



Composite geopolymers of metakaolin and geothermal nanosilica waste



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HIGHLIGHTS

- Geothermal nanosilica was used to produce metakaolin-geopolymers.
- The addition of the nanosilica reduced the demand of the sodium silicate.
- High contents of nanosilica increased the porosity and reduced the strength.
- Crystallization of nepheline and albite was observed at high temperatures.
- The XRD amorphous halo shifted to higher angles due to geopolymerization.

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ABSTRACT

This paper investigated the effect of a nanometric silica from a geothermal waste on the compressive strength, microstructure, and reaction products formed on geopolymers cured at room temperature; the thermal stability was evaluated after exposure up to 800 °C. The geothermal silica waste replaced 0–20% of the metakaolin and was suspended in the alkaline solution, to avoid agglomeration. Sodium silicate and sodium hydroxide were used as alkaline solutions and added to adjust the molar ratios of SiO₂/Al₂O₃ at 2.8, 3.0 and 3.2, Na₂O/SiO₂ = 0.32 and H₂O/Na₂O = 10. The compressive strength of the geopolymers was evaluated up to 60 days of curing. The pastes were characterized by X-ray diffraction, Fourier transformed infrared spectroscopy, and scanning electron microscopy. X-ray diffraction showed the formation of amorphous reaction products, with a shifting in the amorphous halo related to the raw materials; on the other hand, a shift in the characteristic band of aluminosilicates in the infrared spectra also evidenced the geopolymerization process. While the addition of the geothermal silica waste reduced slightly the strength at room and high temperature due to the formation of porosity, its use diminished the demand of waterglass and therefore obtaining low-cost and more sustainable binders.

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1. Introduction and background

Concrete made with Portland cement (PC) is the second most commercialized material by mankind, only after water [1]; it provides several economical and technological advantages compared to other construction materials such as steel, aluminum or glass. Nevertheless, the production of PC is associated with the discharge of considerable amounts of CO₂ to the atmosphere [2]; 4.3 billion tons of cement are produced yearly, emitting nearly four billion tons of CO₂ (i.e. 6–7% of the anthropogenic CO₂ emissions) [3]. Moreover, the demographic and economic growing pace, indicates that from now to the year 2050 the population in urban areas will expand in 1.3 billion people in urban areas [4], so that the increased demand of PC will push the industry to generate around

5.8 billion tons [5], unless alternative green binders satisfy the demand in a sustainable way.

The challenges for alternative binders could be approached by: 1) the partial replacement of PC, 2) the production of alternative low energy cements, e.g. calcium sulfoaluminates, or (3) the formulation of new binders such as alkali activated materials (AAM). Within the latter type are classified the geopolymers, which have an extensive range of application as substitutes for PC due to their properties, namely enhanced mechanical properties, and the ability to withstand both, elevated temperatures and chemical attack by acids and salts [6]. The development and use of AAM as an alternatives to PC for construction and other applications has advanced rapidly in the past years; there are cases of commercial deployment in various countries, in infrastructure, general construction and paving, nuclear waste immobilization, and various other niche applications. Hybrid binder formulations, making use of concepts and components from both alkali-

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activation and Portland cement chemistry, are also becoming appealing due to their potential of offering more robust and less expensive pathway to AAM [7].

Deventer et al. [8] indicated that the production of geopolymers represents a reduction of around 80% in CO₂ relative to PC, although others have suggested caution when stating such figures [9,10]. In the geopolymeric systems, aluminosilicate powdered precursors dissolve under the chemical attack of an aqueous alkaline activator, further polycondensation of the dissolved species form strong solids constituted by an amorphous aluminosilicate gel [11,12]. This process leads to the production of inorganic binders with excellent physical and chemical properties suitable for construction and other purposes.

Two precursors are commonly used for geopolymers [13]: those calcined, such as metakaolin, and other pozzolanic wastes [14,15], and noncalcined materials, such as kaolinite, feldspars, rock-type aluminosilicate or silicate minerals, and mine tailings [16]. Previous studies showed that geopolymers obtained from calcined materials exhibit a higher early compressive strength due to faster reactions; however, the geopolymerization of noncalcined materials results in an increase in compressive strength at later stages related to a lower reactivity [14–16]. Following from that, this investigation analyzes the properties of geopolymers produced using metakaolin (MK) and a noncalcined raw material such as a nanosilica from geothermal wastes (GSW).

The GSW is a byproduct from geothermal power plants, in which the water and steam extracted from the underground are used to generate electricity; approximately 50,000 tons of such waste are discarded yearly, without a systematic application [17,18]. The GSW consists mainly of amorphous nanosilica, with sodium and potassium chlorides as minor components. This residue could be used as source of silica for alkaline silicate solutions for alkali activated binders. There are also reports on the use of GSW as replacement of PC with and without other supplementary materials, and in some cases with the aid of alkaline activation [17–20]. Nevertheless, investigations on its use in geopolymer systems are very limited. Hajimohammadi et al. [21] synthesized geopolymers using GSW and sodium aluminate powder, but the authors did not report the mechanical properties. Escalante-Garcia et al. [22] studied the addition of GSW in alkali activated slag binders and reported an increase in the reactivity of the activated systems; enhanced strength was noted for activation with NaOH, while for activation with waterglass of modulus of 2 the strength was reduced in the presence of GSW. The temperature effect on this type of geopolymer is not reported yet.

Based on the above, this paper presents the results of the synthesis and characterization of geopolymers based on MK partially replaced with 0–20% GSW, activated with alkaline solutions of sodium silicate and sodium hydroxide. The aim was to analyze the effect of the GSW on the mechanical properties and the amount of sodium silicate demanded. The binders were characterized by X ray-diffraction, Fourier transformed infrared spectroscopy, and scanning electron microscopy, after normal curing and also after exposure to temperatures up to 800 °C.

2. Materials and experimental methods

The geothermal nanosilica waste was obtained from the Cerro Prieto Geothermal Power Plant in Baja California Mexico. In order to reduce the chlorides content, the GSW was homogenized and washed with water at 90 °C and then filtered-dried at high temperature. The dry GSW was de-agglomerated by pulverization and sieved to pass the 88 µm mesh. The physical properties of this material are given elsewhere [18]; the particle size, as measured by transmission electron microscopy, was in the range of 20–50 nm [23]. The chemical composition of the washed GSW was predominantly SiO₂, with small amounts of sodium and potassium chlorides (see Table 1).

A commercial metakaolin (MK) was used as the base raw material; its chemical composition, shown in Table 1, indicated a molar ratio SiO₂/Al₂O₃ (S/A) of 1.2. The MK was mostly amorphous with only small amounts of crystalline phases, as kaolinite and quartz. Laser diffraction (Coulter model LS-100Q) indicated an average particle size of 0.8 µm.

The alkaline activator solutions used consisted of mixtures of sodium silicate (waterglass with a modulus, Ms = 3.1), sodium hydroxide flakes and distilled water. The MK was replaced by GSW at 0, 5, 10, 15 and 20% (mass); the required amount of GSW for each mixture was previously dispersed making a suspension with the alkaline solutions, prior to the addition of MK. The alkaline compounds were added to adjust the molar ratios of SiO₂/Al₂O₃ (S/A) at 2.8, 3.0 and 3.2, Na₂O/SiO₂ (N/S) = 0.32 and H₂O/Na₂O (H/N) = 10. The molar ratios of the prepared pastes were calculated taking into account: (a) SiO₂ from the MK, waterglass and GSW, (b) Na₂O from the waterglass and NaOH, and (c) H₂O from waterglass and the additional distilled water. The pastes were prepared by mixing the MK powder with the GSW suspension for 5 min, casting them afterwards in cubic molds of 5 cm. The curing consisted of two stages: the samples were first exposed at 60 °C for 2 h and then kept at 20 °C for 24 h in the molds. Such curing conditions were established as a result of preliminary tests, in which two temperatures of 20 and 60 °C were used for intervals of 2, 4, 6, 12 and 24 h; it was noted that the specimens cured at 20 °C and those cured for more than 2 h at 60 °C were brittle and weak after 3 days, indicating that the curing time was an important parameter. Due to the small particle size of the raw materials, i.e. MK and specially the GSW, the water demand increased, which is known to reduce the strength; superplasticizers were not intended to be used, since they are generally unstable under the strong alkaline conditions of the AAM.

After demolding, the samples were stored in plastic and sealed containers at room temperature up to 60 days. Compressive strength tests were carried out at various ages and pieces from the center of crushed specimens were dried in a vacuum oven at 50 °C during 24 h, and then ground in an agate mill to pass the mesh of 88 µm for further characterization. X-ray diffraction (XRD) analyses were performed on powdered samples using Cu Kα radiation produced at 40 kV and 30 mA (Siemens model D5000). Pellets from such powders were prepared by mixing and pressing the samples with KBr to obtain Fourier transformed infrared (FTIR) spectra (Perkin Elmer model Paragon 1000PC). For the microstructural analysis, the specimens were cold mounted in epoxy resin, ground, polished and gold coated prior to their examination in a scanning electron microscopy (SEM); the equipment used was a JEOL model JSM 6510LV.

The thermal stability of the geopolymeric systems was also evaluated by exposing cubic specimens cured for 28 days at 200, 400, 600 and 800 °C during 2 h, cooling them afterwards at room temperature. The samples were characterized using the techniques previously described.

3. Experimental results and discussion

3.1. Results of the systems cured at room temperature

3.1.1. Compressive strength

Fig. 1 shows that the compressive strength increased with the curing time for all of the systems, evidencing the progress of the reactions with time. The geopolymers with 0% GSW developed the highest strength, which was reduced, but not drastically, with the increase on the GSW content. Such strength reduction could have three possible causes related to the addition of GSW.

The first one could be attributed to the nanometric character of the GSW, in that the water has to wet all of the surface of the particles in order to have a fluid paste; as water was adsorbed on the GSW particles, the workability of the pastes was reduced (the amount of water was kept constant) and air bubbles remained trapped during mixing and casting (this will be further discussed with the microstructural characterization (Fig. 4)).

Second, in the presence of GSW, the amounts of alkaline compounds had to be modified in order to maintain the already mentioned S/A and N/S ratios, i.e. the amount of waterglass had to be reduced; this compensation lowered the modulus from Ms = 1.2 for the geopolymer with 0%GSW to Ms = 0.23 for that with 20% GSW, while keeping the same amount of Na₂O in the activating solution. This had a two-fold effect. On the one hand, the amount of waterglass was reduced in 80% and this affected the rheology of the pastes, in addition to the effect mentioned above; this observation would also suggest that the dissolution of the nanometric GSW was not as fast as expected. On the other hand, the reduction in the Ms means that less SiO₂ was available in the starting alkaline solution, so the activation process started with solutions rich only

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