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Research paper

Effect of metakaolin on natural volcanic pozzolan-based geopolymer cement

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ABSTRACT

This work describes the effect of metakaolin on the microstructure and compressive strength of geopolymeric systems based on natural volcanic pozzolans. The effect of the molar ratios of $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ and the percentage of metakaolin present in the mixture was studied. The proportion of MK varied between 0 and 30%. A mixture of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) was used as an alkaline activator. The mechanical strength of the geopolymeric pastes and the microstructural properties were evaluated, and techniques such as X-ray diffraction (XRD), infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) were used. The optimum proportion of MK was 20%, incorporated as a replacement of the natural pozzolan. The results obtained in the present study reveal the possibility of obtaining a geopolymeric cementing material with a compressive strength of up to 68 MPa at 28 days of curing at room temperature ($25 \pm 3^\circ\text{C}$).

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1. Introduction

Approximately 11.6% of the Colombian territory, mainly located in the Andean region of central and southern Colombia, is covered with soil deposits derived from volcanic ashes or natural pozzolans (NP) that are mainly composed of sodic-calcic plagioclase feldspars, volcanic glass, amphiboles, pyroxenes and quartz (Lizcano et al., 2006). The possibility of using NP in the synthesis of geopolymers cured at room temperature to replace traditional Portland cement in some applications is of great interest in countries that have large deposits of these materials, such as Colombia. In general, these materials have elevated $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratios (≥ 6.0) and low percentages of CaO and reactive Al_2O_3 , which, along with their semicrystalline nature, hinder their use as a primary precursor in obtaining geopolymers at room temperature ($25 \pm 3^\circ\text{C}$) (Tchakoute et al., 2013a). Consequently, the use of thermal activation treatments and the addition of secondary sources that provide CaO and/or Al_2O_3 , such as granulated blast furnace slag (GBFS) and metakaolin (MK), are common to improve the NP reactivity (Djjobo et al., 2014; Kani and Allahverdi, 2009; Tchakoute et al., 2012; Robayo et al., 2016). Specifically, MK has been used as precursor and an alumina source in different geopolymeric systems, such as the ones based on granulated blast furnace slag (GBFS) and fly ash, with satisfactory results (Villaquiran-Cacedo et al., 2015; Kuenzel et al., 2013; Zhang Z.H.

et al., 2014; Zhang et al., 2016; Bernal et al., 2010, 2011; Rajamma et al., 2012; Zhang H.Y. et al., 2014; Luna-Galiano et al., 2015).

In the specific case of volcanic pozzolans, Djjobo et al. (2014) added MK at between 5% and 25% mass to the two NP from Cameroon to compensate for their reactive Al_2O_3 deficiencies, thus obtaining geopolymers cured at room temperature with compressive strengths of up to 68.8 MPa at 28 days. Moreover, Tchakoute et al. (2012) were able to increase the compressive strength of an alkali-activated NP by 32% with the addition of 40% mass MK. The special characteristics of this NP from Western Cameroon allowed Tchakoute et al. (2012) to obtain a geopolymer 100% based on this material with a compressive strength of up to 36 MPa at 28 days of curing at room temperature. In a previous complementary study, Tchakoute et al. (2013b) used this alkali fusion technique to increase the pozzolan reactivity and investigate the effect of metakaolin additions (30–60% mass), reporting strengths between 41.5 and 68.8 MPa and a considerable decrease in the mixture setting time (7–15 min). Conversely, Bondar et al. (2011) added kaolin to an NP of Iranian origin in increments of up to 40%, confirming that the presence of reactive Al promotes a more stable geopolymeric gel.

Based on the above, the NP become a more viable alternative from the industrial point of view for geopolymer technology at a large scale (Palomo et al., 2014) because they have more homogeneous chemical and mineralogical compositions compared to the industrial sub-products commonly used in the production of geopolymers, such as fly ash which is a highly variable material as a consequence of the use of different sources of coal, poorly controlled combustion processes and inappropriate technologies (Duxson, 2009; Mejía et al., 2015). In

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this sense, the goal of the present article is to evaluate the design conditions required to develop binary geopolymeric systems based on two natural volcanic pozzolans (NP1 and NP2) and MK. The objective of adding MK is to increase the content of alumina in the mixture and the proportion of amorphous phase. The effect of the $\text{SiO}_2/\text{Al}_2\text{O}_3$ and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ ratios and the MK addition percentage on the compressive strength of both geopolymeric systems was studied at both 7 and 28 days of curing at room temperature ($25 \pm 3^\circ\text{C}$). This analysis was complemented with the observation of the final product microstructure.

2. Materials and experimental methodology

2.1. Materials

Two natural volcanic pozzolans (NP1 and NP2) from Colombia were used as primary sources. High purity commercial MK (Metamax) was used as an oxide source. The chemical composition of these materials, presented in Table 1, was determined by X-ray fluorescence (XRF) using a MagiX-Pro PW-2440 Phillips spectrometer, equipped with a Rhodium tube and with a maximum power of 4 kW. The $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratios are high (6.79 and 6.48 for NP1 and NP2, respectively) and the content of alkaline components present is low (CaO, Na_2O and K_2O); conversely, MK presents a low $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio (1.93) due to its elevated content of Al_2O_3 (44.63%). The particle size analysis was performed by a Mastersizer-2000 laser granulometer from Malvern Instruments with a Hydro2000MU dispersion unit, and distilled water was used as the dispersing medium. The average particle size $D[4;3]$ obtained for NP1, NP2 and MK was $20.63\ \mu\text{m}$, $17.73\ \mu\text{m}$ and $7.76\ \mu\text{m}$, respectively.

Fig. 1 shows the X-ray diffraction pattern obtained for the natural pozzolans (NP1 and NP2*). The content of amorphous phase of the natural pozzolans was determined by Rietveld refinement (XRD) using corundum (Al_2O_3) as standard. Quantitative analysis results are presented in Table 2 which shows that the content of amorphous phase of NP1 and NP2 is 25.5% and 23.8% mass, respectively. The crystalline fraction of both natural pozzolans is composed primarily (>50% mass) of feldspars and plagioclase. In addition, other minerals can be identified such as quartz, amphiboles and for the case of NP2*, montmorillonite. The latter, part of the smectite family of the phyllosilicate mineral group, exhibited the highest water adsorption among the clay minerals due to its laminar structure and the charge distribution patterns inside its structure. To reduce the demand of water in the mixing process, it was necessary to treat the material at 700°C , which was performed at a heating rate of $14^\circ\text{C}/\text{min}$ and maintained for 15 min. Then, the obtained material, named here NP2, was left to cool slowly. The effect of this treatment is reflected in the XRD patterns (Fig. 1), which indicate a remarkable reduction of the interlayer spacing of the montmorillonite, due to the 001 reflection was shifted from 5.7° to 9.8° ($2<\theta>$) (Tironi et al., 2012).

Although there have been investigated industrial by-products (rice husk ash, waste glass, silica fume and spent diatomaceous earth) as alternative sources of silica in replacement of commercial sodium silicate with highly satisfactory results (Mejía et al., 2013; Puertas and Torres-Carrasco, 2014; Torres-Carrasco et al., 2014; Bernal et al., 2015; Villaquiran-Caicedo et al., 2015; Mejía et al., 2016), in the present study was selected a mixture of sodium hydroxide (NaOH) and

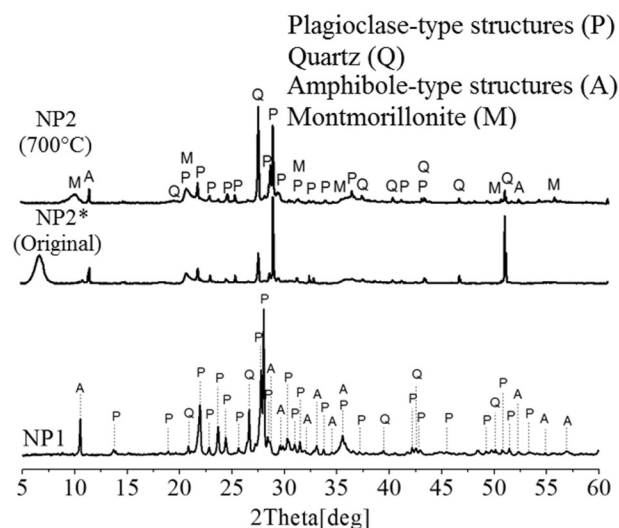


Fig. 1. X-ray diffractogram of the natural pozzolans (NP1 and NP2*: original pozzolans; NP2: after thermal treatment at 700°C).

commercial sodium silicate (Na_2SiO_3) ($\text{SiO}_2 = 32.09\%$, $\text{Na}_2\text{O} = 11.92\%$, $\text{H}_2\text{O} = 55.99\%$) as the activating solution to obtain a solution modulus (M_s : $\text{SiO}_2/\text{Na}_2\text{O}$) in the range of 0.55 to 2.78. These are activators traditionally used in alkaline activation processes.

2.2. Mixture design

The $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio and the MK % mass were evaluated as study factors with three levels each for the generation of 10 treatments in each pozzolan (Table 3). These treatments were randomly performed under equivalent conditions. The response variable was the compressive strength at 7 and 28 days of curing at room temperature ($25 \pm 3^\circ\text{C}$). In addition, the effect of the $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ ratio on the compressive strength of the geopolymeric systems that contained 20% MK was evaluated, as shown in Table 4.

The geopolymeric pastes were obtained in a Hobart mixer with a mixing time of 10 min. Adequate workability of the mixtures was achieved with a liquid/solid ratio of 0.20 and 0.32 for NP1 and NP2, respectively. The fresh pastes were placed in cubic moulds of 20 mm along each side and vibrated for 30 s in an electric vibrating table to remove the trapped air. Afterwards, the moulds were covered with a polyethylene film to control the evaporation of the mixing free water and were cured at room temperature ($25 \pm 3^\circ\text{C}$) for 24 h. After this time, the specimens were demoulded and taken to a curing chamber that ensures a relative humidity above 90% until the test age was reached. The compressive strength was evaluated on three specimens per mixture at 7 and 28 days of curing in an INSTRON 3369 universal testing machine with 50 kN of compression capacity and a deformation rate of 1 mm/min.

The following techniques were used for the microstructural study of the mixtures:

- X-ray diffraction (XRD), using a PanAnalytical X'Pert-MRD X-ray diffractometer with $\text{Cu}\alpha$ adiation and a step size of 0.020 for a 2θ range between 5 and 60° .

Table 1
Chemical compositions of the raw materials (oxides %).

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	Na_2O	MgO	K_2O	TiO_2	LOI	Molar ratio $\text{SiO}_2/\text{Al}_2\text{O}_3$
NP1	61.99	15.52	7.33	5.19	4.07	2.49	1.59	0.77	0.48	6.79
NP2	64.53	16.93	6.03	3.02	1.12	3.59	0.62	0.55	3.32	6.48
MK	50.72	44.63	0.37	–	–	–	–	1.76	0.86	1.93

LOI: loss on ignition.

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