CERAMIC AND COLLOIDAL PROCESSING - EXERCISES

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Exercises 2

1. What are the physical characteristics that can be used to describe a powder?

Physical properties:

- Size and size distribution
- Morphology and form factor
- Specific surface area
- Density (Absolute, Apparent)
- Porosity
- Purity

There are also other characteristics that can be used to describe a powder: chemical composition, crystalline phase, homogeneity.

2. What are the 2 parameters that describe a distribution (of size) and what is the difference between the mode, the mean and the median of a distribution?

A central tendency (mean, mode or median) and the width of the distribution (standard deviation, span)

3. Calculate the diameters d_{ns}, d_{nv}, d_{sv} and d_{vm} for the distribution below, using a total number of particles 10^6. Distribution by number

Cumulative Diameter (microns)	Cumulative % (less than)	Frequency Diameter (microns)	%Frequency (in each class)
4.0	100.0	3.75	0.4
3.5	99.6	3.0	4.1
2.5	95.5	2.175	10.3
1.85	85.2	1.675	13.6
1.5	71.6	1.35	17.3
1.2	54.3	1.125	9.2
1.05	45.1	0.975	12.3
0.9	32.8	0.825	12.4
0.75	20.4	0.675	9.0
0.6	11.4	0.55	5.1
0.5	6.3	0.425	3.9
0.35	2.4	0.175	2.4

Median diameter dn50 = 1.12 µm, σn50 = 0.55 µm

By considering the diameters (d_i) from the frequency distribution, we can calculate each of the different summations over all the size classes needed to calculate the various diameters.

$\sum_{i=1}^{n} N_i$	$\sum_{i=1}^{n} d_i N_i$	$\sum_{i=1}^{n} d_i^2 N_i$	$\sum_{i=1}^{n} d_i^3 N_i$	$\sum_{i=1}^{n} d_i^4 N_i$
	[µm]	[µm^2]	[µm^3]	[µm^4]
1000000	1258675	1991337	3796393	8402790

With these values we can then calculate the following diameters

$d_{ns} = \sqrt{\frac{\sum_{i=1}^{n} d_i^2 N_i}{\sum_{i=1}^{n} N_i}} = 1.411 \mu m$	$d_{vs} = \frac{\sum_{i=1}^{n} d_i^3 N_i}{\sum_{i=1}^{n} d_i^2 N_i} = 1.906 \mu m$
$d_{nv} = \sqrt[3]{\frac{\sum_{i=1}^{n} d_i^3 N_i}{\sum_{i=1}^{n} N_i}} = 1.560 \mu m$	$d_{vm} = d_{4,3} = \frac{\sum_{i=1}^{n} d_i^4 N_i}{\sum_{i=1}^{n} d_i^3 N_i} = 2.213 \mu m$
$d_{sl} = \frac{\sum_{i=1}^{n} d_i^2 N_i}{\sum_{i=1}^{n} d_i N_i} = 1.582 \mu m$	

4. How do you measure a size distribution of a powder,

i) From 25 to 1000 µm ii) <10 µm iii) < 300 nm

What types of diameters are measured by these methods, briefly describe the method and what are the limitations of these methods?

i) Powder size from 25µm to 1000µm:
Method: laser diffraction: the diameter corresponding to the volume of the particle is measured. A laser passes through a powder suspension and diffracts light producing a diffraction pattern. The diffraction pattern is a convolution of all particle sizes. An optical model uses the refractive index of the powder and dispersing liquid to compute a diffractogram for an arbitrary size distribution. The difference between the measured and calculated diffraction pattern is minimised to give the resulting particle size distribution
Limitations:

- broadened distributions for non-spherical particles,
- if the particles are elongated poor results as the particle shape is assumed to be spherical
- when the particles are finer than 1µm, Mie's theory must be applied, this is also true if the difference between the refractive index of the powder and the liquid is small (e.g. polymer particles in water)- …

ii) Powder size <10µm

Method: Sedimentation-centrifugation using powder suspension e.g. centrifugal particle size analyser (CPS).

The measured diameter is the Stokes diameter and projected area (CPS) to get the concentration of particles in each time class (=size class).

Limitations:

CPS

- have to use a standard powder to calibrate size (but it is reliable)
- spherical particle shape assumption

iii) Powder size < 300 nm

Method: Dynamic Light Scattering (DLS, PCS) – the measured diameter is a hydrodynamic diameter. Laser interaction with particles in suspension. The scattered light intensity as a function of angle depends on both the relative refractive index, the wavelength of light and the particle size. The intensity as a function of time at a given angle is related by an autocorrelation function to the diffusion coefficient from random thermal fluctuations (Brownian movement).

Limitations:

- Broad size distributions tend to bias the larger size fraction because scattered light intensity is proportional to r^6
- Laplace transform of ACF is ill defined - images of particles needed for best interpretation when distribution is broad or multi-modal.
- Difficult to measure particle > 500 nm because of very slow movement of large particles which make collection of a good quality ACF difficult.

5. How can we find an agglomeration factor, FAG, less than 1.

Agglomeration factor : $F_{AG} = \frac{d_{V50}}{d_{BET}}$

$F_{AG} < 1 \Leftrightarrow d_{V50} < d_{BET}$ with $d_{BET} = \frac{6}{S_{BET}\rho}$

If we decrease the density of the powder without changing its specific surface area (e.g. closed pores within the particle), the d_{BET} diameter will increase while the median diameter in volume will remain the same (since S_{BET} will not change => V will not change)

We may therefore have $d_{BET} > d_{V50}$ => FAG <1 → closed porosity.