Fused Beads for Better XRF Analytical Results in Cement Application

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CHAPTER 1 XRF BASIC PRINCIPLES

1. 1. SPECIMEN PREPARATION

Analysis by fluorescence of x-rays (XRF) is a technique essentially based on comparison with standards, the accuracy and the reliability of XRF depends largely on the SPECIMEN preparation. The preparation of the specimen is a process to obtain from the sample an entity having the same overall chemical and physical composition.

1.2. METHODS OF SPECIMEN PREPARATION

Two techniques are largely used: confection of pressed tablets and preparation of beads or glass disks by fusion.

- The fusion technique consists in dissolving the specimen in a solvent called a flux at high temperature (>1000°C) in a platinum/gold crucible and to cast it in a casting-dish. The most current used fluxes are borate such as lithium tetraborate, sodium tetraborate, lithium metaborate, or sometimes polymetaphosphates. The result of the fusion is the production of a homogeneous glass disks or bead.

The choice of one of these methods depends of the level of accuracy expected in function of the speed required from the results; this is the case sometimes in industrial process.
The cost and the speed related to the accuracy plays also a predominant role in the final choice.

The two methods are briefly described with both the advantages and inconvenient of these specimen preparation techniques.

1.3. LIMITATION of XRF ANALYSIS.

Only a small portion of the specimen will be analyzed by XRF, because of the relatively low penetration dept of x-rays. In general only the first layer of grains is seen in case of powdered pressed tablets.
It is therefore very important that this layer is representative of the whole specimen.
The analyst has to bear in mind the various effect of heterogeneity and the limitations of the pressed tablet method.

Grain size effect:
With large particles, x-rays gives information about the composition of this single particle, while with small particles the intensities give information about the overall composition.
Particle size distribution:
How good the grinding is carry on; one could avoid particles size distribution. One of the biggest problems is to obtain standards of the same composition and with the same size of distribution. The emitted x-ray Intensities varies in function of the particle size. This is quite important for silicon and aluminium. (in cement application)