

# Assessing the reactivity of magnesium oxide calcinated at different temperatures for understanding its role in degradation processes

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**Motivation**

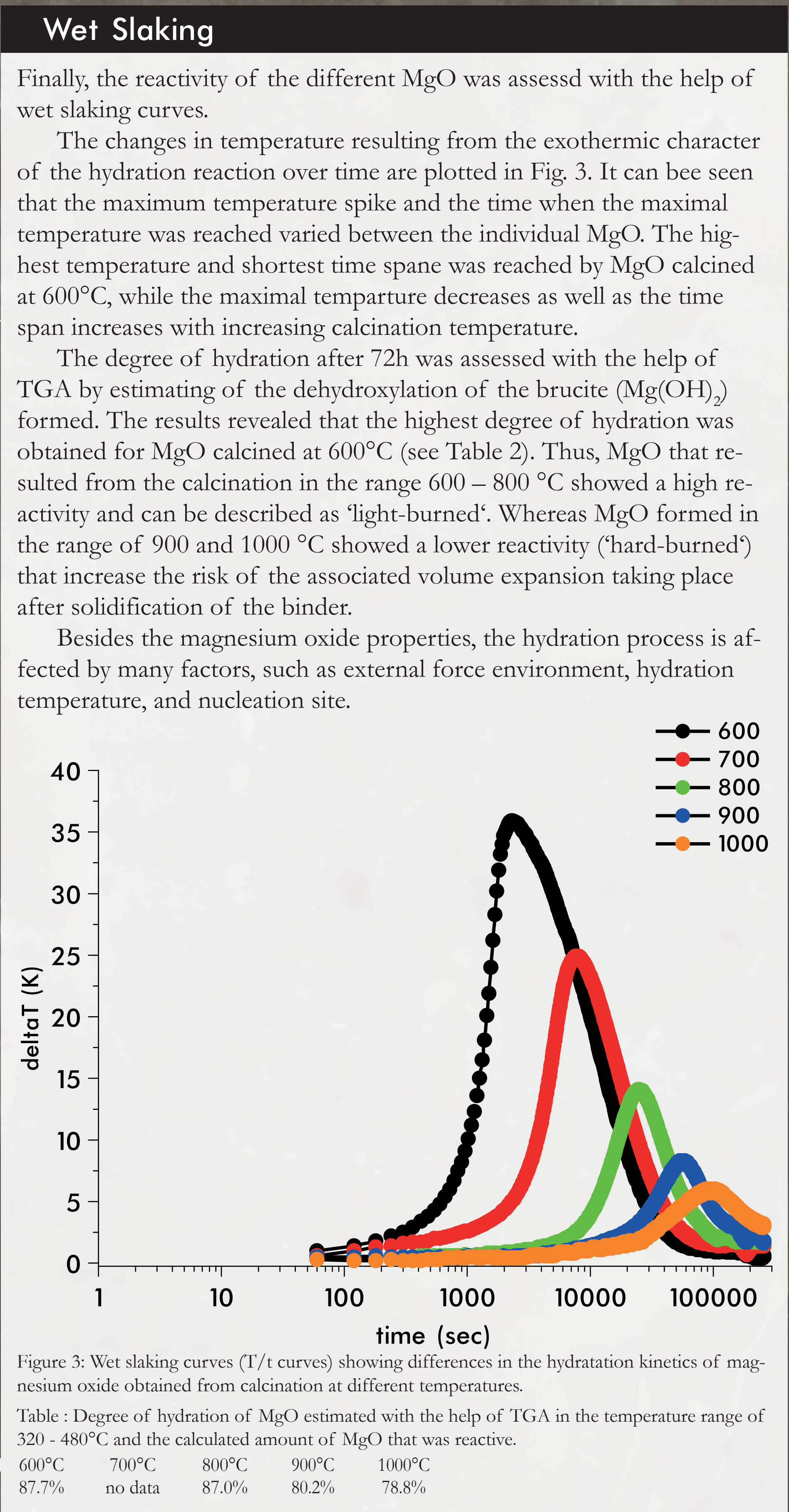
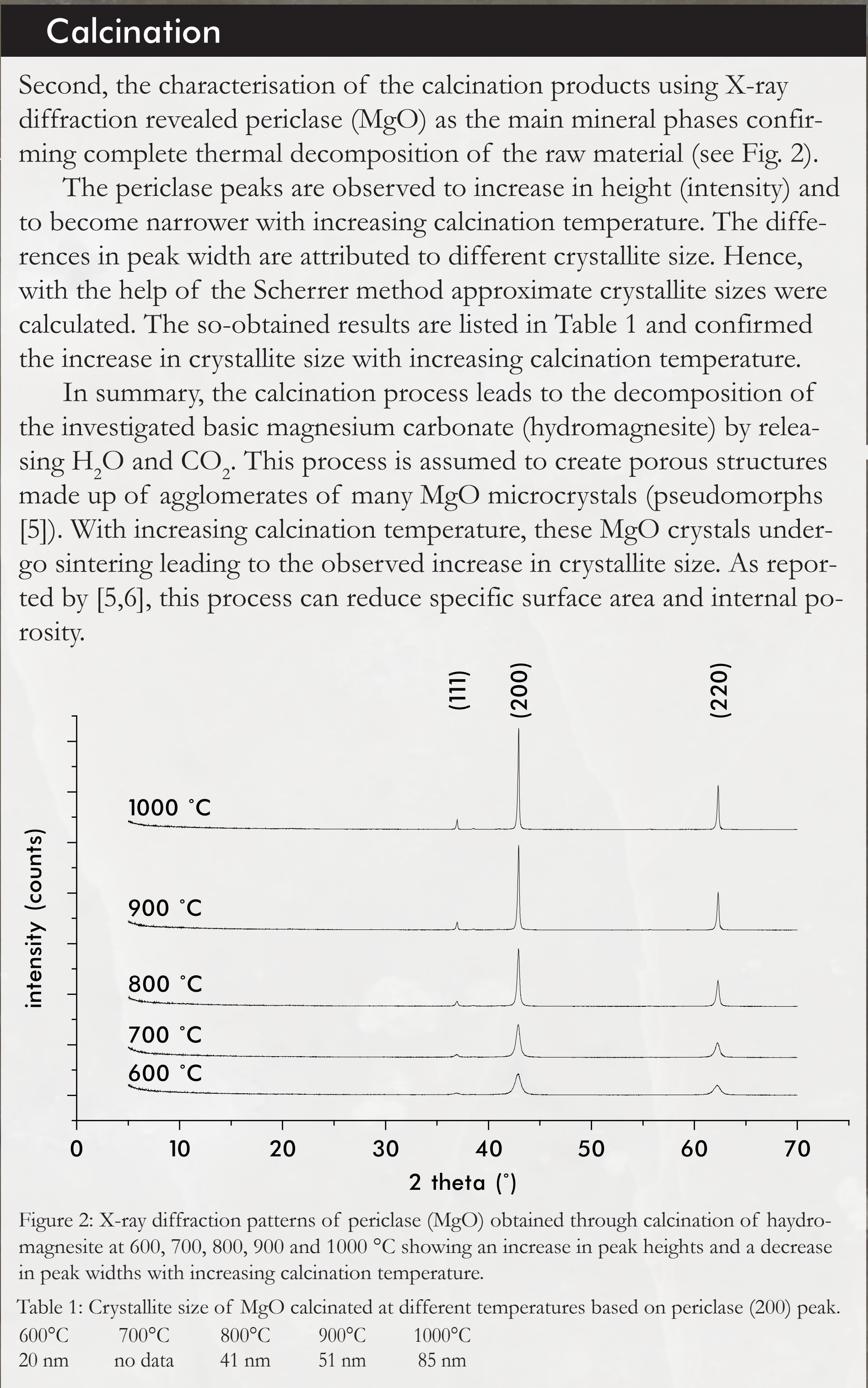
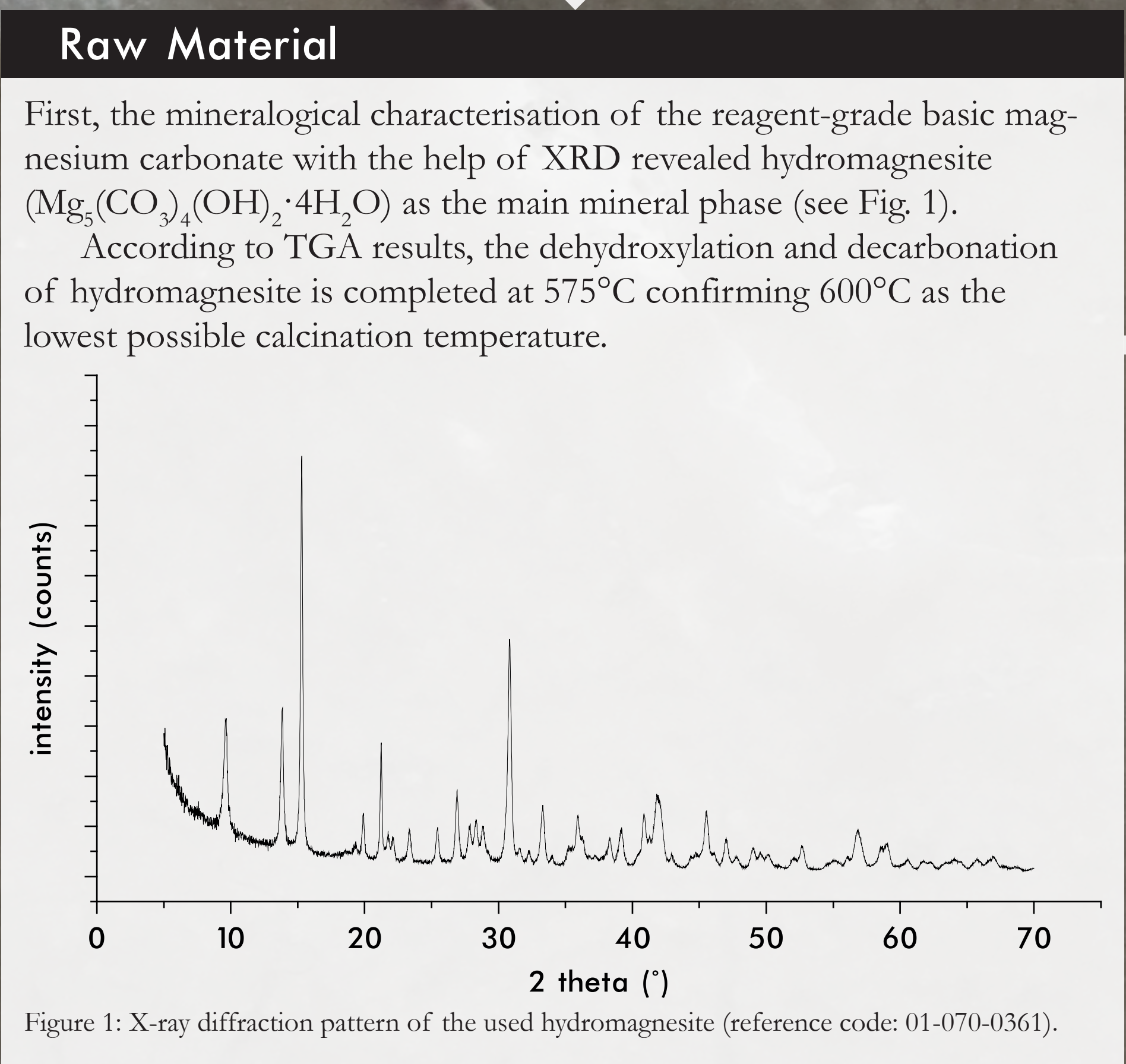
In Tyrol, Austria, dolomite rock was commonly used as raw material for the production of historic mortars and plasters [1]. During calcination of dolomite rock, almost equal amounts of calcium oxide (CaO) and magnesium oxide (MgO) are produced, which later will be wet slaked. While CaO hydrated rapidly, historic mortars occasionally contain non-hydrated MgO suggesting incomplete slaking. Over a large time span, these relicts of MgO react slowly with water to magnesium hydroxide. The associated volume expansion of about 118% takes place after the solidification of the binder leading to local spalling. The goal of this study is to assess the influence of the calcination temperature on the reactivity of MgO during wet slaking (= degree of MgO hydration). The gained insights are believed to be beneficial for a deeper understanding of hydration kinetics of dolomitic quicklime that can be later helpful for the use of light-calcinated dolomite rock as supplementary cementitious material (SCM) [2,3].

**Approach**

The present study assess the reactivity of magnesium oxide calcinated at different temperatures by recording the wet slaking curves. For minimising the influence of e.g. impurities or particle size on the calcination process, very fine powder of reagent-grade heavy magnesium carbonate basic (calcium <0.75% according to certificate of analysis) supplied by Sigma-Aldrich was used. The magnesium oxide was calcinated in steps of 100°C from 600 to 1000°C, subsequently quenched in air and characterised using X-ray diffraction (XRD) analysis. The wet slaking process was performed in a cylindrical dewar under continuous stirring (as proposed by [4]) and constant monitoring of the temperature. The degree of hydration was estimated XRD analysis and thermogravimetric analysis (TGA).

**Experimental Setup**

Calcination of the raw material for one hour was done in a laboratory electric muffle furnace (Nabatherm) with a heating-rate of 15 K/min. Wet slaking was performed with a MgO to water ratio of 1:4 (25 g MgO, 100 g deionised water) under constant mechanical stirring (500 rpm) in a cylindrical dewar (KGW-ISOTHERM) at ~20°C. The changes in temperature were recorded every 60 sec over a total time span of 72 h. XRD analyses were performed with the help of powder diffractometer Empyrean (Panalytical) working with Cu-K $\alpha$  radiation generated at 40 kV and 40 mA. Spectra were taken from 5 to 70° 2 theta with a step size of 0.013° and a counting time of 40 sec/step. Thermogravimetric analyses (TGA) was performed using Netzsch 449 F5 Jupiter from 25 to 1000°C with a temperature gradient of 10°C in a static air atmosphere (specimens dried at 105 °C before analyses).



**Conclusion**

The aim of the present study was to investigate the effect of the calcination temperature on the reactivity of the so-obtained MgO during the wet slaking process. The results revealed that the calcination temperature influences the degree of hydration. For the used experimental setup (permanent stirring at 500 rpm), the highest degree of hydration was achieved for calcination temperatures of 600 to 800 °C. For calcination temperatures of 900 and 1000 °C, sintering of MgO occurred, which is assumed to reduce, both, the internal porosity and the specific surface area resulting in lower reactivities (= lower degree of hydration). A low reactivity can increase the risk of non-hydrated MgO relics that induce damaging due to delayed expansion. This might limit the use of MgO obtained from light-burnt dolomite rock as SCM. At this point, it should be noted that these preliminary results were obtained using reagent-grade magnesium carbonate basic as raw material and, thus, further work on the reactivity of 'natural' MgO is required.

**Outlook**

Next steps comprises the hydration of MgO obtained from calcination of natural dolomite rock. Besides the presented experimental methods, scanning electron microscopy (SEM) will be employed to study morphological characteristics of the so-obtained MgO. The gained insights will be a first step to assess the use of light-burnt dolomite rock as SCM. Later, accelerated carbonation experiments will be carried out to examine the conversion of hydrated MgO (brucite) into magnesium carbonate hydrate phases.

**References**

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