Analysis of metakaolin for cost-effective decarbonized cement using ARL X'TRA Companion X-ray Diffractometer

Author

Dr. Simon Welzmiller, Application Specialist XRD

Introduction

Metakaolin, a highly reactive pozzolanic material derived from the thermal treatment of kaolin clay, plays a pivotal role in the development of decarbonized cement. Its incorporation into cement formulations significantly enhances the mechanical properties and durability of concrete while reducing the carbon footprint associated with traditional Portland cement. Metakaolin contributes to the pozzolanic reaction, consuming calcium hydroxide and forming additional calcium silicate hydrates, which improves the microstructure of the cement matrix.

X-ray diffraction (XRD) is an essential analytical technique used to characterize metakaolin. XRD provides detailed information on the crystalline phases present in the material, ensuring the quality and consistency of metakaolin used in cement production. By identifying and quantifying the mineralogical composition, XRD helps in optimizing the pozzolanic reactivity and performance of metakaolin in cementitious applications. By applying sophisticated methods like PONKCS (Partial or No Known Crystal Structure), quantification of even amorphous parts of a sample is possible in a routine analysis. This synergy between metakaolin utilization and advanced analytical techniques like XRD is crucial for advancing sustainable construction materials.

Instrument & software

The Thermo Scientific™ ARL™ X'TRA Companion X-ray Diffractometer (c.f. Figure 1) is a simple, easy-to-use benchtop XRD instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion Diffractometer uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by

Figure 1: ARL X'TRA Companion X-ray diffraction system.

divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available as an option. Thanks to the innovative solid state pixel detector (55 x 55 µm pitch), the ARL X'TRA Companion Diffractometer provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS (Laboratory Information Management System).

Experimental

Samples of kaolin clay and metakaolin calcined at 600, 700, 800 and 900 °C were ball milled and manually pressed into top loading sample cups. Measurements in reflection mode were performed using Cu Kα (1.541874 Å) radiation spinning sample (c.f. Figure 2). A calibrated peak (PONKCS) was used for quantification of the amorphous phase.

Results

XRD data of metakaolin calcined between 600 °C and 900 °C and kaolin is shown in Figure 2, highlighting major phases (see Table 1 for details). The different calcination temperatures have distinct influences on the phase composition and the amorphous content of the samples. Tracking these changes is crucial for the assessment of optimal process conditions and the product quality. Figure 3 shows the trends of some selected phases for the various temperatures.

One can see that during calcination at 600 °C microcline reacts but not all the kaolinite is activated, thus yielding a suboptimal result. At 700 °C, all the kaolinite is activated, also visible in the quartz and amorphous phase concentrations. Calcination at 800 °C also activates some of the mixed layer clay yielding higher quartz and amorphous phase concentrations and therefore increased pozzolanic activity. It is known that illite starts to collapse at temperatures above 800 °C which forms mullite. At 900 °C this reaction can be observed. Additionally, clinochlore and magnetite form, while biotite decomposes, overall resulting in a reduction of the amorphous phase.

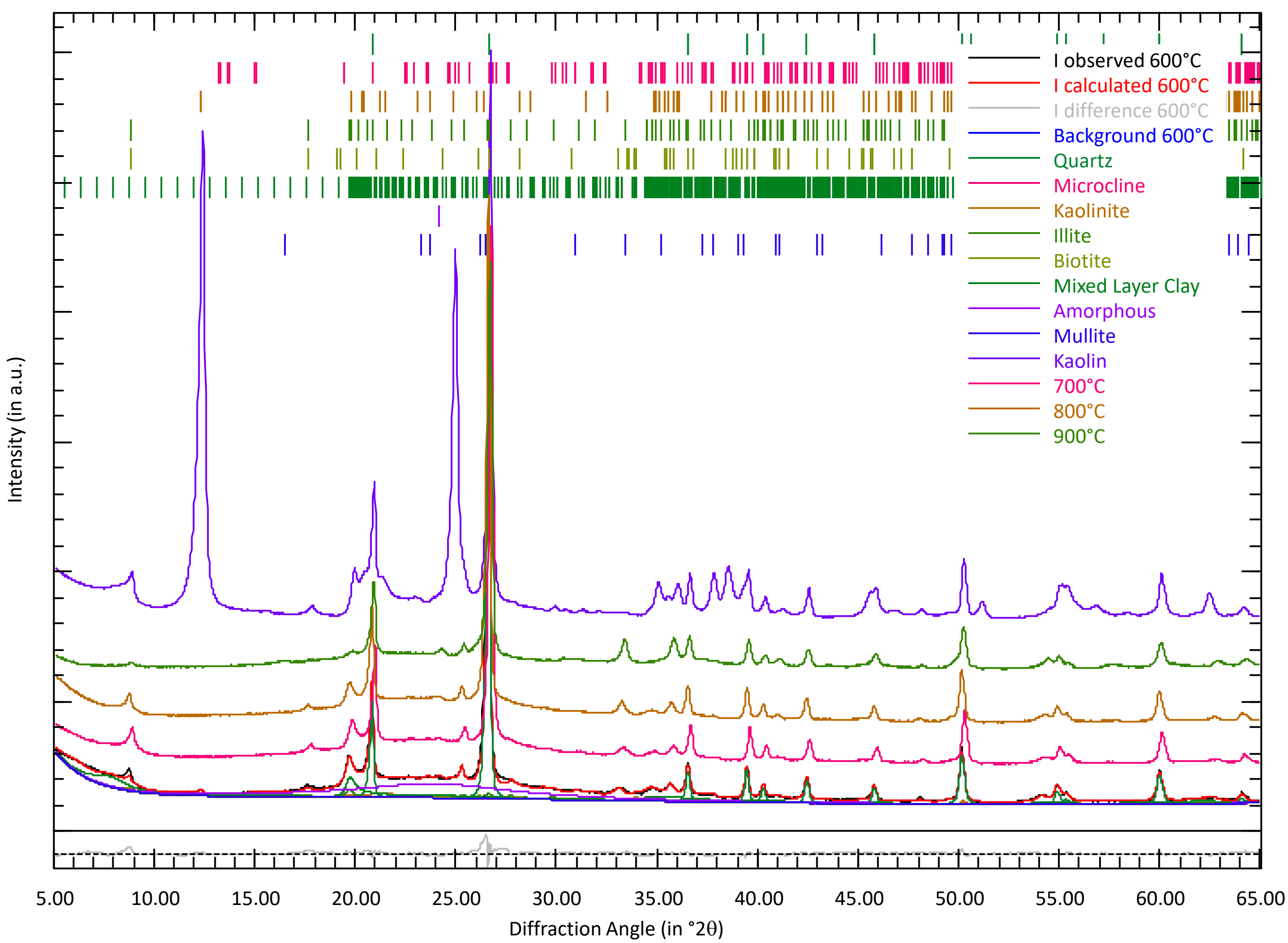

Figure 2: XRD patterns of kaolin clay (purple) and its products (metakaolin) calcined at 600 (black), 700 pink), 800 (yellow) and 900 °C (green); a fit for the 600 °C sample is shown; the refinements include up to 42 phases but only mayor phases are shown.

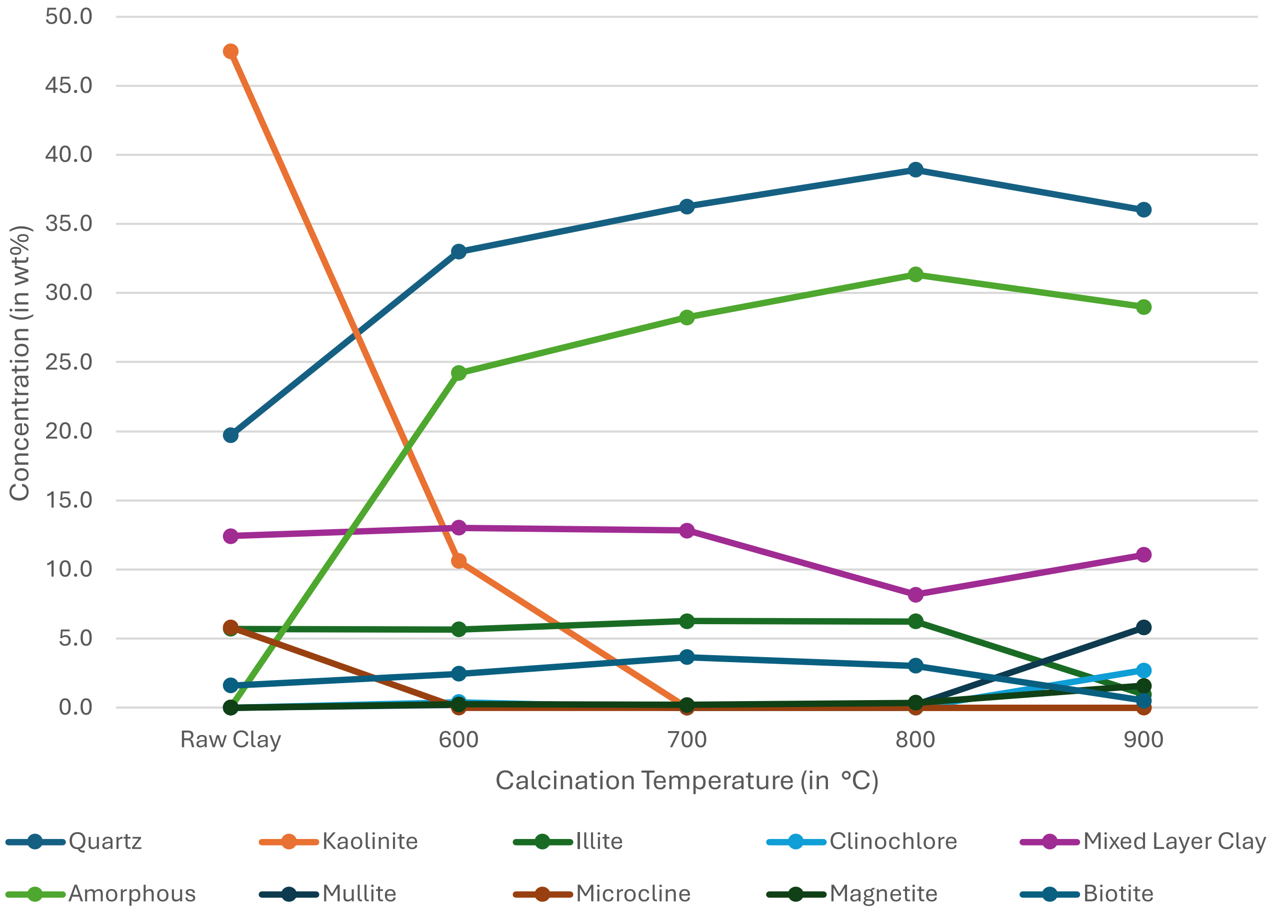

Figure 3: Evolution of phases in Kaolin clay and calcined samples using different calcination temperatures.

Table 1: Results of Rietveld refinement of kaolin and metakaolin samples calcined at 600, 700, 800 and 900 °C. Only major phases are shown.

Quantity (in wt%)	Quartz	Microcline	Kaolinite	Illite	Mullite	Mixed layer clay	Amorphous	Biotite	Magnetite	Clinochlore
Kaolin	19.7	5.8	47.5	5.7	0	12.4	0	1.6	0	0
600 °C	33	0	10.6	5.7	0.2	13	24.2	2.4	0.2	0.4
700 °C	36.3	0	0	6.3	0	12.8	28.3	3.7	0.2	0
800 °C	38.9	0	0	6.2	0.2	8.2	31.4	3	0.4	0
900 °C	36	0	0	0.9	5.8	11.1	29	0.5	1.6	2.7

Your benefits

The ARL X'TRA Companion XRD instrument yields data perfectly suited to analyze kaolin and metakaolin samples. Utilizing **one-click Rietveld refinement** for full quantification, enables **ease of use** for operators and **reliefs training constraints**. Following the evolution of key phases for different calcination conditions is crucial for **process optimization and assessment of the calcination stage** which **saves cost**.

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