Quick Guide

Sibelco Quick Guide to:
Measuring particle size

1. Why do we measure particle size?

Particle size measurement plays a significant role in process and product control and design in many industries.

In ceramics, variances in the size of particles during manufacture can not only affect the minerology of a final product, but also its rheology, casting rate, drying shrinkage and strength.

Such is the impact of even the slightest change, it is critical that ceramics manufacturers measure even the smallest particles.

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2. How do we measure particle size?

The sedimentation technique

The ceramics industry traditionally uses sedimentation techniques to measure particle size. From the basic Andreasen pipette methods, to the more sophisticated instruments based on X-Ray attenuation (such as Sedigraph™), measure the settling rate and obtain the equivalent spherical diameter (e.s.d) from Stokes Law for settling spherical particles.

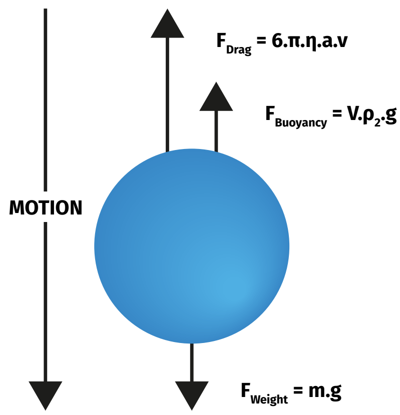

FIGURE 1: SCHEMATIC DIAGRAM SHOWING FORCES ACTING ON A SPHERE IN A FLUID

What is Stokes' Law?

Stokes' Law is the mathematical description of the force required to move a sphere through a quiescent, viscous fluid at a specific velocity:

The buoyancy force is simply the weight of the displaced fluid:

$$F_{buoyancy} = m_{fluid}\, g = V\rho_2 g$$

Balancing Forces: $$F_{drag} + F_{buoyancy} = F_{weight}$$

$$(6\pi\eta a\, v) + (V\rho_2 g) = mg$$

Rearranging gives the Stokes settling equation:

$$v = \frac{2a^2(\rho_1 - \rho_2)g}{9\eta}$$

where: v is the rate of settling of a particle of volume V, density ρ_1 and an equivalent spherical radius of a in a fluid of density ρ_2 and viscosity η.

But while Stokes' Law is straightforward, it is subject to some limitations.

Specifically, the relationship is only valid for Laminar flow – engineers distinguish between this and turbulent flow by using a parameter known as the Reynold's number. The Buckingham Pi Theorem relates the Reynold's number (NRE) to the viscous forces within a fluid:

Using the Stokes' and Reynold's equations, Reynold's number can be plotted against an equivalent spherical diameter.

$$N_{RE} = \frac{2\rho_2 va}{\eta}$$

Figure 2 shows a plot for a selection of common minerals in water at 20°C.

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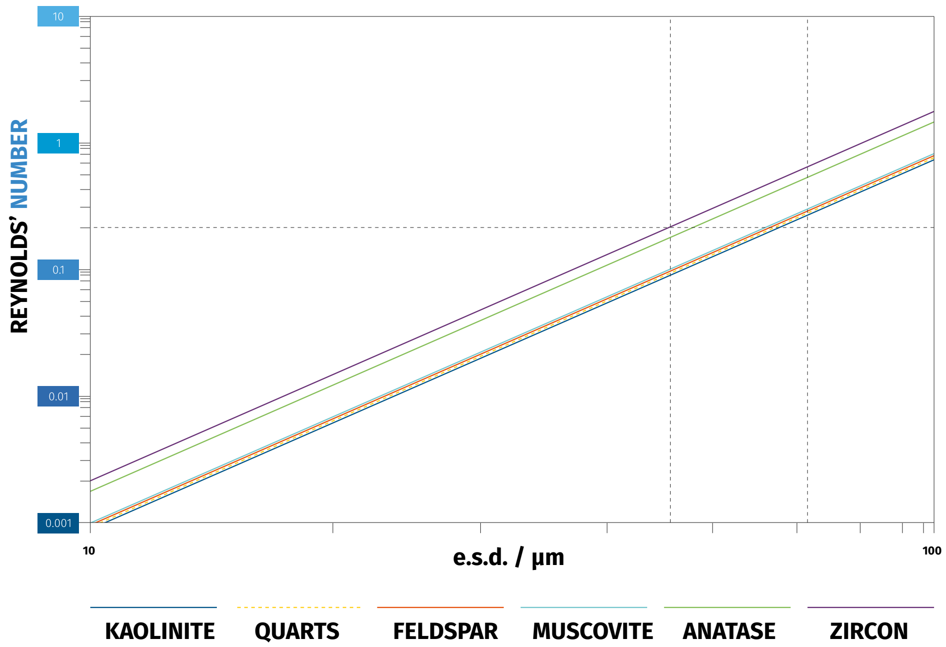
A Reynold's number greater than '1' denotes full turbulent flow and less than ~0.2 full laminar flow. Using a Reynold's number of 0.2 as an absolute upper limit, it can be seen that typical 'heavy' minerals such as zircon or anatase cannot be reliably measured above ~46 µm e.s.d; this upper measurable limit is increased slightly to ~61 µm for lighter minerals such as feldspars and clay minerals. Note that if a sample contains particles greater than these sizes the turbulent flow created will affect the result for the finer particles.

FIGURE 2: DEPENDENCY OF REYNOLDS NUMBER ON E.S.D. FOR SELECTED MINERALS AT 20 °C IN WATER



Brownian motion plays a major part in settling experiments at small particle sizes; particles of 2 µm e.s.d. will almost certainly be affected and particles less than 1 µm significantly so – possibly to the point that they do not settle at all, giving an apparently finer result.

Two further factors that can influence the result of a settling experiment are worth noting:

i) Hindered settling: in general this has an effect at solids level greater than ~1% by mass, and hence settling experiments should be conducted at concentrations less than this.

ii) Mixed materials with differing densities such as zircon and feldspar in a glaze slurry: the density used for the calculations is an average for the system, thus giving an 'averaged' combined result for the e.s.d.

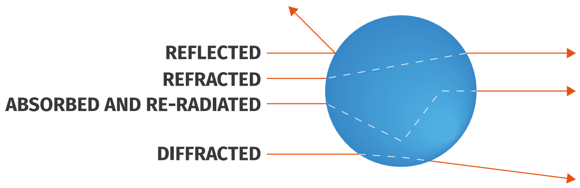
Low Angle Light Scattering – an alternative to sedimentation

The main alternative to sedimentation techniques; Low Angle Light Scattering (LALS) until recently, had serious limitations. Among others, i) the wavelength of the helium neon red laser (633 nm) used as a light source limited the minimum particle size measurable, and ii) absorption or refraction of light by the particles under evaluation is greater at smaller diameters and was not accounted for in the Fraunhofer model, originally used to calculate the equivalent spherical diameter.

With the introduction of blue lasers (with shorter wavelengths) and improved detector electronics in instruments such as those produced by Malvern™, particles down to 0.02 μm can now be detected.

In addition, increased computing power allows the highly complex, but more accurate Mie theory of light scattering to be more readily solved. The theory incorporates the Fraunhofer model, but also takes into account other factors such as the refractive index and absorption of the particle. The choice of refractive indices and absorption differences in mixed mineral systems can cause problems.

FIGURE 3: SCHEMATIC DIAGRAM SHOWING LIGHT SCATTERING MECHANISMS



What are the differences between results obtained from sedimentation and light scattering methods?

Stokes' settling experiments make the assumption that particles are smooth and spherical. However in reality, 'normal' particles have a much higher surface area that that of the equivalent sized sphere, meaning the drag is much greater and the particles settle much more slowly.

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Light scattering experiments use the data gathered to model the volume of the particle and then calculate the equivalent spherical diameter for that volume. For example, a ‘real’ rectangular particle, (illustrated in fig.4) has the same volume as that of the sphere.

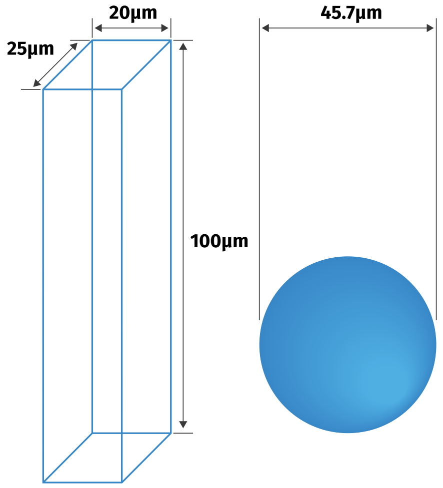
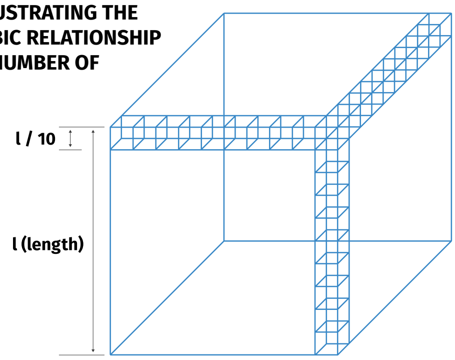


Figure 4: Illustration of a rectangular particle and the e.s.d sphere

A LALS experiment would report the equivalent spherical diameter, however, the rectangle has a surface area ~50% greater than that of the equivalent volume/ mass sphere, and therefore greater drag – the sedimentation experiment would determine the rectangular particle as finer as it will settle slower than predicted. Thus, both methods have their inherent differences and limitations, but in general a settling experiment result normally appears finer than the equivalent light scattering

What is the significance of smaller particles?

FIGURE 5: SCHEMATIC DIAGRAM ILLUSTRATING THE CUBIC RELATIONSHIP IN NUMBER OF



Take a cube of unit length (representing the size of a larger particle). Now imagine smaller cubes (or smaller particles) occupying the same volume as the larger cube.

On this basis, 1,000 of our smaller cubes could fit into the same volume as the original.

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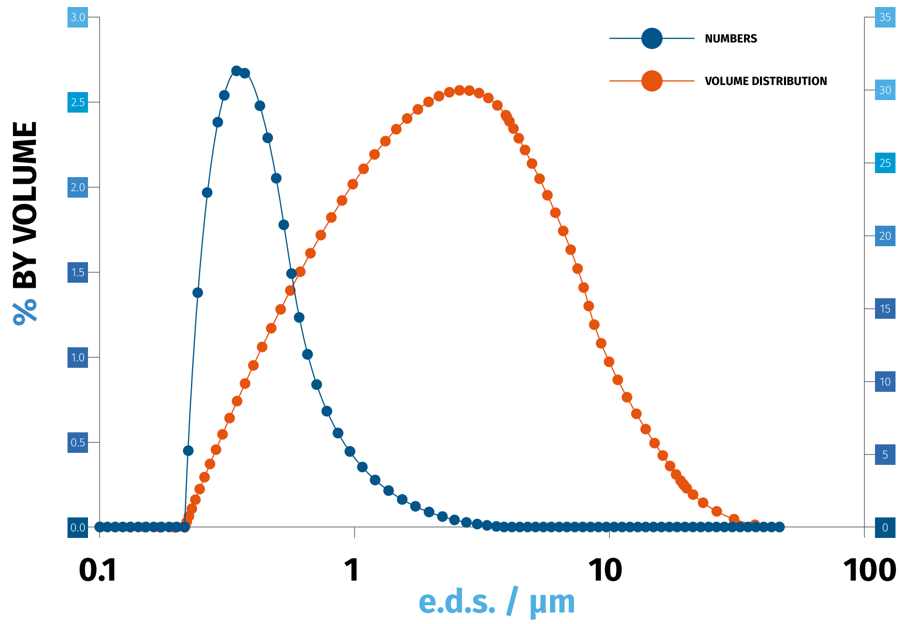
Particle size distributions are typically reported in volume terms, and a typical distribution covers a size range between 100-0.1 µm, three orders of magnitude. Under these conditions, the number of finer particles would be dominant and their influence on the product, significant. See figure 6 for the results in a natural clay sample.

FIGURE 6: LALS PARTICLE SIZE DISTRIBUTION FOR A PLASTIC CLAY AND THE



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