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Development of Total Reflection X-ray fluorescence spectrometry quantitative methodologies for elemental characterization of building materials and their degradation products

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ABSTRACT

In this work, a Total Reflection X-ray fluorescence (TXRF) spectrometry based quantitative methodology for elemental characterization of liquid extracts and solids belonging to old building materials and their degradation products from a building of the beginning of 20th century with a high historic cultural value in Getxo, (Basque Country, North of Spain) is proposed. This quantification strategy can be considered a faster methodology comparing to traditional Energy or Wavelength Dispersive X-ray fluorescence (ED-XRF and WD-XRF) spectrometry based methodologies or other techniques such as Inductively Coupled Plasma Mass Spectrometry (ICP-MS). In particular, two kinds of liquid extracts were analysed: (i) water soluble extracts from different mortars and (ii) acid extracts from mortars, black crusts, and calcium carbonate formations. In order to try to avoid the acid extraction step of the materials and their degradation products, it was also studied the TXRF direct measurement of the powdered solid suspensions in water. With this aim, different parameters such as the deposition volume and the measuring time were studied for each kind of samples. Depending on the quantified element, the limits of detection achieved with the TXRF quantitative methodologies for liquid extracts and solids were set around 0.01-1.2 and 2-200 mg/L respectively. The quantification of K, Ca, Ti, Mn, Fe, Zn, Rb, Sr, Sn and Pb in the liquid extracts was proved to be a faster alternative to other more classic quantification techniques (i.e. ICP-MS), accurate enough to obtain information about the composition of the acidic soluble part of the materials and their degradation products. Regarding the solid samples measured as suspensions, it was quite difficult to obtain stable and repetitive suspensions affecting in this way the accuracy of the results. To cope with this problem, correction factors based on the quantitative results obtained using ED-XRF were calculated to improve the accuracy of the TXRF results.

Keywords: Total Reflection X-ray fluorescence spectrometry, Cultural Heritage, building materials, solid suspensions.
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