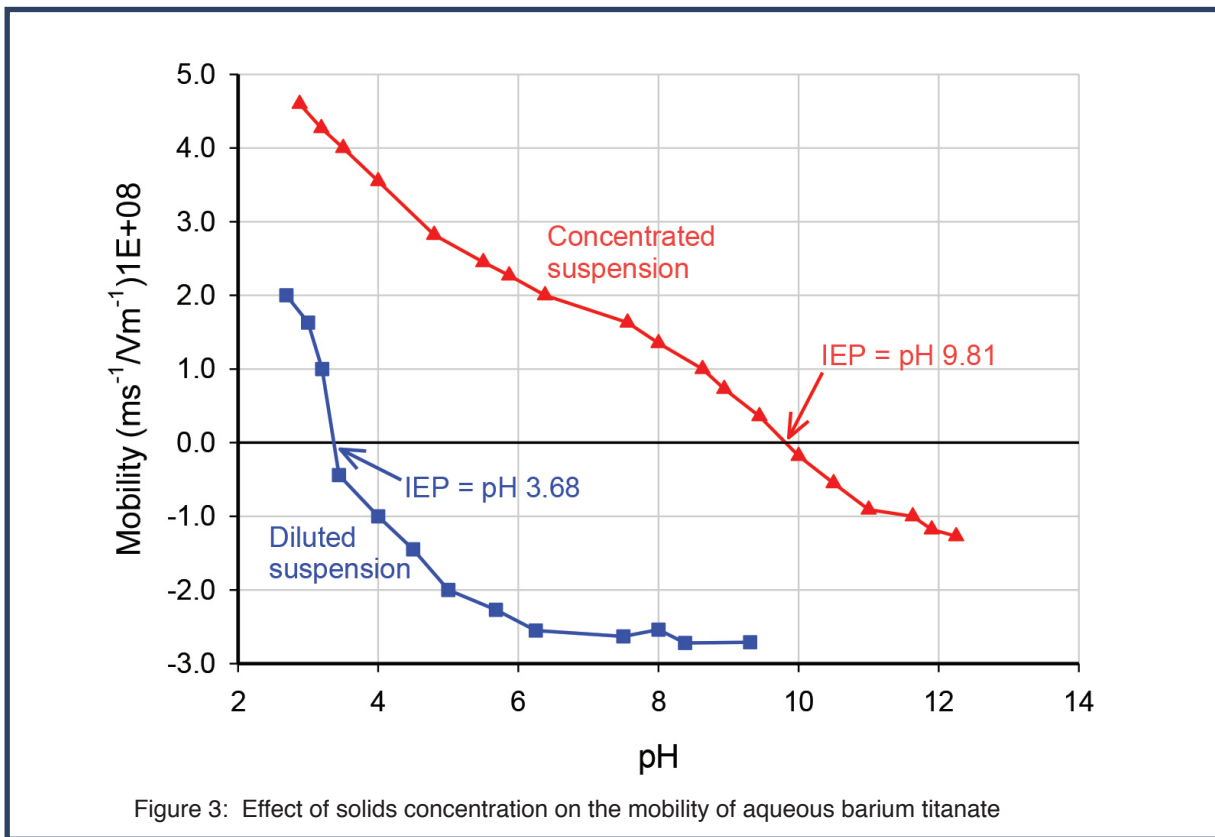


Figure 3: Effect of solids concentration on the mobility of aqueous barium titanate

It is clear that not just the shapes of the mobility vs pH profile, but also the iso-electric point (IEP), are totally different for the two suspensions. The IEP is the pH at which the material will have no surface potential (charge) and so such a suspension will be inherently unstable, and all the particles will eventually aggregate. This can have serious consequences as it impacts, among other things, subsequent formulation – for example, the addition of ionic additives such as

surfactants, dispersants and polyelectrolytes – as well as long-term stability.

Further examination of the data is beyond the scope of this Technical Note. However, the important take-away point is that dilution is never an innocuous process. The consequence is that you will get a value – from whatever characterization technique used – that is not representative of the original concentrated suspension

and so may not translate into a useful performance metric. To avoid this issue, where possible, it is always preferable to make measurements on suspensions or slurries at their *use* concentration.

This does not mean that you *cannot* dilute any suspension. There are ways to minimize (but not completely eliminate) the shift in chemical equilibrium that occurs, for example by using the mother liquor from a centrifuged sample of the original suspension but this is often difficult to obtain in practice. More discussion of this issue is beyond the subject of this short note.

Formulations, though initially prepared as concentrates, are sometimes subsequently diluted to obtain the commercial product. However, dilution, if not performed correctly, can lead to suspension instability which can

affect, for example, long-term shelf storage. Crucially, if it is necessary to dilute a suspension, then it is essential to check the linearity of any metric as a function of the dilution process.

Techniques based on nuclear magnetic resonance (NMR) relaxation avoid these dilution problems, and the Mageleka *MagnoMeter* XRS is able to make measurements on suspensions and slurries prepared at almost any industrially relevant concentration. This not only removes the practical need for dilution – no matter what the application – but also allows monitoring of any dilution process. Importantly, the technique is non-invasive and non-destructive so that samples – at whatever concentration – can be saved and re-measured at some future date. This allows examination of any time-dependent behavior.

*For more information, to send samples, to arrange a demonstration of the MagnoMeter at your facility, or to talk to one of Mageleka's technical applications specialists, please email [roger@mageleka.com](mailto:roger@mageleka.com)*