

Particle Size Analysis of Calcium Carbonates by Laser Diffraction

CALCIUM CARBONATES FULFILL A VARIETY OF DIFFERENT FUNCTIONS IN A WIDE RANGE OF INDUSTRIES:

- **Adhesives and sealants:** as fillers and viscosity controller.
- **Animal and pet feeds:** as a source of calcium and a digestion aid.
- **Construction:** as a filler for concrete, grout, asphalt, and roofing materials.
- **Fertilizers:** as a filler and a source of calcium.
- **Foods:** as an additional source of calcium.
- **Household and hygiene products:** as a mild abrasive.
- **Mining:** as a bridging component in drilling muds.
- **Paints and surface coatings:** as a pigment, filler, extender and finishing aid.
- **Paper:** as a filler, coating pigment, and acidity controller.
- **Pharmaceuticals:** as a filler or bulking agent.
- **Plastics:** as a filler and rheology controller.
- **Rubber and elastomers:** as a filler and extender.

MEASURING THE PARTICLE SIZE OF CALCIUM CARBONATES

To provide this range of functionality, calcium carbonates are available in a wide range of different particle sizes, and with different surface coatings. The Toppers laser diffraction system can measure particle size from nanometers to millimeters, with measurements over the range being achieved in only a few minutes. This range and measurement speed enables the particle size of calcium carbonates to be tracked during production processes as well as in quality control of a final product.



HOW TO DISPERSE CALCIUM CARBONATES

The Topsizer has a range of accessories that enable measurements using liquid or dry dispersion. The processing method and particle size of the calcium carbonate will determine whether liquid or dry dispersion is preferable. Dry dispersion allows larger amounts of sample to be measured, providing reproducible results for coarser particle sizes. However, for the finest grades, liquid dispersion offers more control over the state of dispersion, and consequently produces more reproducible results. The flexibility to quickly change between liquid and dry dispersion units means that a wide range of samples types can be analyzed using a single Topsizer installation.

The DPF-110 dry powder disperser uses compressed air to disperse the sample, and the appropriate air pressure must be selected to ensure dispersion. In liquid dispersion, using the SCF-108A dispersion units, calcium carbonates are usually measured in water. A short duration of ultrasound is often required to disperse agglomerates, and additives, such as sodium hexametaphosphate or sodium pyrophosphate, are used to stabilize the dispersion.

Figure 1 shows the particle size distributions for a calcium carbonate sample measured using both liquid (after 30 seconds of ultrasound) and dry dispersion (at 2bar). These results show good agreement between liquid and dry dispersion which verifies that the sample is fully dispersed in both states.

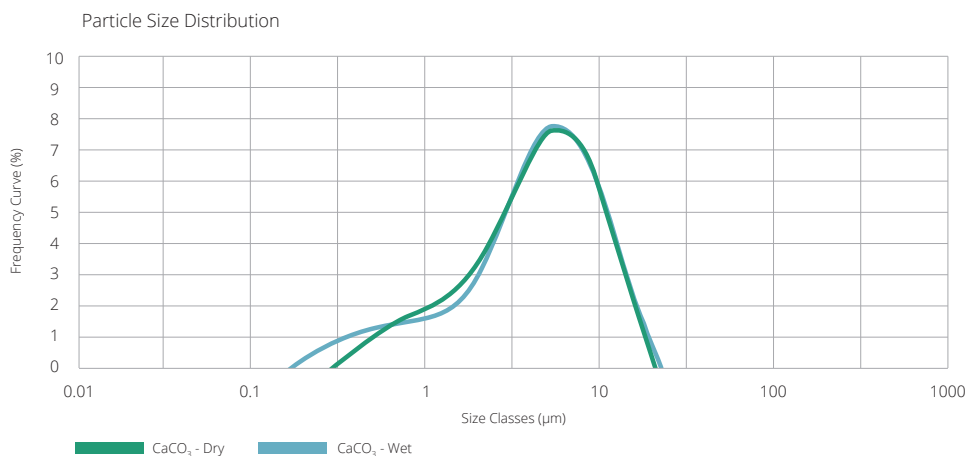


FIGURE 1: Overlay of a calcium carbonate sample measured in liquid (wet) and dry dispersion

SPECIFYING THE PARTICLE SIZE OF CALCIUM CARBONATES

Laser diffraction produces volume-based particle size distributions, from which a range of parameters can be specified. For example, the 97th percentile (D97) can be used to track the coarse particles in the distribution. Calcium carbonates are also commonly specified on the percentage material in certain size ranges, for example the percentage below 2µm for the finer grades and the

percentage above 10µm or 45µm at the coarser end of the range. Figure 2 shows the particle size distributions for four typical calcium carbonates covering Nano-calcium carbonates, precipitated calcium carbonates (PCC) and ground calcium carbonates (GCC) with a size range from 40nm up to 200µm, and Table 1 shows the parameters for these samples.

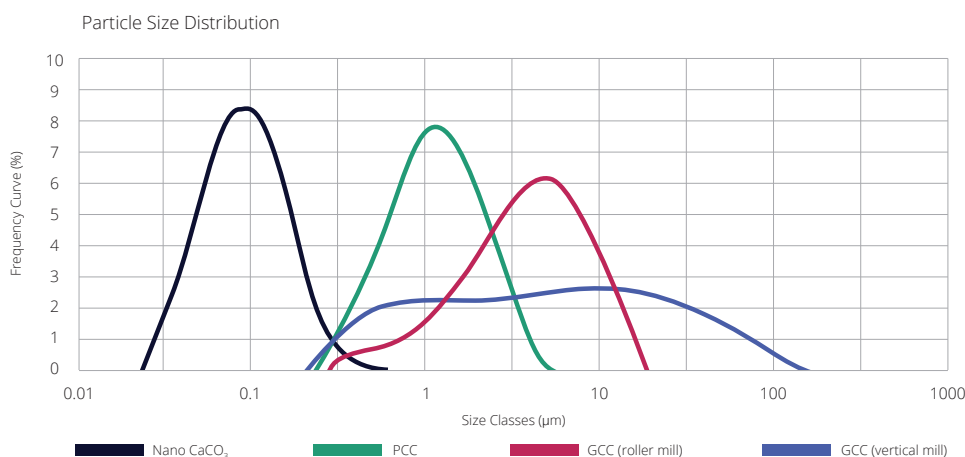


FIGURE 2: Particle size distributions for four types of calcium carbonate

Sample	D10 (µm)	510 (µm)	D97 (µm)	%V <2µm	%V >10µ	%V >45µ
Nano CaCO ₃	0.066	0.130	0.350	100.0	0	0
PCC	0.673	1.440	3.960	71.25	0	0
GCC (roller mill)	1.353	4.638	14.38	17.04	12.95	0
GCC (vertical mill)	0.763	6.012	83.14	28.46	38.60	10.29

TABLE 1:
Particle size parameters for four types of calcium carbonate

REPEATABILITY AND REPRODUCIBILITY

The ground calcium carbonate (GCC) produced by vertical mill has been a challenge for many laser diffraction systems due to very wide particle size distribution. With an optimised product design, the TopSizer can offer customers highly accurate, repeatable and reproducible data. Figure 3 and Table 2 show repeatable results of three aliquots of the same GCC (vertical mill) sample, measured by the same TopSizer instrument. Figure 4 and Table 3 then reveal reproducible particle size distribution of the same batch tested by three different TopSizer instruments.

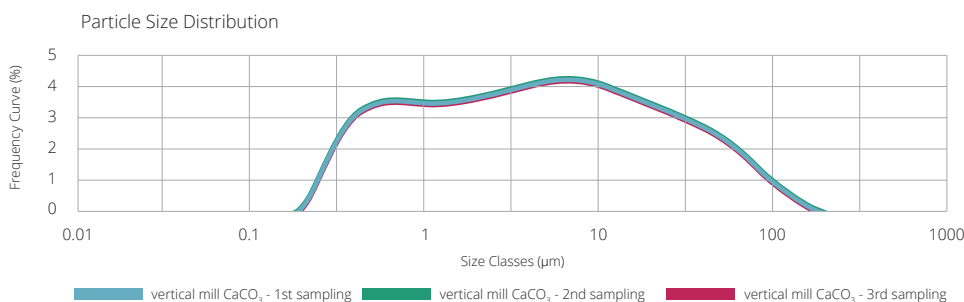


FIGURE 3:
The method repeatability: particle size distribution of three different samples from the same GCC batch measured by the same TopSizer instrument

Sample	D10 (µm)	D50 (µm)	D97 (µm)	%V <2µm
1	0.756	5.769	80.41	28.61
2	0.759	5.750	78.71	28.54
3	0.756	5.797	81.14	28.63
Average	0.757	5.772	80.09	28.59
RSD %	0.23	0.41	1.55	0.17

TABLE 2:
Particle size distribution of three aliquots from the same GCC batch measured by the same TopSizer instrument

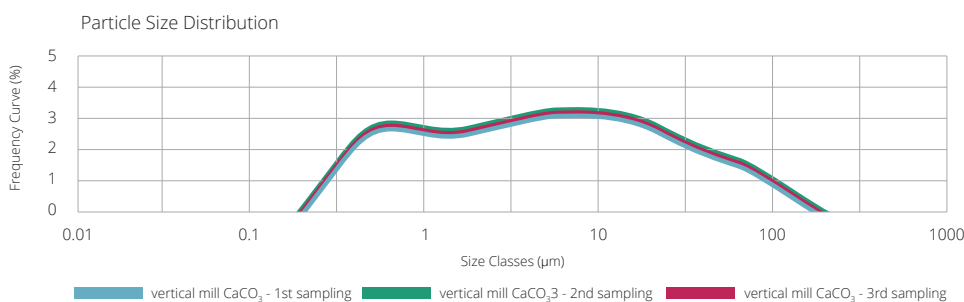


FIGURE 4:
The system reproducibility: particle size distribution of the same GCC batch measured by three different TopSizer instruments

Instrument	D10 (µm)	D50 (µm)	D97 (µm)	%V <2µm
1	0.756	5.769	80.41	28.61
2	0.775	5.896	81.65	27.92
3	0.757	5.840	83.26	28.47
Average	0.763	5.835	81.77	28.33
RSD %	1.40	1.09	1.75	1.29

TABLE 3:
Particle size distribution of the same GCC batch measured by three different TopSizer

SUMMARY

Laser diffraction is a widely used technique for the analysis of calcium carbonate due to its wide dynamic range, flexible dispersion options, speed of measurement and reproducibility of results.