



# Recycling of geopolymer waste: Influence on geopolymer formation and mechanical properties



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## ABSTRACT

Geopolymers are new binders resulting from the activation of an aluminosilicate source by an alkaline solution. These binders are economically and environmentally profitable since they have the advantage of reusing recycled waste and industrial by-products as aluminosilicate sources. In this context, this paper focuses on the geopolymer wastes incorporation in different formulations and their effect on geopolymer formation and the properties of the final materials. For this purpose, the geopolymer wastes were at first characterized. Three compositions differing in the used alkaline solution and the amount of metakaolin added were investigated. A feasibility study allowed retaining 20% as the waste percentage added or substituted to the metakaolin to still obtain geopolymer materials. Moreover, it was shown that the incorporation of the geopolymer waste may disturb the polycondensation rate which was proven to strongly depend on the solid to liquid ratio and the Si/K ratio of the alkaline solution. Finally, relationships were demonstrated between the compressive strengths and the chemical compositions of the different samples. The low reactivity of geopolymer waste can be compensated with the use of highly reactive alkaline solution or the increase of the amount of metakaolin in the mixture.

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## 1. Introduction

In recent years, the growth of waste production associated with the awareness of the environmental problems and the need of sustainable development make waste management a priority [1]. Recycling has drawn great interest as a way to solve waste problems, reduce environmental pollutions and preserve natural resources. For example, the glasses are reused in glass or enamel compositions as cullet and allow lowering the energy and the raw material consumption [2]. Others examples are concretes or ceramics which are crushed and used as aggregates in new concretes [3,4]. In this context, geopolymer materials are a new class of binders having the advantage of using industrial byproducts and recycled waste. These binders are generated from the activation of an aluminosilicate source with an alkaline solution [5,6]. Their formation implies the dissolution of aluminosilicate species in an alkaline environment to form an amorphous three-dimensional geopolymer network by polycondensation reaction. Based on such a unique structure, geopolymers may exhibit good mechanical, chemical and thermal properties making them a promising alternative for a variety of applications [7]. Diverse industrial by-products were proven to be suitable for producing geopolymer materials such as fly ash [8,9], furnace slag [9,10,11], red mud [12], mine waste mud [13], waste concrete [14] and construction and demolition waste [15]. He et al., [12]

established a comparative study between two types of geopolymers based on metakaolin and a mixture of red mud and fly ash. The lower strength obtained for a mixture of red mud and fly ash sample were attributed to the reactivity of the raw materials. The final materials were composed of a geopolymer binder and unreacted phases present as inactive fillers. This fact was also evidenced by Komnitsas et al. [15], in the case of construction and demolition waste-based geopolymers. Indeed, they observed a heterogeneous matrix containing grains of various sizes and attributed this to the partial reaction of the initial concrete. Similarly, Gao et al. [16], demonstrated that the compressive strength firstly increased up to 20% of fly ash content and then decreased as the fly ash content increased into metakaolin-slag blends.

Nazari and Sanjayan [17] proved the possibility of producing geopolymers using aluminum and cast iron slags. They highlight that the silica to alumina ratio is the most important parameter governing the mechanical properties. Onutai et al. [18] evidenced that 40 wt.% of Al waste content in geopolymer mixture lead to a dense structure and therefore optimal compressive strength. Moreover, investigation study about the influence of different types of aggregates, such as lime stone, schist and granite, on the properties of geopolymeric mine waste mud binder was undertaken. It was demonstrated that the aggregate dimensions affect the tensile strength. Ferone et al. [19] have shown the suitability of calcined clay sediments for geopolymer synthesis and evidenced the role of heat treatment temperature on the reactivity. In addition to that, the immobilization of heavy metals in municipal solid waste incineration fly ash based geopolymer was evidenced [20]. The mechanical properties

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and microstructure of the resulting materials were directly linked to the alkaline solution dosage and the Si/Al molar ratio. According to these studies, there are different remaining problems to consider. Indeed, the authors evidenced that the use of wastes implies a decrease of the compressive strength, a contamination coming from the wastes and the low reactivity of these materials. In order to improve the results, it seems to be necessary to determine the optimum amount of wastes for the addition or the substitution of raw materials.

So far, as extensive research on geopolymer has been conducted, the generation of geopolymer waste increases. In this context, an innovative use of geopolymer waste is their incorporation in different geopolymer formulations which is in accordance with the “cradle to cradle” concept. Moreover, reusing these geopolymers allow reducing the amount of raw materials used. Recycling of waste and their use as aluminosilicate sources seems to be profitable since it economical and environmental benefits, leading to greener manufacturing and global sustainable development. Recent investigations using recycling aggregates produced in laboratory point out to the fact that the use of fine recycled aggregates must not exceed 30%, otherwise the performance could be at risk [14]. Current recycled aggregates have particles of impurities such as soil, plastics, wastepaper wood, metals and organic matter. Organic matter leads to lower mechanical performance and lower concrete durability. Moreover, Park and Noguchi [21] studied concrete containing metal impurities and it was found that aluminum caused performance degradation.

The present work aims to evaluate the suitability of using crushed geopolymer in addition or substitution of metakaolin to produce geopolymers materials. For this, the used raw materials were characterized. Then, the feasibility of consolidated materials was evaluated. Several samples were prepared by varying the proportion of geopolymer waste. The structural evolution of the reactive mixtures was monitored by FTIR spectroscopy. Finally, the consolidated materials were characterized by compression tests.

## 2. Experimental part

### 2.1. Raw materials and sample preparation

The consolidated materials were prepared by mixing the metakaolin (MK) and the geopolymer, crushed and sieved at 80  $\mu\text{m}$ , in an alkaline solution (AS) as described in Fig. 1 [22,23]. The reactive mixtures were placed in open polystyrene molds at room temperature for 7 days. Three formulations, differentiated by the amount of metakaolin and the starting silicate solution were studied. The two silicate solutions, denoted as S1 and S3, differ in terms of the Si/K molar ratio (1.7 for S1 and 0.7 for S3) and the water contents (79% for S1 and 59% for S3) [24,25].

For each composition, the effect of the substitution of an amount of metakaolin (MK) by crushed geopolymer (CG) or the addition of this compound was investigated. So, three sets are synthesized. Samples

were synthesized either by adding the crushed geopolymer to metakaolin or substituting an amount of metakaolin by crushed geopolymer. Samples are denoted as  ${}^x\text{SiG}^y$  or  ${}^x\text{SiG}^{\text{Sy}}$  where  $x$  refers to the quantity of metakaolin, Si is the type of the used silicate solution,  $y$  the percentage of crushed geopolymer added to the mixture and  ${}^{\text{Sy}}$  the percentage of crushed geopolymer which substitutes an amount of metakaolin mass. For example  ${}^{12\text{S1}}\text{G}^{\text{S20}}$  refers to the geopolymer obtained from the substitution of 20% of 12 g of metakaolin by crushed geopolymer using S1 as a silicate solution. The nomenclature and the composition of the samples analyzed are presented in the Table 1.

### 2.2. Technical characterization

The chemical composition of the precursors was determined using X-ray fluorescence (XRF). This technique allows the quantification of the atomic elements.

The particle size distributions of the raw materials were measured using a laser particle size analyzer (Mastersizer 2000). The mixture contained 1 g of aluminosilicate precursor in 20 ml of water, mixed by ultrasound to eliminate any aggregation. The measured particle sizes are in the range 0.05–880  $\mu\text{m}$ . Additionally, the concentration of the solution should not be too large (obscuration < 35%).

Powder BET surface areas were determined by  $\text{N}_2$  adsorption at  $-195.85\text{ }^\circ\text{C}$  using a Micrometrics Tristar II 3020 volumetric adsorption/desorption apparatus. Prior to the measurement, the samples were degassed at  $200\text{ }^\circ\text{C}$  under vacuum for 4 h.

Bulk density was measured by pycnometer method. Numerically, it represents the mass per unit volume of matter. The SI unit of density is  $\text{kg}/\text{m}^3$ .

The wettability (water demand) of a powder is the volume of water that can be absorbed by 1 g of powder until saturation. This quantity depends directly on the particle size, the specific surface and the morphology of powder. One gram of powder is weighted and then deposited on a glass slide. Using a micropipette, the water is added to the powder (microlite by microlite) until visual saturation of the granular.

The pH values were measured using a Schott Instrument Lab860 pH-meter at  $25\text{ }^\circ\text{C}$  during the first 400 min of the geopolymer formation. A 2.4 g sample was immersed in 30 mL of osmosed water, which provided a solid to liquid ratio of 0.08 [26].

The mineral phases were identified by powder X-ray diffraction (XRD) with a BRUKERAXS D8 Advance powder diffractometer using  $\text{CuK}\alpha$  radiation ( $\lambda\text{K}\alpha = 0.154186\text{ nm}$ ). The analytical range used was between  $5^\circ$  and  $70^\circ$  with a step size of  $0.04^\circ$  and an acquisition time of 2 s. JCPDS (Joint Committee Powder Diffraction Standard) files were used for phase identification.

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 ThermoFisher Scientific Nicolet

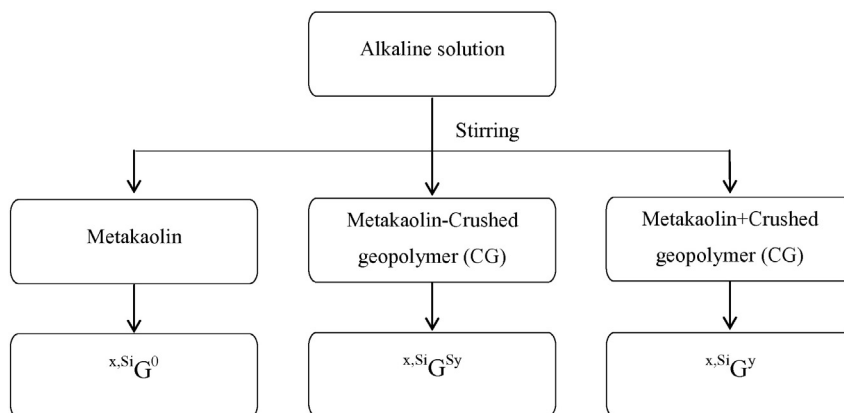


Fig. 1. Synthesis protocol of the various consolidated samples.

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