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Mechanical characterization of large sized geopolymer samples

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Abstract

The development of geopolymers, as mass distribution materials, is increasing rapidly thanks to their relative ease of processing on site. However, the great diversity of their properties, in particular their mechanical resistance, can restrain the use of these materials because of the duration of the samples' characterization by traditional means. The objective of this work was therefore to develop a faster way to estimate the mechanical resistance of geopolymers, whatever their composition, size or shape. The results were studied for different samples, whether they were alkali-activated geopolymers synthesized from pure metakaolin, composites or mortar-type geopolymers. In addition, a method for comparing samples with different sizes and shapes, however not suited for conventional laboratory work, has been developed, allowing a fast, inexpensive and requiring little equipment evaluation of the mechanical properties of geopolymers.

1. Introduction

Alkali-based geopolymers are well-known materials [1] which result from the dissolution of an aluminosilicate source in an alkaline silicate solution [2]. Their main interest comes from their relatively easy synthesis method, their low environmental impact [3] and high number of applications [4]. Moreover, they show good mechanical properties, which makes them good candidates in building materials for example. The parameters controlling the formation, the structure and properties of geopolymers have been studied [5, 6, 7]. It appears that controlling the geopolymerization reaction, and thus the properties, requires the knowledge of the raw materials and in particular their reactivity [2].

From the point of view of their properties of use, they present some similarities with ceramic materials such as resistance to climate or chemical attacks as well as to moderate thermal shocks. Like ceramic materials, however, they remain brittle due to a lack of elasticity. In order to increase the mechanical properties of geopolymers [8, 9] different types of reinforcements can be added during the synthesis. These can be of two types. In the first case, mineral reinforcements can improve the mechanical properties of geopolymers. Several authors have indeed shown that quartz, stable in an alkaline medium [10], can play the role of reinforcement and improve the mechanical properties of a geopolymer matrix [11, 12]. In the second case, the use of fibers, natural or not [13], can also help stabilize the mechanical properties [14].

Recently, the use of geopolymers as an alternative to natural stones has been considered, due to their ease of processing and shaping. One of the targeted applications is the funeral industry, which imposes some restrictions. Indeed, the samples are typically large in size and low in height, and unlike sole construction materials, their impact resistance is largely as important as their compressive strength. This work therefore reports on ways to improve the mechanical properties of geopolymer-based materials to enhance their durability. In addition, the shapes of the samples being unconventional, a method allowing them to be compared was defined to quickly identify the most effective mixtures.

2. Experimental part

2.1. Synthesis of the different formulations

The geopolymer binders considered in this study were prepared from an aluminosilicate and a solution of potassium silicate. The metakaolin used as the aluminosilicate source had an Si / Al ratio of 0.8 and its characteristics were detailed in a previous publication [15]. The activation solution was prepared by mixing a silicate solution with potassium hydroxide, until reaching an Si / K composition of 0.7. The aluminosilicate source was then added to the solution to make a binder. Additives, such as fibers or sands, can later be added to the geopolymer binder, in order to obtain a composite or a mortar, respectively. The protocol for the synthesis of these different mixtures is presented in Figure 1a, and the experimental setups are shown in Figure 1b and c for small scale and large scale mixing, respectively. They were then placed in molds of different sizes and shapes and then were consolidated at room temperature until complete solidification.

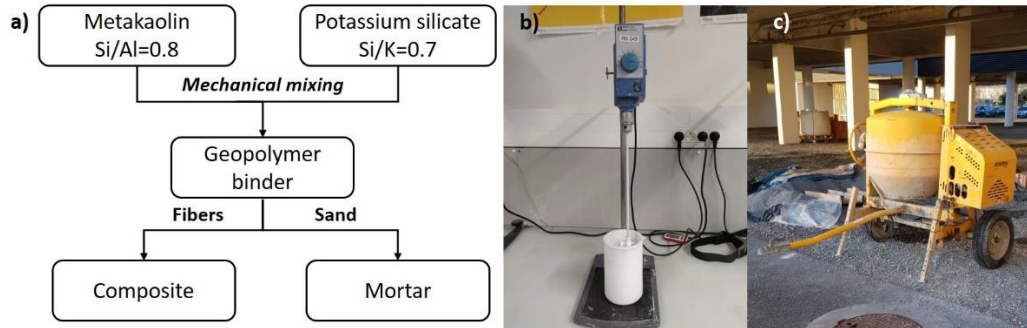


Figure 1: a) synthesis protocol of the different samples, b) experimental setup for small sized samples, c) experimental setup for large sized samples.

2.2. Samples characterization

2.2.1. Compressive strength

Compression tests [16] were performed using an Instron 5969 testing machine equipped with a 50 kN transducer and under the Bluehill3 software. The reaction mixture was previously placed in 15mm in diameter cylindrical jars and was removed from the mold on the day of the test. The specimens were then ground in order to have a 30 mm height as well as flat and perfectly parallel surfaces. The test pieces were systematically measured (height, diameter and mass) in order to determine their density. The sample was then placed at the center of the press until it broke. For repeatability of the measurements, the tests were carried out on five specimens, tested after 7 days of endogenous consolidation, leading to the reported compressive strength value as their average.

2.2.2. Impact resistance

The impact resistance tests were carried out based on standard NF EN 14617-9 [17]. A steel ball of a predetermined mass was dropped from successive heights on the surface of a sample placed on a 20 cm thick bed of sand. The ball was first released from a height of 6 cm (measured from the base of the ball and the impact surface) up to a height of 121 cm in increments of 5 cm, as can be seen in Figure 2a. The degradation of the sample was observed after each impact. Figure 2b and Figure 2c show a sample before and after the test, respectively.

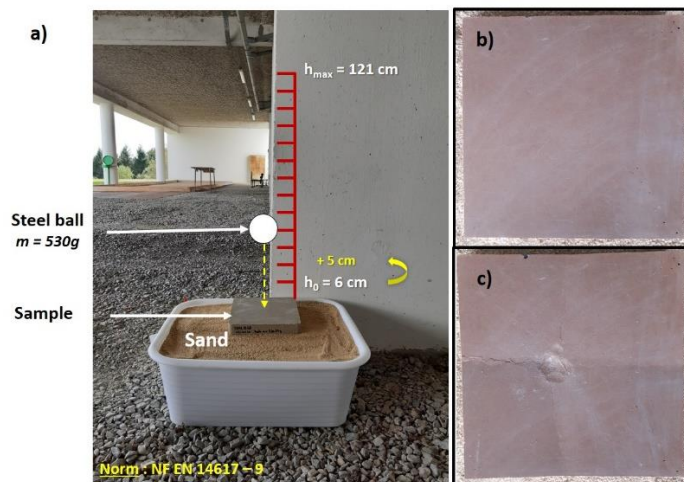


Figure 2: a) impact resistance principle. Test condition of a 20x20x3 cm plate (b) before and (c) after the impact resistance test.

The maximum height of the drop necessary for the sample to break is measured, in which case it allows the energy stored in the sample to be calculated according to the equation:

$$E_{tot} = \sum_{n=1}^{n=n_{max}} (m * g * h_n) , \text{ with :}$$

- n the number of successive drops of the ball during the test,
- n_{max} the number of drops necessary to break the sample
- m the weight of the ball, $m = 0.530$ kg
- g the gravitational acceleration on earth, $g = 9.81$ m.s⁻²
- h_n the height of the nth drop, $h_1 = 0.06$ m, $h_2 = 0.11$ m, etc

In order to better understand the mechanical behavior of our materials when faced with repeated shocks, the shape and, in particular, the thickness of the sample have been modified. Thus, four series of samples were tested: i) 20x20 cm plates with thicknesses ranging from 2 to 4 cm, ii) 10x10 cm blocks with thicknesses ranging from 2 to 5 cm, iii) rectangular plates 60x19 cm with thicknesses of 2 and 4 cm and iv) round plates with a 19 cm diameter and a thickness ranging from 2 to 4 cm. For a standard comparison, the tests were also carried out on a 10x10x3 cm block of white granite.

3. Results

3.2. Mechanical behavior of the samples

The compressive strengths of cylindrical test pieces of the three types of samples (binder, mortar and composite) were measured. This was then linked to their impact resistance, measured on 10x10x3 cm blocks. These data can be seen in Figure 3, which represents the evolution of the fracture energy under impact, or impact strength, as a function of the compressive stress for the different types of samples. The compressive stress value of the binder is high, 68,3MPa, which was already observed in previous studies [18]. Besides, these materials are not resistant to shocks, which is a typical behavior of ceramics, rigid and brittle materials. The addition of sand to the composition, while it greatly increases the impact resistance, decreases the compressive resistance of the sample. Finally, the composite presents the best compromise in terms of properties, but for a higher manufacturing cost.

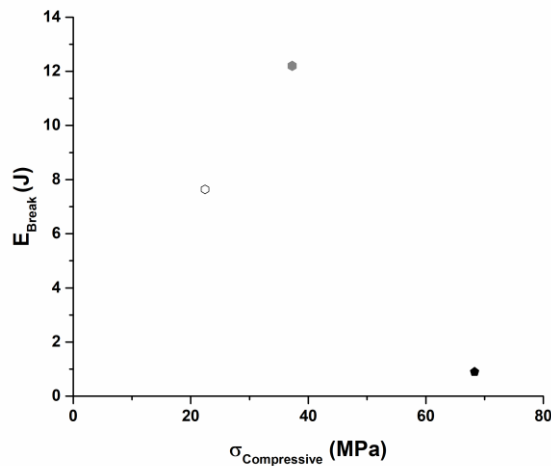


Figure 3: evolution of the stored energy on impact as a function of the compressive stress for the geopolymer binder (◆), composite (●) and mortar (○).

3.3. Improved impact resistance

For the purpose of an application in the funeral industry, considering the manufacturing cost requires the use of mortar. Thus, in order to improve the impact resistance of the developed materials, the effect of additions was evaluated. For this purpose, impact resistance characterizations were carried out on 20x20x3 cm mortar plates without addition, and then with the addition of a fiberglass fabric placed at the center of the plate when the mixture is poured. As shown in Figure 4, for a plate of the same volume, produced with the same composition, the addition of a fiberglass fabric allows the plate to withstand the entire test (ball released successively up to a height of 121 cm) without the crack crossing the sample (see Figure 4,

left), which is the case for a plate without additions (right) after a maximum drop height of the ball of 76 cm. The fiberglass fabric channels the crack in the center of the sample and allows the sample not to break entirely and to maintain a suitable hold for handling.

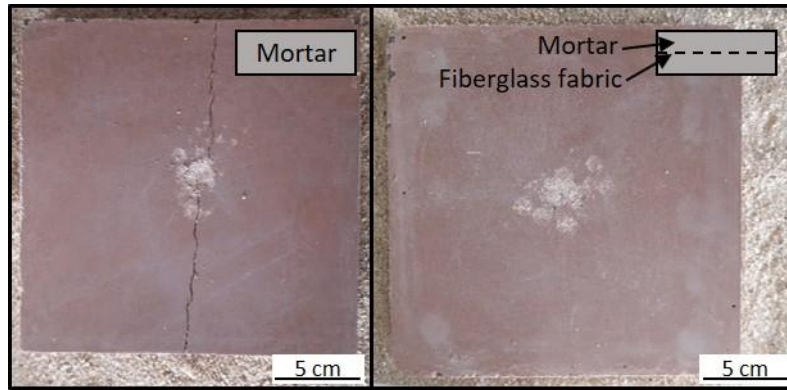


Figure 4: 20x20x3 cm plate of mortar (a) without addition and having undergone successive drops from a ball up to 76 cm in height and (b) with addition of a fiberglass fabric in the center and having undergone successive drops of a ball up to 121 cm according to standard NF EN 14617 - 9 [17].

3.4. Influence of size and shape

Different mortar samples were analyzed to define their suitability for their final application. The different shapes tested are visible in Figure 5. The shapes and sizes of the samples make it difficult to compare them directly. To facilitate this work, a dimensionless factor was defined from a selection of samples and then applied to all the mortar blocks measured. This factor also made it possible to quickly compare the mortar samples to the reference granite block. Its expression is as follows:

- for square or rectangular shapes: $Shape\ factor = \sqrt{2} * S * \frac{e}{s} * \frac{2*(l+l)}{40}$
- for round shapes: $Shape\ factor = \varnothing * \frac{e}{s} * \frac{2*\pi*r}{40}$

with: l the width in cm, L the length in cm, e the thickness in cm, S the impact surface in cm², r the radius in cm, and \varnothing the diameter in cm.

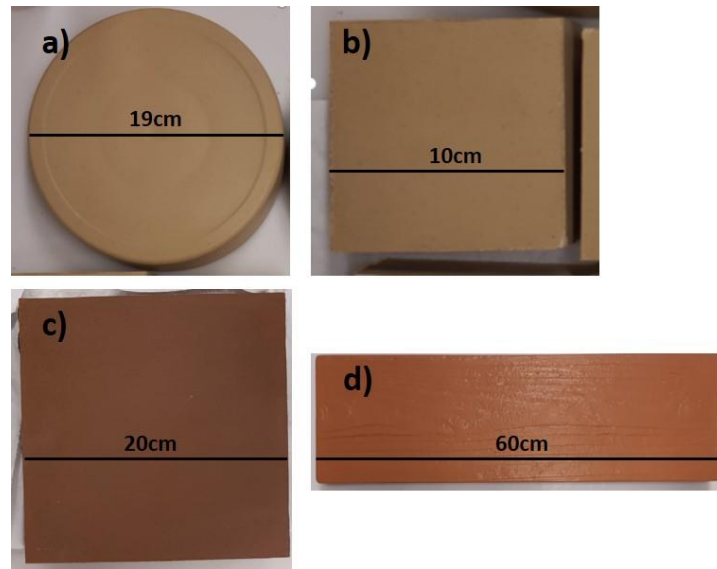


Figure 5 : (a) round shape diameter 19 cm, (b) block 10x10 cm, (c) plate 20x20 cm and (d) rectangle 60x19 cm tested for impact resistance.

Figure 6 gathers all the tests carried out on all the mortar samples. The shape factor makes it possible to compare the different forms with each other by considering the dimensions of each of the samples, as compared to the dimensions of the 10x10 cm blocks. Thus, it makes it possible to highlight a linear relationship between the shape and the total energy stored before the sample breaks, for the same composition of mortar. The shape and especially the thickness of the sample play an important role in impact resistance. Indeed, for a given sample shape, the greater the thickness, the greater the energy stored until rupture. Moreover, if we compare the mortar with the reference sample (shown in white in the figure) we notice that for the same dimension and a similar thickness, the granite stores a greater energy before its rupture than

a 10x10x3 cm block in geopolymer (31.98 J for the geopolymer against 50.38 J for the white granite). However, in order to compare them, it is necessary to take into account the density of the sample $\rho = \frac{m_{\text{sample}}}{V_{\text{sample}}} [g.cm^{-3}]$ with: m_{sample} the mass of the sample (575, 48 g for the geopolymer and 790.71 g for the granite) and V_{sample} the volume of the sample ($10 * 10 * 3 = 300 \text{ cm}^3$). This allows to calculate a specific resistance. Which is equal to 19.12 J.cm³/g for granite and 12.69 J.cm³/g for geopolymer based materials. Although less resistant than granite to repeated impacts, the samples still seem to have a mechanical behavior sufficiently close to be used as flat and large-sized plates.

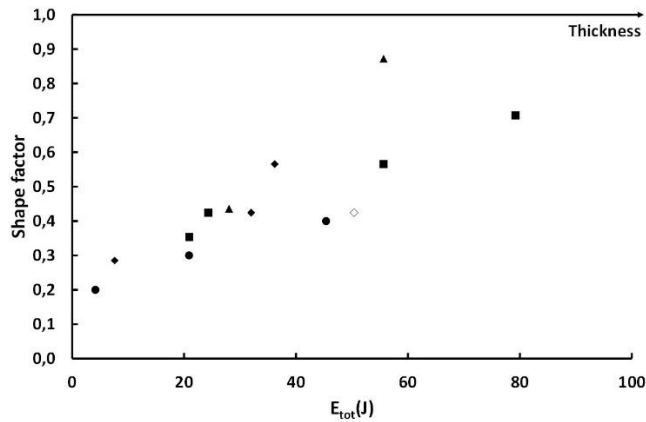


Figure 6 : Energy stored until the rupture of mortar samples with different shapes, with (■) the 10x10 cm blocks, (◆) the 20x20cm plates, (▲) the rectangular plates 60x19 cm, (●) the round plates of diameter 19 cm and (□) the 10x10 block in white granite

4. Conclusion

The aim of this work was to improve the mechanical properties of geopolymer-based materials for their use in the funeral field. As the geometries of the samples, with a low thickness and large surface, are not adapted for traditional characterization methods, a rapid method has been implemented to be able to select the samples which are not suitable for end use. The one presented here, not requiring complex equipment, can be used on the production site in a quality control process and has shown that mortars are good candidates for replacing granite. In addition, the insertion of a fiberglass fabric at the time of pouring the mixture, which is not possible for stone-type materials, considerably strengthens the impact resistance of the plates.

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Declaration of Conflict of Interests

The authors declare that there is no conflict of interest. They have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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