



Characterization of early-age hydration processes in lime-ceramic binders using isothermal calorimetry, X-ray diffraction and scanning electron microscopy



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ABSTRACT

Early-age hydration processes in a lime-ceramic-water system are analyzed within the whole range of possible lime/ceramic ratios. The isothermal calorimetry shows a substantial effect of the heat of wetting on the total heat evolved, ranging from 10 J/g for lime to 3.9 J/g for ceramic. The highest specific hydration heat of 63 J/g during the analyzed 300-h hydration period exhibits the blended binder containing 70% ceramic and 30% lime which correlates well with the highest compressive and bending strengths of the paste prepared using this blend. Portlandite, ettringite and calcite are the main phases identified by the X-ray diffraction analysis after the hydration of ceramic-rich blends. According to the results of scanning electron microscopy, the initial course of pozzolanic reaction is for this type of binders characterized by the transition from tobermorite-like calcium-silicate-hydrate structures into jennite-like structures within the first 48 h. Blends with the ceramic content lower than 70% show a high portion of portlandite, calcite is present in low amount, and the jennite-like structures are observed after 48 h, following the initial formation of components with a very high Ca content. The favorable properties of the ceramic-rich blended binders can be explained by the partial hydraulic character of the ceramic. With the specific hydration heat of 29 J/g after 300 h and compressive strength of the paste 2.8 MPa after 30 days, the applied waste ceramic dust from the hollow brick production has good prerequisites for the application in lime-based binders with satisfactory engineering properties and good economical parameters for the building practice.

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1. Introduction

Lime plasters with the addition of pozzolanic additives have been frequently used as surface layers of building envelopes, in the past. They were utilized for both ordinary buildings and representative castles and palaces, including, e.g., the Roman catacombs [1]. Up to the 19th century most plasters were based on lime. Ordinary lime plasters could though not reach good mechanical properties. Therefore, they needed to be improved by additives which were of either natural or artificial origin. In Europe, volcanic rocks and ashes belonged to the most frequently used natural pozzolans [2,3] and powdered/crushed ceramics were the best known representatives of the artificial ones [4–6]; in ancient China natural organic compounds were preferred [7].

Today, lime-pozzolan plasters are used mainly for the reconstruction of historical buildings, where Portland cement is not desirable because of its incompatibility with the original materials. Metakaolin is probably the most often used pozzolan for this type of plasters [8,9] but quite a few applications of other potential additives such as siliceous waste materials [10], sticky rice [11], wastes of the sugar industry [12], rice husk ash [13], tuff [14], zeolite [15], burnt clay shale [16], or enamel glass [17] were reported by various investigators. The apparent advantages of ceramic powder as a pozzolanic additive to lime, which were well known from the ancient times, were confirmed in a recent study [18] as well.

The hydration processes in lime-pozzolan-water systems were investigated by various methods in the past. Thermal analysis was frequently used for that purpose as the principal method, sometimes in a combination with X-ray diffraction analysis. The studied types of binders were lime-silica fume [19], lime-fly ash [20], lime-metakaolin [21,22], lime-natural pozzolan [23], and also lime-ceramic [24,25], to give a couple of characteristic examples.

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Less traditional methods applied for the analysis of hydration processes in lime-based systems were Raman spectroscopy [26], nuclear magnetic resonance spectroscopy [27], or electrochemical impedance spectroscopy [28]. Calorimetric studies were reported for the lime-metakaolin [22], lime-glass [29] and lime-zeolite [30] binders. Investigations of the hydration heat development in lime-ceramic blends were not found in the common literature sources.

In this paper, the hydration processes in the lime-ceramic-water system are analyzed using a combination of isothermal heat flow calorimetry (IHFC), X-ray diffraction analysis (XRD), scanning electron microscopy (SEM), and mechanical tests. IHFC is applied as the primary method. Monitoring the time development of hydration heat and analyzing the measured time variations of the specific hydration heat power (it characterizes the rate of hydration heat generation) makes possible to determine the basic time frame of the reactions occurring in the system. XRD and SEM are then used for the identification of the most important hydration products in the characteristic time intervals indicated by IHFC. The specific hydration heat as an integral quantity (integral of the specific hydration heat power) provides the information on the total heat evolved in the system up to a selected time. If a sufficiently long time period is chosen, this value can be considered as a measure of the extent of the pozzolanic reactions; thus it can help to identify the prospective lime-ceramic ratios. The results of the IHFC measurements are then utilized in yet another way. Using the specific hydration heat power and specific hydration heat measured for different lime-ceramic ratios, the difference specific hydration heat power and difference specific hydration heat per 1 g of ceramic are calculated. This data enable a more detailed analysis of the course of pozzolanic reactions in the system. It results in the identification of additional time intervals where the material characterization by XRD and SEM experiments is to be done. The effectiveness of the ceramic as a pozzolan for various lime-ceramic ratios can also be estimated in this way. Finally, the mechanical properties of lime-ceramic pastes are determined to assess the applicability of the analyzed blends in the building practice.

2. Experimental methods

The hydration heat development in the analyzed lime-ceramic-water systems was determined by a large-volume isothermal heat flow calorimeter KB01, which was developed at the Department of Materials Engineering and Chemistry, Faculty of Civil Engineering, Czech Technical University in Prague [31]. The reason for its application was the presumably slow hydration heat development; in such cases this type of calorimeter can achieve a higher accuracy than standard calorimeters working with small samples [22]. Before the experiment, the lime and ceramic were mixed together in the required ratio, weighted, placed in a polyethylene bag and closed tightly by a rubber band. Water was stored in a tight polyethylene bottle. Then, the temperature field in the calorimeter containing separated components was equilibrated for 24 h. Afterwards, the components were taken out of the calorimeter and mixed together for 3 min in the polyethylene bag, which was then sealed, weighted and put into the measuring vessel of the calorimeter. The output signal was recorded during the whole process of removing components, mixing, reinserting it and closing the apparatus. The duration of the initial phase of the procedure could thus be identified from the record. The measurement itself began about three and a half minutes after mixing the components, which was necessary for the specimen's fixing in the calorimeter. The time dependence of the hydration heat power (in mW) obtained as the measurement output was then recalculated to the specific hydration heat power N (in mW g^{-1}) and integrated to obtain the total hydration heat (in J) or the specific hydration heat Q

Table 1
Chemical composition of applied materials.

Material	Ceramic dust	Lime hydrate
SiO ₂	51.3	0
Al ₂ O ₃	20.0	0
Fe ₂ O ₃	6.0	0
CaO	11.5	99.3
MgO	4.5	0.5
K ₂ O	3.2	0
Na ₂ O	1.3	0
TiO ₂	0.8	0
SO ₃	1.0	0.1

(in J g^{-1}) as a function of time. The mass of the studied samples was ~ 350 g and the monitoring of hydration processes was performed for two weeks in an air-conditioned laboratory at the temperature of $(25 \pm 0.5)^\circ\text{C}$.

The chemical composition of applied materials was examined by the X-ray fluorescence method. A Thermo ARL 9400 XP (Thermo ARL, Switzerland) device was used in the measurements. The X-ray diffraction analyses were performed using a PANalytical X'PertPRO diffractometer (PANalytical, Spectris plc, Egham, Surrey, England) equipped with $\text{CoK}\alpha$ X-ray tube (40 kV, 30 mA). Diffractograms were collected in the range of 2θ 5–41° with a step of 0.017° at 1000 s/step (scan time 5 h). Soller slit of 0.02 rad, divergence slit of 0.5 and anti-scatter slit of 0.5° were used in the primary beam, Soller slit of 0.02 rad and Fe beta filter in the diffracted beam. A fast linear detector X'CeLerator was applied in the experiments. The quantification was performed using an internal standard (zincite ZnO). The data were evaluated by HighScorePlus software package (version 3.0.5) and JCPDS PDF2 database. A JSM-6510 (JEOL Ltd., Tokyo, Japan) device was used for the SEM studies. Samples were coated by carbon, accelerating voltage was 20 kV, backscattered electrons were detected by EDS SDD x-act 10 mm² INCA detector (Oxford Instruments).

A standard three-point bending test [32] was utilized for the determination of bending strength of lime-ceramic pastes, using an MTS QTest 100 (MTS Systems Corporation, Cary, North Carolina) machine for samples with the dimensions of 40 mm × 40 mm × 160 mm. The span length between the supports was 100 mm, the loading rate 0.15 mm/min. The compressive strength was determined on the fragments of specimens left over after the bending test.

3. Materials and samples

A commercially produced CL90-S lime hydrate ($d_{50} = 8.1 \mu\text{m}$, Blaine specific surface 1374 m²/kg) and waste ceramic dust ($d_{50} = 7.5 \mu\text{m}$, Blaine specific surface 665 m²/kg) originating from grinding of hollow bricks were applied for sample preparation. The chemical composition of both materials, as obtained by the XRF fluorescence method, is shown in Table 1. The high amounts of silica and alumina in the ceramic dust and high purity of the lime presented good prerequisites for their successful mixing in a blended binder. The XRD analysis of phase composition (Table 2) showed that the ceramic dust consisted of quartz, feldspars (albite, microcline) and muscovite as the main minerals but also a significant fraction of amorphous compounds was observed, resulting from the clay thermal decomposition during the brick production. The lime hydrate contained over 96% $\text{Ca}(\text{OH})_2$, 2% calcite, less than 1% $\text{Mg}(\text{OH})_2$ and less than 1% $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$.

The lime: ceramic ratio in the studied systems was 0: 1 (it will be denoted as CD in what follows), 0.1: 0.9 (CL90), 0.3: 0.7 (CL70), 0.5: 0.5 (CL50), 0.7: 0.3 (CL30), and 1: 0 (LM), the water/binder ratio was equal to 0.6 in all cases.

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