PARTICLE SIZE ANALYSIS OF CEMENT

Measuring and controlling the particle size distribution of cement is important both in order to achieve the desired product performance and to control manufacturing costs. Historic techniques of sieve and air permeability are still in use, but laser diffraction is becoming a more popular method to determine the particle size distribution of cement. The laser diffraction technique is quick, easy, reproducible, and provides a complete picture of the full size distribution.

Introduction

Cement is made by heating limestone with small quantities of other materials such as clay or sand to 1450°C in a kiln. The resulting 'clinker', is then ground with a small amount of gypsum into a powder to make Portland cement. The most common use for Portland cement is in the production of concrete – a composite material consisting of cement, aggregate (gravel and sand), and water. The relationship between the particle size distribution of Portland cement and hydration rate and strength has been studied and published (1-5). As a general rule reducing the particle size increases rate of hydration and strength. On the other hand, grinding cement to smaller sizes requires additional energy costs, so it is critical to measure and control the size of the final product.

Measurement Techniques

Historic techniques to determine the particle size and surface area of cement includes sieves (6-7) and air permeability, or Blaine tests (8). More recently laser diffraction is being deployed in cement laboratories to perform particle size analysis. Laser diffraction has the advantages of being quick, easy, and reproducible. In addition, laser diffraction provides a more complete description of the particle size distribution. Although it is possible to correlate laser diffraction results to sieve and Blaine values, the correlation typically falls apart if the size distribution changes, which is likely to happen in a plant upset condition. Vendors of laser diffraction analyzers therefore promote the use of the volume distribution results calculated directly in the instrument software.

Experimental

Cement can be measured in laser diffraction particle size analyzers either as a dry powder dispersed in air, or as a suspension dispersed in alcohol, usually IPA. Method development for wet or dry cement measurements follows the basic guidelines described in other HORIBA application notes (9,10).

Choosing the refractive index to use for cement requires consideration since cement is made up of so many different chemical species. The chemistry of cement is complex, but the basic ingredients include tricalcium silicate, dicalcium

silicate, tricalcium aluminate, tetracalcium aluminoferrite, and calcium sulfate (gypsum). The NIST 114 q standard certificate suggests using 1.70 and 1.0 i, so this value was chosen for the results presented here.

Dry results for cement shown in this document were collected on the LA-950 Particle laser diffraction analyzer (seen below). Since cement is a hard, robust material the measurements were made at the highest air pressure setting for the PowderJet dry powder feeder. The PowderJet keeps a constant mass flow rate through the measurement zone through an automatic feedback loop controlling the vibration rate based on the transmissions readings.

The system settings used for the dry measurements are presented below:

PowderJet:	Small nozzle, high P
Iteration number:	15
Refractive index:	1.70-1.0i, 1.000 (air)

The LA-950 Partica

The wet results presented here were collected on both the LA-950 and LA-300 laser diffraction particle size analyzer. The LA-950 has a broader dynamic range (0.01-3000 μm) compared to the LA-300 (0.1-600 μm), but since these and most cement samples contain little to no powder below 0.1 mm, either system is capable of measuring cement. The LA-950 does contain additional benefits of higher levels of automation and more sophisticated software most labs would find helpful.

Note that all measurements were made in IPA. It is important to watch the optical background when using IPA as thermal fluctuations may cause additional scattering leading to inconsistent results. When encountering this phenomenon all that is typically required is recirculating the IPA until a stable background is achieved.

The system settings used for the wet LA-950 and LA-300 measurements are presented below:

Iteration number:	15
Refractive index:	1.70-1.0i, 1.390 (IPA)
Ultrasound:	60 sec @ level 7
Circulation speed:	6
Acquisition time:	5000 ms

Results

The results from three measurements of Portland cement measured on the LA-950 are shown below in Figure1 and Table1. Note that the results are extremely reproducible, with the coefficient of variation well below those suggested is ISO 13320-1 (11). The coefficient of variation calculations can now be performed directly in the LA-950 software. The HORIBA applications support team recommends all customers utilize this approach to testing reproducibility before accepting results.

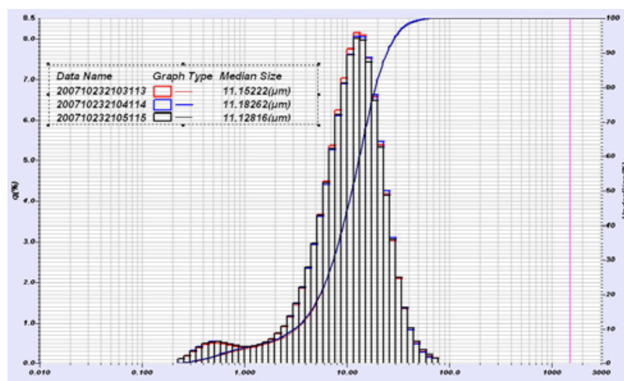


Figure 2: LA-950 Dry Results

File Name	Material	10.00%	50.00%	90.00%
200710232103113.NGB	Portland Cement	3.256	11.152	24.586
200710232104114.NGB	Portland Cement	3.116	11.183	24.671
200710232105115.NGB	Portland Cement	3.112	11.128	24.92
Average		3.161	11.154	24.726
Std. Dev.		0.082	0.027	0.173
CV (%)		2.589	0.245	0.701

Table1: LA-950 Dry Results

Results from Portland cement run in IPA on the LA-950 are shown below in Figure 2 and Table 2. These results are also extremely reproducible and meet ISO 13320-1 guidelines.

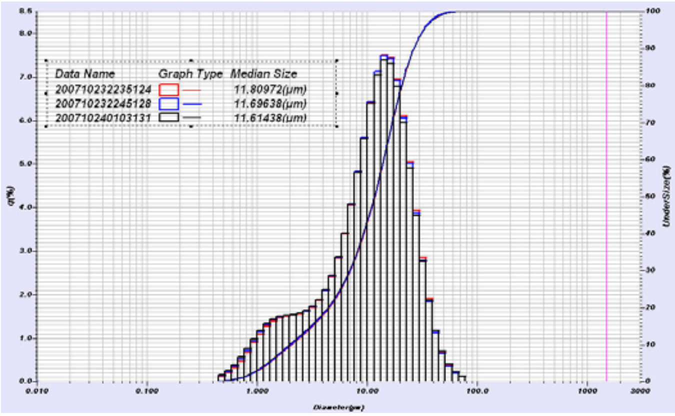


Figure 2: Wet LA-950 Results

File Name	Material	10.00%	50.00%	90.00%
200710232235124.NGB	Portland Cement	2.122	11.81	27.047
200710232245128.NGB	Portland Cement	2.058	11.696	26.743
200710240103131.NGB	Portland Cement	1.999	11.614	27.001
Average		2.06	11.707	26.93
Std. Dev.		0.062	0.098	0.164
CV (%)		2.996	0.838	0.607

Table2: Wet LA-950 Results

Portland cement was also measured wet on the LA-300 laser diffraction system (seen below). This instrument only measures suspensions, but is well suited for routine QC measurements and is smaller, more portable, and less expensive than the LA-950.

The LA-300 System

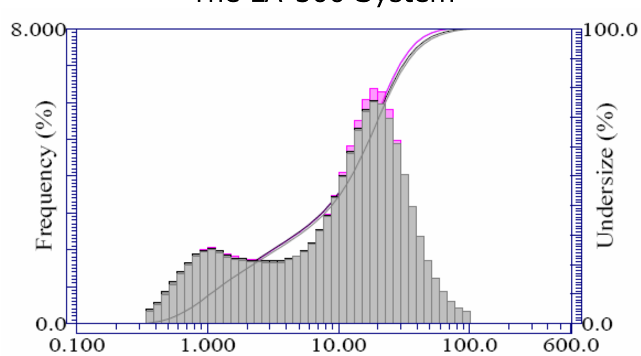


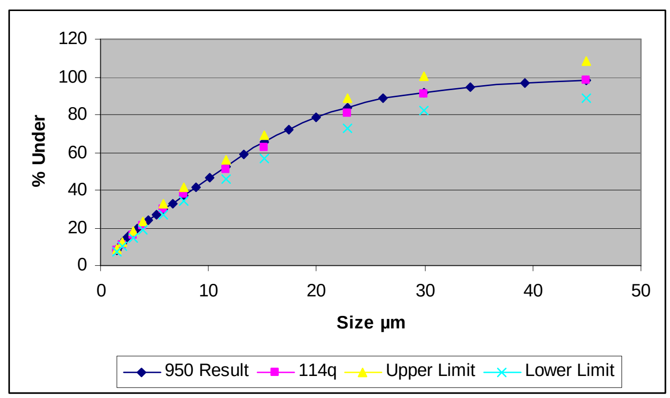
Figure 3: Wet LA-300 Results

File Name	Material	10.00%	50.00%	90.00%
3 - 2 min EUS - 2 - A	Portland Cement	1.034	12.05	31.573
3 - 2 min EUS - 3 - A	Portland Cement	1.015	12.112	33.649
3 - 2 min EUS - 4 - A	Portland Cement	1.052	12.533	32.235
Average		1.034	12.232	32.486
Std. Dev.		0.019	0.263	1.060
CV (%)		1.790	2.148	3.264

Table 3: Wet LA-300 results

NIST 114q Standard

The cement industry uses the NIST Standard Reference Material 114q in order to calibrate fineness testing equipment according to ASTM Standard Methods. This material has also been used as a source material for round robin studies using laser diffraction to measure particle size distribution. NIST 114q was analyzed on the LA-950 in order to asses the response compared to the NIST documentation. Graph 4 below shows the LA-950 result along with the certified 114q values, and the lower and upper limits of accepted results. The results fell within the accepted range.



Graph 4: Dry LA-940 Results for NIST 114q

Conclusions

Laser diffraction is becoming the preferred method for particle size analysis. Many customers prefer measuring cement as a dry powder which eliminates the need to use and recycle alcohol. Other customers still prefer to use IPA as the dispersant. Both the LA-950 and LA-300 can be used successfully for research or routine QC requirements.

References:

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3. Pommersheim J, Effect of particle size distribution on hydration kinetics. Materials Research Society Symposium Proceedings, **85**, Mat Res Soc, Pittsburgh, 1987, pp. 301-306.
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10. AN154 Dry Method Development, available at www.HORIBALAB.com
11. ISO13320-1 Particle Size Analysis – Laser Diffraction Methods, Part 1: General Principles.

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