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Fast quantitative analysis of the amorphous content with the Rietveld method

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The characterization of the mineralogy and chemical composition of multi-phase mixtures is of chief importance in many different contexts, from industry to basic research. In the case of industrial processes, it is often necessary to perform fast and reliable quantitative phase analyses (QPAs) in large amounts of samples that contain amorphous phases. Rietveld refinement from powder x-ray diffraction (XRD) data is widely employed for this purpose. The quantification of the amorphous content with the Rietveld method is usually performed by spiking the samples with an internal standard, and this implies increased processing times. Alternatively, in samples exhibiting the typical, broad XRD signal from the amorphous (glassy) matrix, a poorly-crystalline structure can be used to represent the amorphous phase during the Rietveld analysis [1]. This procedure provides highly consistent data, but is limited because the particular crystal structure used for the QPAs is strongly sample dependent. Recently, it has been shown that fast Rietveld QPAs of coal fly ashes can be carried out with no sample spiking by initially calibrating the XRD signal from the glass [2]. In this work, we evaluate the usefulness of the calibration Rietveld-based approach on two different types of samples: fly ashes from coal combustion plants, and volcanic ashes. While the mineralogy of the fly ashes considered here is relatively simple (they mainly contain quartz, mullite and glass), the volcanic ashes contain sizable amounts of crystalline compounds with both simple and complex structures, including quartz, feldspars, biotite, pyroxene or iron oxides. We show that the calibration approach provides a suitable method to assess in a fast and consistent manner the amount of crystalline and amorphous phases in both types of samples. This method may be extended to industrial characterization processes involving large numbers of complex samples, reducing considerably the analytical times.

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