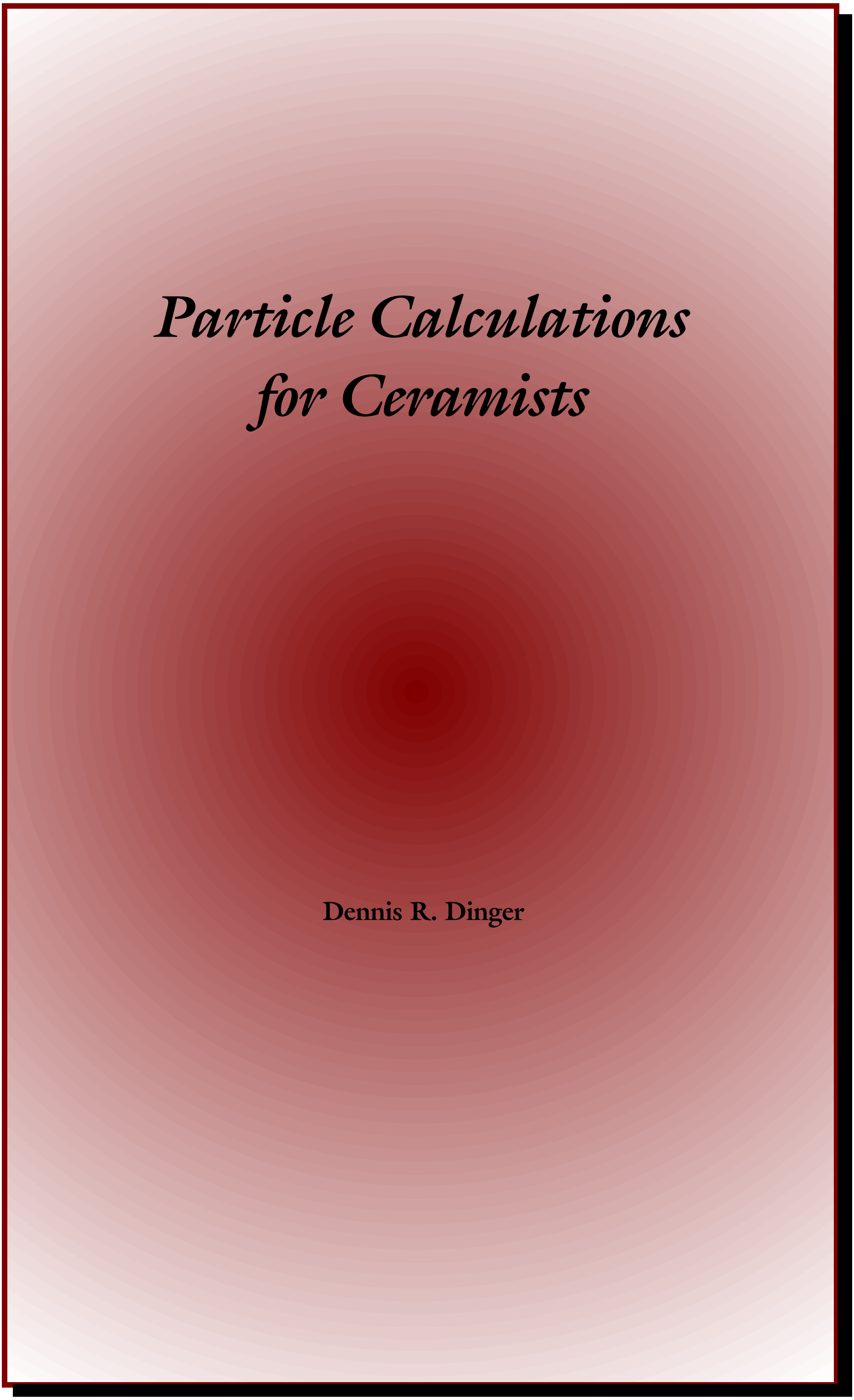

Particle Calculations for Ceramists

Dennis R. Dinger

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Preface

The main goal of this work has been to produce a handy, small reference that contains explanations, examples, and sample calculations for all of the routine calculations performed by ceramists (including engineers, technicians, artists, and students,) as well as materials engineers, technicians, and students, and any others who are or will be working with powdered raw materials.

At the turn of this new 21st Century, ceramic technology (and powder technology in general) is advancing at a rapid pace. Most new ceramics and materials textbooks are written to keep up with that pace, and to document the latest developments in particle science, engineering, and technology.

It appears that a handy reference that can be used on a daily basis by those responsible for the production of ceramic products has been overlooked. If such a book contained explanations for some of the simple phenomena, as well as demonstrations of the calculations, that would be a plus.

Today's students are expected to know more and more about the use and programming of computers when they leave high school and step through the college door. Until only recently, we had to introduce freshmen to the use of computers and appropriate software packages. Now, we assume that they have received such instructions in high school (and they generally have), we expect them to bring their own PCs to college and know how to use them, and we teach *introductory* courses that cover more advanced topics.

Similarly within the fields of ceramics and materials engineering and science, the level of technology has advanced rapidly, but high schools are *not* teaching ceramics fundamentals to all (any?) students.

Within undergraduate ceramics and materials curricula, pressures are being applied to reduce the number of credit hours required for bachelors degrees, as well as to teach all of the cutting-edge methods, principles, and phenomena. Less and less time is available (and less is being spent) to cover necessary fundamental topics, so the more glamorous, advanced topics can be offered.

This handbook is an attempt to help bridge this perceived gap regarding particle size distribution phenomena. Rather than expecting students to be familiar with all of the fundamental particle phenomena, principles, and calculations (or to learn them on their own if they're not), point them to this handbook. Rather than expecting technicians to not ask questions but to just follow the written SOPs (the black box approach), point them to this handbook. Rather than expecting all ceramic and materials engineers to instantly be able to put their fingers on references that explain these topics (which are scattered far and wide throughout the textbooks), point them to this handbook.

I hope you find this handbook helpful on a daily basis.

Dennis R. Dinger
1 August 2001

Nomenclature

[=]	has units of
VSA	volume specific surface area
SSA	mass specific surface area
g	grams
ρ	density
m	meters
cm	centimeters
mm	millimeters
μm	micrometers
nm	nanometers
D_L	largest particle size
D_s	smallest particle size
n	distribution modulus
IPS	InterParticle Spacing
a_i	property value in i^{th} size class or i^{th} component
x_i	mass or volume fraction in i^{th} size class or i^{th} component
vol%	percentage on a volume basis
wt%	percentage on a weight or mass basis

Chapter One

Surface Area

Importance to Ceramics

The surface areas of powders are critically important to the successful production of ceramic wares. The capability of particles to slide against one another (surface against surface) during processing not only affects but controls the viscous behavior of production bodies and suspensions.

Chemical additives, flocculants, deflocculants, plasticizers, binders, etc., are routinely added to ceramic forming bodies and suspensions to adjust viscosities and rheologies, gelation behaviors, and a variety of other green, dry, and fired properties. These additives all interact with the powder surfaces. Some supply lubrication to reduce the friction between interacting particles. Others supply binding properties.

The area of surface present in the powders that form a body and the surface concentrations of coatings on those surfaces control particle/particle friction, electrostatic surface charges, binding strengths, plasticizing properties, etc.

The total surface area of the powders in a body should not only be measured but under constant production control. Additions of constant volumes of chemical additives to bodies with surface areas that vary from batch to batch will produce fluctuating additive concentrations on those surfaces and fluctuating processing properties in the forming bodies.

Practical Examples

Consider two identical black walls to be painted with white paint. If only three gallons of paint are available, and it takes two full gallons to achieve a solid white color on the first wall, what shade of gray will the single gallon produce on the second wall? The thickness of paint on the second wall will be only half that on the first. As a result, the second wall's appearance will differ from the first, even though both walls are coated with the same paint.

Consider also a white drop ceiling panel. What is the fundamental color of the fiber board that makes up such panels? Brown? But ceiling panels are typically painted white; they look white; and they reflect light as if they were white.

Both of these examples demonstrate how the behavior of solids is consistent with the properties and concentration of the applied coatings.

In a suspension or body, a ceramic powder that is only partially coated by an additive will exhibit properties somewhere between those of the clean particle surface and those of the additive. A fully coated ceramic powder will typically behave as if it was a solid particle of the coating (the additive).

As particle surface areas, surface properties, and surface coating concentrations change, processing properties can be expected to fluctuate as well. Examples include the nature of particle/particle interactions which affect forming viscosities, rheologies, and gelation behaviors in suspension, plasticities, green strengths, dry strengths, and porosities and pore size distributions in dry bodies.

As powder surface areas increase or decrease, additive concentrations should be adjusted to achieve constant surface properties. For these reasons, the surface areas of powders, as a function of the total mass of powder present, are of interest.

This leads to the first parameter to be considered: specific surface area.

Specific Surface Area (SSA)

The mass specific surface area of a powder (SSA) is one measure of the surface area present in one (1) gram of powder.

$$\mathrm{SSA} = \frac{\text{surface area}}{\text{unit mass}} \; [=] \; \frac{\mathrm{m}^2}{\mathrm{g}} \qquad (1\text{-}1)$$

Specific surface areas are usually measured by the nitrogen adsorption technique, also known as the BET method.[1] In this technique, a monolayer of nitrogen molecules coats all surfaces. The analytical instruments measure the mass of nitrogen adsorbed and calculate the coverage using the known values of surface area coverage per mass of nitrogen.

Volume Specific Surface Area (VSA)

Similarly, one may refer to the volume specific surface area of a powder (VSA), which is another measure of the surface area present in one (1) true cubic centimeter of the powder.

$$\mathrm{VSA} = \frac{\text{surface area}}{\text{unit volume}} \; [=] \; \frac{\mathrm{m}^2}{\mathrm{cm}^3} \qquad (1\text{-}2)$$

The VSA is commonly used in computer modelling and computer calculations. For example, in particle packing calculations, the volumes occupied by particles are important. It is quite common, therefore, in computer models to tabulate the surface area of the particle size distribution using the VSA. If the SSA is required for later calculations, the conversion from VSA to SSA can be made at that time.

Volume specific surface area is related to the mass specific surface area by the density of the powder:

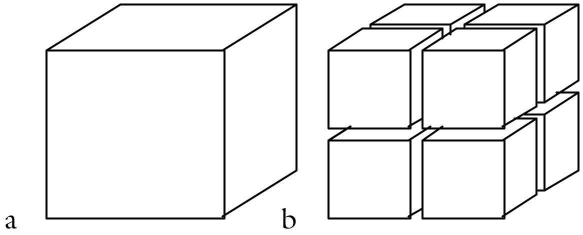

Figure 1-1 (a) A 1 cm³ cube; (b) The same 1 cm³ volume divided into eight smaller, equi-volume cubes.

$$\text{VSA} = \text{SSA} \times \rho \;[=]\; \frac{\text{m}^2}{\cancel{\text{g}}} \times \frac{\cancel{\text{g}}}{\text{cm}^3} = \frac{\text{m}^2}{\text{cm}^3} \qquad (1\text{-}3)$$

Surface Area versus Volume

Consider the powder cube shown in Figure 1-1a. It has a characteristic dimension (an edge length) of 1 cm. It contains 2.60 g of a material; it has a volume of exactly 1 cm^3; and its density is 2.60 g/cm^3. It has six faces with 1.00 cm^2 of surface area each, for a total of 6.00 cm^2 surface area. Its specific surface area is:

$$\frac{6.00\text{cm}^2}{2.60\text{g}} = \frac{2.31\text{cm}^2}{\text{g}} = \frac{0.000231\text{m}^2}{\text{g}} \qquad (1\text{-}4)$$

Now, let's break the cube into smaller cubes, as would occur in a milling operation. We will break it in half perpendicular to each axis so it becomes 8 smaller cubes (as in Figure 1-1b), each with an edge length ½ that of the original and a volume ⅛ that of the original (0.125 cm^3). What is the new total surface area of the 1 cm^3 of material?

The original six outer surfaces are still present, but each inside surface (right and left) of each of the three breakage planes produces a new surface, so the total volume now contains six more 1.00 cm^2 surfaces for a total of 12.00 cm^2. The powder still contains the original 1.00 cm^3 volume, but it now consists of eight particles, each with a characteristic dimension of 0.50 cm and a total of 12.00 cm^2 of surface area. Its SSA has increased:

$$\frac{12.00\text{cm}^2}{2.60\text{g}} = \frac{4.62\text{cm}^2}{\text{g}} = \frac{0.000462\text{m}^2}{\text{g}} \tag{1-5}$$

The new specific surface area is twice that of the original single cube.

If we continue to break the particles into smaller and smaller cubes, greater numbers of particles and increased surface areas will be produced, as shown in Table 1.1.

Table 1.1 Dimensions, Surface Areas, Volumes, and Number of Cubes from 1.00 true cm^3 of powder.

Edge Length of Each Cube	Total Surface Area (m^2)	Total Volume (cm^3)	Total Number of Cubes
1.0 cm	0.0006	1.00	1
0.5 cm	0.0012	1.00	8
1.0 mm	0.006	1.00	10^3
0.1 mm	0.06	1.00	10^6
0.01 mm	0.6	1.00	10^9
1.0 µm	6.0	1.00	10^{12}
0.1 µm	60.0	1.00	10^{15}
0.01 µm	600.	1.00	10^{18}
1.0 nm	6,000.	1.00	10^{21}

Note that the same true volume of material contains more and more particles and ever greater surface areas as the fundamental particle size decreases. This primarily happens to powders during crushing, grinding, and milling operations (i.e., during comminution).

Calculation of Surface Area of a Body or Suspension

To calculate the mass and volume specific surface areas (and quite a few other properties as well) of a body consisting of several powders, one should use the following formula:

$$P = a_1x_1 + a_2x_2 + a_3x_3 + \ldots + a_nx_n \qquad (1\text{-}6)$$

where P = the value of the property for the body or suspension,
a_i = the value of the property for the ith component, and
x_i = depending upon the units of the a_i terms, the x_i terms can be the mass fraction or volume fraction of the ith components in the body or suspension.

Simply stated, this formula shows that the value of a property for the whole body is the sum of the contributions of that same property from each of the body's components in proportion to their mass or volume fractions in the body.

Example 1.1 SSA to VSA

Quartz has a density of 2.60 g/cm^3. A quartz powder has a measured SSA of 4.50 m^2/g. What is the VSA of this quartz powder?

$$VSA = SSA \times \rho = \frac{4.50\text{m}^2}{\cancel{\text{g}}} \times \frac{2.60\cancel{\text{g}}}{\text{cm}^3} = \frac{11.7\text{m}^2}{\text{cm}^3} \qquad (1\text{-}7)$$

Example 1.2 VSA to SSA

The calculated VSA of another quartz powder is 7.90 m^2/cm^3. What is its SSA?

$$\mathrm{SSA} = \frac{\mathrm{VSA}}{\rho} = \frac{7.90\,\dfrac{\mathrm{m}^2}{\cancel{\mathrm{cm}^3}}}{2.60\,\dfrac{\mathrm{g}}{\cancel{\mathrm{cm}^3}}} = 3.04\,\frac{\mathrm{m}^2}{\mathrm{g}} \tag{1-8}$$

Example 1.3 SSA of a Body

A body is to consist of four powders in the composition shown in Table 1.2. What is the SSA of the body? Using (1-6):

$$\mathrm{SSA}_{\mathrm{Body}} = x_1\mathrm{SSA}_1 + x_2\mathrm{SSA}_2 + \ldots + x_n\mathrm{SSA}_n \tag{1-9}$$

where x_i = mass fraction of the i^{th} component in the body
SSA_i = SSA of the i^{th} component in the body

Table 1.2 Body composition and powder properties.

Composition	SSA (m^2/g)	Density(g/cm^3)
40 wt% clay	22.0	2.60
30 wt% feldspar	8.50	2.55
20 wt% quartz	6.38	2.62
10 wt % alumina	4.20	3.96

The mass of each of these components in 1 gram of body is:

$$\begin{array}{lll} \text{40 wt\% clay} & \times\ \text{1 g body} = \text{0.40 g clay} & \\ \text{30 wt\% feldspar} & \times\ \text{1 g body} = \text{0.30 g feldspar} & \text{(1-10)} \\ \text{20 wt\% quartz} & \times\ \text{1 g body} = \text{0.20 g quartz} & \\ \text{10 wt \% alumina} & \times\ \text{1 g body} = \text{0.10 g alumina} & \end{array}$$

Using (1-9), the SSA can then be calculated:

$$
\begin{aligned}
\text{SSA} = {} & \frac{22.0\text{m}^2}{\text{g }\cancel{\text{clay}}} \times \frac{0.40\text{g }\cancel{\text{clay}}}{\text{g body}} + \frac{8.50\text{m}^2}{\text{g }\cancel{\text{feldspar}}} \times \frac{0.30\text{g }\cancel{\text{feldspar}}}{\text{g body}} \\
& + \frac{6.38\text{m}^2}{\text{g }\cancel{\text{quartz}}} \times \frac{0.20\text{g }\cancel{\text{quartz}}}{\text{g body}} + \frac{4.20\text{m}^2}{\text{g }\cancel{\text{alumina}}} \times \frac{0.10\text{g }\cancel{\text{alumina}}}{\text{g body}} \\
& = \frac{13.0\text{ m}^2}{\text{g body}} = 13.0\frac{\text{m}^2}{\text{g}} \qquad \text{(1-11)}
\end{aligned}
$$

Example 1.4 VSA of a Body

What is the VSA of the body in Table 1.2? Using (1-6):

$$\text{VSA}_{\text{Body}} = x_1\text{VSA}_1 + x_2\text{VSA}_2 + \ldots + x_n\text{VSA}_n \qquad \text{(1-12)}$$

where x_i = volume fraction of the i^{th} component in the body
VSA_i = VSA of the i^{th} component in the body

To perform this calculation, one must determine the volume fractions of each powder component present in 1g of body, and then use (1-12):

$$
\begin{aligned}
\text{Volume} = {} & 0.40\cancel{\text{g}}\text{ clay} \div \frac{2.60\cancel{\text{g}}}{\text{cm}^3} + 0.30\cancel{\text{g}}\text{ feldspar} \div \frac{2.55\cancel{\text{g}}}{\text{cm}^3} \\
& + 0.20\cancel{\text{g}}\text{ quartz} \div \frac{2.62\cancel{\text{g}}}{\text{cm}^3} + 0.10\cancel{\text{g}}\text{ alumina} \div \frac{3.96\cancel{\text{g}}}{\text{cm}^3} \\
& = 0.154\text{ cm}^3\text{ clay} + 0.118\text{ cm}^3\text{ feldspar} \\
& + 0.0763\text{ cm}^3\text{ quartz} + 0.0253\text{ cm}^3\text{ alumina} \\
& = 0.374\text{ cm}^3\text{ body} = 1\text{ g body} \qquad \text{(1-13)}
\end{aligned}
$$

The individual volumes of the powder components and the total volume of powder present in 1g of the body, all of which are shown in (1-13), allow the calculation of volume percentages of the constituents in the body:

$$
\begin{aligned}
(0.154\ \cancel{\mathrm{cm}^3} / 0.374\ \cancel{\mathrm{cm}^3}) \times 100\% &= 41.2 \text{ vol\% clay} \\
(0.118\ \cancel{\mathrm{cm}^3} / 0.374\ \cancel{\mathrm{cm}^3}) \times 100\% &= 31.6 \text{ vol\% feldspar} \\
(0.0763\ \cancel{\mathrm{cm}^3} / 0.374\ \cancel{\mathrm{cm}^3}) \times 100\% &= 20.4 \text{ vol\% quartz} \\
(0.0254\ \cancel{\mathrm{cm}^3} / 0.374\ \cancel{\mathrm{cm}^3}) \times 100\% &\underline{= \quad 6.8 \text{ vol\% alumina}} \\
&\qquad 100 \text{ vol\% body}
\end{aligned}
\tag{1-14}
$$

Using the volume percentages calculated in (1-14), the volume of each component in 1 true cm^3 of body can be calculated:

$$
\begin{aligned}
41.2 \text{ vol\% clay} &\times 1\mathrm{cm}^3 \text{ body} = 0.412\ \mathrm{cm}^3 \text{ clay} \\
31.6 \text{ vol\% feldspar} &\times 1\mathrm{cm}^3 \text{ body} = 0.316\ \mathrm{cm}^3 \text{ feldspar} \\
20.4 \text{ vol\% quartz} &\times 1\mathrm{cm}^3 \text{ body} = 0.204\ \mathrm{cm}^3 \text{ quartz} \\
6.8 \text{ vol\% alumina} &\times 1\mathrm{cm}^3 \text{ body} = 0.068\ \mathrm{cm}^3 \text{ alumina}
\end{aligned}
\tag{1-15}
$$

Using (1-12), the VSA can then be calculated:

$$
\begin{aligned}
\mathrm{VSA} = {} & \left(\frac{22.0\mathrm{m}^2}{\cancel{\mathrm{g}}} \times \frac{2.60\cancel{\mathrm{g}}}{\cancel{\mathrm{cm}^3}} \times \frac{0.412\cancel{\mathrm{cm}^3} \text{ clay}}{\mathrm{cm}^3 \text{ body}} \right) \\
& + \left(\frac{8.50\mathrm{m}^2}{\cancel{\mathrm{g}}} \times \frac{2.55\cancel{\mathrm{g}}}{\cancel{\mathrm{cm}^3}} \times \frac{0.316\ \cancel{\mathrm{cm}^3} \text{ feldspar}}{\mathrm{cm}^3 \text{ body}} \right) \\
& + \left(\frac{6.38\mathrm{m}^2}{\cancel{\mathrm{g}}} \times \frac{2.62\cancel{\mathrm{g}}}{\cancel{\mathrm{cm}^3}} \times \frac{0.204\ \cancel{\mathrm{cm}^3} \text{ quartz}}{\mathrm{cm}^3 \text{ body}} \right) \\
& + \left(\frac{4.20\mathrm{m}^2}{\cancel{\mathrm{g}}} \times \frac{3.96\cancel{\mathrm{g}}}{\cancel{\mathrm{cm}^3}} \times \frac{0.068\ \cancel{\mathrm{cm}^3} \text{ alumina}}{\mathrm{cm}^3 \text{ body}} \right) \\
& = \frac{35.0\ \mathrm{m}^2}{\mathrm{cm}^3 \text{ body}} = 35.0 \frac{\mathrm{m}^2}{\mathrm{cm}^3}
\end{aligned}
\tag{1-16}
$$

Chapter Two

Volume and Porosity

True Volume

True volume refers to a volume completely filled by a particle's solid mass with no internal pores. The density of kaolinite is 2.60 g/cm^3. This indicates that 1 true cm^3 of kaolinite, that is, 1 cm^3 with no internal pores, is a 2.60 g mass.

Apparent Volume

The apparent volume of a porous solid is the volume that it *appears* to have when its internal, closed porosity is allowed to contribute to its volume. If one measures the volume of a fired ceramic that contains internal (closed) porosity, the measurement will be an indication of its apparent volume.

The term *apparent volume* usually applies to solid objects, rather than to systems of particles. When this term is applied to particulate systems, it refers to the bulk volume of the system of particles.

Apparent volume, when applied to ceramic ware, is usually measured using the Archimedes' principle. This states that the buoyancy, the difference between the dry and submerged weights of the solid, is equal to the mass of the water displaced. Using the density of water at the measurement temperature, the volume of the displaced water and the volume of the ceramic can be calculated. The formula using the Archimedes' principle is:

$$\textbf{Apparent Volume} = \frac{W_{\mathrm{dry}} - W_{\mathrm{submerged}}}{\rho_{\mathrm{Water}}} \qquad (2\text{-}1)$$

where W_{dry} = mass of dry ware
$W_{submerged}$ = mass of ware submerged
ρ_{Water} = density of water

Apparent volume usually does not include the open porosity in a ceramic ware. To perform the measurements, the ceramics are usually boiled for two hours or soaked for 24 hours to insure that the ware is saturated and all open pores are filled with water. The submerged mass in (2-1), in such cases, is known as the *saturated, submerged mass*.

Example 2.1 Calculate Apparent Volume of a Solid

A piece of fired ceramic has a dry mass of 105.07g. After soaking, its submerged mass is 58.13g. The water temperature is 25°C, and water density at that temperature is 0.99707g/cm^3. What is the apparent volume of the ware?

$$\text{Apparent Volume} = \frac{(105.07\cancel{g} - 58.13\cancel{g})}{\dfrac{0.99707\cancel{g}}{cm^3}} = 47.08\ cm^3 \qquad (2\text{-}2)$$

Bulk Volume

The bulk volume of a powder is the true volume of the powder plus the volume of any internal pores and the volume of external, interparticle pores.

When powders are poured into a container (a bag or a silo, for example), the powder particles will not pack perfectly, but they will pack in random arrangements that define interparticle porosity. As a rule of thumb, random dense packing of similarly sized particles (a monodispersion) will contain about 40 vol% porosity. Quartz has a density of 2.6 g/cm^3. One kg of quartz occupies 384.6 cm^3 true volume:

$$\frac{1000\cancel{g}\text{ quartz}}{\frac{2.6\cancel{g}}{\text{cm}^3}\text{ true density}} = 384.6\text{ cm}^3\text{ true volume} \qquad (2\text{-}3)$$

One (1) kg of quartz in the form of coarse quartz particles, however, will fill a 641cm^3 container if it packs to produce 40vol% interparticle porosity:

$$\begin{array}{ll} \text{40vol\% porosity x 641 cm}^3 & = 256.4\text{cm}^3 \\ \underline{\text{60vol\% solids x 641 cm}^3} & \underline{= 384.6\text{cm}^3} \\ \text{100vol\% of the container} & = 641.0\text{cm}^3 \end{array} \qquad (2\text{-}4)$$

The true volume of the quartz particles is 384.6cm^3. The 641.0cm^3 bulk volume required to hold the quartz particles, however, is much larger.

Although bulk volume can be predicted by guessing how much porosity will be contained when powders are poured into containers, it is safest to measure the bulk volume of powders. To do this, one can simply pour a known mass of a powder sample into a volumetric cylinder, and measure the volume in the cylinder occupied by the powder.

For a constant mass of particles, the bulk volume of a powder typically increases as the fundamental size of the particles decreases. As particle sizes decrease, surface areas increase (as discussed in Chapter 1). As surface areas increase, sliding friction from particle/particle contacts increases, the interparticle porosity increases, and bulk volume increases.

Coarser particles, with greater mass to surface area ratios, can slide more easily over one another to achieve relatively dense packing configurations. The friction between fines and their lower mass to surface area ratios prevent them from sliding easily over one another to achieve dense packing configurations. Even with vibration, packs of fine particles are usually much more open, that is, they contain much more interparticle porosity, than packs of coarser particles.

Tap Volume

Tap volumes and tap densities are frequently measured to determine how particles will pack. The term *tap volume*, is not usually used, but *tap density* is common.

To measure the tap density of a powder, one measures the tap volume, that is, the bulk volume of the powder after it has been subjected to vibrations. The measurement starts as a bulk volume measurement, but the volumetric cylinder containing the powder must be subjected to vibrations (usually using a vibrating table). The bulk volume of the powder after vibration will typically be less than the bulk volume of the powder before vibration. The tap density of the powder after vibration should be greater than the bulk density of the powder without vibration.

The tap density of a powder is:

$$\text{Tap Density} = \frac{\text{mass of powder}}{\text{bulk volume after vibration}} \; [=] \; \frac{\text{g}}{\text{cm}^3} \qquad (2\text{-}5)$$

Bulk volumes should be used to calculate the volumes of silos or storage hoppers for powders – tap densities should not. The reasoning is simple: subjecting powder in a volumetric cylinder to intense vibrations may be easy to perform in the lab, but subjecting a 1000^+ liter hopper or silo in the plant to the same intensity of vibration is not. If vibration is not going to be used on the storage hoppers in the plant, vibration should not be used in the lab to measure bulk volume. If, however, vibration is used in the plant, a similar intensity of vibration can be used in laboratory tests.

Example 2.2 Measuring Bulk Volume

One (1) kg of a powder (ρ = 3.96g/cm^3) is poured into a 1 liter volumetric cylinder. The powder fills 495 ml of the cylinder. What are the true volume and bulk volume of the powder?

$$\text{True Volume} = \frac{1000\cancel{g}}{\left(\frac{3.96\cancel{g}}{\text{cm}^3}\right)} = 253\ \text{cm}^3 \qquad (2\text{-}6)$$

$$\text{Bulk Volume} = \text{measured volume} = 495\text{ml} = 495\text{cm}^3 \qquad (2\text{-}7)$$

Example 2.3 Calculate Silo Volume – I

How much volume is required for a silo to hold 20 metric tons of the powder in Example 2.2?

$$\text{Silo Volume} = \frac{\text{total mass}}{\text{bulk density}} = \frac{\text{total mass to store}}{\left(\frac{\text{test mass}}{\text{test bulk volume}}\right)} \qquad (2\text{-}8)$$

$$\frac{20{,}000\cancel{\text{kg}}}{\left(\frac{1\cancel{\text{kg}}}{495\text{cm}^3}\right)} = 9.9 \times 10^6 \cancel{\text{cm}^3} \times \frac{1\text{m}^3}{10^6\ \cancel{\text{cm}^3}} = 9.9\text{m}^3 \qquad (2\text{-}9)$$

Internal Porosity

Porosity in ceramics falls into two categories: open and closed. Fired ware, raw materials, and agglomerates can have both types of porosity.

Open pores are accessible from the surface. Surface area measurements by the nitrogen adsorption technique will include the external surface area of a ceramic plus all of the internal surface areas of open pores.

Closed pores are not accessible from the surface of the material. Surface area instruments will not pick up the surfaces within closed pores. Volume measurements, however, cannot distinguish between solid material and closed pores, so the closed pore volume is normally included in the reported volumes and densities of solids.

Example 2.4 Estimating Internal Porosity

The apparent volume of a 1592g insulating brick is 1990cm^3. If the true density of the brick material is 2.60g/cm^3, estimate its internal porosity.

$$\text{Internal Porosity} = \frac{\text{Volume}_{\text{Pore}}}{\text{Volume}_{\text{Apparent}}} \times 100\% \qquad (2\text{-}10)$$

$$\text{Volume}_{\text{True}} = \frac{1592\cancel{g}}{2.60\,\frac{\cancel{g}}{\text{cm}^3}} = 612\ \text{cm}^3 \qquad (2\text{-}11)$$

$$\text{Volume}_{\text{Pore}} = \text{Volume}_{\text{Apparent}} - \text{Volume}_{\text{True}}$$

$$= 1990\text{cm}^3 - 612\text{cm}^3 = 1378\text{cm}^3 \qquad (2\text{-}12)$$

$$\text{Internal Porosity} = \frac{\text{Volume}_{\text{Pore}}}{\text{Volume}_{\text{Apparent}}} \times 100\%$$

$$= \frac{1378\cancel{\text{cm}^3}}{1990\cancel{\text{cm}^3}} \times 100\% = 69.2\% \qquad (2\text{-}13)$$

Interparticle Porosity

When systems of particles are processed, random arrangements of those particles will define interparticle porosity, which consists of the pores between the compacted particles. The bulk volume of a powder includes both the particle volume and the interparticle pore volume, i.e., the interparticle porosity.

Interparticle porosity is an important parameter in ceramic processing because it affects green and dry densities, and firing shrinkages of compacts.

Minimum Porosity (or Packing Potential)

The capability of a particle size distribution to pack to achieve a desired value of interparticle porosity is a function of the particle size distribution, the surface area and roughness of the powders, the state of flocculation/deflocculation of the suspension, and the nature of the additive chemicals in the suspension, to name a few.

We often refer to the minimum porosity of a powder compact, even though the powder is in suspension and not actually compacted. In such cases, we want to know how densely the system of particles could pack (if they were forced to do so). The packing capability of a system of particles affects the viscosity and rheology of suspensions of particles.

Viscosity and Rheology

Interparticle fluid provides particulate suspensions with their fluidity. Fluid must first fill all interparticle porosity before it can begin to separate particles so flow can occur. A system of particles must have the capacity to pack to a denser volume percent solids than the desired solids content of those particles in suspension.

If a process suspension is to have 70vol% solids (i.e., 30vol% fluid), it is important that a compact of those particles can pack denser than 70vol% to achieve an interparticle porosity less than 30vol%.

If such a system of particles can only pack to 70vol% (i.e., 30vol% interparticle porosity), when 30vol% fluid is added, the system will still behave as a solid. All particles will be in contact and the fluid will simply fill all of the interparticle pores.

If a different system of particles can pack to 80vol% (i.e., 20vol% interparticle porosity), when 30vol% fluid is added, 20vol% will fill the pores, and the remaining 10% fluid can separate the particles and impart fluidity to the suspension.

The denser a system of particles can pack, the lower the viscosity of a suspension of those particles at a fixed solids content and in a constant additive chemical environment.

Using Viscosity as an Indication of Packing Potential

If an exact porosity percentage is not needed, but only relative packing capabilities of different particle size distributions are desired, packing capabilities can be compared by measuring suspension viscosities. At constant solids content and in a constant chemical additive environment, the lower the viscosity of a suspension, the better the packing capabilities of the particles in that sample.

The denser a distribution can pack, less fluid is required to fill pores and more fluid is available to separate particles and reduce viscosities. Tests like this are easiest to perform when suspensions are in a state of maximum deflocculation.

These phenomena help to show why particle size distributions of the solids are so important to ceramic forming technology. As particle size distribution changes, the interparticle porosity in a dense compact will change, and the viscosity and rheology of the forming body or suspension will change as well.

An algorithm that can be used to estimate interparticle porosity of particle size distributions will be discussed in a later chapter in this book.

Porosity & Pore Size Distribution

The pore size distribution in a powder compact is a description of the interparticle porosity. But unlike the porosity, which is simply the total percentage present, the pore size distribution describes the size distribution of the pores comprising the porosity.

Assuming the pores are filled with water, the interparticle porosity defines the amount of water present and the total drying energy required to remove all of the water from a green body. The pore size distribution, however, controls how easily the green ware will dry. If the pores are really small, a body will cling to its moisture. If the pores are large, a body will give up moisture rather easily.

Particulate systems can have identical porosities but totally different pore size distributions. The simplest example of this uses basketballs and BBs. Consider a room filled totally with basketballs. If they are in a random dense packing arrangement, they will fill about 60vol% of the room, leaving about 40vol% pores. If the room had been filled instead with BBs in a similar random dense packing arrangement, they would also fill about 60vol% of the room, leaving about 40vol% pores. The two systems just described have similar packing densities, similar porosities, but totally different pore size distributions. It should be relatively obvious that the pores within densely packed basketballs would be much larger than the pores within densely packed BBs.

Both of these systems could hold similar volumes of gases or liquids in the porosity, but the ease of movement of those fluids through each pack would be totally different. Fluid could move easily through the basketballs within the larger pores, but fluid would flow with much more difficulty through the much smaller pores within the pack of BBs. The flow of fluids within ceramic powder compacts defined by coarse and fine powders are similar to the behaviors in this simple example.

Pore size distributions generally correlate with the permeability of compacts. During drying, liquid water and water vapor flow through the pores. During firing, oxygen enters the compact through its pores and gaseous decomposition products escape through the pores. The ease with which these liquids and gases flow in and out of compacts is dependent on the pore size distribution of the interparticle porosity.

Dry Pressing Operations

Companies performing dry pressing operations are faced with the calculation of several of the parameters discussed in this chapter. Stored raw materials are slurried and spray dried. Spray dried granules are then stored in silos awaiting pressing operations. Bulk volume calculations may be employed for storage of the raw materials as well as storage of the spray dried granules.

Controls that affect the bulk volume of granules are important to achieving consistency during die filling operations.

Those same controls will affect the packing capabilities of the powders, which in turn will affect the densities and shrinkages during firing/sintering of the pressed pieces.

In most cases, the control variables that affect bulk volumes of granules and packing behaviors during pressing are variables that must be applied during batching of the suspensions. Spray drying parameters will also affect these properties. In most cases, however, changes have to be made to the particle size distributions, solids loadings, and chemical additive treatments when formulating and mixing the suspensions before spray drying to effect desired properties such as bulk densities of the spray dried granules during storage and die filling.[2]

Example 2.5 Die Fill Volume during Dry Pressing

Spray dried granules with bulk densities of 0.900g/cm^3 are made from powders with 2.6g/cm^3 true density. What mass of granules is required to fill a square die cavity that is 5cm × 5cm × 3cm?

$$\text{Volume of die cavity} = 5\text{cm} \times 5\text{cm} \times 3\text{cm} = 75\text{cm}^3 \qquad (2\text{-}14)$$

$$\text{Granule mass} = \text{die volume} \times \text{granule bulk density} \qquad (2\text{-}15)$$

$$= 75\cancel{\text{cm}^3} \times 0.900\frac{\text{g}}{\cancel{\text{cm}^3}} = 67.5\text{g} \qquad (2\text{-}16)$$

Example 2.6 Applied Load during Pressing

What load must be applied during pressing to achieve 200 psi when pressing the ware described in Example 2.5?

$$\text{Pressing Pressure} = \frac{\text{Load}}{\text{Pressing Area}} \qquad (2\text{-}17)$$

$$\text{Load} = \text{Pressing Pressure} \times \text{Pressing Area} \qquad (2\text{-}18)$$

$$= 200\ \text{psi} \times 25\cancel{\text{cm}^2} \times \left(\frac{1\text{in}}{2.54\cancel{\text{cm}}}\right)^2 = 775\text{lb load} \qquad (2\text{-}19)$$

Example 2.7 Interparticle Porosity in a Pressed Pellet

If a die is used to press cylindrical pellets, one can estimate the interparticle porosity achieved during pressing. The mass of powder in the pellet, the volume of the pellet, and the true density of the powders are required. For example, if an alumina pellet is 2.54cm diameter, 1.20cm thick, 20.0g mass, and the alumina has a true density of 3.96g/cm^3, the interparticle porosity can be calculated as follows:

$$\text{Interparticle Porosity} = \frac{\text{Volume}_{\text{Pellet}} - \text{Volume}_{\text{True}}}{\text{Volume}_{\text{Pellet}}} \times 100\% \qquad (2\text{-}20)$$

$$\text{Volume}_{\text{Pellet}} = \pi r^2 h = \pi(1.27\text{cm})^2(1.20\text{cm}) = 6.08\ \text{cm}^3 \qquad (2\text{-}21)$$

$$\text{Alumina Volume}_{\text{True}} = \frac{20.0\cancel{\text{g}}}{3.96\dfrac{\cancel{\text{g}}}{\text{cm}^3}} = 5.05\ \text{cm}^3 \qquad (2\text{-}22)$$

$$\text{Pore Volume} = 6.08\ \text{cm}^3 - 5.05\ \text{cm}^3 = 1.03\ \text{cm}^3 \qquad (2\text{-}23)$$

$$\text{Interparticle Porosity} = \frac{1.03\cancel{\text{cm}^3}}{6.08\cancel{\text{cm}^3}} \times 100\% = 16.9\% \qquad (2\text{-}24)$$

Example 2.8 Expected Shrinkage during Sintering

What shrinkage might one expect during sintering of the cylindrical pellets described in Example 2.7? If the pellets are sintered to zero (0%) porosity, the 16.9% interparticle porosity will be eliminated. If shrinkage is isotropic, i.e., uniform in all directions, the new pellet volume will be 5.05 cm^3, which equals the true volume of the alumina. Shrinkage can then be calculated as follows:

$$\text{Volume of sintered pellet} = \pi r_s^2 h_s \tag{2-25}$$

where subscript ‘s’ refers to sintered dimensions,
subscript ‘u’ refers to unfired dimensions,
l.s.% = linear shrinkage percentage,
l.s. = linear shrinkage expressed as a fraction,
r_s = sintered radius = r_u - (l.s.% × r_u) =
= (100% - l.s.%) r_u = (1 - l.s.) r_u
h_s = sintered height = h_u -(l.s.% × h_u) =
= (100% - l.s.%) h_u = (1 - l.s.) h_u

$$\pi r_s^2 h_s = \pi [(1 - \text{l.s.})r_u]^2 [(1 - \text{l.s.})h_u] \tag{2-26}$$

$$= \pi\ r_u^2 h_u (1 - \text{l.s.})^3 = 5.05\ \text{cm}^3 \tag{2-27}$$

$$= 6.08\ \text{cm}^3\ (1 - \text{l.s.})^3 = 5.05\ \text{cm}^3 \tag{2-28}$$

$$(1 - \text{l.s.})^3 = 5.05\ \cancel{\text{cm}^3} / 6.08\ \cancel{\text{cm}^3} = 0.831 \tag{2-29}$$

$$1 - \text{l.s.} = 0.831^{1/3} = 0.940 \tag{2-30}$$

$$\text{l.s.} = 1.000 - 0.940 = 0.060; \qquad \text{l.s.\%} = 6.0\% \tag{2-31}$$

Note: The actual shrinkage may be less then this value, but it cannot be greater than this value.

Chapter Three

Density

True Density

The true density of a powder is the mass of a perfect sample of the material per unit volume. True densities of most common materials are listed in materials handbooks.

Consider alumina, which has a true density of $3.96 g/cm^3$. One (1) true cm^3 of alumina, with no internal porosity, will have a mass of 3.96g. Any alumina sample that has a measured density less than this value contains some porosity, impurities, and/or imperfections.

Pycnometer Measurements

True densities are measured using pycnometry. Glass pycnometer bottles have traditionally been used to make such laboratory measurements. Electronic helium pycnometers are also now available. Measurements using both types of pycnometers are based on the same principles.

Pycnometers are designed to precisely and reproducibly define constant volumes. Glass pycnometer bottles have ground joints that precisely accept their glass stoppers. Each stopper has a capillary tube along its axis. When stoppers are inserted into the bottles, the volume of the bottle including the full length of the capillary tube defines a constant volume. Stoppers can be removed and replaced numerous times and the same volume will be reproducibly defined.

To measure the true density of powders, the powders must be finely ground, usually using mortar and pestle. Coarse powders frequently contain internal pores. Proper fine grinding breaks such particles through their pores and exposes all of the pores as external surfaces. If grinding is insufficient and internal porosity remains, measured densities will be lower than recorded handbook values.

To perform true density measurements, four masses must be measured and recorded:

w_e = empty bottle plus stopper,
w_f = bottle plus stopper filled with water,
w_p = bottle, stopper, and dry powder, and
w_{pf} = bottle plus stopper and powder, filled with water.

To calculate true density, one needs to measure the temperature of the water and use its precise density at that temperature (ρ_w). True density is then defined as follows:

$$\text{True Density} = \frac{\text{mass of powder}}{\text{volume of powder}} \tag{3-1}$$

$$= \frac{(w_p - w_e)}{\left[\dfrac{(w_f - w_e) - (w_{pf} - w_p)}{\rho_w}\right]} \tag{3-2}$$

This equation makes more sense when the various terms are considered. The numerator is equal to the mass of the powder:

$$\text{Mass}_{\text{Powder}} = \text{Mass}_{\text{Powder + Bottle}} - \text{Mass}_{\text{Bottle}} = w_p - w_e \tag{3-3}$$

The denominator calculates the volume of the powder by calculating the volume of the full bottle (the first term) and the volume of the fill water, i.e., the volume within the bottle that is **not** occupied by the powder (the second term):

$$\text{Volume}_{\text{Bottle}} = \frac{(\text{Mass}_{\text{Bottle full of water}} - \text{Mass}_{\text{Bottle Empty}})}{\text{Density}_{\text{Water}}} \tag{3-4}$$

$$\text{Volume}_{\text{Fill Water}} = \frac{(\text{Mass}_{\text{Bottle+Powder+Water}} - \text{Mass}_{\text{Bottle+Powder}})}{\text{Density}_{\text{Water}}} \tag{3-5}$$

The difference between the volume of the bottle and the volume of fill water is then equal to the volume of the powder.

$$\text{Volume}_{\text{Powder}} = \text{Volume}_{\text{Bottle}} - \text{Volume}_{\text{Fill Water}} \tag{3-6}$$

Example 3.1 Pycnometer Density

The pycnometer density of quartz powder is to be measured. The four masses are as follows: $w_e = 43.017\text{g}$, $w_f = 105.822\text{g}$, $w_p = 88.456\text{g}$, $w_{pf} = 133.920\text{g}$. The temperature of the water is 25°C, and water density at that temperature is 0.99707g/cm^3. What is the true density of this quartz sample?

$$\text{Mass}_{\text{Powder}} = w_p - w_e = 88.456\text{g} - 43.017\text{g} = 45.439\text{g} \tag{3-7}$$

$$\text{Volume}_{\text{Bottle}} = \frac{(w_f - w_e)}{\rho_w} \tag{3-8}$$

$$= \frac{(105.822\cancel{g} - 43.017\cancel{g})}{0.99707\frac{\cancel{g}}{\text{cm}^3}} = 62.990\text{cm}^3 \tag{3-9}$$

$$\text{Volume}_{\text{Fill Water}} = \frac{(w_{pf} - w_p)}{\rho_w} \tag{3-10}$$

$$= \frac{(133.920\cancel{g} - 88.456\cancel{g})}{0.99707\frac{\cancel{g}}{\text{cm}^3}} = 45.598\text{cm}^3 \tag{3-11}$$

$$\text{True Density} = \frac{\text{Mass}_{\text{Powder}}}{\text{Volume}_{\text{Powder}}}$$

$$= \frac{\text{Mass}_{\text{Powder}}}{\text{Volume}_{\text{Bottle}} - \text{Volume}_{\text{Fill Water}}} \tag{3-12}$$

$$= \frac{45.439\text{g}}{62.990\text{cm}^3 - 45.598\text{cm}^3} = \frac{45.439\text{g}}{17.392\text{cm}^3} = 2.61\frac{\text{g}}{\text{cm}^3}$$

Bulk Density

Bulk density is calculated as the mass of powder per bulk volume. The bulk volume takes into account all internal and interparticle porosity. Bulk volume and bulk density are used to calculate the volume of a silo required to hold dry raw material powders or spray dried body granules.

$$\text{Bulk Density} = \frac{\text{Powder Mass}}{\text{Bulk Volume}} \qquad (3\text{-}13)$$

Example 3.2 Measuring Bulk Density

One (1) kg of a powder ($\rho = 3.96\text{g/cm}^3$) is poured into a 1 liter volumetric cylinder. The powder fills 495 ml of the cylinder. What is the bulk density of the powder?

$$\text{Bulk Density} = \frac{1\text{kg}}{495\ \text{cm}^3} = \frac{1000\text{g}}{495\ \text{cm}^3} = 2.02\,\frac{\text{g}}{\text{cm}^3} \qquad (3\text{-}14)$$

Example 3.3 Calculate Silo Volume – II

What silo volume is needed to hold 20 metric tons of the powder in Example 3.2?

$$\text{Silo Volume} = \frac{\text{Powder Mass}}{\text{Bulk Density}} = \frac{20\ \text{tons}}{2.02\,\frac{\text{g}}{\text{cm}^3}}$$

$$= \frac{20\times10^6\cancel{\text{g}}}{2.02\,\frac{\cancel{\text{g}}}{\text{cm}^3}} = 9.9\times10^6\cancel{\text{cm}^3}\times\frac{1\text{m}^3}{10^6\,\cancel{\text{cm}^3}} = 9.9\text{m}^3 \qquad (3\text{-}15)$$

Apparent Density

Apparent density of a porous ceramic is the density of the powder with its internal porosity included as part of its volume. To calculate apparent density, one needs to measure the mass of the dry sample, and its submerged, saturated mass. This measurement requires the use of the Archimedes' method, that is, soaking and saturating the open pores with water, followed by measuring the mass of the ware submerged in water.

To measure the saturated, submerged mass, one must boil the ware for two hours, or soak it in water for 24hr. Then, the buoyancy, which is the difference between the dry mass and the submerged, saturated mass, is equivalent to the volume of water displaced. The volume of water displaced is equivalent to the apparent volume of the ware.

Apparent density is then equal to:

$$\text{Density}_{\text{Apparent}} = \frac{\text{Mass}_{\text{Dry}}}{\left(\frac{\text{Buoyancy}}{\rho_{\text{W}}}\right)} = \frac{\text{Mass}_{\text{Dry}}}{\left(\frac{\text{Mass}_{\text{Dry}} - \text{Mass}_{\text{Submerged}}}{\rho_{\text{W}}}\right)}$$

$$= \frac{\text{Mass}_{\text{Dry}}}{\text{Volume}_{\text{Apparent}}} \tag{3-16}$$

Example 3.4 Calculate Apparent Density

What is the apparent density of the sample in Example 2.1?

$$\text{Density}_{\text{Apparent}} = \frac{\text{Mass}_{\text{Dry}}}{\text{Volume}_{\text{Apparent}}} = \frac{105.07\text{g}}{47.08\ \text{cm}^3} = 2.232\frac{\text{g}}{\text{cm}^3} \tag{3-17}$$

Compaction Density

The compaction density of a dry pressed body is a measure of the bulk density of the compact. If the pressed sample is a simple shape, its dimensions can be measured. A measurement is recommended because small errors will be introduced when die dimensions are used for this calculation. Die dimensions should be used for this calculation as a first estimate only.

Usually, some spring back occurs when the ware is removed from the die and the amount of spring back will be a function of pressing pressure, the type, concentration, and properties of the polymeric surface additives within the compact, raw material composition, single or double pressing action within the die cavity, and direction of the measured dimension relative to the pressing direction (parallel or perpendicular).

Example 3.5 Compaction Density in a Pressed Pellet

In Example 2.7, the interparticle porosity in a pressed pellet was calculated. An alumina pellet with 2.54cm diameter, 1.20 cm thickness, and 20.0g mass was the compact. The true density of the alumina powder is 3.96g/cm^3. The pressed pellet volume is 6.08 cm^3 (Equation 2-21) and the interparticle porosity was determined to be 16.9% (Equation 2-24). What is the compaction density of the sample?

$$\text{Density}_{\text{Compaction}} = \frac{\text{Mass}_{\text{Pellet}}}{\text{Volume}_{\text{Pellet}}} = \frac{20.0\text{g}}{6.08\text{cm}^3} = 3.29\,\frac{\text{g}}{\text{cm}^3} \qquad (3\text{-}18)$$

Chapter Four

Suspension Densities

Many ceramic bodies are batched in blungers as suspensions and then the body slips are stored in aging tanks until they are used in processing. Ceramic process engineers are frequently called upon to batch, adjust, and control such suspensions. Several different units are available to characterize their solids contents and compositions.

Weight Percentage

Just as the composition of each constituent in a body can be defined using weight percentages, solids contents and moisture contents of suspensions can also be defined as weight percentages. Weight percentages of a constituent, or of the total solids, or of the fluid can all be defined as follows:

$$\text{Weight \% Composition} = \frac{\text{Mass}_{\text{Individual Constituent}}}{\text{Mass}_{\text{Total Body}}} \times 100\% \qquad (4\text{-}1)$$

$$\text{Weight \% Solids Content} = \frac{\text{Mass}_{\text{Total Solids}}}{\text{Mass}_{\text{Total Suspension}}} \times 100\% \qquad (4\text{-}2)$$

$$\text{Weight \% Moisture Content} = \frac{\text{Mass}_{\text{Fluid}}}{\text{Mass}_{\text{Total Suspension}}} \times 100\% \qquad (4\text{-}3)$$

Example 4.1 Definition of a Suspension with Weight Percentages

A 2.5 metric ton batch is to contain 0.6 tons clay, 0.9 tons feldspar, and 1.0 ton alumina. The suspension is to be mixed in the production blunger at 48wt% solids. What is the composition of this body in wt%? How much water must be added to the batch to achieve the desired solids content? What is the moisture content in wt%?

Body composition using (4-1):

$$\begin{array}{ll} \dfrac{0.6\text{ tons clay}}{2.5\text{ tons body}} \times 100\% & = 24\text{wt\% clay} \\[2ex] \dfrac{0.9\text{ tons feldspar}}{2.5\text{ tons body}} \times 100\% & = 36\text{wt\% feldspar} \\[2ex] \dfrac{1.0\text{ tons alumina}}{2.5\text{ tons body}} \times 100\% & = 40\text{wt\% alumina} \\ \hline 2.5\text{ tons body} & 100\text{wt\% body} \end{array} \tag{4-4}$$

Mass of Water to be added using (4-2):

$$48\text{wt\% solids} = \frac{2.5\text{ tons solids}}{\text{Mass}_{\text{Total Suspension}}} \times 100\%$$

$$\text{Mass}_{\text{Total Suspension}} = \frac{2.5\text{ tons solids}}{48\cancel{\text{wt\%}}\text{ solids}} \times 100\cancel{\%} = 5.2\text{tons suspension} \tag{4-5}$$

5.2tons Total Suspension Mass - 2.5 tons solids (4-6)
= 2.7 tons water to be added

Moisture Content in wt% using (4-3):

$$\frac{2.7\cancel{\text{tons}}\text{ water}}{5.2\cancel{\text{tons}}\text{ Total Suspension}} \times 100\% = 52\text{wt\% Moisture} \tag{4-7}$$

Volume Percentage

Similarly, body compositions, solids contents, and moisture contents can be defined as volume percentages:

$$\text{Volume \% Composition} = \frac{\text{Volume}_{\text{Individual Constituent}}}{\text{Volume}_{\text{Total Body}}} \times 100\% \qquad (4\text{-}8)$$

$$\text{Volume \% Solids Content} = \frac{\text{Volume}_{\text{Total Solids}}}{\text{Volume}_{\text{Total Suspension}}} \times 100\% \qquad (4\text{-}9)$$

$$\text{Volume \% Moisture Content} = \frac{\text{Volume}_{\text{Fluid}}}{\text{Volume}_{\text{Total Suspension}}} \times 100\% \qquad (4\text{-}10)$$

Example 4.2 Definition of a Suspension with Volume Percentages

What is the composition of the body in Example 4.1 expressed as volume percentages? What is the volume of the water to be added? What is the moisture content in vol%? What is the solids content in vol%? To do these calculations, the densities of the raw materials are required. Densities are: Clay = Feldspar = 2.6g/cm^3; Alumina = 3.96g/cm^3.

Body composition using (4-8):

$$0.6\ \cancel{\text{tons}}\ \text{clay} \times \frac{10^6\cancel{\text{g}}}{\cancel{\text{ton}}} \times \frac{1\text{m}^3}{10^6\cancel{\text{cm}^3}} \times \frac{\cancel{\text{cm}^3}}{2.6\cancel{\text{g}}} = 0.23\ \text{m}^3\ \text{clay} \qquad (4\text{-}11)$$

$$0.9\cancel{\text{tons}}\ \text{feldspar} \times \frac{10^6\cancel{\text{g}}}{\cancel{\text{ton}}} \times \frac{1\text{m}^3}{10^6\cancel{\text{cm}^3}} \times \frac{\cancel{\text{cm}^3}}{2.6\cancel{\text{g}}} = 0.35\text{m}^3\ \text{feldspar} \qquad (4\text{-}12)$$

$$1.0\cancel{\text{ton}}\ \text{alumina} \times \frac{10^6\cancel{\text{g}}}{\cancel{\text{ton}}} \times \frac{1\text{m}^3}{10^6\cancel{\text{cm}^3}} \times \frac{\cancel{\text{cm}^3}}{3.96\cancel{\text{g}}} = 0.25\text{m}^3\ \text{alumina} \qquad (4\text{-}13)$$

$$2.5\ \text{tons total solids} \quad = \quad 0.83\ \text{m}^3\ \text{total body solids} \qquad (4\text{-}14)$$

$$\frac{0.23\text{m}^3\ \text{clay}}{0.83\text{m}^3\ \text{total body}} \times 100\% = 28\text{vol\% clay} \qquad (4\text{-}15)$$

$$\frac{0.35\text{m}^3\ \text{feldspar}}{0.83\text{m}^3\ \text{total body}} \times 100\% = 42\text{vol\% feldspar} \qquad (4\text{-}16)$$

$$\frac{0.25\text{m}^3\text{ alumina}}{0.83\text{m}^3\text{ total body}} \times 100\% = 30\text{vol\% alumina} \qquad (4\text{-}17)$$

Volume of Water to be added:

2.7tons water to be added [from (4-6)]

$$2.7\cancel{\text{tons}}\text{ water} \times \frac{10^6\cancel{g}}{\cancel{\text{ton}}} \times \frac{1\text{m}^3}{10^6\cancel{\text{cm}^3}} \times \frac{\cancel{\text{cm}^3}}{1\cancel{g}}$$

$$= 2.7\text{m}^3\text{ water to be added} \qquad (4\text{-}18)$$

Moisture Content in vol% using (4-10):

2.7m^3 water + 0.83m^3 solids [from (4-14)]

$$= 3.53\text{m}^3\text{ body} \qquad (4\text{-}19)$$

$$\frac{2.7\cancel{\text{m}^3}\text{ water}}{3.53\cancel{\text{m}^3}\text{ total}} \times 100\% = 76\text{ vol\% Moisture} \qquad (4\text{-}20)$$

Solids Content in vol% using (4-9):

$$\frac{0.83\cancel{\text{m}^3}\text{ solids}}{3.53\cancel{\text{m}^3}\text{ total}} \times 100\% = 24\text{ vol\% Solids} \qquad (4\text{-}21)$$

Density

Overall suspension density can be defined simply in units of g/cm^3 or lb/ft^3. Each of the raw materials and water have their own densities, listed in Example 4.2.

Suspension densities, as a function of solids content, can also be similarly defined.

$$\text{Suspension Density} = \frac{\text{Mass Suspension}}{\text{Volume Suspension}} \qquad (4\text{-}22)$$

Example 4.3 Suspension Density in g/cm^3

What is the overall suspension density in g/cm^3 of the suspension described in Examples 4.1 and 4.2? Data needed to answer this question can be found within those sample calculations. Also, recall that these examples were suspensions at 48wt% (24vol%) solids content.

$$5.2 \text{ tons total body} = 3.53 \text{ m}^3 \text{ total body} \qquad [\text{from}(4\text{-}5{,}19)]$$

$$\text{Density } [=] \frac{\text{g}}{\text{cm}^3} = \frac{\text{Mass}}{\text{Volume}} \tag{4-23}$$

$$= \frac{5.2 \cancel{\text{tons}} \text{ total}}{3.53 \cancel{\text{m}^3} \text{suspension}} \times \frac{10^6\text{g}}{\cancel{\text{ton}}} \times \frac{\cancel{\text{m}^3}}{10^6\text{cm}^3} = 1.47\frac{\text{g}}{\text{cm}^3} \tag{4-24}$$

$$= 1.47 \frac{\text{g}}{\text{cm}^3} \text{ @ 48wt\% or 24vol\% solids content} \tag{4-25}$$

Example 4.4 Suspension Density at a Different Solids Content

What is the density of the suspension in Examples 4.1-3 at a different solids content, that is, at 55wt% solids? Note from (4-14 & 18) that at 48wt% solids, the total suspension contained 2.5 tons of solids and 2.7 tons of water, which is 0.83m^3 of solids and 2.7m^3 of water.

Total Suspension Mass using (4-2):

$$\frac{2.5 \text{ tons solids}}{\text{Total Suspension Mass}} \times 100\% = 55\text{wt\% solids} \tag{4-26}$$

$$\frac{2.5 \text{ tons solids}}{55\text{wt\% solids content}} \times 100\% = 4.55 \text{ tons suspension} \tag{4-27}$$

$$\text{New Moisture Content} = 4.55 \text{ tons total - } 2.5 \text{ tons solids}$$
$$= 2.05 \text{ tons water} \tag{4-28}$$

$$2.50\text{ tons solids} = 0.83\text{m}^3\text{ solids} \tag{4-29}$$

$$2.05\text{ tons water} = 2.05\text{m}^3\text{ water} \tag{4-30}$$

$$4.55\text{ tons total suspension} = 2.88\text{m}^3\text{ total suspension} \tag{4-31}$$

Solids Content using (4-2):

$$\frac{2.5\ \cancel{\text{tons}}\text{ solids}}{4.55\ \cancel{\text{tons}}\text{ suspension}} \times 100\text{wt\%} = 55\text{wt\% solids content} \tag{4-32}$$

Solids Content using (4-9):

$$\frac{0.83\cancel{\text{m}^3}\text{ solids}}{2.88\cancel{\text{m}^3}\text{ suspension}} \times 100\text{vol\%} = 29\text{vol\% solids content} \tag{4-33}$$

Suspension Density:

$$\frac{4.55\ \cancel{\text{tons}}\text{ total}}{2.88\cancel{\text{m}^3}\text{total}} \times \frac{10^6\cancel{\text{g}}}{\cancel{\text{ton}}} \times \frac{1\cancel{\text{m}^3}}{10^6\cancel{\text{cm}^3}} = \frac{4.55\text{g}}{2.88\text{cm}^3} \tag{4-34}$$

$$= 1.58\frac{\text{g}}{\text{cm}^3} @\ 55\text{wt\% or }29\text{vol\% solids content} \tag{4-35}$$

Specific Gravity

Density can also be defined as specific gravity, which is the ratio of suspension or solid density relative to the density of water (1.0 g/cm^3).

$$\text{Specific Gravity} = \frac{\text{Density of Solid or Suspension}}{\text{Density of Water}} \tag{4-36}$$

Example 4.5 Specific Gravities

What are the specific gravities of the suspensions in Examples 4.1-4? Note that all required data were calculated earlier.

$$48\text{wt\% solids} = 24\text{vol\% solids} = 1.47\text{g/cm}^3 \qquad [\text{from}(4\text{-}25)]$$

$$55\text{wt\% solids} = 29\text{vol\% solids} = 1.58\text{g/cm}^3 \qquad [\text{from}(4\text{-}35)]$$

Specific Gravity at 48wt% solids:

$$= \frac{1.47\frac{\cancel{g}}{\cancel{c}m^3}}{1\frac{\cancel{g}}{\cancel{c}m^3}} = 1.47 \qquad (4\text{-}37)$$

Specific Gravity at 55wt% solids:

$$= \frac{1.58\frac{\cancel{g}}{\cancel{c}m^3}}{1\frac{\cancel{g}}{\cancel{c}m^3}} = 1.58 \qquad (4\text{-}38)$$

Liter Weight

Some companies express the densities of their suspensions in terms of liter weight, that is, the weight in grams of a liter of suspension.

$$\text{Liter Weight} = \text{Mass of 1 liter of suspension}$$
$$= 1000\text{cm}^3 \times \text{Suspension Density} \qquad (4\text{-}39)$$

Example 4.6 Liter Weights

What are the liter weights of the suspensions in Example 4.5 at 48wt% and 55wt% solids? Since a liter is 1000ml, or 1000 cm^3, the liter weight is calculated by multiplying the density in g/cm^3 by 1000cm^3.

Liter Weight at 48wt% solids:

$$1.47 \frac{g}{\cancel{cm^3}} \times 1000\cancel{cm^3} = 1470 \text{ g} \qquad (4\text{-}40)$$

Liter Weight at 55wt% solids:

$$1.58 \frac{g}{\cancel{cm^3}} \times 1000\cancel{cm^3} = 1580 \text{ g} \qquad (4\text{-}41)$$

Example 4.7 Tank Volume

What volume tank is required to hold the 5.2 metric ton batch in Example 4.1? The answer is a tank large enough to hold the volume of the suspension which was calculated in (4-19): 3.53 m^3. We will start this example, however, from the data given in (4-25): 48wt% solids = 24vol% solids = 1.47g/cm^3.

$$\text{Tank Volume} \geq \text{Suspension Volume} \qquad (4\text{-}42)$$

Volume of Suspension:

$$\text{Suspension Volume} = \frac{\text{Total Suspension Mass}}{\text{Suspension Density}} \qquad (4\text{-}43)$$

$$= \frac{5200\text{kg suspension}}{1.47 \frac{g}{cm^3}} = \frac{3540\cancel{kg}}{\frac{\cancel{g}}{\cancel{cm^3}}} \times \frac{1000\cancel{g}}{\cancel{kg}} \times \frac{1m^3}{10^6\cancel{cm^3}} \qquad (4\text{-}44)$$

$$= 3.54 \text{ m}^3$$

Adjusting Solids Content in a Tank of Suspension

To increase the solids content in a storage tank, one must evaporate water, or add more solids. Another way is to filter press some of the slip, throw the cakes back into the tank, redisperse the suspension, and re-adjust the solids content. Increasing the solids content is never easy to do.

To decrease the solids content in a storage tank, one only needs to add more fluid (water) to the tank.

How much water must be added? Assuming the tank is well mixed, and suspension densities are uniform from top to bottom of the tank, a sample can be taken, dried in a drier, and the solids content can be calculated.

$$\text{Solids Content} = \frac{\text{Mass of Solids}}{\text{Mass of Suspension}} \qquad (4\text{-}45)$$

$$\text{Mass of Suspension} = \frac{\text{Mass of Solids}}{\text{Solids Content}} \qquad (4\text{-}46)$$

Example 4.8 Measuring Tank Solids Content – I

A representative suspension sample must be taken from a well stirred tank. A known weight of sample should be dried in a drier, and the dry weight should be measured. From these values, the solids content can be calculated. For example, if a 22.45g sample from a production tank is placed in a drier, and after drying, it weighs 12.83g, what is the solids content of the production suspension?

$$\frac{12.83\cancel{g}\text{ dry solids}}{22.45\cancel{g}\text{ suspension}} \times 100\% = 57.15\text{wt\% solids} \qquad (4\text{-}47)$$

Example 4.9 Measuring Tank Solids Content – II

An alternative way to measure solids content is to utilize a flask that reproducibly holds a constant, known volume of suspension. If the flask should exactly hold 1 liter volume, then the mass of sample contained would be exactly the liter weight, which is one way solids contents can be expressed. If the flask holds some other volume, the mass of suspension held by the flask divided by the volume contained in the flask will give the density of the suspension.

A stainless steel volumetric cylinder, when completely filled, holds 642.5cm^3 of fluid or suspension. When this flask was filled with the production slip tested in Example 4.7, it held 1039.5g of suspension. What was the solids content of the suspension?

$$\frac{1039.5\text{g suspension}}{642.5\text{cm}^3} = 1.618\,\frac{\text{g}}{\text{cm}^3} \qquad (4\text{-}48)$$

Example 4.10 Comparing Solids Contents – I

How do the two samples in Examples 4.8 (57.15wt% solids) and 4.9 (1.618g/cm^3) compare? To complete this calculation, more information is needed about the suspension. This suspension is the same body composition discussed in Examples 4.1 and 4.2. The necessary information can be found there:

2.5 tons total solids= 0.83 m^3 total body solids [from(4-14)]

$$\frac{2.5\cancel{\text{tons}}}{0.83\cancel{\text{m}^3}} \times \frac{10^6\text{g}}{\cancel{\text{ton}}} \times \frac{\cancel{\text{m}^3}}{10^6\text{cm}^3} = 3.012\,\frac{\text{g}}{\text{cm}^3} \qquad (4\text{-}49)$$

Now, consider 100g of suspension from Example 4.8:

$$100\text{g} \times 57.15\text{wt\% solids} = 57.15\text{g solids} \qquad (4\text{-}50)$$

This 100g contains 57.15g solids and 42.85 g water. Using the density of the solids from(4-49):

$$\frac{57.15\cancel{\text{g}}\text{ solids}}{\left(\dfrac{3.012\cancel{\text{g}}\text{ solids}}{\text{cm}^3\text{ solids}}\right)} = 18.97\text{ cm}^3\text{ solids} \qquad (4\text{-}51)$$

$$\begin{aligned}&42.85\text{cm}^3\text{ water} + 18.97\text{cm}^3\text{ solids}\\&\qquad = 61.82\text{cm}^3\text{ suspension}\end{aligned} \qquad (4\text{-}52)$$

$$\frac{100\text{g suspension}}{61.82\text{cm}^3\text{ suspension}} = 1.618\,\frac{\text{g}}{\text{cm}^3}\text{ suspension} \qquad (4\text{-}53)$$

The calculated density of the suspension in Example 4.8 is $1.618 \mathrm{g/cm^3}$ (4-53), which is the stated density of the sample from Example 4.9. The two samples in Examples 4.8 and 4.9 appear to be two samples taken from the same production batch.

Example 4.11 Comparing Solids Contents – II

How do the two samples in Examples 4.8 (57.15wt% solids) and 4.9 ($1.618 \mathrm{g/cm^3}$) compare? Example 4.10 based the calculation on the wt% solids results from Example 4.8. This calculation will start from the suspension density results of Example 4.9. Recall from (4-49) that the average density of the solids is 3.012g solids/$\mathrm{cm^3}$ solids.

Consider 100 $\mathrm{cm^3}$ of suspension sample from Example 4.9:

$$100\ \mathrm{cm^3}\ \text{suspension} \times 1.618 \frac{\mathrm{g}}{\mathrm{cm^3}} = 161.8\mathrm{g}\ \text{suspension} \qquad (4\text{-}54)$$

Assuming X grams of solids and Y grams of water, two equations are required to solve this problem:

$$\frac{X\ \text{solids}}{3.012 \frac{\mathrm{g}}{\mathrm{cm^3}}} + \frac{Y\ \text{water}}{1.000 \frac{\mathrm{g}}{\mathrm{cm^3}}} = 100\ \mathrm{cm^3}\ \text{suspension} \qquad (4\text{-}55)$$

$$X\ \text{solids} + \quad Y\ \text{water} \quad = 161.8\ \mathrm{g}\ \text{suspension} \qquad (4\text{-}56)$$

Rearranging (4-56) produces:

$$X = 161.8\mathrm{g} - Y \qquad (4\text{-}57)$$

Substituting (4-57) into (4-55) produces:

$$\frac{161.8\mathrm{g} - Y}{3.012 \frac{\mathrm{g}}{\mathrm{cm^3}}} + \frac{Y\ \text{water}}{1.000 \frac{\mathrm{g}}{\mathrm{cm^3}}} = 100\ \mathrm{cm^3}\ \text{suspension} \qquad (4\text{-}58)$$

Multiplying all terms in (4-58) by 3.012g/cm^3 and simplifying:

$$(161.8\text{g} - \text{Y}) + 3.012\ \text{Y} = 301.2\text{g} \tag{4-59}$$

$$-\text{Y} + 3.012\ \text{Y} = 301.2\text{g} - 161.8\text{g} \tag{4-60}$$

$$2.012\ \text{Y} = 139.4\ \text{g} \tag{4-61}$$

$$\text{Y} = \frac{139.4\text{g}}{2.012} = 69.28\text{g water} \tag{4-62}$$

Substituting into (4-57):

$$\text{X} = 161.8\ \text{g suspension} - 69.28\text{g water} \tag{4-63}$$

$$\text{X} = 92.52\ \text{g solids} \tag{4-64}$$

Calculating weight percent solids from (4-2):

$$\frac{\text{Mass Solids}}{\text{Mass Suspension}} \times 100\text{wt\%} \tag{4-2}$$

$$= \frac{92.52\cancel{\text{g}}}{161.8\cancel{\text{g}}} \times 100\text{wt\%} = 57.18\text{wt\%} \tag{4-65}$$

Example 4.8 showed 57.15wt% solids in the sample. Again, the two results appear to be samples taken from the same production batch.

Example 4.12 Adjusting Solids Contents

When the solids contents of tanks full of suspensions need to be adjusted, using the solids content of the tank in wt% makes the calculation relatively simple. The weight percentage solids can be used with the total suspension mass to calculate the mass of solids, and it can easily be used with the mass of solids to calculate the total suspension mass.

How much water must be added to a tank containing 3.80metric tons of suspension at 57.15wt% solids (Example 4.8) to reduce the solids content to 48.00wt%?

Currently, the tank contains:

$$3.80 \text{ tons suspension} \times 57.15\text{wt\% solids} = 2.17 \text{ tons solids} \tag{4-66}$$

At the new solids content, using (4-2), the tank should contain:

$$\frac{2.17\text{tons solids}}{48.00\text{wt\% solids}} = 4.52 \text{ tons suspension} \tag{4-67}$$

The difference between the new, adjusted suspension mass and the current suspension mass is the water to be added:

$$\begin{aligned} &4.52 \text{ tons new suspension - } 3.80 \text{ tons current suspension} \\ &= 0.72 \text{ tons water to be added} \end{aligned} \tag{4-68}$$

Mixtures

$$\text{Density}_{\text{Mixture}} = \frac{\text{Total Mass Mixture}}{\text{Total Volume Mixture}} \tag{4-69}$$

$$\text{Solids Content}_{\text{Mixture}} = \frac{\text{Total Mass Solids}}{\text{Total Mass Mixture}} \times 100\% \tag{4-70}$$

$$\text{Moisture Content}_{\text{Mixture}} = \frac{\text{Total Mass Water}}{\text{Total Mass Mixture}} \times 100\% \tag{4-71}$$

Example 4.13 Mixtures of Suspensions

What will be the solids content of a suspension formed by mixing 0.850 metric tons (52.4wt% solids) of today's production batch with 0.745 metric tons (57.3wt% solids) of yesterday's batch?

The solution to this type problem is to solve individually for the solids and water contained in each suspension, sum all solids, sum all water, and then recalculate the new solids and moisture contents of the combination.

Mass of solids in each:

$$\begin{aligned}&0.850\text{tons} \times 52.4\text{wt\%} + 0.745 \text{ tons} \times 57.3\text{wt\%} \\ &= 0.445 \text{ tons solids} + 0.427 \text{ tons solids} \\ &= 0.872 \text{ tons total solids}\end{aligned} \qquad (4\text{-}72)$$

Mass of fluid in each:

$$\begin{aligned}&0.850\text{tons} \times 47.6\text{wt\%} + 0.745 \text{ tons} \times 42.7\text{wt\%} \\ &= 0.405 \text{ tons water} + 0.318 \text{ tons water} \\ &= 0.723 \text{ tons total water}\end{aligned} \qquad (4\text{-}73)$$

Total suspension:

$$\begin{aligned}&0.872 \text{ tons solids total} + 0.723 \text{ tons water total} \\ &= 1.595 \text{ tons total suspension}\end{aligned} \qquad (4\text{-}74)$$

Solids and moisture contents in the mixture are:

$$\frac{0.872\,\cancel{\text{tons}}\text{ solids}}{1.595\,\cancel{\text{tons}}\text{ suspension}} \times 100\% = 54.7\text{wt\% solids} \qquad (4\text{-}75)$$

$$\frac{0.723\,\cancel{\text{tons}}\text{ water}}{1.595\,\cancel{\text{tons}}\text{ suspension}} \times 100\% = 45.3\text{wt\% moisture} \qquad (4\text{-}76)$$

Chapter Five

Description of Particle Size Distributions

The analysis of particle size distributions is one of the most important functions a ceramic process engineer must perform. Control of the particle physics properties of ceramic suspensions and forming bodies is accomplished by controlling the individual and collective particle size distributions of the powder ingredients. Following particle size analysis, proper presentation of the results can aid in our understanding of the distributions.

Particle size distribution measurements will be the subject of the next chapter. This chapter will deal with the fundamentals of particle size distributions and their graphical presentation.

Histograms (Frequency Distributions)

The first form of distribution is called a histogram, or frequency distribution. Histograms characterize particle size distributions by showing the percentage of particles contained in each of several precisely-defined size classes that cover the range from the smallest particles, D_S, to the largest particles, D_L.

Histogram amounts are labeled "% in Class" because they show the percentage of particles in each size class over which the distribution extends.

Figure 5-1 and Table 5.1 show the histogram of a particle size distribution that was measured by sieve analysis. Each of the vertical lines of each histogram bar corresponds to the size of one of the sieves used in the analysis. The amount shown by the height of each bar is the percentage of particles in the distribution that lie

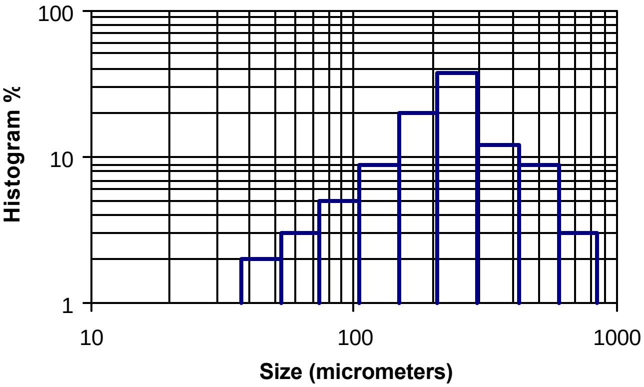

Figure 5-1 Histogram of a Particle Size Distribution

Table 5.1 Histogram and CPFT Data

Size (µm)	% in Class	CPFT	CPGT
841		100	0
	3		
595		97	3
	9		
421		88	12
	12		
298		76	24
	37		
210		39	61
	20		
149		19	81
	9		
105		10	90
	5		
74		5	95
	3		
53		2	98
	2		
37		0	100

within the size bounds for that bar. For example, the smallest size class between 37 and 53 micrometers contains 2% of the distribution (2% in Class). The largest size class between 841 and 595 micrometers contains 3% of the distribution, etc. The sum of all of the percentages (% in Class) in a histogram equals 100%.

Since both axes in Figure 5-1 are logarithmic (log) axes, this type of graph is known as a log-log presentation.

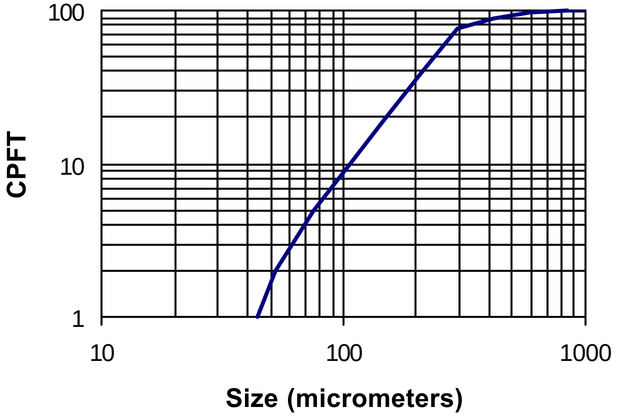

Figure 5-2 CPFT Curve for the Particle Size Distribution

Cumulative Presentations

Figure 5-2, and the third column in Table 5.1, show the corresponding CPFT (cumulative percent finer than) distribution, for the histogram in Figure 5-1. The CPFT graph is also presented in log-log format. Note that when a cumulative distribution is drawn, it is draw as straight line segments connecting the CPFT values at each size. Each CPFT value is the sum of all histogram values for particles smaller than that size. This can be seen in Table 5.1, where the CPFT values are the summation of the '% in Class' values from smallest to largest sizes.

There are no particles in the distribution smaller than 37 μm, so the CPFT value at 37 μm is zero. Two percent of the particles are smaller than 53 μm (the 2% in the smallest size class), so the CPFT at 53μm is 2%. The 5 CPFT at 74μm is the sum of the 3% and 2% in the two smallest size classes. The 10 CPFT at 105μm (10%) is 2% + 3% + 5%, etc. At the upper size limit in the distribution, D_L, the value should always be 100 CPFT.

Note that if all particles are smaller than some size, D_L, all particles are smaller than all sizes larger than D_L. Consistent with

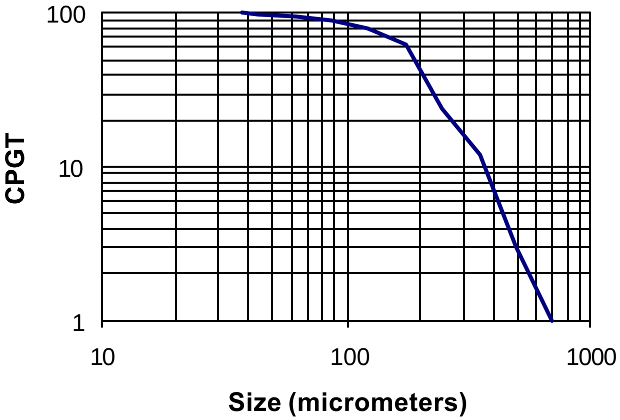

Figure 5.3 CPGT Curve for the Particle Size Distribution

this, the CPFT at 1000μm for this distribution is 100%, as is the CPFT at all sizes larger than 841μm. Similarly on the fine end of the distribution, if the CPFT at 37μm is 0%, the CPFT at any smaller size will also be 0%. The corresponding horizontal lines are not usually drawn, but they are understood.

The fourth column in Table 5.1 shows the corresponding CPGT (cumulative percent greater than) distribution. This form of presentation is less common, but it does occur. Figure 5-3 shows the CPGT chart for this same particle size distribution. Its calculation is similar to the CPFT calculation, but the summation moves from zero (0%) at the coarsest size to 100% at the finest.

Recommended Presentation

All three forms of particle size distribution (PSD) graphs are common within ceramics. Which one is best? Which one is recommended? There are two ways to answer these questions:

The answer from the first point of view says to let tradition dictate the form to be used. All three forms contain the same information. All three are acceptable. As long as particle size distributions are being used to control ceramic production processes, it does not really matter which form is used. Any of the three is better than none.

Two additional questions, however, lead to the second point of view: Which form is easiest to understand? Which form is easiest to use? From this point of view, the histogram is the best choice. True, it contains all of the same information as the CPFT and CPGT presentations, but it presents the information in a straightforward, unmasked form. The accumulation processes which produce the two cumulative curves partially hide important features of the data.

Whenever one is considering production options concerning particle size distributions, it is much easier to see, think through, and predict what will happen to a production body or suspension using histogram information rather than either cumulative form.

Because histograms present the actual amounts in each size class, comparing the amounts in two different size classes, or combining the amounts in a particular size class of two different distributions, is relatively easy to visualize with the histogram presentation. These same tasks, of course, could be accomplished using the cumulative presentation forms, but with greater difficulty.

The following question demonstrates this point: Which class size in the distributions shown in Figures 5-2 and 5-3 contains the greatest mass of particles? The answer is obvious in Figure 5-1, but it is not obvious at all in Figures 5-2 or 5-3.

My suggestion to those who have been traditionally using either cumulative form of particle size distribution is to learn to use histograms. It may take some time until they become a comfortable form to use, but it is a worthwhile effort.

Cumulative/Histogram Conversions

Conversions between any of these forms of particle size distribution presentation are relatively simple to perform.

The difference between the cumulative values at any two particle sizes is equal to the histogram value within that size range.

The cumulative value at any size is the sum of all particles greater than (CPGT) or less than (CPFT) that size.

The CPFT at any size is equal to 100% minus the CPGT at that size. The CPGT at any size is equal to 100% minus the CPFT at that size.

Example 5.1 Histogram to CPFT Conversion

Calculate the cumulative percent finer than 210μm for the histogram shown in Figure 5-1 and Table 5.1.

$$\begin{aligned} \text{CPFT } 210\mu\text{m} &= \text{Summation of all size classes} \\ &\quad\ \text{less than } 210\mu\text{m} \\ &= 20\% + 9\% + 5\% + 3\% + 2\% \\ &= 39\% \text{ finer than } 210\mu\text{m} \end{aligned} \tag{5-1}$$

Example 5.2 CPFT to Histogram Conversion

Calculate the histogram value for the 149μm to 105μm histogram size class from the CPFT data shown in Figure 5-2 and the third column in Table 5.1. The CPFT values at 149μm and 105μm are 19% and 10%, respectively.

$$\begin{aligned} \text{Histogram } \% &= \text{Difference between CPFT values} \\ &= \text{CPFT } 149\mu\text{m} - \text{CPFT } 105\mu\text{m} \\ &= 19\% - 10\% = 9\% \end{aligned} \tag{5-2}$$

Example 5.3 CPFT to CPGT Conversion

If the CPFT value for a distribution at 105μm is 23%, what is its CPGT? The relationship between CPFT and CPGT is:

$$100\% = \text{CPFT} + \text{CPGT} \tag{5-3}$$

Rearranging (5-3) and substituting:

$$\begin{aligned} \text{CPGT } 105\mu\text{m} &= 100\% - \text{CPFT } 105\mu\text{m} \\ &= 100\% - 23\% = 77\% \end{aligned} \tag{5-4}$$

Combining Distributions

In almost all cases, several powders are mixed to form ceramic production bodies and suspensions. The particle size distribution of the whole body can be calculated from the particle size distributions of the ingredient powders. An equation of the form of (1-6) can be used to perform this calculation:

$$P_j = a_1x_1 + a_2x_2 + a_3x_3 + \ldots + a_nx_n \tag{5-5}$$

where P_j = the histogram %, CPFT, or CPGT of the body in the j^{th} size class,
a_i = percentage of i^{th} ingredient in the body
x_i = the histogram, CPFT, or CPGT of the i^{th} ingredient in the j^{th} size class

Equation (5-5) must be used to calculate the particle size distribution value for each particle size (or particle size class) in the distribution. This equation can be used with any of the three forms of particle size distributions. Particle size distributions for each of the ingredient raw materials must be in the same form, and the resulting PSD of the body will be in that same form as well.

Example 5.4 Combining CPFT Distributions

The particle size distributions of A and B powders are given. What will be the particle size distribution of a body that consists of 70% A and 30% B? Equation (5-5) must be used once for each size class in this distribution; $a_A = 0.7$; $a_B = 0.3$; and the x_{Aj} and x_{Bj} values are the CPFT values shown for each size class in Table 5.2.

Table 5.2 PSD Data

Size(µm)	CPFT A	CPFT B
149	100	100
105	60	100
74	40	50
53	0	20
37	0	0

$P_j = a_A \times x_{Aj} + a_B \times x_{Bj} = \mathbf{CPFT_j}$

$P_{149} = 0.7\times 100\% + 0.3\times 100\% = \mathbf{100\%}$

$P_{105} = 0.7\times 60\% + 0.3\times 100\% = \mathbf{72\%}$

$P_{74} = 0.7\times 40\% + 0.3\times 50\% = \mathbf{43\%}$

$P_{53} = 0.7\times 0\% + 0.3\times 20\% = \mathbf{6\%}$

$P_{37} = 0.7\times 0\% + 0.3\times 0\% = \mathbf{0\%}$

To make this example easier to follow, the equations, calculated CPFT values, and table values for each size are aligned.

Example 5.5 Combining Histograms

The histograms of A and B powders are given. What will be the particle size distribution of a body that consists of 40% A and 60% B? Again, Equation (5-5) must be used once for each size class in this distribution; $a_A = 0.4$; $a_B = 0.6$; and the x_{Aj} and x_{Bj} values are the Histogram values shown for each size class in Table 5.3.

Table 5.3 Histogram Data

Size(µm)	A	B
595	24	6
421	36	24
298	30	42
210	10	28
149		

$P_j = a_A \times x_{Aj} + a_B \times x_{Bj} = \mathbf{\%\ in\ Class_j}$

$P_{595\text{-}421} = 0.4\times 24\% + 0.6\times 6\% = \mathbf{13.2\%}$

$P_{421\text{-}298} = 0.4\times 36\% + 0.6\times 24\% = \mathbf{28.8\%}$

$P_{298\text{-}210} = 0.4\times 30\% + 0.6\times 42\% = \mathbf{37.2\%}$

$P_{210\text{-}149} = 0.4\times 10\% + 0.6\times 28\% = \mathbf{20.8\%}$

Again, to make this example easier to follow, the equations, calculated histogram values, and table values for each size are aligned.

The presentation of histogram values in Table 5.3 associates each value with a single size, although the actual size classes are defined by each size and its next smaller neighbor. If one size is used to define a size class, it can be the upper size, the lower size, or a mean size. Each lab, or company, should standardize their data using one of these forms. Using the upper size limit as the label for that class size happens to be the author's standard procedure. For this reason, there are no 149µm histogram values in Table 5.3, nor calculations for a body value at that size.

Linear vs Log Scales

Particle size distributions can be presented on linear, semi-log, or log-log scales. Simple preference, of course, usually defines the type of scales used when presenting particle size distributions. But again, there are good technical reasons for using log-log presentations.

Figures 5-4, 5-5, and 5-6 show linear, semi-log, and log-log charts of the body histogram from Example 5.5. All sizes in these histograms correspond to standard sieve sizes. Notice that in the linear presentation in Figure 5-4, the size classes all appear to have different sizes (the bars are different widths). Standard sieve size classes form a geometric progression and they all have equally sized bars when plotted on log axes as shown in Figures 5-5 and 5-6.

When using log scales, very broad ranges of particles sizes can be depicted legibly on a single chart. Figure 5-1 shows two decades of particle sizes (from 1000µm to 10µm) while Figures 5-5 and 5-6 each show only a single decade of particle sizes (from 100 to 10µm). If the range of sizes needs to include sizes from 10,000µm down to 0.1µm (5 decades of width), a log scale would allow all values, both large and small, to be easily distinguished – a linear scale would not.

When a linear scale is used, as in Figure 5-4, the smaller sizes all merge into one indistinguishable mass. It would be impossible to draw or distinguish a value at 0.1µm from a value at 0.2µm on a linear scale. This is one of the main reasons why linear scales are seldom used for the size axis of a particle size distribution plot.

Similar considerations apply to the histogram and the CPFT axes (the Y-axis) of particle size distribution plots. With a linear axis, the details of the minor constituents in the distribution will

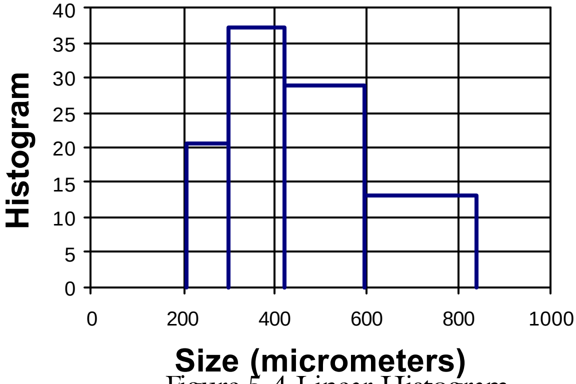

Figure 5-4 Linear Histogram

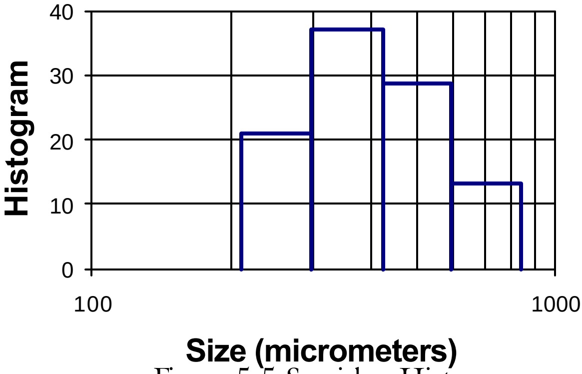

Figure 5-5 Semi-log Histogram

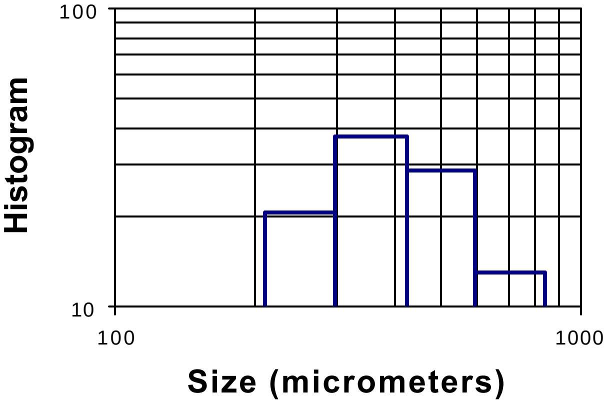

Figure 5-6 Log-Log Histogram

be obscured by the requirement that the size axis must extend from large sizes down to zero.

There is no zero, however, on a log axis. All decades on log axes (from 10,000μm to 1,000μm, from 1,000μm to 100μm, from 100μm to 10μm, etc.) are the same width. Each succeeding decade along the axis approaches zero, but zero will never appear.

Considering the importance of the colloidal particles to the control of ceramic production bodies, it is necessary not only to measure sub-sieve and submicron particles, but to present the particle size distribution results in a manner that allows engineers to see, understand, and utilize the data in these fine size ranges.

There is another reason for using log-log axes for particle size distributions. Several of the fundamental particle size distribution equations for particle packing and particle comminution by Andreasen[3], Dinger-Funk[4], and Gaudin-Schuhmann[5], are power law equations (Arrhenius equations) that produce straight line plots on log-log axes. This applies to the histograms of all three equations and the cumulative forms of the Andreasen[3] and Gaudin-Schuhmann[5] equations.

When one is trying to produce a body with a particle size distribution that packs well, or when one is trying to alter milling parameters to change the product particle size distribution, the results can be more easily deciphered when the distributions are plotted log-log. One simply needs to consider the straightness of the line, or the slope of the line, to determine whether the experiments were successful or not.

The author recommends that all particle size distributions be plotted as log-log histograms.

Size Classes & Series

The complete set of sieves for measuring particle size distributions follow a *fourth root of two* series ($\sqrt[4]{2} = 1.1892$). Any two adjacent sieve sizes are a factor of 1.1892 apart. That is, 1.1892 times the smaller size equals the larger size. When the sieve series consists of every other available sieve size, a *square root of two* series

$(\sqrt{2} = 1.4142)$ is produced. In fact, two different square root of two series can be produced, depending on the size of the first screen selected. Four different *factor of two* series can also be produced by using every fourth screen. Table 5.4 shows examples of these series.

Table 5.4 Sieve Series[6]

Mesh No.	2:1	$\sqrt{2}$	$\sqrt[4]{2}$
16	1000	1000	1000
20			850
24		710	710
28			600
32	500	500	500
35			425
42		355	355
48			300
60	250	250	250
65			212
80		180	180
100			150
115	125	125	125
150			106
170		90	90

Particle size distribution presentations should always show size classes that follow one of these sieve series. When plotted on log axes, each size class in the series will be the same width. If such a series were plotted on linear axes, or if screens were randomly missing from a distribution plotted on log axes, all of the histogram size class bars would appear as different widths.

Even when one of the modern particle size analyzers is being used to measure particles in the sub-sieve size ranges, the data should always be presented as equal width size classes (i.e., in one of these series).

If they do not already use such a series for size classifications, most new analyzers have an option that allows the operator to specify the sizes to be used when presenting the measured data. Sub-sieve size classes should be a continuation of the sieve series size classes used for the coarser particles. Even when sieves are not being used, particle size distribution data should always be presented in this way.

When complete size series are used, it is much easier to compare amounts in adjacent size classes and to make appropriate engineering decisions concerning the particle size distribution results.

The underlying problem, solved by using appropriately chosen size series, deals with our willingness to take shortcuts when presenting particle size data. All of the histograms presented so far in this chapter, Figures 5-1, 5-4, 5-5, and 5-6 show the *% in Class* results as bars. The shortcut is to simply plot a single point that represents the histogram value for each bar, and then to connect the dots forming a line. When this occurs, it is difficult to see the width of each size class in the distribution. It is normal to assume that all class sizes are the same width, but when they are not, major errors can be made. Histograms, plotted as bars will show the widths of each size class very clearly.

When a complete *square root of two*, or *fourth root of two* series is used, histograms can be plotted as bars, or lines, without detrimental consequences.

When sieves, or size classes, are randomly skipped during routine particle size analyses, or random-width size classes are used, confusion (chaos?) and misleading results will be produced.

The simplest remedy to this potential problem is to always use the same, complete size classification series. Each lab should specify a complete size series to be used as a laboratory standard (including sieve sizes and size categories for sub-sieve analytical instruments, as appropriate), and then that size classification should be used consistently for all particle size distribution measurement techniques.

Example 5.6 Particle Size Series Calculations – I

The smallest available standard sieve is 400 Mesh, which is 38µm. What are the next five smaller size classifications following a $\sqrt[4]{2}$ size series?

$$\frac{\mathrm{Size}_{\mathrm{Larger}}}{\mathrm{Size}_{\mathrm{Smaller}}} = \sqrt[4]{2} = 1.1892 \qquad (5\text{-}6)$$

$$\mathrm{Size}_{\mathrm{Smaller}} = \mathrm{Size}_{\mathrm{Larger}} / 1.1892 \qquad (5\text{-}7)$$

$$\begin{aligned}
\text{Size}_1 &= 38\mu\text{m} / 1.1892 = 31.95\mu\text{m} = 32\mu\text{m} \\
\text{Size}_2 &= 31.95\mu\text{m} / 1.1892 = 26.87\mu\text{m} = 27\mu\text{m} \\
\text{Size}_3 &= 26.87\mu\text{m} / 1.1892 = 22.59\mu\text{m} = 23\mu\text{m} \\
\text{Size}_4 &= 22.59\mu\text{m} / 1.1892 = 19.00\mu\text{m} = 19\mu\text{m} \\
\text{Size}_5 &= 19.00\mu\text{m} / 1.1892 = 15.98\mu\text{m} = 16\mu\text{m}
\end{aligned} \tag{5-8}$$

The next five $\sqrt[4]{2}$ sizes smaller than 38μm are 32, 27, 23, 19, and 16mm.

Example 5.7 Particle Size Series Calculations – II

What are the next five smaller size classifications below 38mm following a $\sqrt{2}$ size classification?

$$\frac{\text{Size}_{\text{Larger}}}{\text{Size}_{\text{Smaller}}} = \sqrt{2} = 1.4142 \tag{5-9}$$

$$\text{Size}_{\text{Smaller}} = \text{Size}_{\text{Larger}} / 1.4142 \tag{5-10}$$

$$\begin{aligned}
\text{Size}_1 &= 38\mu\text{m} / 1.4142 = 26.87\mu\text{m} = 27\mu\text{m} \\
\text{Size}_2 &= 26.87\mu\text{m} / 1.4142 = 19.00\mu\text{m} = 19\mu\text{m} \\
\text{Size}_3 &= 19.00\mu\text{m} / 1.4142 = 13.44\mu\text{m} = 13\mu\text{m} \\
\text{Size}_4 &= 13.44\mu\text{m} / 1.4142 = 9.50\mu\text{m} = 10\mu\text{m} \\
\text{Size}_5 &= 9.50\mu\text{m} / 1.4142 = 6.72\mu\text{m} = 7\mu\text{m}
\end{aligned} \tag{5-11}$$

The next five $\sqrt{2}$ sizes smaller than 38μm are 27, 19, 13, 10, and 7μm.

Particle Size Distribution Equations

Andreasen's Equation[3] for particle packing, and Gaudin's & Schuhmann's Equation[5] for comminution are the same equation:

$$\frac{CPFT}{100\%} = \left(\frac{D}{D_L}\right)^n \qquad (5\text{-}12)$$

where D = particle size,
D_L = largest particle in the distribution, and
n = distribution modulus.

The Dinger-Funk Equation[4] is:

$$\frac{CPFT}{100\%} = \frac{D^n - D_S^n}{D_L^n - D_S^n} \qquad (5\text{-}13)$$

where D_S = smallest particle in the distribution, and
all other variables are as defined above.

Andreasen's packing experiments showed that best packing occurs when the distribution modulus, n, has values between ⅓ and ½. Dinger & Funk showed that best packing occurs when n = 0.37, which is in agreement with Andreasen's results.

Gaudin & Schuhmann showed that the Andreasen packing equation also applies to comminution. Typical distribution moduli after dry milling are 0.6 and greater. The distribution moduli of wet-milled powders, however, can be less than 0.6 by proper control of the mill parameters. Mixtures of powders can have large, small, or even negative distribution moduli.

Dinger & Funk derived their equation from Andreasen's, by adding the concept of a smallest particle size, D_S. According to Andreasen's theory, his equation includes infinitely small particles. Practically, this is impossible – actual particle size distributions don't contain infinitely small particles. The addition of the D_S to the equation solves this problem.

The histograms of all three of these equations, and the cumulative distributions of Andreasen's, and Gaudin and Schuhmann's equations all plot as straight lines on log-log axes.

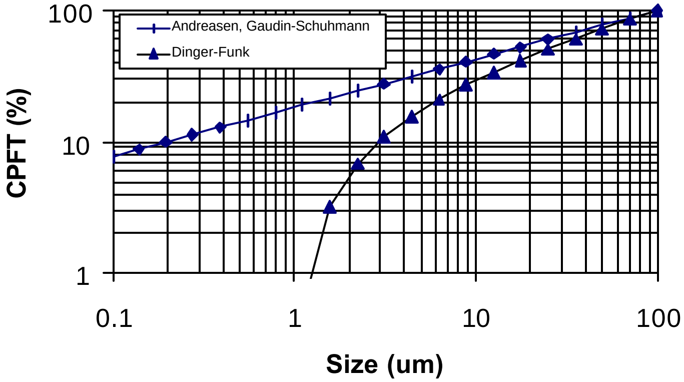

Figure 5-7 Andreasen, Gaudin-Schuhmann, and Dinger-Funk CPFT Distributions

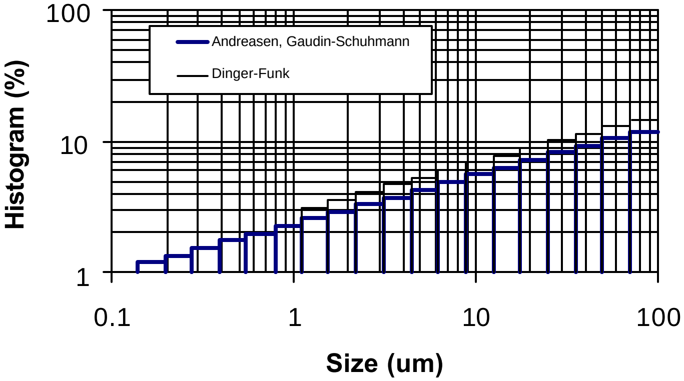

Figure 5-8 Andreasen, Gaudin-Schuhmann, and Dinger-Funk Histograms

Figure 5-7 shows examples of the cumulative forms of these equations, and Figure 5-8 shows examples of the histograms. All of the distributions shown in these two figures have distribution moduli, n, equal to 0.37. Note that the Andreasen and Gaudin-Schuhmann distributions in both figures plot as straight lines. The CPFT version of the Dinger-Funk distribution is a curved line, typical of most particle size distributions, but the histogram also plots as a straight line. These distributions would not appear as straight line plots on linear or semilog charts.

Graphical Interpretation of Distribution Moduli

The distribution modulus, n, in these equations is frequently referred to as the *slope* of the distribution line. This refers to the slope of the line, just as *m* is the slope of the straight line:

$$y = mx + b \qquad (5\text{-}14)$$

The fundamental meaning of the slope of a particle size distribution comes from the alternative presentation of log-log plots. If one plots the logarithms (base 10) of the histogram values versus the logarithms (base 10) of the particle sizes on linear axes, the slope of the line through the histogram bars (or through the CPFT curve) is equal to the distribution modulus.

Figure 5-9 shows a replot of Figure 5-8. In this case, the logs of the histogram values are plotted versus the logs of the particle sizes on linear axes. The two figures are identical — only the axes and axis labels have been changed.

The solid black line in Figure 5-9 has been drawn parallel to the upper edges of the histogram bars in the figure. The equation for this line, in the form of Equation (5-14) is:

$$y = 0.37x + 0.63 \qquad (5\text{-}15)$$

With X and Y defined as shown on the chart, the complete equation for this line is:

$$\log\text{(histogram)} = 0.37\ (\log\text{ particle size}) + 0.63 \qquad (5\text{-}16)$$

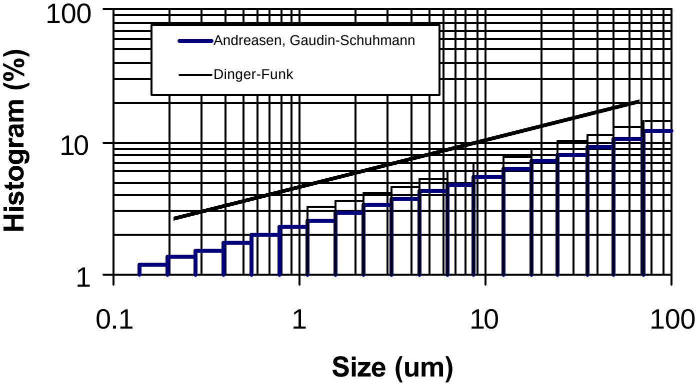

Figure 5-9 Log of Andreasen, Gaudin-Schuhmann, and Dinger-Funk Histogram Values vs Log Size

Both forms of this equation (5-15 and 5-16) show that the slope of the straight line in Figure 5-9 is 0.37. This is exactly the value of the distribution modulus used to draw the histograms and CPFT distributions in all three of these figures (5-7, 5-8, & 5-9).

The distribution modulus of a histogram, or a straight-line CPFT distribution, is equivalent to the slope of the straight line when the logs of the histogram or CPFT values are plotted versus the logs of the particle sizes on linear axes.

The steeper the slope of this line, the greater the value of the distribution modulus. The less steep the slope of this line, the lower the value of the distribution modulus. In fact, distribution moduli can be negative.

When a particle size distribution has a negative distribution modulus, it means that greater masses of particles are contained in the smaller size classes than in the larger size classes.

Negative Distribution Moduli

Distributions with negative distribution moduli are not typical of milled powders. They are easily formed when several powders are mixed to produce a body and greater percentages of the fine powders are combined with lesser percentages of the coarse powders.

Negative distribution moduli are relatively common in ball clays and kaolins which, in their sedimentary forms, are mixtures of many different particle size distributions containing more fine particles and fewer coarse particles.

Example 5.8 Calculation of Distribution Modulus

Figure5-10 shows part of the histogram from a ball milled powder. Estimate the distribution modulus of this particle size distribution between ~3μm and ~50μm.

The straight line has been added to the figure to highlight the slope of the histogram in this size range. This line passes through two grid points on the chart, (3μm, 0.3%) and (70μm, 5%). These two points can be used to calculate the distribution modulus:

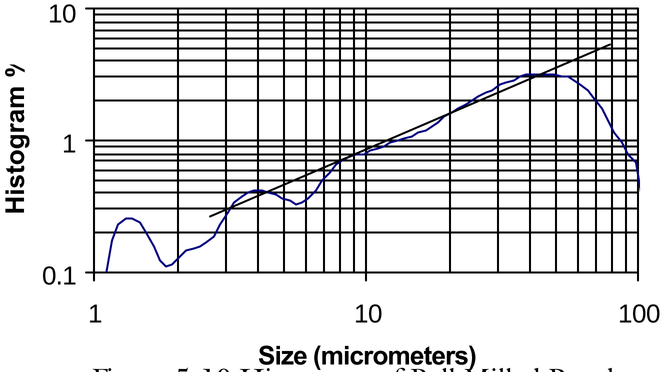

Figure 5-10 Histogram of Ball Milled Powder

$$\text{Distribution Modulus} = n = \text{slope of the line} = \frac{\Delta Y}{\Delta X}$$

$$= \frac{\log 5.0\% - \log 0.3\%}{\log 70\mu m - \log 3\mu m} = \frac{0.699 - (-0.523)}{1.845 - 0.477}$$

$$= \frac{1.222}{1.368} = 0.893 = n \qquad (5\text{-}17)$$

The slope that defines the distribution modulus is the slope calculated with the logs of the histogram and size values when plotted on log histogram vs log size axes as in Figure 5-8. Therefore, the slope is calculated using the differences (ΔY and ΔX) between the logs of the values, as shown in Equation (5-17).

The distribution modulus calculated in this example, n=0.893, is typical of ball milled powders(n > ~0.6).

Chapter Six

Particle Size Distribution Measurements

Several types of particle size analysis techniques are commonly used in industrial practice today. Sieve analysis and sedimentation are traditional methods. Automated particle size analyzers that can measure particle sizes in the sub-sieve range (<~38µm) are also frequently used. Common automated techniques include static sedimentation, centrifugal sedimentation, microscopy, electrical resistance, and laser scattering.

Most of the automated techniques are computer-driven, so no calculations are required to obtain particle size distribution results. Depending on the instrument and the system defaults, it may be desirable to specify a particle size series for data presentation (as discussed in Chapter 5) to insure that all particle size data measured at a single company location are comparable.

Sedimentation, Laser Scattering, and Electrical Resistance Methods

All particle size analysis techniques have upper and lower size bounds. Outside these bounds particle sizes cannot be measured. Sedimentation techniques are useful between ~74µm and ~1µm. Particles greater than ~74µm disrupt sedimentation by settling too fast, pulling smaller particles with them. Particles less than ~1µm (the colloidal size range) are affected by Brownian motion and they don't settle as predicted by Stoke's Law.

In sedimentation techniques, particles in the size classes smaller than the instruments' lower size bounds cannot be measured but the total mass of particles in this range is usually measurable. For example, in the Micromeritics SediGraph 5100[*], which uses X-ray absorption to detect particles, the mass of particles smaller than the lower size bounds is recorded with each sample. This instrument measures the cumulative mass percent finer than each size, so when it reaches its lower size limit, the CPFT value at that point is available. This is very useful data.

Laser scattering techniques measure particle sizes from several hundred micrometers down into the sub-micron size ranges. The fundamental physics used to design these instruments is usually based upon scattering (diffraction) off of spherical particles, even though spherical particles are not frequently encountered.

Laser scattering techniques are limited to very specific particle size ranges. Instrument detectors are positioned to receive the signals scattered from specific size classes of particles. Any particles outside these detection limits, such as particles smaller than the instruments' lower size bounds and particles greater than the instruments' upper size bounds, will not (cannot) be measured. Particles that cannot be detected, by definition, simply do not exist. If 50% of a powder sample is smaller than a laser scattering instrument's lower detection limit, the particle size distribution results will give the details (normalized to 100%) of the particles in the measuring range, without giving any suggestion of the percentage of particles not measured. This is a common limitation of the laser scattering technique.

Laser scattering measurements can be performed in relatively short periods of time (seconds), but precision of results increases with the improved sampling statistics of longer analysis times. The accuracies of results are limited by the particle physics assumptions and the sensor limitations mentioned.

The electrical resistance method measures the resistance as particles pass between two electrodes. As particles pass through the gap, the resistance changes as functions of the resistivity and volume

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[*] Micromeritics Instruments Corp., Norcross, GA.

of the particle between the electrodes. Resistance measurements are then converted to particle sizes and the numbers are tabulated. Samples analyzed with this technique must be very dilute to insure that particles pass through the electrode orifice one-at-a-time.

Sieve Analyses

Standard sieves have lower limits, typically at 38μm. Some dry, sonic sieving techniques use small specialty sieves to measure even smaller particles.

Sieve analysis results are affected by the quantity of powder analyzed, the nature and duration of the vibrations (ro-tapping) used during sieve analyses, and the type of analysis performed (wet or dry).

Too little powder will produce poor sampling statistics. Too much powder can cause *blinding* of screens, and inaccurate results. Screens are *blinded* when holes are blocked by large particles, which prevents smaller particles from passing. When *blinding* occurs, sieve analysis results will be incorrect.

Sieve analyses can be performed wet or dry. When performing dry sieving, static charges can cause fine particles to cling to the surfaces of coarse particles which can prevent accurate separations by size. Ro-tapping will usually not dislodge such particles. Some fines will therefore contribute to the masses of particles on coarse screens. Wet sieving can eliminate this problem. The water washes the fines from the surfaces of the coarse particles and allows them to flow to the appropriate finer screens or to the pan.

Wet sieving is frequently performed to analyze particle size distributions of slurries or suspensions. Wet samples do not need to be dried before an analysis, but the sieves and particles need to be dried after analysis before masses of particles can be measured.

Size Definitions

Each type of instrument defines particle size in its own special way. Results from the various types of instruments will differ, although all results from any single instrument (when standard operating procedures are performed with consistency) should be consistent and comparable with one another.

Sieve analyses characterize particles by the size of the smallest square holes through which particles can pass. Non-spherical particles, depending on their orientation, can fit through a variety of hole sizes. Which size characterizes an American football? ... its length from point to point? ... or its diameter at the midsection? Which size characterizes a standard pencil? ... its length? ... or its diameter?

In each of these examples, the smallest characteristic size in each case would be the midsection diameter, but depending on the type and magnitude of vibration, some of the footballs and pencils would report on larger screens than the one characteristic of their midsection diameter. Even if all footballs and all pencils were exactly the same size, each analysis would report a range of sizes for footballs and pencils from their midsection diameters to larger sizes.

For similar reasons, sieve analysis results for fibrous or irregularly shaped materials are difficult to interpret (especially, if the material is not known to be fibrous or irregularly shaped.)

Automated sedimentation techniques scan particles in planes perpendicular to their settling direction. Some systems use visible light to analyze shadows cast by particles. Other systems use X-ray absorption to analyze the mass of particles in the plane of the beam. Signal intensities in each case are converted to indicate the mass of particles in the beam. The time it takes for the each particle size to settle to the plane of the beam defines the particle sizes using Stoke's Law. Sizes are usually specified as equivalent spherical diameters.

Sedimentation techniques require each powder sample to have a single density. A whiteware *body* in which all powders have the same density could be analyzed in a single sedimentation analysis. But a whiteware body containing clays, feldspar, quartz, kaolin, and some added alumina (which is a significantly higher density) could not be analyzed in a single sedimentation run. Each powder ingredient in the alumina body can be analyzed individually, however, and the particle size distribution for the whole body can be calculated.

Laser scattering techniques measure diameters based on the particle physics assumptions used to define the scattering geometries. Laser scattering sizes are defined as equivalent spherical diameters. Particles in laser scattering analyses will be tumbling as they flow through the analysis cells, rather than quiescent as expected in sedimentation techniques.

Laser scattering techniques are not affected by density variations as are the sedimentation techniques. Production *body* samples (regardless of constituent body powder densities) can therefore be analyzed using laser scattering techniques.

Microscopy techniques measure the dimensions of particles lying on flat surfaces. A coin, when dropped onto a hard surface, will almost always lie flat with its round profile perpendicular to the vertical. Similarly, non-spherical particles will typically lie in their most stable orientations, exposing the particles' largest cross-sections to the viewer. Although thickness measurements are possible using SEM techniques, thicknesses are not easily measured by microscopy.

As a result, one should not expect total randomness of particle orientation when using microscopy. Because of non-random orientations, microscopy tends to overestimate the sizes of particles.

Sampling statistics are important when measuring particle size distributions by microscopy. The other types of analyses measure enormous numbers of particles. The numbers of particles analyzed by microscopy tend to be minuscule. An analysis collected from only a few hundred particles is simply insufficient to properly characterize a particle size distribution. Don't attempt to perform particle size analyses manually using microscopy — even under ideal conditions, sampling statistics will be poor. With automated systems, thousands of particles, representing many frames of view, must be analyzed to obtain reasonable counting statistics.

Sampling

The accuracy of sampling always affects particle size analyses. Some analysis techniques are much more precise than others, but when non-representative samples are analyzed, the results will be very precise measurements of bad samples (i.e., results will be useless).

Everybody *always* thinks their samples are representative of the bulk, but the samples frequently are not. Proper sampling must be considered every time a small sample is taken from a large one. Even in the lab, when a sample is prepared in a large beaker, but only a small fraction of its contents is placed into the analysis chamber, proper sampling techniques must be observed, or the sample analyzed will be non-representative.

When samples are not representative, production controls will appear to be inconsistent. Production problems will come and go for no apparent reason. Lots of reasons can be given to explain production problems that mysteriously come and go, but proper sampling should not be one of them.

Importance of Particle Size Analysis

This chapter wouldn't be complete without a brief statement of the importance of particle size distribution controls to ceramic production processes. Regardless of the problems inherent in measuring particle size distributions, control of the particle size distributions of all powders in a ceramic body is critical[2,7].

The overall particle size distribution (PSD) of a body controls rheological properties, that is, viscous behavior during processing. PSD also controls the packing potential of the powders in a body, the overall porosity and pore size distribution within a compact, drying and firing shrinkages, the solids content and flocculant/deflocculant concentrations needed to achieve a target viscosity, the surface area of the powders, the interparticle spacing in suspensions, the number of particles, etc.

Without a doubt, PSD is one of the most important processing controls available to a ceramic engineer.

Example 6.1 Calculate A Sieve Analysis

Sieve analyses should be performed using a complete stack of sieves in a $\sqrt[4]{2}$ or $\sqrt{2}$ series. Do not skip any screens in the series within the range of sizes to be measured. At the completion of the analysis, the results should appear as shown in Table 6.1. Results should include the mesh numbers and the mass of particles contained on each screen.

All powder analyzed should pass through the largest screen used in the analysis. When this occurs, the coarsest screen will contain no material. The reason for this is to allow the results to completely define the range of particle sizes between a largest particle size and the sub-sieve size range.

Table 6.1 Sieve Analysis Data

Mesh No.	Amount (g)
20	0.0
24	1.7
28	2.6
32	7.9
35	12.6
42	18.8
48	12.0
60	9.8
65	3.7
pan	5.2

A sieve analysis can give no indication whatsoever of the size of powders remaining on the top screen (the largest) in the stack. If another sieve analysis had been performed with the same sieves shown in Table 6.1, and 2 grams of particles remained on the 20 Mesh screen, how large would those particles be? Without a larger sieve to delimit their size, we simply wouldn't know. All we would know for sure is that they were larger than 20 Mesh — which covers a large range of possible particle sizes.

At the other extreme, however, particles smaller than the smallest sieve (those in the pan) are considered to be the *sub-sieve* fraction of the analysis. If smaller sieves are available, the powder in the pan can be further analyzed using those sieves. If no smaller sieves are available, the powder in the pan can be further analyzed using one of the automatic particle size analyzers that can measure particle sizes down into the sub-sieve and sub-micrometer ranges.

To complete the sieve analysis calculation, one needs to perform the following steps using the sieve results:

1 – Calculate the total mass of particles in the analysis.

2 – Enter the micrometer sizes corresponding to the sieves.

3 – Convert the masses on each screen to percentages, that is, to the histogram values (*% in Class*).

4 – Calculate CPFTs from the histogram values.

5 – Plot the results on log-log paper.

Table 6.2 shows the complete results of the sieve analysis calculation. The original data from Table 6.1 are shown in the first and third columns of Table 6.2. The particle sizes must be in micrometers before the results can be plotted. Micrometer sizes corresponding to the mesh numbers are listed in the second column of Table 6.2. The masses of particles on each screen were summed and the total mass is shown on the bottom line of the table. According to the data recorded in Table 6.1, this sieve analysis was performed on a total of 74.3 grams of powder.

Table 6.2 Complete Sieve Analysis Data

Mesh No.	Size(μm)	Amount (g)	Histogram Values (% in Class)	CPFT(%)
20	850	0.0	0.0	100.0
24	710	1.7	2.3	97.7
28	600	2.6	3.5	94.2
32	500	7.9	10.6	83.6
35	425	12.6	17.0	66.6
42	355	18.8	25.3	41.3
48	300	12.0	16.1	25.2
60	250	9.8	13.2	12.0
65	212	3.7	5.0	7.0
pan		5.2	7.0	0.0
	Total:	74.3	100.0	

The masses in column 3 are normalized to produce the *% in Class* histogram values in column 4. For example:

$$\text{24 Mesh} \qquad 1.7\text{g} \qquad \frac{1.7\text{g}}{74.3\text{g}} \times 100\% = 2.3\% \tag{6-1}$$

$$\text{28 Mesh} \qquad 2.6\text{g} \qquad \frac{2.6\text{g}}{74.3\text{g}} \times 100\% = 3.5\% \tag{6-2}$$

As a check, the sum of the percentages in column 4 is listed on the bottom line of the table. This sum should, and does, total 100%. Following calculation of the histogram values, the CPFT values in the last column can be calculated.

Note that all of the particles are smaller than 20 mesh. That is, they all passed through this first, coarsest screen. Its CPFT, the cumulative percent finer than 850μm, is 100%. The 1.7g on the 24 mesh screen passed through 20 mesh but those particles were too large to pass through 24 mesh. Those particles form the 20×24 Mesh size category (850-710μm). Those are the only particles in the distribution larger than 710μm; everything else is smaller than 710μm; so the CPFT at 24 mesh (710μm) is:

$$\text{CPFT @ 24 mesh } (710\mu\text{m}) = 100\% - 2.3\% = 97.7\% \tag{6-3}$$

The 24×28 Mesh category (710-600μm) contains 3.5% in class. These 3.5% plus the 2.3% in the 20×24 Mesh category are the only particles coarser than 600μm. The cumulative percent finer than this size can be calculated by subtracting this value (3.5%) from the CPFT at the next larger size (in this case, at 710μm) or by summing the particles on all smaller screens:

$$\text{CPFT @ 28 mesh } (600\mu\text{m}) = 100\% - 2.3\% - 3.5\% = 94.2\% \tag{6-4}$$

or

$$\begin{aligned}\text{CPFT @ 28 mesh } (600\mu\text{m}) &= 7.0\% + 5.0\% + 13.2\% \\ &\quad + 16.1\% + 25.3\% + 17.0\% + 10.6\% = 94.2\%\end{aligned} \tag{6-5}$$

This same procedure is repeated to calculate the CPFTs for all smaller size classes. Note that there can be nothing smaller than the pan, so its CPFT value should be zero (0.0%). The CPFT at the smallest screen size (in this case 65 mesh, 212μm) should be equal to the percentage in the pan. The two different methods to calculate the CPFTs from histogram values, plus these two points can also be used to check that the calculation has been done correctly.

To complete the analysis, the data can be plotted on log-log axes. First, one should mark the size classes on the log-log paper as shown in Figure 6-1. Then, the histogram bars can be drawn at the heights corresponding to the *% in Class* values, and the CPFT values

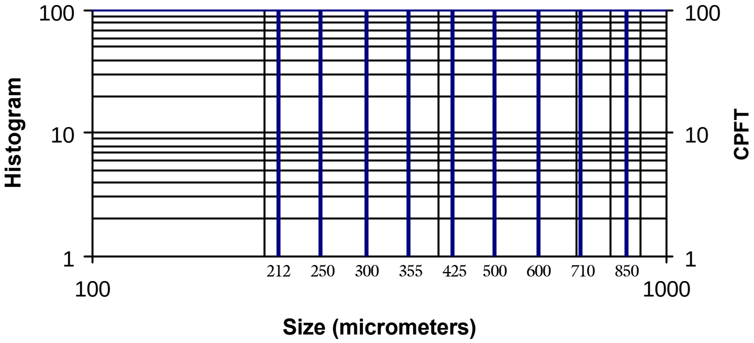


Figure 6-1 Particle Size Distribution Size Classifications

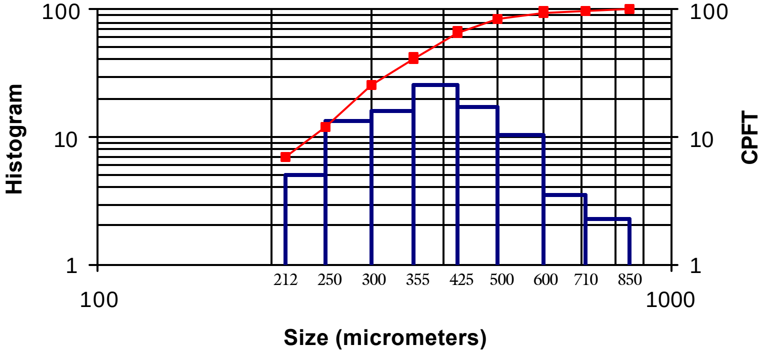


Figure 6-2 Histogram and CPFT Plots

can be plotted with straight lines drawn to connect the points. Both histogram and CPFT distributions are shown in Figure 6-2.

If all size classes form a $^{4}\sqrt{}^{2}$ or $\sqrt{2}$ series, all of the histogram bars (as in Figure 6-2) will be the same width. The CPFT at the smallest size will correspond to the amount in the pan (7.0 CPFT @ 212μm), and there will be no histogram bar for the pan.

Notice that the amount on each screen is drawn as the height of the bar between that screen's size and the next larger screen's size. After all, the material residing on each screen passed through the next larger screen, but could not pass through the screen on which it rests. The histogram bars reflect that. All of these points can be checked in Figure 6-2, and they should all be consistent with the data in Table 6.2.

Example 6.2 Recalculate a CPFT Distribution to Fit a Size Series

Table 6.3 shows a CPFT distribution that was measured using an automated analyzer with variable width size classes. Recalculate this distribution to fit a $\sqrt[4]{2}$ size series.

First calculate a $\sqrt[4]{2}$ size series, starting with the 170 mesh sieve size, which is 90μm.

$\sqrt[4]{2}$ is 1.1892.

90 μm/1.1892= 75.7 μm
75.7μm/1.1892= 63.6μm
63.6μm/1.1892= 53.5μm
53.5μm/1.1892= 45.0μm
45.0μm/1.1892= 37.8μm (6-6)
37.8μm/1.1892= 31.8μm
31.8μm/1.1892= 26.8μm
26.8μm/1.1892= 22.5μm
22.5μm/1.1892= 18.9μm

Table 6.3 Measured CPFT

Size (μm)	CPFT (%)
90	100.
80	98.2
70	95.3
60	81.8
50	53.9
40	25.6
30	7.1
20	1.0

The new $\sqrt[4]{2}$ size series includes the following μm sizes: 90, 75.7, 63.6, 53.5, 45.0, 37.8, 31.8, 26.8, 22.5, and 18.9. The conversion to the new size classes requires that each point be interpolated from a straight line connecting the two points surrounding it in the original distribution. The interpolation should use the straight line on the *log-log* plot.

The largest size in each distribution is 90μm, so the new distribution starts at 100CPFT @ 90μm. The second size in the new distribution, 75.7μm, is between the 70μm and 80μm values in the original data. It can be interpolated from those two points. The third size in the new distribution, 63.6μm, is between the 60μm and 70μm values of the original distribution. It can be interpolated from those two values. The rest of the distribution can be calculated in similar manner. The interpolation process uses the following equations:

$$\frac{\log \mu_{NEW} - \log \mu_{SO}}{\log \mu_{LO} - \log \mu_{SO}} = \frac{\log \%_{NEW} - \log \%_{SO}}{\log \%_{LO} - \log \%_{SO}} \tag{6-7}$$

$$\log \%_{NEW} = \left(\frac{\log \mu_{NEW} - \log \mu_{SO}}{\log \mu_{LO} - \log \mu_{SO}}\right) \times (\log \%_{LO} - \log \%_{SO}) + \log \%_{SO} \tag{6-8}$$

$$10^{\log \%_{NEW}} = \%_{NEW} \tag{6-9}$$

where μ_{NEW} = new size
μ_{SO} = smaller original size
μ_{LO} = larger original size
$\%_{NEW}$ = new CPFT
$\%_{SO}$ = original CPFT at smaller size
$\%_{LO}$ = original CPFT at larger size

$$\log\%_{75.7} = \frac{\log 75.7 - \log 70}{\log 80 - \log 70} \times (\log 98.2 - \log 95.3) + \log 95.3$$

$$\log\%_{75.7} = 1.98677 \qquad \%_{75.7} = 97.0\% \tag{6-10}$$

$$\log\%_{63.6} = \frac{\log 63.6 - \log 60}{\log 70 - \log 60} \times (\log 95.3 - \log 81.8) + \log 81.8$$

$$\log\%_{63.6} = 1.93802 \qquad \%_{63.6} = 86.7\% \tag{6-11}$$

The complete set of calculation results are shown in Table 6.4.

Table 6.4 Original and New CPFT Distributions

Original Size (µm)	New Size (µm)	Original CPFT (%)	New CPFT (%)
90	90	100.	100
80		98.2	
	75.7		97.0
70		95.3	
	63.6		86.7
60		81.8	
	53.5		62.9
50		53.9	
	45.0		37.9
40		25.6	
	37.8		19.9
	31.8		9.2
30		7.1	
	26.8		4.1
	22.5		1.8
20		1.0	
	18.9		1.0

Example 6.3 Convert CPFT Trace to CPFT and Histogram Distributions Fitting a $^{4}\sqrt{2}$ Size Series

If you are handed a complete CPFT distribution trace from an automated sedimentation analysis instrument, calculate the CPFT and Histogram distributions to fit a $^{4}\sqrt{}^{2}$ size series.

This is almost an identical problem to Example 6.2, but rather than receiving the data in tabular form, the data is a CPFT distribution trace from an automated instrumental analysis. The data is shown in Figure 6-3.

Since this is a complete CPFT trace, the CPFT does not need to be recalculated. The size classes of interest must be marked on

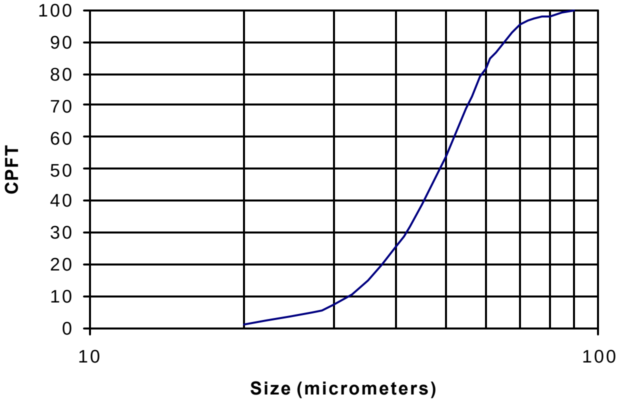


Figure 6-3 CPFT Trace

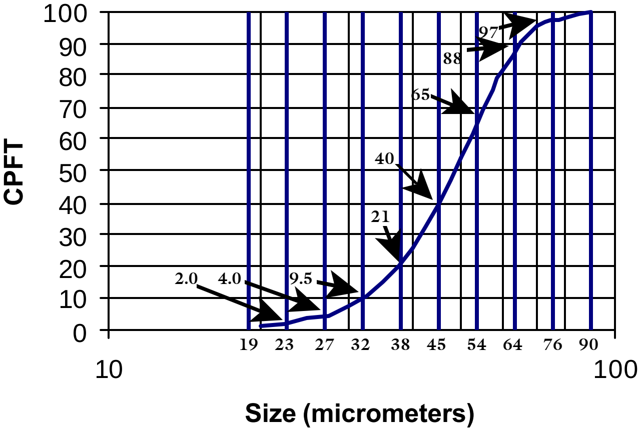


Figure 6-4 CPFT Trace Plus Size Classes

the chart. With the size classes in place, the CPFT values can be read at those sizes. From the newly tabulated CPFT values, the histogram can be calculated.

Figure 6-4 adds vertical lines at each size boundary to the CPFT plot of Figure 6-3. CPFT values, which can be read where the size classification lines cross the CPFT curve, are also shown in Figure 6-4. Histogram values can be calculated from the CPFT values. The new sizes, the CPFT values read from Figure 6-4, and the histogram values are shown in Table 6.5.

Table 6.5. Histogram and CPFT Data from Figure 6-4

Size (µm)	CPFT (%)	Histogram (%)
90	100	3
76	97	9
64	88	23
54	65	25
45	40	19
38	21	11.5
32	9.5	5.5
27	4	2
23	2	2
19	0	

The chart in Figure 6-4 is not particularly large, nor does it contain precise, fine grid lines like an actual CPFT trace from an instrument would contain. The CPFT values read from this chart are not very precise, but the procedures have been demonstrated.

Once the CPFT values are listed, as in Table 6.5, histogram values can be calculated.

Note that the histogram value for the size class between any two adjacent sizes in this table is listed with the larger size. This explains why there is a histogram value of 3% listed with the top size in the table. The largest size class, 90-76µm, contains 3% of the distribution. The second size class, 76-64µm, contains 9%. The smallest size class, 23-19µm, contains 2% of the distribution, which is listed on the 23µm line.

Example 6.4 Correction of Distributions with Missing Size Classes

A particle size analysis has been performed, but some of the sizes classes were missed. How does one correct the data? The data for this problem is shown in Table 6.6. Note that the 76 and 38µm size classes are missing.

Table 6.6 Histogram with missing size classes.

	Size (µm)	Histogram (%)
	90	15
76?—		
	64	18
	54	27
	45	30
38?—		
	32	5
	27	3
	23	2
	19	

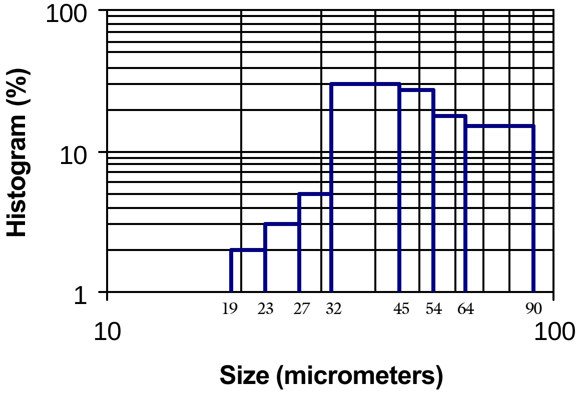


Figure 6-5 Histogram With Variable Size Classes

Figure 6-5 shows a plot of this data. Note that the 90-64μm and 45-32μm size classes are twice as wide as all other size classes, and they each contain more powder relative to the other size classes. The reason for this is that they each contain the amounts from two sieves, had this been a complete sieve series.

This is typical of histograms that contain variable size classes. The broader size classes contain more particles, the narrower classes contain fewer, and it's difficult to make comparisons between the distributions containing differently sized classes.

The general solution to this type of problem is to convert the histogram to a CPFT curve, interpolate the missing CPFT values, and convert back to a histogram. The CPFT values calculated for this distribution at 90, 64, 45, and 32μm are 100%, 85%, 40%, and 10%, respectively. The two missing sizes in this distribution are 76 and 38μm. The CPFT values for these two sizes can be calculated using Equations (6-8) & (6-9):

$$\log\%_{76} = \frac{\log 76 - \log 64}{\log 90 - \log 64} \times (\log 100 - \log 85) + \log 85$$

$$\log\%_{76} = 1.96500 \qquad \%_{76} = 92.3\% \tag{6-12}$$

$$\log\%_{38} = \frac{\log 38 - \log 32}{\log 45 - \log 32} \times (\log 40 - \log 10) + \log 10$$

$$\log\%_{38} = 1.30348 \qquad \%_{38} = 20.1\% \qquad (6\text{-}13)$$

With these two new CPFT values inserted into this particle size distribution, the new histogram appears as shown in Figure 6-6. The single class between 32 and 45μm in Figure 6-5 contains the greatest mass of particles of any class in the distribution. Splitting this one class into two shows that the 45 to 54μm class contains the greatest mass of particles of any in the distribution. Splitting the other larger size class in Figure 6-5 (64 to 90μm) also reduced the mass in each of the two new classes.

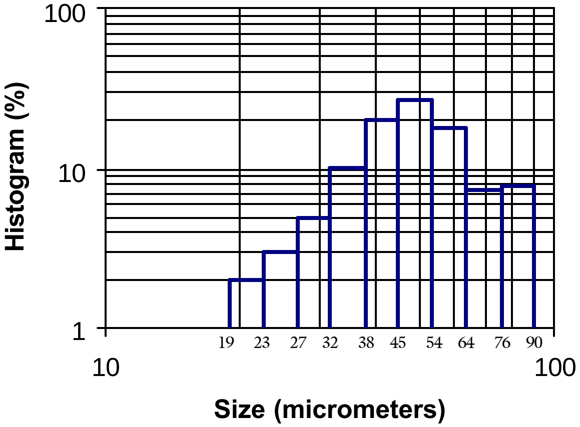


Figure 6-6 Corrected Histogram with Constant Size Classes

It is not recommended to calculate particle size distribution values, like was done in this example, on a routine basis. It is better to include all of the appropriate sieves, or to specify a complete $\sqrt[4]{2}$ or $\sqrt{2}$ size classification at analysis time, than to back-calculate after an oversight. But if necessary, this calculation can be performed to correct a particle size distribution measurement.

Chapter Seven

Calculated Surface Areas, Number of Particles, and Shape Factors

After the completion of particle size analyses, several other particle size distribution characteristics can be calculated. These include surface areas and numbers of particles within each size class as well as for the whole distribution. Shape factors can then be used to adjust the calculated surfaces areas to more appropriately apply to the actual, non-spherical particle shapes.

Shape Factors

The surface area contributed by each size class of particles as well as the volume specific surface area (VSA) can be calculated from a complete particle size distribution. One must make a shape assumption to perform these calculations. The most commonly assumed shape is the sphere. If particles are known to be non-spherical, or some other known shape, surface areas can be adjusted to represent those shapes.

Table 7.1 shows one form of shape factor that can be used to calculate surface areas for a variety of particle shapes.[2] The factors in this table are ratios of the surface area of a unit volume particle of the particular shape to the surface area of a unit volume sphere. For example, a cube that contains the same volume as a sphere will have 1.24 times the surface area of the sphere.

These shape factors are particularly applicable to particle calculations. Most analyzers, after measuring particle volumes or particle masses, express particle sizes as equivalent spherical diameters. Surface areas can then be calculated from the particle size analysis results using the spherical particle assumption and the measured equivalent spherical diameters. These factors can then be used to convert the results to apply to other shapes.

Table 7.1. Surface Area Shape Factors
(Aspect ratios L/t)

Sphere		1			
Tetrahedron		1.48			
Octahedron		1.37			
Square Disks	(31.62/1)	4.40	Hexagonal	(39.24/1)	4.38
	(9.72/1)	2.44	Disks	(13.88/1)	2.41
	(2.83/1)	1.41		(3.51/1)	1.37
[Cube]	(1/1)	1.24		(1.24/1)	1.18
	(1/2.83)	1.38		(1/2.28)	1.30
	(1/11.19)	1.93		(1/9.01)	1.80
	(1/31.65)	2.66		(1/25.51)	2.47

If all particles in the distribution are coarser than the particle size analyzer's lower size boundary, the measured specific surface area (SSA) can be used with the particle size analysis results to calculate the appropriate shape factor for the particles. If some particles in a distribution are smaller than a size analyzer's lower bound, shape factors cannot be calculated – although shape assumptions can be made.

Some companies make careful non-spherical shape assumptions on a mineral by mineral basis. Other companies simply ignore the problem and use spherical shape assumptions and measured equivalent spherical diameters. Each approach has advantages and disadvantages.

The recommendation is to be consistent in the application of whichever techniques are chosen for a particular lab. Consistency in sampling, sample preparation, analysis, and calculation techniques allow comparison of day-to-day results. Random, day-to-day variations in any of these techniques render data that is meaningless. Attempts to interpret meaningless data and apply the results to control processes will only produce problems.

Geometric Mean Size

When calculating surface areas and numbers of particles in a distribution, a mean particle size should be used to represent each size class. Each class in a $\sqrt[4]{2}$ or $\sqrt{2}$ size series covers a range of particle sizes. The use of a mean size allows a single size value to represent all particles in each class. The geometric mean size, which defines the center of each histogram bar when plotted on log axes, is recommended for this use:

$$\text{Geometric Mean Size} = (\text{Upper Size} \times \text{Lower Size})^{1/2} \qquad (7\text{-}1)$$

Example 7.1 Calculate the Geometric Mean Size to Represent a Single $\sqrt[4]{2}$ Size Class

A $\sqrt[4]{2}$ size class includes all particles between 45μm and 38μm. What is the geometric mean size of this class?

$$\text{Geometric Mean Size} = (45\mu\text{m} \times 38\mu\text{m})^{1/2} = 41.3\mu\text{m} \qquad (7\text{-}2)$$

Surface Areas and Number of Particles

With the results of a complete particle size analysis in hand, one can calculate the number of particles in each size class in a distribution using simple geometry and a particle shape assumption. The numbers of particles in each size class can then be used to calculate the surface areas contributed by each size class. The summation of the surface areas in each class produces the calculated surface area for the whole distribution.

The limitations of these calculations, of course, include the accuracy of any shape assumptions made, and the particle size analysis techniques' inability to "see," and measure, all particles present in each distribution.

It is the author's opinion that we need to make the simple shape assumptions and use *measured* particle size analyses results to calculate particle volumes, numbers, and surface areas. The details provided by such calculations can be very useful.

These calculation techniques, ***used with consistency*** within each lab, can provide lots of information about day-to-day variations in powder systems and suspensions. As analysis techniques improve and instruments are upgraded, the results of the measurements and calculations will become even more valuable to our understanding of, and ability to control, ceramic production processes.

Example 7.2 Calculate the Surface Area of a Spherical Particle

With a geometric mean size representing each size class, the surface area of a single particle of each size can be calculated. The most common particle shape assumption is a sphere. Using (10-1) and 41.3μm (representing the 45-38μm size class in Example 7.1), the surface area of a spherical particle is:

$$4\pi r^2 = \pi d^2 = \pi(41.3\mu m)^2 = 5{,}360\ \mu m^2 \qquad (7\text{-}3)$$

Example 7.3 Calculate the Number of Particles in a Size Class

If the 45-38μm particle size class contains 17.0% of a distribution, how many particles are in this size class?

To calculate this number of particles in a single class from its histogram value (that is, from the volume percentage of particles in this size class,) one needs to know the volume of a single particle.

If the particle size analysis results are for a single material with a constant density, volume percent and mass percent histogram and cumulative distribution results are equivalent. This is the case because volumes are proportional to masses for materials with constant density. Volume and mass percentages will be numerically identical because the proportionality will drop out as the percentages are calculated.

In such cases, calculations can be performed assuming a total powder mass of 1 gram, or a total powder volume of 1 cm^3, whichever is appropriate. Results can be scaled to the actual mass or to the actual volume of particles as the final step in the calculation.

The volume approach is appropriate to this problem. We know, therefore, that the whole distribution contains 1 true cm^3 of powder, and this size class contains $0.170cm^3$ of powder.

The number of particles represented in a volume of powder is simply the volume divided by the volume of a single particle. The volume of a single spherical particle, from Equation (10-2), is:

$$4/3\pi r^3 = \pi d^3/6 = \pi(41.3\mu m)^3/6 = 36{,}900\mu m^3 \qquad (7\text{-}4)$$

The number of particles in this size class can now be calculated:

$$\text{Number of Particles} = \frac{\text{Volume in Size Class}}{\text{Volume of One Particle}} \qquad (7\text{-}5)$$

$$= \frac{0.170\cancel{cm^3}}{36{,}900\cancel{\mu m^3}} \times \frac{10{,}000\cancel{\mu m^{\;3}}}{1\cancel{cm}} = 46.1 \times 10^6 \text{ particles} \qquad (7\text{-}6)$$

This result indicates that there are 46.1×10^6 particles in this size class in each 1 true cm^3 of the whole distribution. Note that the number of particles is enormous, even though the volume represented is relatively small.

If the raw material of interest contains 1 true m^3 of powder, the number of particles can be scaled to represent that amount:

$$\frac{46.1 \times 10^6 \text{ particles}}{1\,\cancel{\text{cm}^3}\text{ of powder}} \times \frac{10^6\,\cancel{\text{cm}^3}}{1\text{ m}^3} = \frac{46.1 \times 10^{12}\text{ particles}}{1\text{ m}^3\text{ of powder}} \tag{7-7}$$

Example 7.4 Calculate the Surface Area in One Size Class

This example will continue the problem begun in Example 7.3. Using the number of particles in the size class from (7-6), and the surface area of a single particle from (7-3), the surface area contributed by particles in this size class can be calculated.

$$SA_{\text{in Class}} = \text{Number of Particles}_{\text{in Class}} \times SA_{\text{per Particle}} \tag{7-8}$$

$$= 46.1 \times 10^6\,\cancel{\text{particles}} \times \frac{5360\ \mu\text{m}^2}{\cancel{\text{particle}}} \tag{7-9}$$

$$= 2.47 \times 10^{11}\,\cancel{\mu\text{m}^2} \times \left(\frac{1\text{ m}}{10^6\,\cancel{\mu\text{m}}}\right)^2 \tag{7-10}$$

$$= 0.247\text{ m}^2 \tag{7-11}$$

There are 0.247 m^2 of surface area contributed by the particles in this size class in each 1 true cm^3 of this powder.

Example 7.5 Calculation of a Shape Factor from SSA Data

A sieve analysis has been performed on a clay mineral (density = $2.60 g/cm^3$) and the 325×400 Mesh sample (45-38μm) contains 0.858g of material. A BET surface area measurement of this single size class indicates it has specific surface area, SSA = $0.1603 m^2/g$. What shape factor applies to this powder?

The volume of powder in this sample is:

$$\frac{0.858\cancel{g}}{\frac{2.60\cancel{g}}{\text{cm}^3}} = 0.330\ \text{cm}^3 \tag{7-12}$$

The number of particles in this sample, using the volume of each particle calculated in (7-4), is:

$$0.330\cancel{\text{cm}^3} \times \frac{1\ \text{particle}}{3.69\times10^{-8}\cancel{\text{cm}^3}} = 8.943\times10^6\ \text{particles} \tag{7-13}$$

The calculated surface area of this number of spherical particles, using (7-3), is:

$$8.943\times10^6\ \cancel{\text{particles}} \times \frac{5360\ \cancel{\mu\text{m}^2}}{\cancel{\text{particle}}} \times \left(\frac{1\ \text{m}}{10^6\ \cancel{\mu\text{m}}}\right)^2$$

$$= 0.0479\ \text{m}^3 \tag{7-14}$$

The measured surface area (using the SSA value) of this amount of powder is:

$$0.858\cancel{g} \times \frac{0.1603\text{m}^2}{\cancel{g}} = 0.1375\ \text{m}^2 \tag{7-15}$$

The calculated shape factor for this 325×400 Mesh material is:

$$\text{SF} = \frac{\text{Measured SA}}{\text{Calculated SA}} = \frac{0.1375\cancel{\text{m}^2}}{0.0479\cancel{\text{m}^2}} = 2.871 \tag{7-16}$$

It is not always possible to calculate shape factor because it is not always possible to completely measure all particle sizes in a powder. The particle sizes and surface areas of all powders in a sample must be measured before the shape factor can be calculated. Otherwise, only a best estimate (a guess?) is possible.

Before using the same calculated value for all size classes of a powder, consider these few points:

1) Powder shapes, porosities, and surface roughnesses may change as a function of particle size.

2) Internal porosities can contribute surface area to BET measurements. It is impossible to determine from BET measurements alone whether measured surface areas are internal pore areas, external surface areas, or both.

3) Large agglomerates formed from small particles have fundamentally different structures and densities than single particles of the same size. Surface areas of size classes containing agglomerates and single particles will not (cannot) be represented by a single shape factor.

This example has been shown to demonstrate that it is possible to calculate shape factors from laboratory measurements. The application of shape factors, however, cannot be treated lightly or whimsically. Much thought and consideration should go into any shape factor decisions.

Chapter Eight

Dense Packing

Many engineers are interested in particle size distributions that pack well (i.e., that pack densely). Improving the packing capability of the particle size distribution of a forming body or suspension can make the difference between achieving acceptable rheological properties at the desired solids content, or not being able to form ware at all.

When forming bodies and slips are held at constant solids contents within constant additive chemical environments, improvements to the packing capabilities of the particle size distributions translate into lower viscosities, better rheological properties, and fewer forming problems. Improved packing densities can also produce lower moisture contents in green bodies and lower drying and firing shrinkages.

Improvements to the packing capabilities of powders will only produce improved packing densities when bodies and slips are deflocculated. Reductions in viscosity and improved rheological properties will occur over the full range of flocculation/deflocculation states. But flocculated suspensions, in which particles are tied up by interparticle attractions and gelation, will not produce the expected packing density improvements. Rheological properties of gelled suspensions and high solids plastic forming bodies will be improved, but the packed structures formed from flocculated bodies and slips will be relatively open and highly porous.

Rationale

Improvements to the packing capabilities of particle size distributions bring improved rheological properties and decreased viscosities. The denser a system of particles can pack, the lower will be the porosity in a compact of those particles. When fluid is added to a system of particles to form a suspension, the carrier fluid will first fill any available pores; then it will begin to separate particles. With improved packing capabilities, less pore volume needs to be filled; less carrier fluid is tied up in those pores; and more carrier fluid is available to separate particles and reduce viscosities.

Even though forming bodies and slips do not contain solid particle compacts, the packing capabilities of the powders define the amount of fluid needed to fill pores and the amount remaining to separate particles. The powder packing capability therefore defines the ratio between pore water and dilution water in suspensions.

The packing capability of the particle size distribution is also one of the main parameters controlling the InterParticle Spacing (IPS) which is discussed in the next chapter. The IPS is the average distance between particles in suspension. Generally speaking, the greater the percentage of dilution water, the greater the IPS, and the lower the viscosity of the suspension or forming body.

Viscosities of particulate suspensions are greatly influenced by particle/particle interactions and collisions during flow. The greater the distance between particles (the greater the IPS), the fewer particle/particle interactions and collisions during flow, and the lower the viscosity of those suspensions.

Dense packing particle size distributions, relatively speaking, have minimum numbers of particles in each size class. This helps to minimize collisions, reduce viscosities, and improve rheologies of particulate suspensions and forming bodies. More complete discussions of these phenomena are included in our text[2].

Best Estimates

We cannot precisely calculate packing densities from first principles, but we have made simplifying assumptions which allow relatively simple algorithms to calculate these values. The author and his colleagues have found the resulting calculated packing densities (expected minimum porosities) to be extremely helpful to identify and produce dense packing particle size distributions.

Packing arrangements can be modelled in today's computers by recording the size, shape, position, and orientation of each particle in a compact. There are two fundamental problems to this approach: memory size and time. With ~10^{10} to ~10^{20} particles per true cm^3 of powder in many particulate systems, the enormous sizes of common computer memories are still not large enough to hold data for all particles in only one cm^3 of powder.

The time constraint applies to the number of trials it takes to place a particle in a compact. If particles are randomly positioned (packed) in a compact, how many random attempts need to be allowed to position each individual particle? Since two particles cannot occupy the same volume, each attempt to pack a particle in a compact requires interference tests between the particle to be packed and all particles already packed. The time it takes to pack a single particle, and check all possible interferences, rises exponentially as the number of successfully packed particles increases.

The more difficult question is: How many attempts to pack a particle need to be made before one can accurately conclude that the particle cannot fit into the compact? If truly random procedures are used, this number must be quite large.

Computer capabilities, memory sizes, and speeds continue to improve our ability to accurately model particle packing phenomena, but simplifying assumptions still need to be made to allow the calculations to be completed within reasonable times and within the capabilities of available computers.

Packing Density

Many process applications require the optimization of particle size distributions to achieve dense packing. The goal of some systems, such as in the field of refractories, is to achieve extremely high packing densities in the refractory compacts. Other goals include minimizing viscosities at the highest possible solids contents, improving rheological properties (that is, reducing dilatant effects), and successfully achieving near-net-shape forming processes.

In each of these cases, whether bodies will be extruded, plastically formed, slip cast, tape cast, or processed by some other method, the 'packing potential' of the solid particles is the main parameter of interest.

The algorithm presented in this section is described in detail in the *Predictive Process Control* textbook[2]. The presentation in the textbook covers the details of the derivation, development, and use of the algorithm.

The Equation for Densest Packing

The equation[4] that defines the particle size distribution for densest packing is:

$$\frac{\mathrm{CPFT}}{100\%} = \frac{D^n - D_S^n}{D_L^n - D_S^n} \qquad (8\text{-}1)$$

where D = particle size,
D_L = largest particle in the distribution,
D_S = smallest particle in the distribution, and
n = distribution modulus.

Densest packing occurs when the distribution modulus, n, equals 0.37. The following algorithm to calculate expected minimum porosities is based on this value for densest packing.

Procedure to Calculate Minimum Expected Porosity

The minimum expected porosity for a particle size distribution corresponds to the maximum packing factor for that distribution. The following equation set is used to calculate the minimum expected porosity:

$$
\begin{aligned}
V_{a1} &= a_1x_1 \\
V_{a2} &= x_1 + a_2x_2 \\
V_{a3} &= x_1 + x_2 + a_3x_3 \\
\cdot \quad & \quad \cdot \quad \cdot \quad \cdot \\
\cdot \quad & \quad \cdot \quad \cdot \quad \cdot \\
V_{ai} &= \sum_{j=1}^{i-1} x_j + a_ix_i \\
\cdot \quad & \quad \cdot \quad \cdot \quad \cdot \\
\cdot \quad & \quad \cdot \quad \cdot \quad \cdot \\
V_{an} &= \sum_{j=1}^{n-1} x_j + a_nx_n
\end{aligned}
\qquad (8\text{-}2)
$$

where a_i = apparent volume of a monodispersion of the i^{th} size class, defined by Equation (8-3),

x_i = volume fraction of the i^{th} size class particles,

V_{ai} = apparent volume calculated with reference to the i^{th} size class particles,

n = number of particle size classes in the distribution,

i = 1, corresponds to coarsest size class, and

i = n, corresponds to finest size class in the distribution.

Size classes used for this calculation *must* follow a complete size series as defined in Chapter 5. A complete fourth root of two ($\sqrt[4]{2}$) size series from D_L to D_S is recommended.

The x_i terms in (8-2) are the measured volume fractions of each size class in the distribution. For test purposes, particle size distributions defined according to (8-1) can be used, but for production bodies, particle size distributions must be measured using one of the available automatic particle size analyzers and/or sieve analyses.

The a_i terms in the equation set are the apparent volumes to which each size class in a distribution can pack. All a_i values in this algorithm are for continuous size distributions; they are all the same value; and they are all calculated using the following equation:

$$a_i = \frac{1}{1 - \left(\frac{1}{CSR}\right)^{0.37}} \qquad (8\text{-}3)$$

where CSR = Class Size Ratio = $\sqrt[4]{2}$

Note that the 0.37 exponent in the denominator of Equation (8-3) relates the a_i value to the ideal packing of an n=0.37 distribution.

When a fourth root of two Class Size Ratio is used, all a_i values in Equation Set (8-2) should be:

$$\text{For CSR} = \sqrt[4]{2}\,; \qquad a_i = 16.102 \qquad (8\text{-}4)$$

If a square root of two Class Size Ratio is used, all a_i values in Equation Set (8-2) should be:

$$\text{For CSR} = \sqrt{2}\,; \qquad a_i = 8.3090 \qquad (8\text{-}5)$$

For any other CSR, the a_i value can be calculated using (8-3). A complete size series (from the distribution's D_L to its D_S) of the CSR **must be** used to perform this calculation. If the size series is incomplete, calculated porosities will be inaccurate.

With the measured volume fractions for the particle size distribution and the a_i value in hand, the apparent volumes, V_a, in Equation Set (8-2) can be calculated. One apparent volume, V_a, must be calculated for each size class in the distribution. There will be one equation in Equation Set (8-2) for each size class in the distribution. If there are twenty size classes in the distribution, the full equation set will contain twenty equations for V_{a1} to V_{a20}, for coarse to fine particles, respectively.

The largest resulting V_a from the equation set is then used to calculate the expected minimum porosity for the particle size distribution:

$$\text{Minimum Expected Porosity (\%)} = 40\% \times \left(1 - \frac{1}{V_a}\right) \qquad (8\text{-}6)$$

The 40% constant in this equation corresponds to the expected minimum porosity of a monodispersion. Monodispersions will typically pack to include 40% porosity, that is, to 0.6 packing factor, PF. Broader size distributions are expected to pack more densely than monodispersions. This equation therefore produces values from 40% (poor packing) to 0% (perfect packing).

This calculation procedure is quite easy to implement in a spreadsheet, or in a BASIC or FORTRAN program.

Other Important Relationships

There are some other simple relationships between properties such as porosity and pore fraction, packing percentage and packing factor (PF), apparent volume (V_a) and packing factor, and pore fraction and packing factor. These relationships are reviewed here:

$$\text{Packing Factor} = \text{PF} = \text{Packing \%} / 100\% \qquad (8\text{-}7)$$

$$\text{Apparent Volume} = V_a = 1 / \text{PF} \qquad (8\text{-}8)$$

$$\text{Pore Fraction} = 1 - \text{PF} \qquad (8\text{-}9)$$

$$\text{Porosity \%} = \text{Pore Fraction} \times 100\% \qquad (8\text{-}10)$$

All of these parameters can apply to individual size classes of particles, or to complete particle size distributions.

Example 8.1 Calculate Packing Percentage, Apparent Volume, and Pore Fraction from Packing Factor

A powder sample can pack to a packing factor, PF = 0.6. Another way to express packing factor is as a percentage. From

Equation (8-7), a packing factor (PF) of 0.6 corresponds to a 60% packing percentage.

An apparent volume is a bulk volume. 0.6 PF corresponds to 1.67 bulk volume using Equation (8-8). Another way to say this is that one true volume of powder will occupy 1.67 volumes. To test this, note that 60% (that is, 0.6 PF) of 1.67 volumes = 1 volume.

The pore fraction is the difference between the packing factor and 1.0, so according to (8-9) if the packing factor is 0.6, the pore fraction (or pore factor) is 0.4.

The porosity, expressed in percent, is simply the pore fraction expressed as a percentage (8-10). So a pore fraction of 0.4 is equivalent to a porosity of 40%.

Example 8.2 Calculate the Minimum Expected Porosity for a Particle Size Distribution

Table 8.1 shows the particle size distribution in Figure 6-4. What is the minimum porosity that can be expected from a dense pack of this particle size distribution?

Since this table contains histogram data, and the distribution follows a complete fourth root of two size series, the apparent volumes from Equation Set (8-2) can be calculated. Remember that the x_i terms in the equation set are volume *fractions*, not percentages, so the histogram values in the table must all be divided by 100% before proceeding with the calculation.

Table 8.1. Histogram and CPFT Data from Figure 6-4

Size (µm)	CPFT (%)	Histogram (%)
90	100	3
76	97	9
64	88	23
54	65	25
45	40	19
38	21	11.5
32	9.5	5.5
27	4	2
23	2	2
19	0	

This being a $\sqrt[4]{2}$ size series, 16.102 will be used for all a_i terms in the equation set.

The calculations using Equation Set (8-2) follow. Note again that the calculation proceeds from coarsest (subscript 1) to finest (subscript 9) particles:

$$V_{a1} = a_1x_1 = 16.102 \times 0.030 = 0.483 \quad (8\text{-}11)$$

$$V_{a2} = x_1 + a_2x_2 = 0.030 + 16.102 \times 0.090 = 1.479 \quad (8\text{-}12)$$

$$\begin{aligned} V_{a3} &= x_1 + x_2 + a_3x_3 \\ &= 0.030 + 0.090 + 16.102 \times 0.230 = 3.823 \end{aligned} \quad (8\text{-}13)$$

$$\begin{aligned} V_{a4} &= x_1 + x_2 + x_3 + a_4x_4 \\ &= 0.030 + 0.090 + 0.230 + 16.102 \times 0.250 = 4.376 \end{aligned} \quad (8\text{-}14)$$

$$\begin{aligned} V_{a5} &= x_1 + x_2 + x_3 + x_4 + a_5x_5 \\ &= 0.030 + 0.090 + 0.230 + 0.250 + 16.102 \times 0.190 \\ &= 3.659 \end{aligned} \quad (8\text{-}15)$$

$$\begin{aligned} V_{a6} &= x_1 + x_2 + x_3 + x_4 + x_5 + a_6x_6 \\ &= 0.030+0.090+0.230+0.250+0.190+16.102\times0.115 \\ &= 2.642 \end{aligned} \quad (8\text{-}16)$$

$$\begin{aligned} V_{a7} &= x_1 + x_2 + x_3 + x_4 + x_5 + x_6 + a_7x_7 \\ &= 0.030+0.090+0.230+0.250+0.190+0.115 \\ &\quad +16.102\times0.055 = 1.791 \end{aligned} \quad (8\text{-}17)$$

$$\begin{aligned} V_{a8} &= x_1 + x_2 + x_3 + x_4 + x_5 + x_6 + x_7 + a_8x_8 \\ &= 0.030+0.090+0.230+0.250+0.190+0.115+0.055 \\ &\quad +16.102\times0.020 = 1.282 \end{aligned} \quad (8\text{-}18)$$

$$\begin{aligned} V_{a9} &= x_1 + x_2 + x_3 + x_4 + x_5 + x_6 + x_7 + x_8 + a_9x_9 \\ &= 0.030+0.090+0.230+0.250+0.190+0.115+0.055 \\ &\quad +0.020+16.102\times0.020 = 1.302 \end{aligned} \quad (8\text{-}19)$$

Apparent volumes (V_{a1} through V_{a9}) were calculated for each of the nine size classes, shown in Equations (8-11) through (8-19). The largest value, 4.376, from Equation (8-14) is used to calculate the minimum expected porosity for this distribution using (8-6):

$$\text{Minimum Expected Porosity (\%)} = 40\% \times \left(1 - \frac{1}{4.376}\right) = 30.86\% \qquad (8\text{-}20)$$

The calculated Minimum Expected Porosity value should be positive, between 0% and 40%. Depending on the value of apparent volume (V_a) used in this equation, the calculated percentage can be negative. Such values are ***meaningless***. Some negative percentages usually occur when a spreadsheet program is used to perform the calculation (the recommended method) and when a minimum porosity is calculated for each apparent volume from Equation Set (8-2). Although it requires another step (or three) to program into the spreadsheet calculation, the minimum porosity should only be calculated for the largest apparent volume.

The only meaningful minimum porosity corresponds to the largest calculated apparent volume. If the calculation was performed correctly, the minimum expected porosity will be a positive percentage. Minimum porosities corresponding to all other calculated apparent volumes (other than the maximum) should be ignored.

The minimum expected porosity in this example is 30.86%. This indicates poor packing. Also, the fact that the fourth size class, V_{a4}, produced the largest apparent volume indicates that the distribution's packing is defined by the quantity of particles in the fourth size class. Specifically, too many particles are contained in that size class. To improve the packing of this distribution, that is, to reduce the largest apparent volume, the amount of particles in the fourth size class should be reduced.

In any distribution, the size class that produces the largest apparent volume term is the particle size class in the distribution that defines the minimum expected porosity. If a particle size distribution packs well, the expected minimum porosity will be defined by the smallest size class in the distribution. When a coarser size class produces the largest apparent volume, that size class contains too many particles. It is advisable, therefore, to pay attention to the size class that produces the largest apparent volume term in the equation set. When the identified size class is not the smallest class in the distribution, it is possible to modify the distribution to improve packing.

Distributions that pack well should have minimum expected porosities below 10%. Dense-packing distributions created by the author and his colleagues during the last 20 years routinely had expected porosities in the 5-6% range using this calculation method.

Example 8.3 Densest Packing Distribution

What is the densest packing distribution for this range of particle sizes? The calculation in Example 8.1 was for a distribution with D_L = 90μm and D_s = 19μm. This is a narrow particle size distribution. To describe a distribution as *narrow* means that it doesn't include a broad range of particle sizes. A relatively large calculated minimum porosity of ~30% is an indication that the distribution is indeed narrow.

Some process bodies contain distributions that are narrow and it is almost impossible to broaden them to any useful degree. For example, any distributions with a largest particle size, D_L, near or smaller than 1μm will be narrow. There simply are not enough colloidal size classes available to sufficiently broaden such distributions to achieve dense packing.

If the stated D_L and D_s values for a distribution cannot be changed, what is the particle size distribution that defines densest packing for the available range of particle sizes? ... and what is its expected minimum porosity?

The starting point for any such experiment should be to use Equation (8-1) over the available particle size range with a distribution modulus, n, equal to 0.37. The resulting particle size distribution, following Equation (8-1) with D_L = 90μm, D_s = 19μm, and n = 0.37, is shown in Table 8-2.

Using the calculation procedure demonstrated in Example 8.2, the maximum apparent volume for this distribution is $Va_{max} = 2.28$, and the expected minimum porosity is 22.5%.

This relatively large value of minimum porosity indicates that this distribution will not pack well either. The reason, as mentioned, is that this distribution is still very narrow, only covering a range of particle sizes from 90 to 19μm. This does not even represent one full decade of particle sizes. One full decade of particle sizes would be represented by size classes from 90 to 9μm.

Table 8.2. Histogram and CPFT Data for a Dense-Packing Distribution

Size (μm)	CPFT (%)	Histogram (%)
90.00	100.0	14.2
75.68	85.8	13.3
63.64	72.6	12.5
53.51	60.1	11.7
45.00	48.4	11.0
37.84	37.4	10.3
31.82	27.2	9.64
26.76	17.5	9.04
22.50	8.5	8.48
18.92	0.0	

Monodispersions, and narrow particle size distributions in general, do not pack well. To achieve dense packing, particle size distributions should contain particles covering several decades of sizes. When the largest size in a distribution (the D_L) is 90μm, packing would be much improved if the smallest particles were in the colloidal range, 0.19μm, for example. Such a distribution is actually more typical of a real distribution than one including only particles between 90 and 19μm.

But if the size constraints are 90 to 19μm, as in this example, Table 8.2 shows the densest packing distribution using Equation (8-1).

For comparison purposes, consider a particle size distribution that does cover several decades of particle sizes, extending from D_L = 90μm to D_s = 0.176μm (0.176μm is the nearest fourth root of two size class to 0.19μm), with a distribution modulus, n = 0.37. The calculated maximum apparent volume for this distribution is Va_{max} = 1.11, and the corresponding expected minimum porosity is 3.98%. This distribution would pack really well.

If system requirements dictate that the 90 to 19μm size limitations must stand, then the two histograms (from Tables 8.1 and 8.2) should be plotted together to show the changes necessary to improve particle packing of the distribution in Table 8.1.

Example 8.4 Comparing Packing Capabilities of Distributions

How does one compare the packing capabilities of two different particle size distributions?

To compare the packing capabilities of two particle size distributions, one can perform the calculations, or one can go into the lab to compare properties of actual distribution.

For example, if the distribution in Example 8.2 was modified to include fewer particles in the fourth size class (which was suggested to improve the packing of that distribution), how should one compare the new distribution to the original?

One way would be to measure the new particle size distribution and calculate its minimum expected porosity. The two minimum porosity percentages could then be compared. The smaller expected minimum porosity would indicate the better packing distribution.

To compare the two distributions in the lab, tap densities of the dry powders or viscosities of slurried powders could be compared.

The viscosity method requires that the two distributions be slurried, and that their additive chemistries and solids contents be adjusted to be identical. Then, the slurry with the lower viscosity will indicate the particle size distribution with the denser packing capability.

When tap densities are performed, one would expect that the distribution with the denser tap density should be the distribution with the denser packing capability. Meaningful tap densities, however, are difficult to achieve. Tap densities should only be used on systems of relatively coarse particles, or on systems in which the ware are formed in molds using a process similar to the tap density process.

As powder particle sizes decrease, surface friction forces have more and more influence on tap density results. Tap densities are also subject to the type, intensity, and duration of the vibrations used. Tap density results frequently correlate well with measured surface areas, but poorly with predicted packing densities.

Another possible method to compare packing densities would be to pack suspensions of particles using a piston press with a filter on the bottom to allow the fluid to flow out of the compact. At least two problems exist with this test method: (1) such experimental presses are not very common, and (2) pressing must stop before stresses are reached where particles begin to break.

The simplest method which can easily be performed in most laboratories is the slurry viscosity comparison. The author suggests this comparison method be used whenever possible.

Chapter Nine

InterParticle Spacing

The InterParticle Spacing (IPS) calculation provides an estimate of the average distance between particles in suspension. Its calculation procedure uses two measured analyses and a control parameter: the measured volume specific surface area, the measured particle size distribution, and the solids content of the suspension. The equation[8] for IPS is:

$$\mathrm{IPS} = \frac{2}{\mathrm{VSA}}\left[\frac{1}{\mathrm{Vs}} - \frac{1}{(1 - \text{Pore Fraction})}\right] \qquad (9\text{-}1)$$

where VSA = volume specific surface area,

Vs = volume fraction solids in the suspension, and

Pore Fraction = pore fraction expected in a dense pack of powder.

The fundamental assumption of the IPS calculation is that the function of all non-pore water is to separate particles[8]. The expected minimum porosity calculation (Example 8.2) is used to determine the fraction of water in suspension that is tied up in the pores. The remaining water is assumed to be spread evenly over all particle surfaces where its function is to separate particles.

Example 9.1 Calculate an IPS

The particle size distribution in Example 8.2 and Table 8.1 is that of a quartz sample (density = 2.60 g/cm^3) with measured SSA

= 1.542m^2/g. If this sample is slurried at 60vol% solids, what will be its IPS? The only other parameter needed is its minimum expected porosity, which was calculated in Equation (8-20): 30.86%.

Using (9-1), the IPS is:

$$\mathrm{IPS} = \frac{2}{1.542\,\frac{\mathrm{m}^2}{\cancel{\mathrm{g}}} \times 2.60\,\frac{\cancel{\mathrm{g}}}{\mathrm{cm}^3}} \left[\, \frac{1}{0.6} - \frac{1}{(1 - 0.3086)} \,\right]$$

$$= \frac{2}{4.009\,\frac{\cancel{\mathrm{m}^2}}{\cancel{\mathrm{cm}^3}} \times \frac{\cancel{10^4}\mathrm{cm}^2}{\cancel{\mathrm{m}^2}}} \,(\, 1.667 - 1.446 \,)$$

$$= 0.4989 \times 10^{-4}\,\mathrm{cm}\ (\, 0.221)$$

$$= 0.110 \times 10^{-4}\,\cancel{\mathrm{cm}} \times \frac{10^4\,\mu\mathrm{m}}{1\,\cancel{\mathrm{cm}}} = 0.110\,\mu\mathrm{m} \qquad (9\text{-}2)$$

The IPS value is a useful process parameter because it is derived from two laboratory measurements and one process control parameter: the measured SSA, the measured particle size distribution, and the solids content. As any (or all) of these three change, the IPS changes, the interParticle fluid environment changes, and the viscous, rheological, and forming properties of each suspension change.

Example 9.2 A Negative IPS???

What does it mean when a calculated IPS is negative? A negative calculated IPS means that there is not enough fluid in the sample to fill all pores. In other words, the desired solids content for the suspension requires a denser packing capability than is possible with the available particle size distribution.

In experimental samples, IPS will never be negative, but zero. Particles will be touching; interParticle pores will not be filled with fluid; and viscosities, if they can be measured at all, will correspond to systems of wet particles, rather than to suspensions.

Using the system in Example 9.1 again, assume it is to be slurried, not at 60vol% solids as in that example, but at 70vol% solids. The calculated minimum expected porosity for that sample is 30.86%. This system will require at least 30.86vol% of fluid just to fill the pores, and it will need additional fluid to separate particles and impart fluidity to the suspension. If only 30vol% fluid is added to this particle size distribution, as required by a 70vol% solids system, the pores will not be filled.

The IPS calculation, again using (9-1), will be:

$$\text{IPS} = \frac{2}{1.542\,\frac{\text{m}^2}{\cancel{\text{g}}} \times 2.60\,\frac{\cancel{\text{g}}}{\text{cm}^3}} \left[\frac{1}{0.7} - \frac{1}{(1-0.3086)} \right]$$

$$= \frac{2}{4.009\,\frac{\cancel{\text{m}^2}}{\text{cm}^{\cancel{3}}} \times \frac{10^4\,\cancel{\text{cm}^2}}{\cancel{\text{m}^2}}} \left[1.429 - 1.446 \right]$$

$$= 0.4999 \times 10^{-4}\,\text{cm}\,[\,-1.137]$$

$$= -0.5688 \times 10^{-4}\,\cancel{\text{cm}} \times \frac{10^4\,\mu\text{m}}{1\,\cancel{\text{cm}}} = -0.5688\,\mu\text{m} \qquad (9\text{-}3)$$

As soon as the rightmost term in the square brackets appears as a negative number, the calculation procedure can stop. That could have occurred in the third line of the calculation when the –1.137 value appeared. Particles in process suspensions cannot have negative spacings, so there's no sense proceeding with the calculation. If this calculation were performed in a spreadsheet, however, the calculation would be completed and the results cell would show – 0.5688µm as the IPS value.

The negative result indicates insufficient liquid in the sample. How can this be fixed? Either the moisture content of the body can be increased (and the solids content lowered) or the particle size distribution can be modified to improve packing and produce a smaller minimum expected porosity.

Even though the negative distance value is meaningless, the fact that the value is negative is extremely useful, because it is an early indication that the desired properties for the process body or test suspension cannot be met as specified.

The practical solution to this problem, when it occurs, is to mill some of the particle size distribution and to mix some of that new distribution with the original. When proper proportions are combined, it is usually possible to successfully suspend the body at the desired solids content.

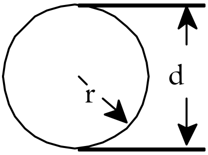
To determine the proper proportions to be combined, the packing potential of the particle size distribution must be improved. This requires using the packing algorithms demonstrated in Chapter 8.

Chapter Ten

Particle Formulas

Spheres

For a sphere with radius, r, and diameter, d, its surface area and volume formulas are:



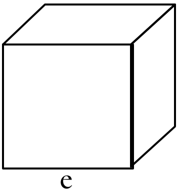
Surface area: $= 4 \pi r^2 = \pi d^2$ (10-1)

Volume: $= 4/3 \pi r^3 = (\pi d^3)/6$ (10-2)

Equation (Cartesian coordinates): $x^2 + y^2 + z^2 = r^2$ (10-3)

Cubes

For a cube with edge length, e, its surface area and volume formulas are:

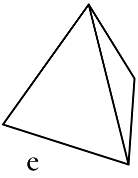


Surface area:
One face: $= e^2$ (10-4)
Total (six faces): $= 6e^2$ (10-5)

Volume: $= e^3$ (10-6)

Tetrahedra

A tetrahedron is a polyhedron with four faces, each of which is an equilateral triangle. Tetrahedra are related to cubes: the six edges of a tetrahedron correspond to six face diagonals of a cube. The surface area and volume formulas of a tetrahedron, with edge length, e, are:



Surface area:

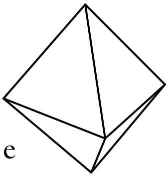
One face: $= (\sqrt{3}/4)e^2$ (10-7)

Total, four faces: $= \sqrt{3}\ e^2 = 1.732051\ e^2$ (10-8)

Volume: $= (\sqrt{2} / 12)\ e^3 = 0.1178511\ e^3$ (10-9)

Octahedra

An octahedron is a polyhedron with eight faces, each of which is an equilateral triangle. Octahedra are also related to cubes: the six vertices of an octahedron sit at the face centers of a cube's surfaces. The surface area and volume formulas of an octahedron, with edge length, e, are:



Surface area:

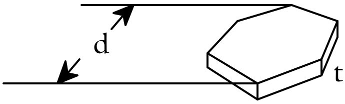
One face: $=(\sqrt{3}/4)e^2$ (10-10)

Total, eight faces: $= 2\sqrt{3}\ e^2 = 3.464102\ e^2$ (10-11)

Volume: $=(\sqrt{2}/3)e^3 = 0.4714045\ e^3$ (10-12)

Hexagonal Plates

Some particles, such as kaolinite, are known to be hexagonal plates. If the vertex to vertex distance across a plate is d and the thickness of the plate is t, the surface area and volume relationships are as follows:



Surface area:

Upper surface: $= (3/8)\sqrt{3}\ d^2 = 0.6495191\ d^2$ (10-13)

Lower surface: $= (3/8)\sqrt{3}\ d^2 = 0.6495191\ d^2$ (10-14)

Edge surface: $= 6(d/2)t = 3dt$ (10-15)

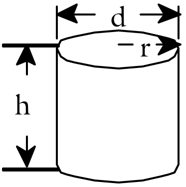
Total: $= 1.2990381\ d^2 + 3dt$ (10-16)

Volume: $= (3/8)\sqrt{3}\ d^2t = 0.6495191\ d^2t$ (10-17)

Although these hexagonal solids were defined as "plates", the aspect ratio, that is the ratio of d to t, can define these particles to be thin hexagonal plates, or at the other extreme, long hexagonal fibers.

Cylinders

Similar to the hexagonal plates, the aspect ratio of cylindrical diameter, d, to height, h, can define cylinders to be thin plates or long fibers. The surface area and volume formulas are:



Surface area:

Upper surface: $= \pi r^2 = (\pi/4)d^2 = 0.7853982\ d^2$ (10-18)

Lower surface: $= \pi r^2 = (\pi/4)d^2 = 0.7853982\ d^2$ (10-19)

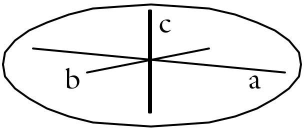
Edge surface: $= 2\pi rh = \pi dh = 3.1415927\ dh$ (10-20)

Total: $= 1.5707963\ d^2 + 3.1415927\ dh$ (10-21)

Volume: $= \pi r^2h = (\pi/4)d^2h$

$= 0.7853982d^2h$ (10-22)

Ellipsoids



The simplest shape typically used for computer modelling of particle packing phenomena is the sphere. A variation on the sphere, which allows the modelling of more realistic particle shapes is the ellipsoid. At one extreme, where the a and b major axes are large and the c minor axis is small, the ellipsoids represent flat plates. At the other extreme, where the a and b minor axes are small and the c major axis is large, ellipsoids can represent long fibers. All of these variations apply to ellipsoids defined by a single equation:

Equation (Cartesian coordinates): $$\frac{x^2}{a^2} + \frac{y^2}{b^2} + \frac{z^2}{c^2} = 1 \qquad (10\text{-}23)$$

In this formula, a, b, and c define the major and minor axes in the x, y, and z directions, respectively.

References

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Particle Calculations for Ceramists

This book was written to aid all ceramists to perform fundamental particle calculations. Discussions and sample calculations cover, for example, particle surface areas and volumes, slip densities and solids contents, particle size distributions, bulk volume and density, comparing and combining particle size distributions, and particle packing. The various particle size distribution presentation forms, i.e., linear, semi-log, and log-log charts, as well as histograms and cumulative distribution plots, are presented and discussed.

Brief explanations accompanying each topic precede the many sample calculations. This book should be a handy reference for ceramic technicians, engineers, scientists, artists, and anyone else involved in the exciting field of fine particle processing and control.

The Author ...

Dennis Dinger is a private consultant in the field of ceramic processing and control, and Professor Emeritus of Ceramic and Materials Engineering at Clemson University. Over the past two decades, he has directed many applied ceramic engineering research projects studying the effects of powder characteristics on the behaviors of production slips and bodies. He is the author of many computer programs and models on particle packing phenomena, and a co-author, with the late Professor James E. Funk, of the ceramic processing textbook *Predictive Process Control of Crowded Particulate Suspensions Applied to Ceramic Manufacturing*.