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The influence of calcium content on the performance of metakaolin-based geomaterials applied in mortars restoration

F. Allali [a], E. Joussein [b], N. Idrissi Kandri [a], S. Rossignol [c,*]

[a] Laboratoire de la Chimie Appliquée, Faculté des Sciences et Techniques B.P. 2202—Route d'Imouzzer, Fes, Morocco
[b] Université de Limoges, Groupement de Recherche Eau Sol Environnement (GRESE) EA 4330, Faculté des Sciences et Techniques, 123 avenue Albert Thomas, 87060 Limoges cedex, France
[c] Université de Limoges, Science des Procédés Céramiques et Traitement de Surface (SPCTS), UMR CNRS 7315, Ecole Nationale Supérieure de Céramique Industrielle (ENSCI), 12 rue Atlantis, 87068 Limoges cedex, France

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ABSTRACT

The architectural heritage needs to be preserved, especially coatings, because they communicate the historical building as it was created. The aim of this work is to develop a stable and durable restore coating mortar which would be compatible with the traditional elements of masonry, based on metakaolin-geopolymer binder mixed with traditional material of restoration. To do this pure metakaolin substitution in the Si-Na and Si-K geopolymers formulation were done by different amount of calcium hydroxide (lime) and calcium carbonate (calcareous sand). The feasibility of their use in synthesizing geopolymers and investigations of their properties were performed using FTIR, XRD, and compressive strength. Briefly, the results traduce the role of $CaCO_3$ as binder quasi without dissolution in alkaline solution whatever the cation used (Na- or K-based), the polycondensation reaction is effective in this case. Using the calcium hydroxide $Ca(OH)_2$, the reaction is dominated by hydration process which clearly limit the polycondensation one for the short time (up to 1 h). The potential use of geopolymer binder as restoration mortar for historical building is then effective since the reaction seems to be quite effective with calcareous sand easily accessible with a low cost.

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1. Introduction

The rehabilitation of historical buildings is very important in terms of history and culture of cities that reflect the characters of each region. According to this, architectural heritage preservation has reached a rising interest for scientists, architects, engineers and archaeologists. Since a long time, the most widely used binder in the construction activity were lime based mortars (e.g. calcium lime, pozzolan lime, hydraulic lime; [1]. So quasi all the historic monuments are lime based binder and need same composition for their restoration one. However, several authors indicated that the use of concrete mortars in our cultural heritage showed several disadvantages. Indeed, the crystallization processes of salts present in the cement-based mortars can induces damage of the stone, or also the presence of structural problems, when movements of building is effective, due to the low flexibility of the cement-based mortars [1,2,3,4]. According to these facts, it will be interesting to obtain a palliative of cement-based concrete having nice properties (acidic resistance, no leaching materials, high compressive strength…). In Morocco, an important number of monuments are inscribed on the World Heritage List. Unfortunately these built heritages are subjected to a number of potentially harmful deterioration agents during their long lifecycle. As well known, the long-term exposition to air pollution, rainfall, freeze–thaw cycles and, most of all, salt crystallization may cause significant damages to the constituent materials of the masonries [5,6], especially coatings because they are the most exposed to environmental degradation factors. It is therefore necessary to develop strategies of monument restoration based on new insights. Moreover, it is well known that the restoration of cultural heritage is realized using natural raw materials which are localized near the monument. So the new concrete can be realized with classical raw material largely found around the world. The kaolin is a well done candidate. One of the most innovative applications for kaolin's is their use as inexpensive raw materials in the synthesis of geopolymers. Indeed, there has been increasing interest in producing eco-friendly geopolymers from natural, inexpensive clayey materials [7,8,9]. Geopolymers are new materials that are obtained through the activation of aluminosilicates and alumina- and silica-rich materials such as calcined clays which are used as precursors [10] and appear to be a promising alternative for conventional cementitious materials due to their hydrothermal stability and resistance to acidic environments and/or reducing agents [11,12]. They are also known for their promising mechanical and thermal properties [13,14].These new materials exhibit similar properties of natural rocks. So, it's could be a good alternative of classical material in restoration work [15]. However, the presence of secondary minerals in kaolin ore deposit is quite effective inducing no pure kaolin except specific

* Corresponding author.
E-mail address: sylvie.rossignol@unilim.fr (S. Rossignol).

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Table 1
Characteristics of the raw materials used.

Raw material	Chemical composition (wt%)
Metakaolin M1	55 SiO_2; 40 Al_2O_3; 0.8 (K_2O + Na_2O); 1.4 Fe_2O_3; 1.5 TiO_2; 0.3 (CaO + MgO)
Calcitic sand	84.9 CaO; 6.4 SiO_2; 4.5 Al_2O_3; 1.4 Fe_2O_3

site. So it is difficult to obtain a constant elemental composition. Moreover, the use of high purity metakaolin in the geopolymer for restoration can induce some differences in term of colors, and aspects which is a disadvantage. One of the solutions is to use not pure natural raw material. The objective of this work is to develop a stable and durable repair coating mortar which would be compatible with the elements of masonry, using a metakaolin-geopolymer binder mixed with traditional material of restoration (lime and calcareous sand: Ca-rich component). Since geopolymer is traditionally devoid of calcium, it is necessary to focus on the effect of pure metakaolin substitution in the Si-Na and Si-K geopolymers formulation by different amount of calcium hydroxide (lime) and carbonate in the formulation in the way to understand the calcium behavior in the geopolymer binder. First, the physical and chemical properties of the raw materials were briefly investigated, and then introduced in the geopolymer formulation. The feasibility of their use in synthesizing geopolymers and investigations of their properties were performed using FTIR, XRD, and their compressive stress was also evaluated.

2. Materials and methods

2.1. Mortar designs and mixing procedures

Two raw materials designed for the coatings restoration were used to perform two sets of consolidate materials: a calcareous sand ($CaCO_3$) and calcium hydroxide ($Ca(OH)_2$). The calcareous sand is not pure calcite and contains quartz and a little amount of dolomite and muscovite. Moreover, a metakaolin M1 supplied by Imerys was also used in the formulation of mortar. The characteristics of the raw materials used in this study are described in Table 1. Various types of geomaterials were synthesized by substitute metakaolin M1 with (i) calcareous sand or (ii) calcium hydroxide in the formulation of an ordinary geopolymer using a Na or K alkaline silicate solution supplied by Woellner. In the way to compare, the same Si/M ratio (Si/M = 1.7; M = Na or K) was used for the two silicate solution. The protocol used is described in Fig. 1. Briefly, pellets of NaOH (VWR, 97% pure) or KOH (VWR, 85.2% pure) were dissolved in sodium silicate or potassium silicate inducing a Si/M ratio near 0.89 and 0.58 respectively. Then, silicate solution synthesized was mixed with metakaolin M1 and alternative materials (calcareous sand or calcium hydroxide) in the way to increase the Ca/Si ratio for each component. The reactive mixture was homogenized and then placed in an opened sealed mold in ambient air (25 °C, 40% R.H.). The nomenclature used for the consolidate materials obtained with calcareous sand is $MK_{1-x}(CaCO_3)_x$ SiNa and $MK_{1-x}(CaCO_3)_x$ SiK for a MK_1 – $CaCO_3$ substitution with sodium or potassium silicate solution, respectively. Same nomenclature is applied with calcium hydroxide – MK substitution i.e. $MK_{1-x}(Ca(OH)_2)_x$ SiNa and $MK_{1-x}(Ca(OH)_2)_x$ SiK.

2.2. Characterization technics

Chemical composition of each raw material was performed from X-ray fluorescence (XRF) analyses using an XMET 5100 device commercialized by OXFORD Instruments. Acquisitions proceeded from pressed pellets for 300 s time analyses. The synthesized materials were characterized from CarMaLim platform (University of Limoges, France) using X Ray diffractometer with a BRUKER AXS D8 Advance powder diffractometer using CuKα radiation ($\lambda K\alpha = 0.154186$ Å) over the 2θ range from 5° to 70° with an acquisition time of 2 s and a step size of 0.04°. Files from the Joint Committee on Powder Diffraction Standards (JCPDS) were used for phase identification. Infrared spectroscopic investigations were realized from a Thermo Fisher Scientific 380 infrared spectrometer (Nicolet) using the attenuated total reflection (ATR) method. The IR spectra were gathered over a range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1}. The acquisition of spectra has started just after the mixing. The atmospheric CO_2 contribution was removed with a straight line between 2400 and 2280 cm^{-1}. To monitor the geopolymer network formation, a software was used to acquire a spectrum (64 scans) every 10 min for 13 h. The software realized a spectral acquisition in an automatic way at regular intervals.

The compressive strength of samples stored for 7 days were determined using LlyodEZ20 machine moving at constant cross-head displacement of 0.2 mm/min. Test tubes, used for the compression tests, were cylindrical in shape with a diameter (Φ) of 15 mm and a height (h) of approximately 35 mm and were aged in closed mold at room temperature. The samples were demolded and rectified just before the tests. Samples were rectified by means of a diamond wheel to obtain perfectly parallel and flat faces. All values presented in the current work are an average of 10 samples. The experimental error is obtained from the average of standard deviation.

3. Results and discussion

3.1. Calcareous sand-based geomaterials

3.1.1. Calcareous sand and metakaolin mortar from sodium silicate solution

The infrared spectra of the various mixtures from metakaolin – calcareous sand with sodium silicate solution are reported in the Fig. 2a.

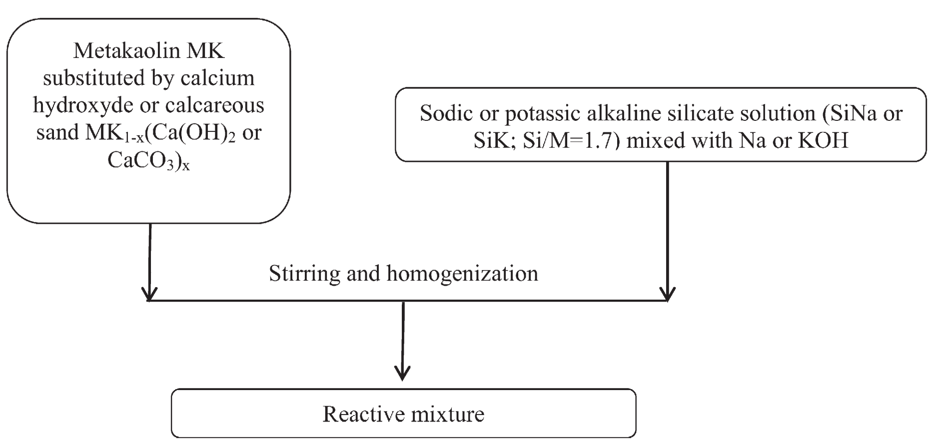

Fig. 1. Synthesis protocol of consolidated materials.

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