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application
note

Particle Sizing of Cement using the Mastersizer 2000

Introduction

Cement has been an integral part of construction since a primitive version was developed by the Romans. Today the most commonly used form of cement is Portland cement, which was first developed in the 19th century.

Portland cement

In general, the production of cement comprises three stages: preparation of a raw mix; production of the clinker; and the grinding of the clinker to form the finished cement.

The raw mix can be made up of materials such as calcium oxide, silicon oxide, aluminium oxide, ferric oxide and magnesium oxide. Most of these minerals can be obtained from local rock, but clay and limestone may also be added. These components are then ground into a raw mix, so that no more than 15% of the volume or mass of particles are larger than 90µm (Figure 1). The proportion of coarse particles in the raw mix must be controlled to ensure that the resulting mix is homogenous and that the sintering of the components within the kiln during the next stage of production is completed within a reasonable time frame.

The raw mix is heated to approximately 1400°C in order to from the clinker. Calcium sulphate may also be added, in order to provide control of the setting time, prior to grinding of the clinker to form the finished cement.

The properties of any cement are dependent on their composition and particle size distribution. For example, the properties of Ordinary Portland Cement can be varied by adding other
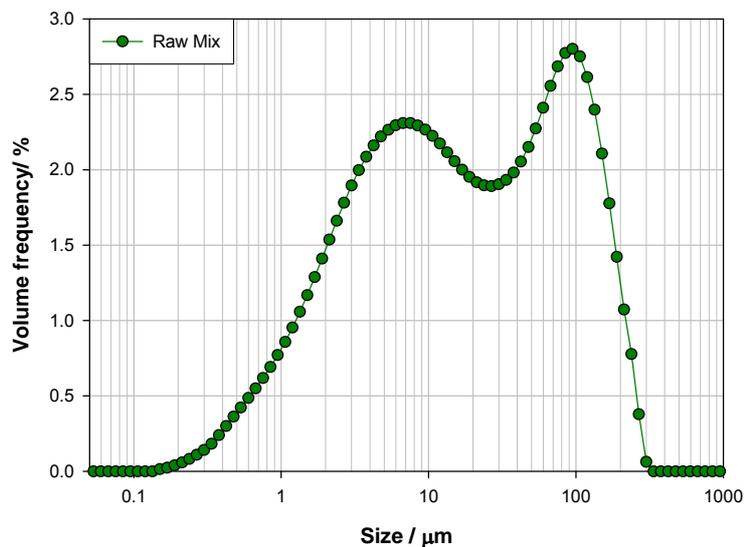

Figure 1: Particle size distribution of a raw mix

components to produce different cement blends. For instance, blast-furnace slag and fly-ash can be added to Portland clinker in order to save cost, but these blends can have poorer early strength. Expansive cements can be produced in order to offset shrinkage during drying by use of expansive clinkers (such as sulpho-aluminate). The aesthetic properties of cement can also be altered by adding pigments and clays to produce coloured or white cements.
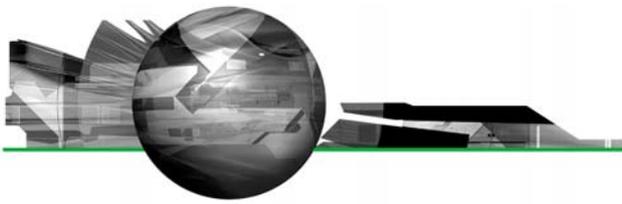
Particle size

The critical strength and curing properties of cement are dependent on the particle size distribution resulting from the grinding phase. The compressive strength and curing qualities are increased as the particle size decreases due to the increase in surface area. For this reason cement

has traditionally been specified using the Blaine number, a surface area based parameter measured by an air permeability technique.

However, additional information can be gained by measuring the particle size distribution. For example, if two cement samples have the same average size or surface area then the sample with the narrower size distribution will have a higher compressive strength. Secondly the Blaine number will be less sensitive to changes in the coarse fraction.

In a general cement, between 60 and 70% of the material should be between 3µm and 30µm in size. Excess large particles (greater than 50µm) can cause problems by reducing strength due to incomplete hydration, where as excess small particles (less than 2µm) can reduce

strength and cause the cement to crack by setting exothermically.

Experimental

In this application note, laser diffraction has been used to measure samples of the raw mix and two cement blends. Particle size information has also been used to determine the efficiency of a separator. Additionally a cross contamination study has been carried out where alternate measurements of two different cement types have been made in order to demonstrate that there is no contamination between measurements.

Measuring the particle size of cement

The particle size distributions of the various cement samples have been measured by laser diffraction using a Mastersizer 2000. Laser diffraction instruments operate by illuminating particles in a collimated laser beam and measuring the scattered light over a range of angles. Particle size information can be obtained as the angle to which particle scatter light is dependent on their size. Therefore by measuring the angular dependence of the scattered light, the particle size distribution can be obtained by using an appropriate scattering model.

The sample is dispersed and passed through the measurement cell using a dispersion unit. Samples can be dispersed wet or dry depending on which is more appropriate to the application. For cement, the dry dispersion unit allows for a larger volume of material to be measured easily and it also avoids the additional cost of the solvent which would be required for a wet dispersion of cement. Therefore, in this report the samples have been dispersed dry, using the Scirocco. The Scirocco operates by vibrating a tray of material that feeds into a venturi which, combined with compressed air,
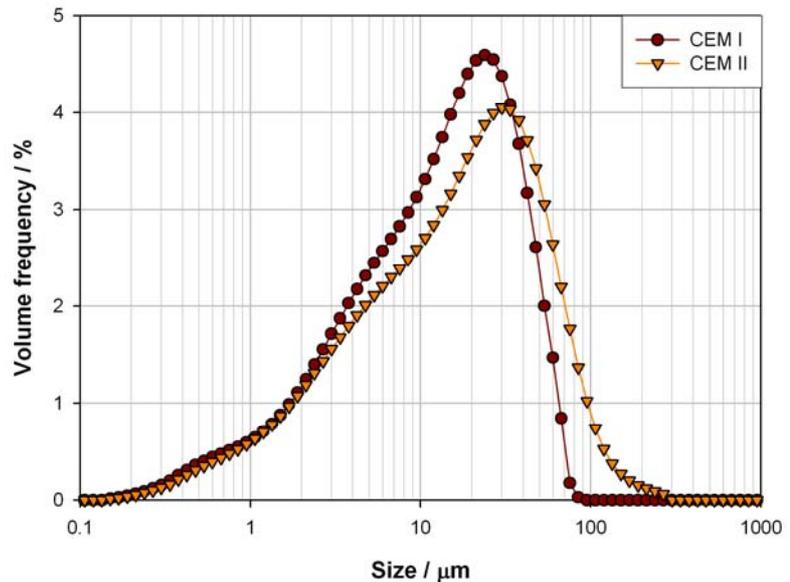

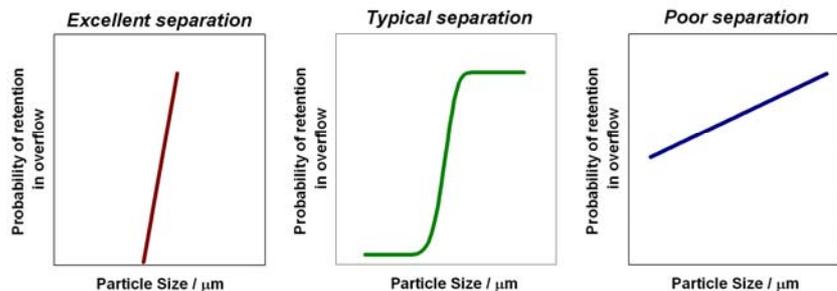**Figure 2:** Particle size distribution of CEM I and CEM II

Figure 3: Tromp curves showing different degrees of separation.

is used to disperse the particles. The Scirocco can be used with several trays for different volumes of sample and an additional hopper, which can take up to 25g. In addition, water-resistant, ceramic dispersing components are available, providing robust operation when handling abrasive materials.

Results

Figure 2 shows the particle size distribution for two cement grades,

CEM I and CEM II. CEM I is general purpose cement containing around 95% clinker where as CEM II contains between 65% and 95% clinker, with fly-ash or limestone added to give moderate sulphate resistance.

From the size distributions it can be determined that 65% of the volume of the CEM I sample is between 3 and 30µm, which reduces to 54% in CEM II. The higher proportion of material between 3 and 30µm in CEM I

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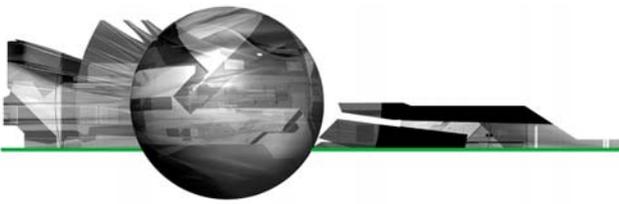
suggest that it will have a higher compressive strength.

Figure 2 shows that the main differences between CEM I and CEM II are in the coarse particle content. This can be quantified by looking at the volume weighted average (D[4,3]) which is sensitive to the coarse particles in the distribution. CEM I has a D[4,3] of 17.8µm and CEM II has a D[4,3] of 26.0µm. These two samples can therefore be clearly distinguished by the D[4,3]. In contrast, the surface area will be insensitive to changes in the coarse particle fraction. CEM I has an SSA of 1.36 and CEM II has an SSA of 1.2. This larger particle size will cause CEM II to have a longer hydration time and to generate less heat during hydration.

Measuring separator efficiency

A separator is essential to the clicker grinding process in order to remove large clinker particles, which require further grinding, from the fine particles, which meet specification. Separation is typically achieved by use of an air classifier. Within the classifier, the milled powder is held within a rotating air stream, forcing coarse particles impact on the walls of the classifier. These coarse particles can then be collected as they drop down due to gravity; whereas fines particles are transported away in the air stream towards the centre of the classifier. Control of the classifier speed, along with the feed rate of material into the classifier, allow the cut-point for the separation process to be controlled.

The efficiency of a separator must be determined to make sure that no oversized particles are present in the final product and that fine particles are not processed any further. This can be measured by plotting Tromp curves. These plots show the probability that a particle of a particular size is separated into coarse (overflow) fraction (figure 3). Ideally, the tromp curve should be as
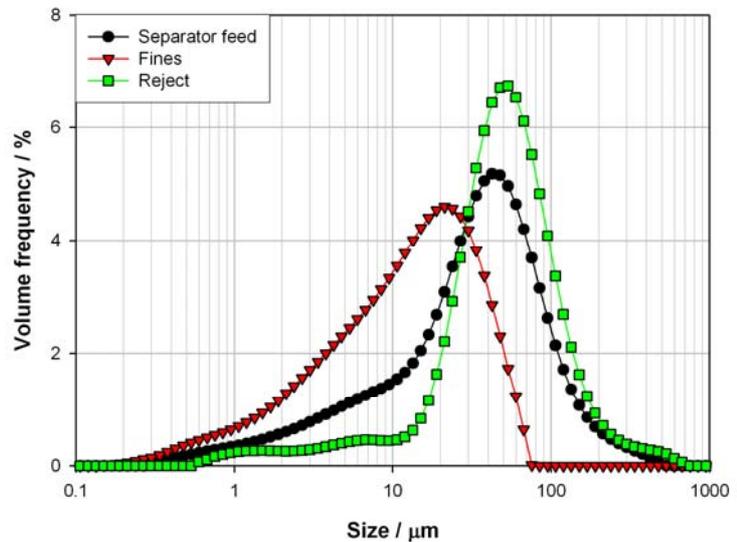

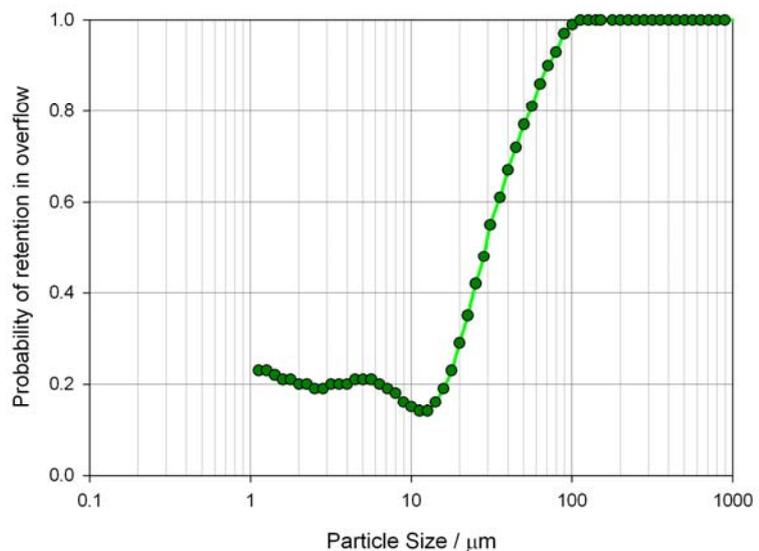**Figure 4:** Separator feed, fines and rejects

Figure 5: Tromp curve from the size distributions in figure 4.

steep as possible, showing excellent separation of the coarse and fine particles. In practice, this is not achievable, and some proportion of fine particles (referred to as bypass material) is typically found with the overflow. This is because fine particles have a low inertia: they therefore tend to follow the air flow within the separator, and are subsequently not subject to classification by either gravitational or centrifugal forces. Poor separation, where a significant fine particle
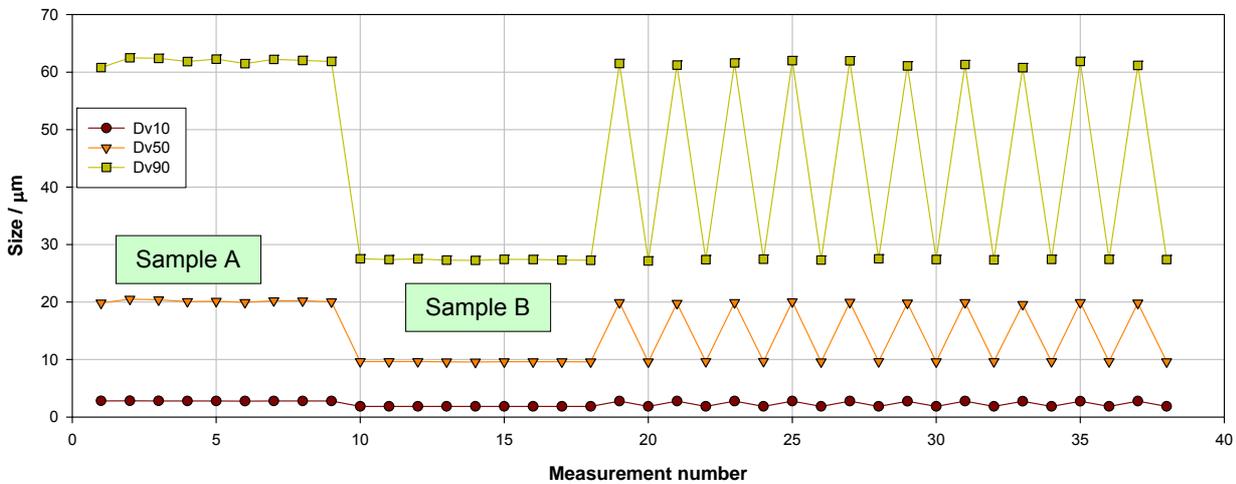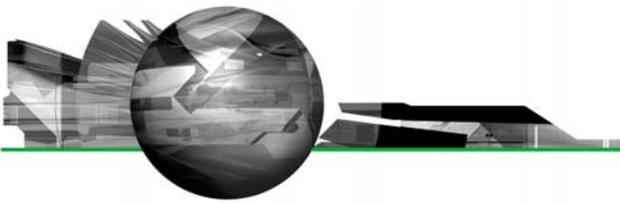
Figure 6: Contamination study using samples A and B

fraction is observed in the overflow, must be avoided, as this can lead to over-milling of the clinker.

To plot a Tromp curve, both the size distribution and feed rate for two of the feed, oversize and undersize are required. Tromp curves can also be determined from the particle size distributions of all three components. Figure 4 shows the particle size distributions for the feed, overflow and underflow from a cement classifier. The grade efficiency can be calculated easily in the Mastersizer software by selecting records for the feed, underflow and overflow and the resulting Tromp curve is displayed on a grade efficiency report (Figure 5). In this case, the slope of the Tromp curve is quite steep, showing that the separator is performing well and is giving clean separation of the product. If this were not the case then remedial steps could be taken and performance re-assessed to reach optimal conditions. The plateau observed at small particle sizes within the Tromp curve relates to the presence of fine particles in the coarse oversize stream. These fines may be bypass material. However, they may also be fine particles which are adhered to coarse particles within

the classifier, preventing them being separated into the underflow stream, but are subsequently dispersed during the laser diffraction measurements.

Cross contamination

The final experiment in this application note was designed to test for cross contamination in dry measurements. This was tested by making consecutive measurements of two different cement samples. Measurements were carried out using the Mastersizer 2000 and the Scirocco dry powder feeder with hopper as this test is particularly interesting for automated laboratories.

The testing process was as follows; 10 repeat measurements were made of sample A, followed by 10 repeat measurements of sample B. This established a reference size for both samples A and B. In order to try and simulate a situation where cross contamination could occur, alternating measurements of sample A and sample B were made. The particle size parameters (Dv10, Dv50 and Dv90) are plotted in Figure 6 for the initial measurements of sample A and sample B as well as the subsequent

alternating measurements to test for contamination.

Figure 6 shows that the particle size remains unaffected by the alternating measurement sequence and therefore there is no cross contamination between consecutive measurements of sample A and sample B. The measurement reproducibility is of the order of 1%, based on the Dv50 measured for each grade, even when making alternate measurements.

In an automated lab the software can monitor the cleanliness of the system it may not be possible to detect the more subtle effects of cross contamination. This experiment has demonstrated that the results of measurements are not affected by cross contamination. This should enable users to have a high degree of confidence in the results obtained from an automated system.

Conclusions

Laser diffraction is a fast and effective technique for measuring the particle size of cement. The size range of the Mastersizer 2000 is ideally suited to measuring material over broad size ranges. For example, measurements

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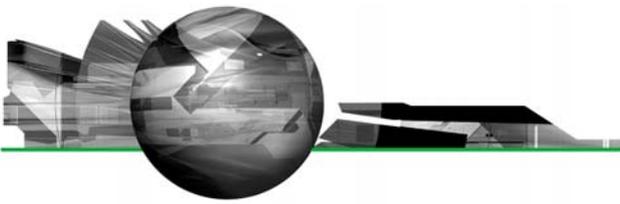
of raw mix to extra fine cement grades can be made without having to change any lenses. Sample can be dispersed either wet or dry and the Scirocco dry powder feeder provides an easy method for dispersing larger volumes of cement samples.

In this application note the particle size distribution of raw mix. CEM I and CEM II have been measured. CEM I contains a greater percentage of material between 3 and 30µm which indicates higher compressive strength. CEM II showed a greater proportion of coarse material indicating a slower hydration time. The efficiency of a separator has been determined from size distribution measurements of the feed, overflow and underflow. Finally a contamination study has been carried out to show that making consecutive measurements of different types of cement has no effect on the measured size distribution.

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