Importance of particle size distribution in cement sampling for X-ray fluorescence

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Introduction

X-ray fluorescence, is the preferred method for analysis of cement samples, due to its rapidity and exactness. It permits the elementary composition of a sample to be determined. Presently, there are two ways to prepare solid samples, pearl and pellet. Both have particular advantages and disadvantages; however, the pearl preparation gives better results, particularly in respect to reproducibility. This method fuses the sample with a fondant in order to obtain a glass. Its amorphous matrix dilutes the sample, thereby reducing interference.
In contrast, use of the pellet method causes problems in terms of reproducibility. This is due to the particle size distribution, which is a limiting parameter in X-ray fluorescence. Although the irradiated surface is quite big (0.01 – 0.1cm^2), the volume is very shallow (0.01 – 0.1cm^3). Consequently the surface must be representative.
Preparation of the pellet consists of grinding and pressing of the sample. The grinding time must be long enough. The grains of all oxides are not the same size, due to oxide differences in hardness. Therefore, in order to obtain a satisfactory particle size distribution, it was necessary to find the optimum grinding time. This was found by studying the evolution of peak intensity for silicium (figure A) and calcium (figure B), as a function of grinding time.

Experimental conditions

Four samples of cement (which were ground at 3, 4, 5 and 6 minutes respectively) were analyzed by X-ray fluorescence, checking Kα lines for calcium and silicium. The control tube was set at 40 kV and 70 mA. The detection device was a FPC detector (Flow Proportional Counter), particularly suited for light elements.

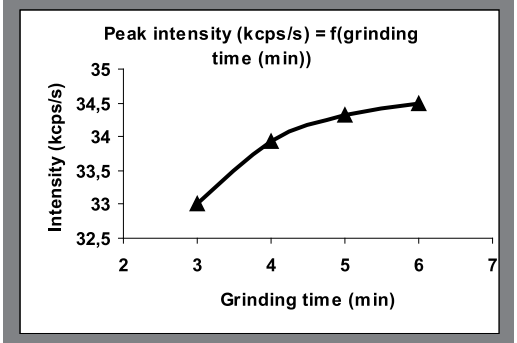
Results and discussion

The graphs showed an increase of the silicium peak intensity, until it reached a limit at approximately six minutes. In contrast, the calcium peak intensity dropped, until it reached its limit at six minutes as well. This shows: the longer the grinding time, the better determination of the representative character of the surface; ultimately, the "true" composition of the sample is ascertained.
This study confirms the hardness of the generated silicium oxide presented a problem for reproducibility in the cement's pellet. It was necessary to grind for at least six minutes to obtain the best possible homogeneity of the sample and thus reproducibility.

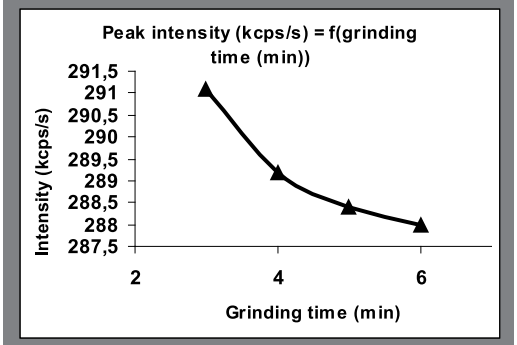
Conclusion

In industry, the main parameter considered is time, which must be optimized in each part of the process. Therefore, it has been decided to prepare a pellet with a grinding time of four minutes. Indeed calibration gave good results for reproducibility with this method of preparation.
In conclusion, compromise must be taken between research and its application within industry.

A) Evolution of silicium peak intensity



B) Evolution of calcium peak intensity



Evolution of intensity (kcps/s) for calcium peak and silicium peak in function of grinding time (min), in a sample of cement by fluorescence X.

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