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Comparative study of the various methods of preparation of silicate solution and its effect on the geopolymerization reaction

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ABSTRACT

This paper is based on the characterization of synthesized geopolymer binders based on either powder or solution silicate, and the amount of water contained in synthesized binders is determined to evaluate their possibility to coat a brick. The structural evolution of the formed geopolymers was investigated using FTIR spectroscopy. The mechanical properties were evaluated using compression tests. The structural evolution ensured that the solutions prepared from silicate powder or liquid had different degrees of polymerization, which modified the polycondensation reaction of the mixture. Nevertheless, the use of aluminosilicate solutions based on powder or liquid display similar behavior in a polycondensation reaction. The obtained materials show good mechanical properties, and it is possible to deposit this binder on the brick depending on the water content.

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Introduction

The building and construction sectors have been identified among the major contributors to a global environmental impact owing to their high energy consumption [1,2]. This sector accounts for approximately 40% of total energy consumption and 38% of CO_2 emissions. Hence, it is necessary to adopt a global approach in this industry for material and building lifecycles and create this approach with a sustainable development perspective. Thus, the development of new composite materials called eco-materials that are composed of earth bricks, wooden frames and geopolymer binders can be considered as a constructive solution [3]. The idea of assembly with wood and earth bricks is used in many existing structures for construction systems owing to their good mechanical properties and the light weight of wood. Earth bricks also provide thermal inertia and stored moisture despite the cracks that may appear at the interface [4]. To inhibit crack formation, an inorganic mineral mortar, such as a geopolymer binder, can also be used because of its ecological properties [5] with respect to this type of structure. Indeed, a preliminary study on a porous geopolymer binder developed in the laboratory showed the ability of the binder to adhere to both wood and earth [6].

This new class of material called geopolymer binder features ill-organized, three-dimensional materials that result from the activation of an aluminosilicate source using an alkaline solution. Many investigations have used common clays [7,8] and industrial waste as raw materials [9,10]. Among these materials, metakaolin is the most commonly used because it is the cheapest aluminosilicate with a good degree of purity and high reactivity [11]. The existing literature [12] has proved that through the dehydroxylation process, the effect of impurities is considered an indicator of metakaolin reactivity. Further, the activating solution is a critical parameter because it governs the reaction kinetics and the working properties of the final materials. In fact, it was demonstrated that the initial Si/M molar ratio of the silicate solution controls the nature and the quantity of the siliceous species; when the Si/M ratio decreases, the solution contains more depolymerized silicate species such as Q° and Q^{1} with the presence of a higher number of non-bridging oxygen atoms [13], inducing different reactivities. There are different methods to synthesize these silicate solutions. Autef et al. [14] prepared their solutions in laboratory by dissolution of amorphous silica in an alkali solution. Gharzouni et al. [15] used commercial solutions with different initial Si/K molar ratios. They demonstrated that the differences in terms of siliceous species and the degree of depolymerization between silicate solutions will induce different reactivities. However, great attention is given to the Na-geopolymers because they present different properties depending on the aluminosilicate source and the used solution [16]. The difference in the behavior between geopolymers based on K or Na can be related to the characteristics of the alkali solution. Thus, some works have related the use of Na and K as

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alkaline solutions [17]. In fact, some publications have addressed the comparison between sodium and potassium solutions. A molecular dynamics study [18] of the interaction of oxygen molecules and oxygen diffusivity in NaOH and KOH at different temperatures and concentrations highlighted that the diffusion coefficients for hydroxide Na^+ and K^+ decrease with increasing solute concentration. However, it is important to remember that the diffusion coefficient of oxygen in NaOH solutions is smaller than that in the corresponding KOH solutions at all concentrations. The common point in all of these works is that they use silicate in solution form, and few formulations with silicate powder are made. However, the use of liquid solutions requires drastic storage conditions [5]. As an alternative, the use of mixtures based on powders with only the addition of water will be considered. In this context, the use of powder-based mixtures seems to be easier to carry out in the samples. The use of silicate powder is rare despite the advantages of powder silicates, including its low price and ease of utilization [19]. Thus, it would be very interesting to conduct a work involving powder or liquid to understand the mobility of species in solution and control the Si/Na molar ratio.

Various reports have demonstrated that the solution preparation method has an impact on silicate species [20]. The authors [12,21] have shown that for the same Si/M ratio, a commercial silicate solution in which KOH pellets were dissolved and a laboratory solution result in the same silicate species in different amounts. For example, the decomposition of Raman spectra [22] of laboratory and commercial solutions with a Si/M ratio of 0.7 result in the same contributions but a different intensity. Brykov et al. [23] showed that the preparation of the solution has little influence on the connectivity of the silicon atoms compared with the solutions provided by dissolving a colloidal silica or a glass. It is suggested that commercial silicate solutions and laboratory-made solutions present differences in reactivity. A study based on various solutions with different cations and Si/M ratios by spectroscopy investigations (FTIR, Raman and NMR) has been conducted by Vidal et al. [24]. This work has demonstrated that the manufacturing process causes a slight variation in the amounts of the different silicate species responsible for the reactivity of the solution. In the same way, it has shown [25] that the nature of the silica introduced and its reactivity have an influence on the viscosity of formed binder. Consequently, the nature of the solution plays a significant role in the final material.

The aim of this study is to synthesize geopolymer materials based on powdered silicate from two different metakaolins and compare them with those synthesized based on commercial silicates. These formulations were analyzed by FTIR spectroscopy and a compressive mechanical test to investigate their later use as deposits on earth bricks.

Experimental part

Raw materials and sample preparation

Before preparing the geo-materials, the alkaline solution is synthesized from a mixture of silicate (powder (P) or liquid (L)), NaOH pellets (97% purity) and water to obtain a solution with a Si/M ratio equal to 0.7. The characteristics of the formed solutions are reported in Table 1. The metakaolins M1 (SiO_2 = 55%, Al_2O_3 = 40%) and M2 (SiO_2 = 55%, Al_2O_3 = 39%) are then added as described in Fig. 1. These two metakaolins are prepared differently: M2 is flash-calcined, whereas M1 is calcined in rotary furnace. The difference in the calcination method of these kaolins induces varying reactivity in the geopolymer reactions [26]. After stirring for 15 min at 700 rpm, the reactive mixture was placed in a sealed polystyrene mold at ambient temperature for 24 h to complete the polycondensation reaction. The samples were then stored for 7 days and then demolded. Owing to the high reactivity of M2, some water must be added to the mixtures [24]. Throughout the entire study, synthesized geopolymers will be denoted according to the MxW_z^y nomenclature; M*x* represents the type of used metakaolin (M1 or M2), W is the type of alkaline silicate (P: powder, L: liquid), and *z* and *y* represent the initial and final Si/Na ratios, respectively. For example, M1 $L_{0.7}^{1.7}$ was synthesized based on M1 metakaolin and a silicate/sodium solution ratio Si/Na = 1.7. Different compositions with varying Si/Na values are summarized in Table 2.

Table 1
Characteristic of different solutions.

Samples	Chemical compositions			pH values
	% SiO_2	% H_2O	% Na_2O	
$P_{0.7}^{1.70}$	27.5	64.2	8.3	14
$L_{0.7}^{1.70}$	27.5	64.2	8.3	14
$L_{0.7}^{1.29}$	31.8	55.5	12.7	14

Technical characterization

Fourier transform infrared (FTIR) spectroscopy in ATR mode was used to investigate the structural evolution of the geopolymer mixtures. The FTIR spectra were obtained using a Thermo Fisher Scientific 380 infrared spectrometer (Nicolet). A drop of the geopolymer reactant mixture was deposited onto the instrument's diamond crystal and protected with a small bell from any environmental pollution during the spectrum acquisition process, which was performed regularly until the end of the geopolymerization process. The IR spectra were gathered over a wavenumber range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1}. The atmospheric CO_2 contribution was removed with a straight line between 2400 and 2280 cm^{-1}. To follow the evolution of the involved bonds within the sample in time, a macro was used to acquire a spectrum every 10 min for 7 h, producing 64 scans in total. To allow comparisons of the various spectra, the spectra were baseline-corrected and then normalized.

The pH values were measured with using a Schott Instruments Lab860 pH-meter at 25 °C for 2500 min. The samples in pellet form were placed in tubes (Ø: 35 mm and H: 70 mm) and covered with water to maintain the ratio of geopolymer weight to water weight equal to 0.08 (m_{geo}/m_{water} = 0.08) for each measurement. At the

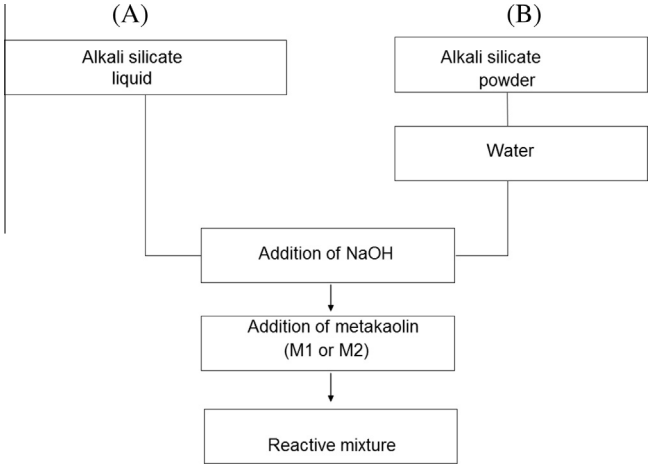

Fig. 1. Synthesis protocol of geo-materials (A) liquid and (B) powder silicate.

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