

MICROTRAC

MEB

PARTICLE CHARACTERIZATION

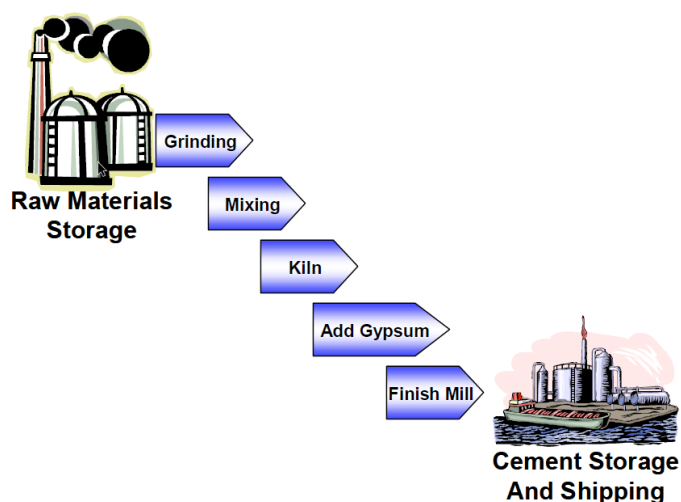
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Particle Size Measurement of Cement by Laser Diffraction

Background

The manufacture of Portland cement depends upon many factors, including chemical composition of raw materials, firing temperatures, and particle size. Of particular interest in this note is particle size measurement, since it can affect the efficiency of process grinding and quality of the final product. While there is continuing progress in automated process control, many plants throughout the world control grinding manually and measuring particle size by Blaine air permeability, Wagner photo-sedimentation, and sieve particle size techniques. Blaine and Wagner values are generally related empirically to surface area, which is an indicator of fineness while sieve values provide information on the coarser portion of the particle distribution. These methods do not lend themselves to timely measurements and, therefore, lag various stages of production. The Microtrac particle size analyser has been used for over 20 years to shorten the time between measurement of particles and process intervention indicated by particle size while aiding the quality control efforts.

The Process



Raw Mill

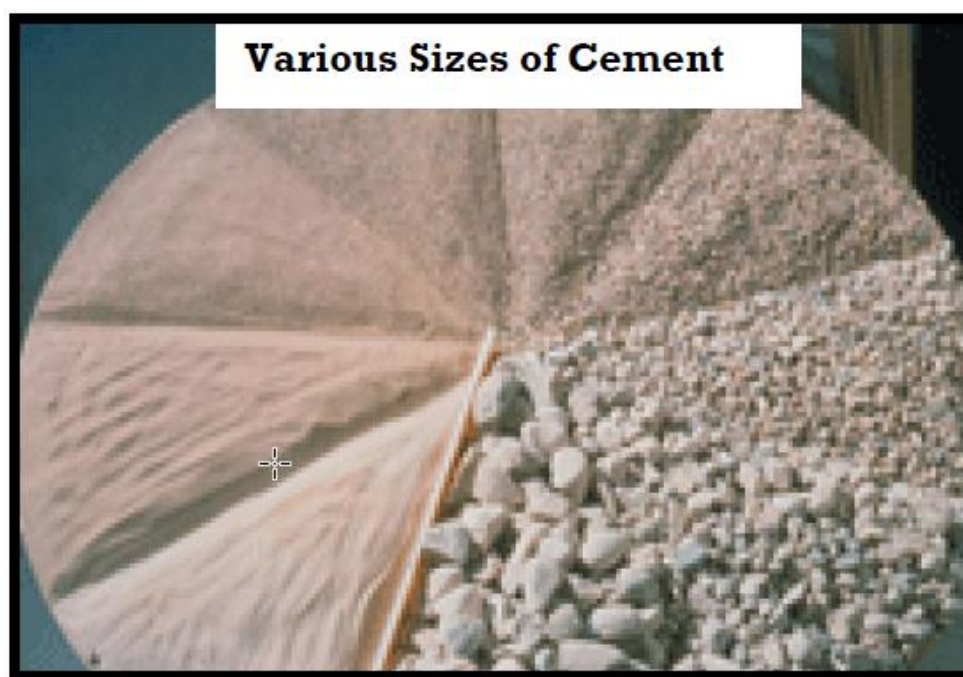
The raw mill (typically a roller mill) reduces the particle size of a mixture of the raw material (limestone) with clays, shale, and blast-furnace slag, which add silica, alumina, iron, magnesia, and sulphur trioxide. Globally, the mixture can vary as influenced by the composition of locally available raw materials. The milling may be either wet or dry and affords Microtrac the first opportunity for measurement prior to the mixture being pumped to the kilns. Typical particle size data, for the mixture discharged from the raw mill, is given as percent passing 50-mesh sieve. This size screen corresponds to 300 microns and is well within the measuring range of the Microtrac analysers. The size of the mixed particles is important because the material is forwarded to the kiln for drying (in the case of wet milling) and formation of the "clinker". Drying is a highly energy-intensive (and therefore expensive) process. Small particles dry more rapidly than large particles, where the rate is roughly proportional to the inverse of the particle size. The size is also important to the formation of the clinker as a homogeneous mixture of the raw materials. Thus, particle size at the raw mill step affects efficiency of later process steps, such as kiln drying, and ultimately the final quality of the cement.

The Kiln

In the kiln, the temperature of the homogenized mixture from the raw mill is raised to approximately 1600°C to produce a material that is slightly hard but easily broken apart by the finish mill. This clinker product from the kilns is a chemically homogenized, physically stable material for storage, in-process transport, and ultimate finish milling to produce the end product.

Finish Mill

The clinker is transported to the finish mill, where gypsum (calcium sulphate) is added for final product milling. The purpose of the finish mill is to reduce the clinker to a size that relates to both the early set and the ultimate strength characteristics of the final Portland cement. Clinker size directly affects the thermodynamics of the chemical reactions required for setting and curing characteristics. Cement that is too fine can react quickly causing excessive heat evolution and expansion of the cement/concrete. As cooling occurs, the concrete can then become unstable and crack or loose strength. If cement particles are too large, insufficient chemical heat generation can occur that is required for reaction completion, causing diminished strength.



In addition, many studies have been conducted examining the relationships between Portland cement particle size distribution (PSD) and hydration and hardened paste strength properties. Within certain

limits and for a fixed water-to-cement (w/c) ratio, a reduction in median particle size generally results in an increased hydration rate and, therefore, improved early properties such as high early strengths. Typical particle measurements include a sieve measurement (desirable value approximately 90% smaller than 325 mesh), Blaine determination (desirable value 4000 cm²/gm), and Wagner empirical surface area (typical value 2000 cm²/gm). The Wagner determination is far too slow for real-time control but because it is more sensitive than Blaine it is used to adjust final Blaine fineness values on a daily or more infrequent basis to achieve final specification.

Particle Size Distribution using Microtrac S3500

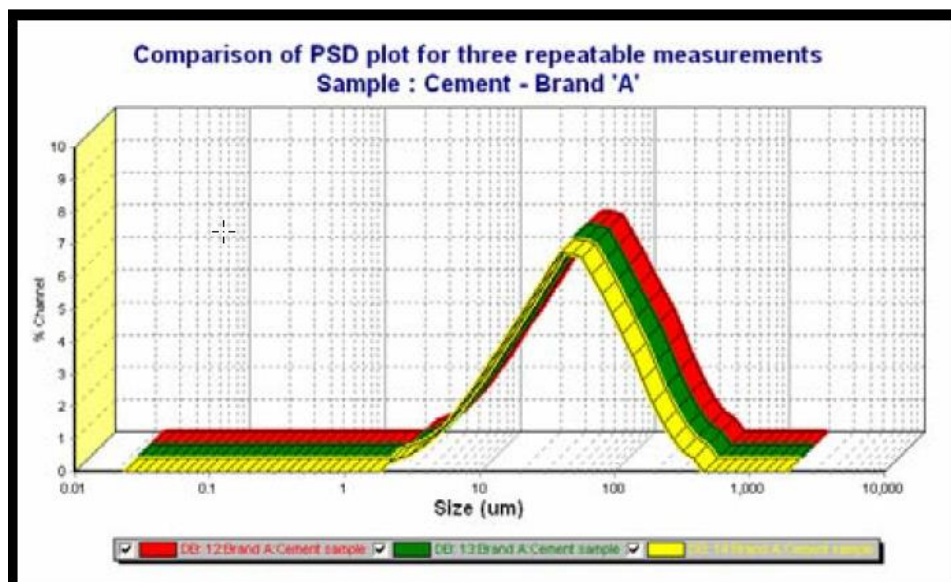
The Microtrac S3500 complies with ISO 13320 and consists of different modules for Wet and Dry mode analysis. Sample Delivery Controller (SDC) provides for measurement using water or any organic solvent. As desirable, the instrument can be converted to use the Turbotrac Dry Powder Dispersion module. In either situation, operation is fully, computer software controlled and provides easy, user-friendly operation regardless of skill level. Additionally, the Turbotrac offers the One-Shot accessory allowing very small quantities to be measured. The S3500 employs patented Tri-laser (solid state diodes) technology to illuminate the particles from which the scattered light is detected at 0.02 to 163 degrees. This permits full range measurement from 20nm to 2000 microns. An alternate lens permits measurement to 2860 microns. The system uses Microtrac FLEX intuitive software to perform well established Mie scattering theory to calculate the PSD of samples. It also offers key features like Data emulation, PSD in 3-D view, Statistics of measurements, Trend analysis, High resolution of data (128 Channel values), Mean volume, number & area distributions, and easy options to create SOPs.



Particle Size Measurements

Three separate brands of cement were obtained for particle size measurement using the Microtrac s3500 in both the wet (fluid dispersion) mode and Turbotrac (dry powder) dispersion mode. Repeatability, reproducibility and fluid/dry powder data were compared graphically and mathematically.

Fluid (wet) mode



For fluid measurement the SDC is automatically filled with dispersion medium which for cement can be water or organic solvent (isopropyl alcohol) depending upon user's demands. Conversion to organic solvents does not require any tubing or other changes to the system as it contains stainless steel, glass cell and Teflon components. Prior to the measurement, a blank measurement is made. Cement powder is then transferred to the SDC and measurement started. Repeatability for one sample addition is shown by the graphic above for one brand of cement. This experiment tests the instrument ability to repeat the same data report on the same sample circulating in the system. It is clear and convincing from the graph that the Microtrac S3500 instrumentation provides bubble-free, highly repeatable measurements. Reproducibility was also tested by performing separate transfers from the same sample bag to the SDC. An acceptable value for RSD % (standard deviation/mean of measurements) is 3% while international methods require 10%. The reproducibility for 2 samplings measured for all three materials is shown in the table below.

Brand	Parameter	Mean / μm	Standard deviation	RSD / %
Brand A	D10	8.75	0.05	0.516
	D50	38.55	0.19	0.503
	D90	134.20	1.60	1.170
Brand B	D10	7.07	0.09	1.210
	D50	27.77	0.23	0.811
	D90	77.88	0.29	0.369
Brand C	D10	6.84	0.08	1.100
	D50	28.30	0.19	0.663
	D90	81.60	0.95	1.160

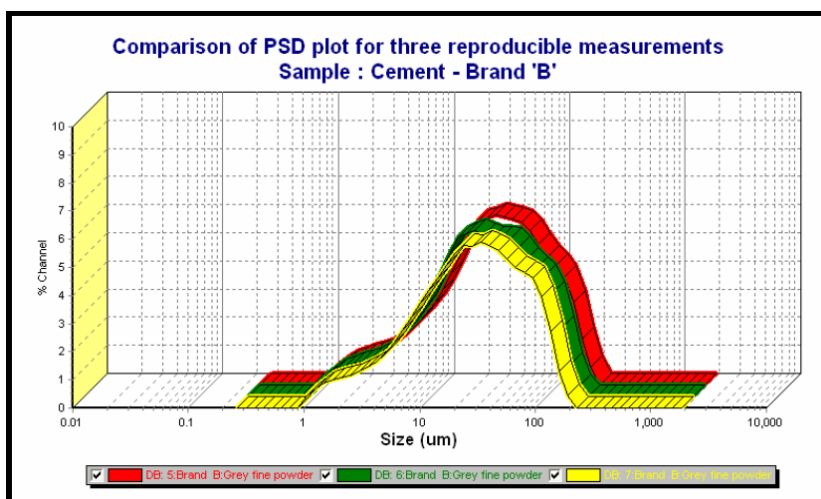
Note that the reproducibility for all three materials is well below the desired upper limits explained above. As well, it is interesting that the three materials show as much as a 30% difference in median (50%) particle size suggesting that the materials were manufactured under different specifications or may not conform to quality standards established for the type of Portland cement being manufactured. Regardless, the S3500 clearly shows differences of particle size distribution among the three products.

Dry powder measurement mode using Turbotrac

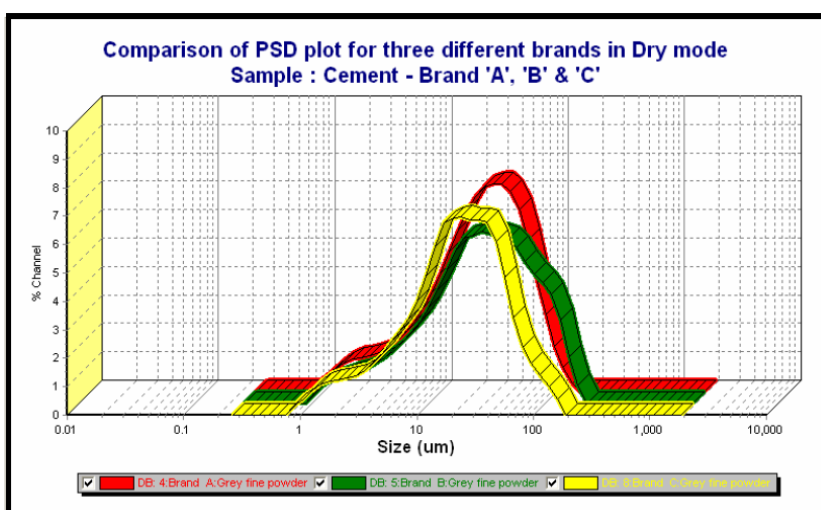
Dry powder measurements were performed using the Microtrac S3500 with the Turbotrac accessory. Sample is transported to the optical section by means of an aspiration tube, applied air and a vacuum which cause plume of cement powder to be developed. In the optical section, the aerosolized powder interacts with the laser beam whereupon light scattering (diffraction detection) occurs. Turbotrac measurement is automatic and self-cleaning and requires only one click of software to activate automatic blank measurement followed by measurement under the direction of a prescribed SOP located in software. Method parameters for measuring Brand B were:

- Air pressure =30psi; Refractive index (required for Mie scattering calculations) = 1.60

A comparative graph for Brand B is shown as well as the statistical values for measurement of three separate measurements.



Above is a comparative graph of the three cement samples measured using the Turbotrac accessory on a Microtrac S3500. Notable is that Brands A and C are very similar but are different in distribution from Brand B. Brand B contains a larger percentage of coarser particles larger in size than 45 microns.



Often there are questions concerning the comparison of Microtrac data to Blaine and Wagner, fineness tests. The data in the figures below show the linear correlation of these measurements to Microtrac data. Note that the unit notation for the Microtrac CS (calculated specific surface area in units M^2/CC) value has been converted to standard cement units of cm^2/gm by multiplying the value by 10^4 and then dividing by the density of cement ($3.15 gm/cc$). The correlation with Microtrac values allows easy conversion to Blaine and Wagner and display using Microtrac FLEX software.

