Quality Control of Cement – from sample crushing and fine grinding to elemental analysis

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RETSCH offers a range of instruments suitable for sample preparation during the complete production process, from the quarrying of the raw materials to the final product. The typical sample preparation process involves preliminary size reduction, sample division and fine size reduction before the sample can be submitted to further analyses.

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Primary Size Reduction

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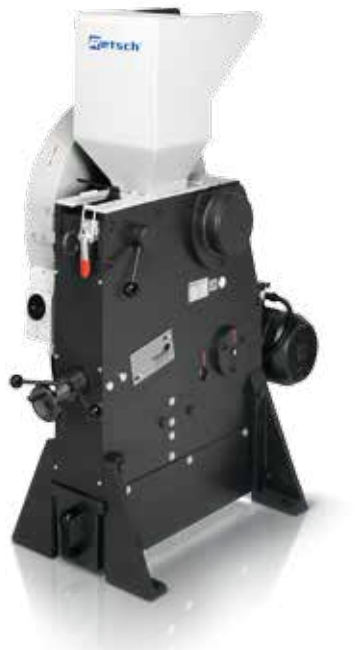

RETSCH Jaw Crusher BB 200

Sample Division

After pre-crushing the material has to be divided further to obtain a representative part sample. The qualitative composition of the part sample has to match the quality of the original sample as closely as possible. The number and quantity of part samples to be taken depends on the total quantity, its homogeneity and the maximum grain size contained. Random sampling is not advisable due to the high error rate of around 20%. The error rate can be decreased down to 0.5% by using a sample divider such as RETSCH's Sample Divider PT 100 or Rotating Tube Divider PT 200. Automatic material feed via a synchronized feeder allows for easy operation and ensures reproducible results. The dividers accommodate feed sizes of up to 10 mm and divide the sample into 1-3 or 6-10 representative part samples according to DIN 51 701.

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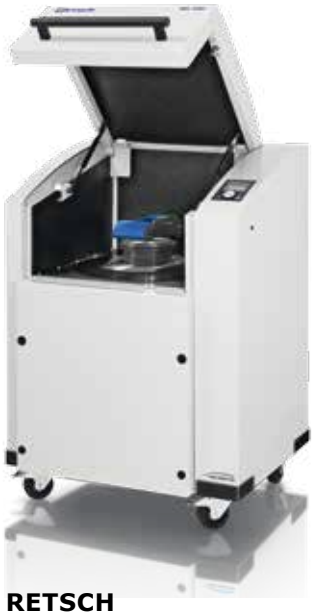
Fine Grinding

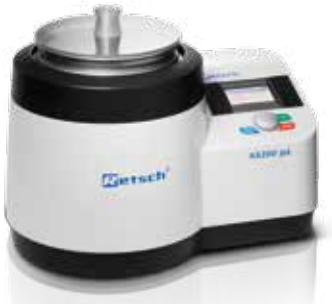
Now the part sample can be ground to sizes of 100 µm and smaller, either in a ball mill or in a vibratory disc mill such as RETSCH's RS 200. This mill is typically used for sample preparation to XRF analysis and delivers grind sizes down to 100 µm and smaller in as little as 30 to 40 seconds. The grinding results of the RS 200 are highly reproducible. The pulverised sample can now be analyzed by XRF or elemental analysis. Solid, high-quality pellets are an important precondition for reliable and meaningful XRF analysis. With the PP 40, RETSCH offers a pellet press which produces strong pellets with a smooth surface.



RETSCH
Vibratory Disc Mill
RS 200

Determination of the particle size

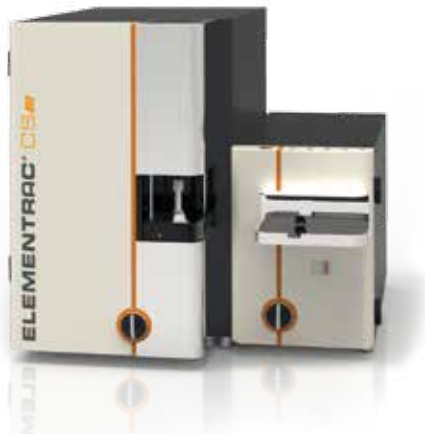
RETSCH also provides a wide range of sieving equipment covering a size range from 10 µm to 125 mm, using a variety of sieving motions. With vibratory sieving, the sample is subjected to a 3-dimensional movement, i.e. a circular motion superimposes the vertical throwing motion. Due to this combined motion, the sample material is spread uniformly across the whole sieve area. For sieve cuts of powdered substances which tend to agglomerate, a 3-dimensional movement is not suitable. Therefore, RETSCH has developed the Air Jet Sieving Machine AS 200 to sieve agglomerating powders in a size range from 10 µm to 4 mm. The material on the sieve is moved by a rotating jet of air: A vacuum cleaner which is connected to the sieving machine generates a vacuum inside the sieving chamber and sucks in fresh air through a rotating slit nozzle. When passing the narrow slit of the nozzle the air stream is accelerated and blown against the sieve mesh, dispersing the particles. Above the mesh, the air jet is distributed over the complete sieve surface and is sucked in with low speed through the sieve mesh. Thus the finer particles are transported through the mesh openings into the vacuum cleaner or, optionally, into a cyclone. RETSCH also offers the software EasySieve® for easy evaluation and documentation of the results.



RETSCH
Air Jet Sieving Machine
AS 200 jet

Measurement of carbon and sulphur concentrations

A reliable determination of the carbon and sulphur content in cement is an essential part of the quality control process. If the sulphur content is too high, the cement could be destroyed as a result of the chemical reaction of sulphuric acid. The carbon content is usually certified as CO_2 and sulphur content is given as SO_3. In general, elemental analysers combust the sample and measure the released gases. Inorganic samples with a low carbon and sulphur content such as cement are usually combusted in an induction furnace at temperatures up to 2200°C. Table 1 shows typical results for CO_2 and SO_3 concentrations in a cement sample and also in wood and coal which are both used as fuels in many cement factories. The results were obtained with ELTRA's CS-2000 analyzer, with very low standard deviations. The system works reliably and quickly with a typical measurement time of only 60 to 120 seconds.



ELTRA
CS-*d* analyzer

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Analytical values of 10 measurements of cement, wood and coal

	Cement	Wood	Coal
% CO_2	6.01 +- 0.04		
% SO_3	2.61 +- 0.04		
% C		49.86+- 0.18	
% S		0.02 +- 0.001	
% C			64.42 +- 0.07
% S			0.56 +- 0.002

Secondary fuels

A constantly growing number of cement plants not only use fossil fuels for the cement clinker burning process but also secondary fuels. Secondary fuels either consist of residues from a wide range of manufacturing and production areas or of residues with a high calorific value which are usually obtained from heterogeneous waste. These secondary fuels must meet all the requirements of primary fuels with regards to product quality and environmental safety. Therefore, they have to be prepared and analyzed in the same way and, in addition, they have to be analyzed for their gross calorific value to ensure a stable and reproducible combustion process.

Sample preparation and analysis

As secondary fuels are often soft and elastic materials, primary size reduction has to be effected with cutting and shearing forces such as are applied in cutting mills. Here, the same grinding procedure as for hard and brittle material has to be followed: Pre-cutting followed by sample division and fine grinding. Since the secondary fuels usually are very heterogeneous, the sampling process becomes evident for correct analytical results. RETSCH's heavy-duty cutting mill SM 300 easily handles inhomogeneous sample materials with different breaking properties and is especially safe to operate. It is available with heavy-metal-free grinding tools for neutral-to-analysis sample preparation which makes it ideal for processing secondary fuels. With the three exchangeable cutting rotors and its high powered drive, textile and leather parts, plastic caps and ductile metal cans are cut up just as effectively as abrasive electronic scrap and non-metallic car-shredding parts. As there are limits to this cutting mechanism, i.e. steel and iron parts should be separated by a magnetic separator before the cutting process. The final fineness is mainly determined by replaceable bottom sieves with defined aperture sizes. Depending on the sample material, final particle sizes < 2 mm can be achieved. A cyclone increases the material throughput and helps to dissipate the grinding heat. For the fine grinding down to 200 µm, the Ultra-Centrifugal Mill ZM 200 from RETSCH is the ideal tool. It grinds extremely fast and thanks to the patented cassette system it is very easy to clean which allows for high sample throughput. The sample size again is determined by an exchangeable sieve. Experience shows that aperture size of 0.5 or 1 mm is fully adequate for obtaining analytical fineness. Very elastic material like rubber has to be embrittled with liquid nitrogen before pre cutting and final grinding to avoid melting of the sample.

Due to their different combustion behaviour (compared to inorganic material), secondary fuels cannot be processed in an induction furnace. The most suitable choice is a measurement with a resistance furnace at a temperature of 1,300°C. As cement factories use different types of fuels with different calorific values, it is essential to measure the carbon content as a perfect indicator for the calorific value of a material. Sulphur, on the other

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hand, is an important parameter for processing the combustion gas desulphurisation. Whereas coal has a rather high sulphur content of up to 5%, it is only around 0.02% for secondary fuels such as wood or biomass. With ELTRA elemental analyzers samples of greatly varying sulphur content can be examined precisely and reliably.

Secondary fuel samples

Conclusion

Quality control is an important aspect of cement production. Sample preparation is an essential part of it, because only a representative and reproducible processing of the sample material ensures reliable and meaningful analysis results. Retsch offers a range of instruments for dividing, crushing, grinding and sizing all materials which are involved in the production process of cement, including secondary fuels. To ensure the right choice of instrument for the right sample material, Retsch offers free-of-charge sample testing in application laboratories all over the world.

Elemental analysers based on combustion technology are a useful addition to XRF analysis for the quality control of cement and related products, ensuring fast, precise and reliable determination of carbon and sulphur. With its offering of analyzers using resistance or induction furnaces or both, ELTRA covers a wide range of applications for C and S determination in organic and inorganic samples. The product range is ideally suited to the variety of analytical applications in a cement plant.

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