

CERAMIC AND COLLOIDAL PROCESSING - EXERCISES

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Exercises 6 - Solutions

1. What are the main methods of classifying a powder?

What are their advantages and disadvantages (limitations)?

How to classify a powder with sizes between

5000 to 50 μm

50 to 5 μm

<5 μm

<100nm

The two main methods of classification are sieving and air classification. Screening or sieving is a popular, 'inexpensive' and easy to design method. Indeed, the system consists of generally square meshes. On the other hand, such square meshes have the disadvantage of often underestimating the size of the particles, as the size measurement methods assume that the particles are spherical, elongated particles give an equivalent spherical diameter larger than the sieve opening. Another disadvantage is that the agglomeration of particles can distort the results especially for sieve openings smaller than 44 μm (ASTM standard), when the powders are otherwise characterised in suspension (e.g. via laser diffraction). In this case, it is necessary to add lubricants to the powder mixture or to do a wet sieving.

The air classifier has the advantage of classifying a large amount of powders in a short time. On the other hand, the apparatus is more expensive than sieving and consumes a significant amount of energy by supplying pressurized air.

Classification: size limits

- Powders from 5000 to 50 μm : the most suitable method is sieving
- Powders from 50 to 5 μm : Air classifiers also cover this size range (1000 to 0.1 μm) and in case the powders can be suspended in a liquid then, sieving is also suitable.
- <5 μm : it is necessary to use air classifiers as the wet sieving limit is around 5 μm :
- <100 nm: in this case centrifuges are used, and even ultracentrifuges to obtain a resolution of a few nm.

2. What type of machine would you use to reduce the size of particle to the following dimensions:

- to mm

- alumina <100 μm

- alumina <1 μm

- to mm: this type of coarse particle can be obtained by simple crushing (rotary, jaw, roller or even hammer mills)
- Alumina <100 μm : a rotary ball mill is sufficient.
- Alumina <1 μm : for fine milling, it is necessary to use other types of mills such as vibratory mills, stirred or agitated ball mills (e.g. attritors) or even the fluid impact mill, depending on the structure of the powders (defects, grain size, porosity, state of agglomeration).

3. What are the differences between wet milling and dry milling?

Wet milling uses particles in suspension in the milling chamber (usually the liquid used is water, for economic and ecological reasons, but it depends on the nature of the powder to be milled). Compared to the dry method, wet milling consumes less energy but causes much faster wear of the milling balls and can work with smaller sizes. Wet milling uses less energy to achieve the same size reduction, but if you have to dry for transport or application it can become more expensive. Handling and pumping of slurries of fine particles is much easier than in the dry state and avoids dust formation which, depending on the material in question, can be to some degree a health hazard. If the forming or shaping process is a wet process, e.g. slip casting, then wet milling is advantageous.

Dry milling is generally used when materials react with water other fluids are too expensive, or the material must be used in a dry state and energy used to dry the ground slurry adds excessive expense to the overall process.

4. Could you describe a milling circuit?

First, the raw material is loaded into the milling chamber (and the liquid in the case of wet milling). Milling can then begin. A classification step is generally present in order to remove the particles having reached the desired size. The other particles return to the milling chamber. Finally, when necessary, a final separation step makes it possible to remove the carrier liquid or carrier fluid (gas), e.g. spray drying.

5. Table 1 below shows the results of pure sand size reduction by a ball mill:

x (μm)	R (1h) %	R (5 h)	R (10 h)	R (15 h)	R (20 h)
2	97.5	92	81	75	78
3	96.5	88	75	68	75
4	95.5	83	72	61	73
5	95	81	69	55	70
10	92	70	44	35	60
20	88	55	18	27	50
30	81	45	7	10	40
40	77	30	3	0	0
50	72	20	0	0	0
100	60	1	0	0	0
200	20	0	0	0	0

R(h) = cumulative weight of powder retained on the sieves (greater than cumulative distribution, e.g. Rosin-Rammler type distribution) for each of the of the sieves (h = hours of milling)

- How long does it take to reach the milling limit?

For times between 10 and 15hrs, we observe that the size retained increases for sieves $>20\mu\text{m}$ and then between 15 and 20h for all sizes. So the milling limit is reached around 15hrs.

- Why is there a milling limit?

There is an intrinsic size limit for each material (perfect single crystal) below where it is no longer possible to accumulate enough elastic energy in a particle to break crystal bonds and it

can only deform plastically. At this point the energy of powder-bead collisions has a tendency to form agglomerates between the particles. If the cohesive strength is sufficient.

- How can we modify the milling limit?

We can use additives that prevent the agglomeration to help refine the milling. A lubricant such as stearic acid is often used. Also, it might be possible to change the size of the milling balls, or even the type of mill e.g. attrition or wet milling using a dispersant like polyacrylic acid, to limit agglomeration.