# EVALUATION OF MECHANICALLY ACTIVATED KAOLIN AS ALKALI-ACTIVATED MATERIAL PRECURSOR

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Key Words: Mechanical activation, non-thermal dehydroxylation, kaolin, alkali-activated precursor.

The cement industry is one of the most polluting worldwide, therefore the finding of new binding materials is mandatory. In this regard, alkali-activated materials (AAMs) rise as one of the most promising alternatives to ordinary Portland cement. AAMs are produced through the reaction of aluminosilicate solid precursors with alkaline activator solutions, forming a gel that hardens resulting in a binder material. Many precursors have been assessed, and recently, the trend is to use waste or by-products as precursors since aside from reducing the carbon footprint in the cement production, second life is given to residues that otherwise would probably be disposed of. However, the use of waste and by-products presents some drawbacks, e.g., the variability of the composition of the waste depending on the place and the season, as well as the availability of waste. The huge demand of raw materials for the cement industry could not be reached with residues. Hence, the optimal precursor depends on the local availability of suitable raw materials [1]. Clays and clay minerals are worldwide available and can easily meet the raw materials' demand of the cement industry [2]. Nonetheless, the reactivity of clay precursors is low, and a previous activation of the material must be performed. The most common activation process is carried out through a thermal process, where the clay structure undergoes a dehydroxylation stage to remove the hydroxyls and amorphize the structure. Depending on the clay type and properties, the thermal dehydroxylation requires temperatures between 500 and 900 ºC. The energy consumption during this process is an important downside in front of waste-based precursors. Therefore, the activation of the clay precursor should be achieved with low energy-requiring processes. In this sense, mechanical activation (MA) arises as a feasible activation method, which could reduce the required energy compared to thermal dehydroxylation. Nevertheless, the reactivity of mechanically activated clays is barely studied. This work aims to assess the reactivity of mechanically activated kaolin for its use as precursor for AAMs production.

With this objective, commercial kaolin was exposed to a series of mechanical treatments using a planetary ball mill at different milling times (30, 60 and 120 min) and rotation speeds (250, 300 and 350 rpm). Moreover, thermal activation of the same raw material was performed to obtain a typical metakaolin (MK) as reference material. The obtained materials were studied through X-ray diffraction (XRD) with amorphous content calculation, infrared spectroscopy (IR) with band deconvolution, $^{27}$Al and $^{29}$Si nuclear magnetic resonance (NMR), and chemical attacks with NaOH 8 M to determine whether if their reactivity was equivalent to MK or not. The XRD analysis of raw kaolin showed that the main crystalline phases were kaolinite and quartz, with some impurities of illite and microcline. Through MA, first illite, then kaolinite and lastly microcline were transformed into amorphous phase, reaching in the samples treated with the most aggressive MA an estimated amorphous content higher than in MK. NMR revealed that the Al coordination is reduced from VI (octahedral sheet in kaolinite) to V and IV with MA, but with still some VI-coordination Al remaining. The deconvolution of the Si-O-T (T=Si, Al) band in IR enlightened the change in the kaolin's structure with MA, seen with a decrease of kaolinite bands' intensity together with the raising of new bands corresponding to the vibrations of the amorphous phase. Finally, the chemical attacks exhibited that the amount of Si and Al dissolved (potentially reactive for AAMs formation) for the mechanically treated samples is high but not higher than for the MK. The insoluble residue of the chemical attack revealed that only traces of kaolinite remained unreacted in some samples with less energetic MA, together with quartz and microcline. For the MK, the illite phase was not amorphized, and therefore is detected in its dehydroxylated form before and after the chemical attack in XRD.

In conclusion, mechanically activated kaolin presented high reactivity and therefore can be used as precursor for AAMs formulations. The increase of milling time and rotation speed leads to a more reactive material, mainly due to the collapse of the kaolinite structure.

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