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Comparative properties of a lime mortar with different metakaolin and natron additions

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HIGHLIGHTS

- Lime increases (10–30%), on natron-MK adjusted mix, improve resistance.
- In lime-natron-MK adjusted mortars, C-H has not been detected.
- Lime increases on initial geopolymer result in more homogeneous microstructure.

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ABSTRACT

The heterodox hypothesis about the construction of the pyramids in the Old Kingdom of Egypt with cast stone has served as a starting point in the design of several blends of aerial slaked lime mortar for their potential use in restoration events. Aerial slaked lime mortar characteristics with added metakaolin and sodium carbonate were analysed. Increments in the ratio of lime on adjusted initial proportions, corresponding to a theoretical geopolymer, were then made. Seven blends were characterized: two of them corresponding to different a water/binder ratio geopolymer and the rest to 5, 10, 30 and 60% lime increments. Finally, a lime mortar remained without additions.

10 and 30% lime increases on an adjusted specimen of sodium carbonate, metakaolin and slaked lime mainly improve Shore C hardness and compressive strength when compared both to the theoretical geopolymer and to conventional lime mortars. These become the most influential factors within a month, and values in this interval are suggested to improve these characteristics. TGA/DSC, XRD and SEM/EDS analyses show absence of portlandite and an improvement in the continuity of the microstructure in these values, yielding closer results to a polymer rather than to a conventional lime mortar.

1. Introduction

1.1. Background

Searching for suitable mortars for the conservation of heritage masonry has become a commonplace research area in recent years. In this sense, a main streamline is the study and use of lime mortars, assuming they are usually more appropriate and similar, in terms of their permeability and hardness, to the heritage mortars to be restored. Such studies range from the analysis of heritage mortar specimen components [1–5] to the characterization of new mixes in anticipation of alternative uses [6–9]. The present study focuses on the analysis of aerial slaked lime mortars with added metakaolin (MK) and sodium carbonate for a resulting material potentially suitable for the restauration of traditional stone masonry.

Heritage masonry buildings undergo degradation due both to environmental factors and to construction materials (brick, stone, mortars). This makes the intervention on the physical or chemical source of these lesions necessary. Lime mortars in such buildings have, indeed, proved compatible with other materials for long time periods; these mortars have also shown their efficiency under environmental and mechanical stress, which makes it interesting to simulate them instead of using other industrial materials which have caused more severe damage than the damage they were to restore [10]. Mortar intervention techniques in building restoration nowadays (injection, grouting…) are, however, understood as essentially different from the mortar techniques in place at

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the time of construction [11]. Deadlines in the construction process do also differ in these restoration events, as lime mortars are to be added for more compressive strength in shorter time periods.

Lime mortar designs currently used for heritage building restoration may mirror as a starting point the technology sustained by preceding cultures, updating, however, some of their characteristics both to new uses as well as to implementation. In this regard, chemistry of natural pozzolans used in combination with lime-mortars in ancient civilizations is well known, such as Santorin earth, in Greece, or Vesuvius pozzolans, in Italy, described by Vitruvius in the 1st century CE. Our attention was drawn, however, by the heterodox constructive hypothesis developed by French scientist Joseph Davidovits, where he described a cast stone construction system for the pyramids of the Old Kingdom of Egypt; he aimed at a global explanation, based on science, experimentation, archaeology, religion and hieroglyphics. In terms of construction materials, he concluded that they consist of natural limestone disaggregated by the water of the River Nile, which was subsequently re-agglomerated by means of a binder derived from lime, natron and kaolinitic clay, as if it were "concrete" [12].

The hypothesis for the present paper has given rise to all sorts of news, editorials and research articles, either from supporters or opponents, in the last three decades [13–21]. It has, in turn, become a pioneering step towards a hybrid or mixed construction theory, which puts forward the use of both carved stone and cast stone. The latter was used in combination with diatomaceous earth instead of the kaolin used in the genesis theory [22–24]. In opposition to the mixed or hybrid construction hypothesis, which did not specify clearly the process to obtain the artificial stone, Davidovits suggested the materials which might have been in use for the production of such conglomerate, along with the chemical reactions obtained. These were, in turn, used as the theoretical foundation for the experiment. The resulting binder was an inorganic polymer known as geopolymer, a term proposed by Davidovits which suits the purpose of the present paper, even when it could be taken as a trade name within alkali-activated binders.

1.2. Theoretical basis

According to Davidovits [12], the binder used to manufacture the conglomerate limestone of the Khufu pyramid would be constituted by natron (sodium carbonate anhydrous), kaolinite clay, slaked lime (calcium hydroxide) and carnallite (magnesium chloride). When these raw materials are mixed in water, they develop four basic chemical reactions, resulting in a geopolymer composed by hydrated feldspathoid, mica-chlorite, magnesite, calcite and halite (Fig. 1).

Alkali-activated aluminosilicate inorganic polymers are formed by joint polymerization of alumina and silicate species under the following defining empirical formula [25]:

$$M_n(-(SiO_2)_Z - AlO_2)_n, wH_2O$$

where M stands for an alkali cation (Na^+, K^+, Li^+, Ca^{++}, Ba^{++}, NH_4^+, H_3O^+), n stands for the degree of polycondensation, z usually stands for 1, 2 or 3, although it may be higher and w stands for the number of water molecules.

1.3. Preliminary considerations

Reactions (3) and (4) (Fig. 1) show that the addition of magnesium chloride (carnallite) decompose in magnesium, less reactive and soluble than sodium, and mononegative chlorine ions that reduce the pH of the mix; this allows the handling of the fresh mortar without protection. The appearance of salts in the specimens was avoided and finally magnesium chloride was not used. Therefore, the two basic chemical reactions in the experimentation would be (1) and (2).

Another factor has been considered is the theoretical determination of the binder in order to generate the mixes corresponding to a "pure" geopolymer. The stoichiometric calculation is done taking into account that the transformation of substances in chemical reaction is a phenomenon that occurs molecule by molecule; therefore, in the second reaction the caustic soda initially originated should merge completely with kaolin and disappear.

The replacement of kaolin of the initial hypothesis (Fig. 1) by metakaolin, a derivative with greater reactivity, was eventually adopted. Its pozzolanic activity makes it particularly interesting for the production of mortars and concrete.

Starting from a mix which is in accordance with the theoretical base, lime amounts are increased in the following mixes while sodium carbonate and MK are reduced proportionately; by doing so, all sodium carbonate combines in the chemical reactions. Were only lime amounts increased without compensating at the same time the MK percentage with natron, the increase in the binder proportion would result in lime and MK increases; thus, the transition between the geopolymer and conventional lime mortars could not be assessed, as an actual comparison between the geopolymer and conventional lime mortar with added MK would be obtained. The aim for the analyzed lime mortars is, first, to show

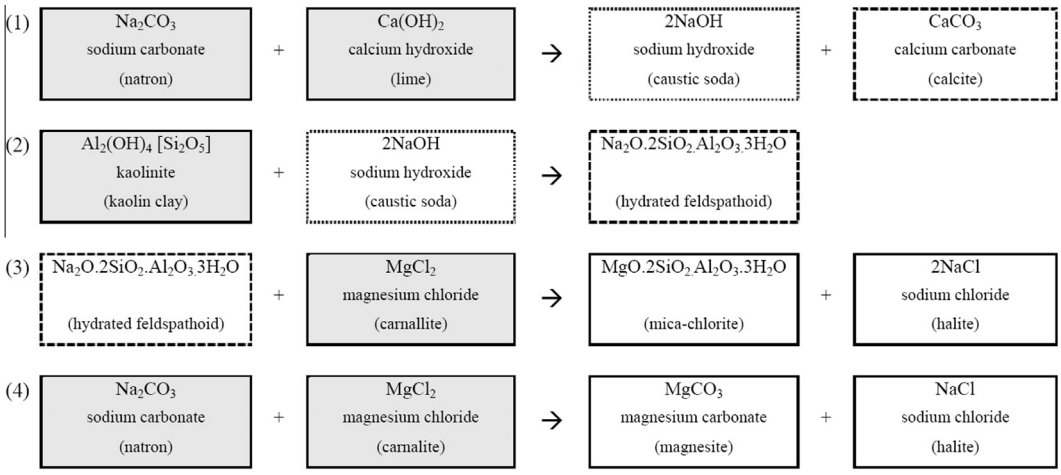

Fig. 1. Reactions of the historical constructive hypothesis according to Davidovits [12].