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Importance of granulometry on phase evolution and phase-to-phase relationships of experimentally burned impure limestones intended for production of hydraulic lime and/or natural cement

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In contrast to modern ordinary Portland cement production from finely ground raw material blends, ancient burning of hydraulic lime was conducted by burning larger pieces of natural raw material. Due to natural variability of raw material composition, exploitation of different beds from even one formation can result the product with significantly different composition and/or properties. Prague basin (Neoproterozoic to pre-Variscan Palaeozoic of the central part of the Bohemian Massif – the so-called Barrandian area, Czech Republic) represents a classical example of the limestone-rich region with long-term history of limestone burning for quick lime and/or various types of hydraulic binders. Due to the fact that burning of natural hydraulic lime has been abandoned in this region at the turn of 19th/20th c., significant gap in knowledge on the behavior of various limestone types and on the influence of minor variance in composition on the quality of burned product is encountered. Moreover, the importance of employment of larger pieces of raw material for burning for the development of proper phase-to-phase relationships (i.e. development of hydraulic phases below sintering temperature at mutual contacts of minerals) has not been examined before. To fill this gap, a representative specimens of major limestone types from the Prague basin have been selected for experimental study: Upper Silurian limestone types (Přídolí and Kopanina Lms.), and Lower Devonian limestones (Radotín, Kotýs, Řeporyje, Dvorce-Prokop, and Zlíchov Lms.).

Petrographic character of the experimental material was examined by polarizing microscopy, cathodoluminescence, scanning electron microscopy with an energy dispersive spectrometer (SEM-EDS), and X-ray diffraction (XRD) of insoluble residue. Based on the data from wet silicate analyses, modal composition of studied impure limestones was computed.

Experimental raw material was burned in laboratory electric furnace at 1000 and 1200°C for 3 and/or 6 hours. Burned samples were examined by XRD for phase composition and by SEM-EDS for phase-to-phase relationships due to the burning. Based on our data it is evident that larnite-belite (dicalcium-silicate) is dominant phase in burned silica-rich limestones (represented by e.g. Dvorce-Prokop, Přídolí and/or Kopanina Lms.). In clay-rich limestones containing kaolinite and illite, gehlenite and other calcium aluminates and aluminosilicates were detected (represented by Kosoř, Řeporyje, and/or a portion of Dvorce-Prokop Lms.). Due to higher proportion of Fe-oxihydroxides in the Řeporyje Lms., brownmillerite (calcium aluminoferrite) forms as a typical minor phases during burning. Free-lime (plus its hydrated form – portlandite) makes dominant phase in limestones exhibiting low non-carbonate admixture (Kotýs and/or a portion of Kopanina Lms.). These results clearly demonstrate that presence of certain non-carbonate minerals governs formation of certain hydraulic phases in burned product, whilst mutual proportions of individual minerals in raw materials influence amount of newly formed phases.