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J. Hormes, A. Diekamp, W. Klysubun, G.-L. Bovenkamp, N. Börste

PII: S0026-265X(15)00304-5
DOI: doi: 10.1016/j.microc.2015.11.034
Reference: MICROC 2333

To appear in: *Microchemical Journal*

Received date: 14 August 2015
Revised date: 11 November 2015
Accepted date: 17 November 2015

Please cite this article as: J. Hormes, A. Diekamp, W. Klysubun, G.-L. Bovenkamp, N. Börste, The characterization of historic mortars: A comparison between powder diffraction and synchrotron radiation based X-ray absorption and X-ray fluorescence spectroscopy, *Microchemical Journal* (2015), doi: 10.1016/j.microc.2015.11.034

The characterization of historic mortars: a comparison between powder diffraction and synchrotron radiation based X-ray absorption and X-ray fluorescence spectroscopy

J.Hormes[a,b*], A. Diekamp[c], W. Klysubun[d], G.-L. Bovenkamp[b], N. Börste[e]

[a] Institute of Physics, Bonn University, Nussallee 12, D-53115 Bonn, Germany
[b] Center for Advanced Microstructures and Devices (CAMD), Louisiana State University, 6980 Jefferson Hwy., Baton Rouge, LA 70806, USA
[c] Material Technology Innsbruck, University of Innsbruck, Innsbruck, Austria
[d] Synchrotron Light Research Institute, 111 University Ave., Muang, Nakhorn Ratchasima 30000, Thailand
[e] Faculty for Theology Paderborn, Kamp 6, D-33098 Paderborn, Germany

Abstract

Three mortar samples from different construction phases of the Cathedral of Paderborn (Germany) between ~1015 and ~ 1220 AC have been investigated using three different and partly complementary techniques: conventional X-ray diffraction (XRD), synchrotron radiation based X-ray fluorescence (SR-XRF), and X-ray absorption near edge structure (XANES) spectroscopy. Samples were sieved and then investigated according to size: smaller and larger than 75 µm. For the small samples XRD detected $CaCO_3$ (calcite) and SiO_2 (quartz) as the most abundant compounds and $Na(AlSi_3O_8)$ (albite) as a minor component. No compounds containing transition metals (e.g. Fe) were observed by XRD. XRF results indicate just a marginal enrichment of binder compounds in the small samples as compared to the large ones. By XRF, in all samples Fe was observed at a very high concentration together with Ti and Zn at high concentrations and some other metals with lower concentration indicating that these metals are in an amorphous form and thus “invisible” for XRD. XANES spectra confirm the XRD result that most Ca exists as calcite, however at least in one sample (the “youngest” one) where the S-concentration is quite high also $Ca(SO_4)$ was detected via Ca-K and S-K-XANES spectra. Fe-K-XANES spectra indicated that Fe exists in the mortar samples with valency +3 as Fe_2O_3. Most likely, the SiO_2 grains are coated with iron oxides that have a high adsorption capability for trace metals explaining in this way the XRF detection of several metals at low concentration.

1. Introduction

Historical buildings are important landmarks of the architecture, culture and history of a specific region and/or community. Though of significant importance for the conservation and restoration of these buildings, investigations are still quite rare that provide detailed information about materials and technologies used for the fabrication of cements and mortars. This is particularly notable as mortars can also be an additional good source for dating of buildings and for determining potential past interventions [1 - 3].