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An investigation of the effect of migratory type corrosion inhibitor on mechanical properties of zeolite-based novel geopolymers

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ABSTRACT

The effects of migratory type corrosion inhibitor and curing time on the thermal stability and mechanical properties of Ecuadorian natural zeolite-based geopolymers were evaluated. Geopolymer samples were prepared by alkali activation of the natural zeolite by 8 M NaOH solution and calcium hydroxide Ca(OH)₂ 1–3 wt%, with an activator/binder ratio of 0.6. The geopolymer samples cured for 24 h at 40 °C and then for 6 days more at room temperature showed the compressive strength values in a range of 3–5,5 MPa. Mineralogical analysis of natural zeolite obtained by XRD is as follows: Mordenite (~67%), quartz (~27%) and amorphous (~6%). SEM-EDS micrographs analysis of geopolymers revealed the presence of Na and Ca which proves the incorporation of the activators, NaOH and Ca(OH)₂. The compressive strength values obtained indicate that the use of alkali activation of natural zeolites is an effective method for the synthesis of geopolymers. The mechanical properties of geopolymers were slightly but not adversely affected by the addition of the migratory corrosion inhibitor, MCI-2005 NS. These results will be used in future research on geopolymer concrete with embedded reinforcing steel.

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1. Introduction

Ordinary Portland cement (OPC) concrete has traditionally been used as building material worldwide, reaching an annual global cement production of 2.8 million tons, and is expected to increase to about 4 million tons per year [1,2]. Studies predict that by 2050 world demand for OPC production will sharply rise to 5.5 Gt/year [1,3]. This would imply significant large environmental impacts and massive consumption of natural resources [1,2,4].

It is calculated that ca. 0.54 tons of CO_2 per ton of clinker is released during cement production and 0.46 tons of CO_2 emitted as the result of burning combustible to supply the energy needed for this process. So it can be said that only cement production can contribute approximately 7% of human-driven global CO_2 emissions [1,4–8].

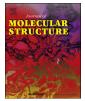
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Global warming problem and greenhouse gas effect lead to numerous investigations in attempts to develop new binders without cement to reduce CO_2 emissions in the production of OPC, replacing the clinker with supplementary cementitious materials giving rise to the development of new alternative materials [8]. These new materials are called geopolymers and first developed by Joseph Davidovits in 1982 [9]. Geopolymers are tetrahedral threedimensional inorganic aluminum silicate inorganic polymers composed of [AlO₄] and [SiO₄] which are mainly prepared from aluminum silicates or of industrial wastes such as fly ash [3,10–12], natural pozzolana [13,14], metakaolin [15,16] and natural zeolites [4,17–19], etc. Geopolymers are prepared by the activation of aluminum silicates mentioned by an alkali silicate solution in a highly alkaline condition [9,20,21].

Geopolymers are a class of aluminosilicate binder materials synthesized by a condensation reaction called geopolymerization of aluminosilicate minerals (such as industrial solid waste, calcinated clays, natural minerals and so on) with solutions of alkaline activator at or above room temperature [22]. They have been considered gradually to be potentially revolutionary materials in







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recent years to obtain great advantages like replacement material for OPC, in terms of its thermal, mechanical and chemical resistance properties are usually similar and even superior [15].

Various authors have studied the use of corrosion inhibitors in OPC concrete, mainly for reinforced steel corrosion embed in concrete, from coastal regions where the climate is characterized by contaminants such as chlorine ions (Cl^{-}) and carbon dioxide (CO_{2}) [23-28], this affects in the detachment an delayering of steel and as a consequence, the detriment of concrete in reinforced structures. The use of corrosion inhibitors is a method to prevent and delay the apparition of corrosion in concrete reinforced bars [29,30]. One of the possible definitions of corrosion inhibitors is given by the ISO 8044: 1989 that establishes that the material is a chemical substance that, in certain quantity, slows down the corrosion rate without a change in the concentration of any other corrosion agent [31]. Recently, the corrosion inhibitors are easy to implement for the protection of reinforced steel in concrete but, to be considered viable, these additives must not only prevent or delay the apparition of the corrosion, but also must not have any harming effect over the properties of concrete like power, curing time, workability and durability [27,32].

Over the last decades, new types of mixtures and additives have been developed in order to reduce the risk of steel corrosion in reinforced concrete. Some corrosion inhibitors can be mixed into fresh concrete, giving greater corrosion protection. Other types of corrosion inhibitors may be applied to the surface of existing concrete structures [33]. The great majority of investigations that have been mentioned above mainly focus on the study of corrosion and physical-chemical properties in reinforced concrete beams of OPC.

The effect of the addition of corrosion inhibitors on the properties of geopolymers has not been investigated yet. However, this contribution will only investigate whether the migratory type corrosion inhibitor has an effect or not in three types of geopolymer blends prepared. Therefore, the main objective of the present study is to examine the effects of addition of amine carboxylate migratory corrosion inhibitor (MCI-2005 NS) on mechanical properties of geopolymers synthesized by alkali activation of Ecuadorian natural zeolite-rich tuff with sodium hydroxide NaOH and Ca(OH)₂ (1–3 wt %). All the synthesized new geopolymer samples were characterized by TGA/DSC (different heating rates), XRD, SEM-EDS methods.

2. Experimental part

2.1. Materials

Ecuadorian natural zeolite-rich tuff was supplied by Zeolitas S.A. Company, which is located in the city of Guayaquil-Ecuador. Zeolite was received as small rocks, which were milled to obtain a particle size of 200 μ m and dried at 60 °C for 24 h. After this process, the samples were pulverized to a particle size of <45 μ m. The zeolite powder obtained was used without any further treatment.

The amine carboxylates technology based migratory type corrosion inhibitor, MCI-2005 NS was gently supplied by Cortec[®] Corporation. MCI-2005 NS is a liquid additive that protects reinforcing steel, carbon steel, galvanized steel and other metals embedded in concrete from corrosion induced by carbonation, chlorides and atmospheric attack. This inhibitor has the following specifications: pH: 11–12 (1% solution), non-volatile content: 25–30% and density: 9.9–10 lb/gal.

Alkaline activators were selected according to the literature in which they mention high compressive strength [34-38], the use of NaOH and Ca(OH)₂ as moisturizing at different percentages. NaOH was used as pallets with a purity of 99.0% supplied by Merck Millipore and Ca(OH)₂ powder with a purity grade of 97.2% supplied by J.T. Baker origin.

2.2. Synthesis, testing and characterization

The natural zeolite was activated with an 8 M NaOH solution with different percentages of Ca(OH)₂, (1–3 wt% of the mass of zeolite) to prepare each geopolymer sample (ZG). The reactive and zeolite were mixed by adding the MCI-2005 NS corrosion inhibitor in the amount of 1 $1/m^3$, according to product specifications. Samples were prepared at the constant activator/binding ratio of 0.6. A HOBART model N-50 mechanical mixer with mixing speeds and agitator type according to the standard ASTM C305 [39], was used to mix the components and prepare the geopolymer samples. The geopolymer obtained paste was poured into $50 \times 50 \times 50$ mm cubic molds according to standard ASTM C109/C109 M-05 [40,41] and covered with a plastic packing to prevent dehydration, excessive moisture loss and thermal stress in the geopolymer structure [42], cured for 24 h at 40 °C, and then further demolded and cured at room temperature for 6 days more.

To determine the particle size reliability, a random sample of zeolite powder was analyzed on the Mastersizer 2000, Hydro SM equipment. The distribution of the natural zeolite particle size is presented in Fig. 1. According to this figure, the natural zeolite studied has particles in the range of $1 - 45 \,\mu\text{m}$. Most of these particles are in the range of $10 \, to \, 20 \,\mu\text{m}$ and approximately 90% of the particles have a size of $> 12 \,\mu\text{m}$.

Compressive strength tests were performed using a SHIMADZU Universal Testing Machine model UTM-600KN to the cubes for the aforementioned curing times in order to evaluate the compressive force according to ASTM C109/C109 M-05 [40].

The microstructure, elemental analysis and mineral content of the zeolite as well as the activated materials was carried out by scanning electron microscopy (SEM) equipped by energy dispersive spectrometer (EDS), and X-ray diffractometry methods, respectively. SEM-EDS micrographs were obtained using a Scanning Electron Microscope equipped with a FEI brand Dispersive Energy Spectrometer model Inspect S. For the SEM-EDS analysis, an aluminum sample holder was used on an adhesive carbon disk to improve the electron transfer. No metal coating was used on the sample for analysis, an acceleration voltage of 12.5 KV and an atmosphere of 80 Pa of water vapor was used, with two spot sizes for the images and a spot size of 4 for The EDS analysis.

A PANalytical[®] X'Pert PRO X-ray diffractometer (XRD) was used in conjunction with X'Pert High Score Plus[®] software to quantify the mineral and amorphous contents of both zeolite and geopolymers. The operating conditions were 40 mA and 45 KV, with step size of 0.02°, a conventional X-ray tube (Cu_Kα radiation) and a multi-channel X'Celerator detector with anti-dispersion protection. In addition, the Rietveld method was used to quantify the mineralogical and amorphous content according to experimental

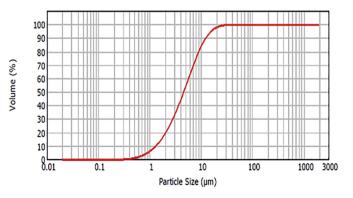


Fig. 1. Particle size distribution of natural zeolite powder prepared.

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