



Relations between structural characteristics and compressive strength in volcanic ash based one-part geopolymer systems

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ABSTRACT

Relations between structural characteristics and compressive strength in volcanic ash based *one-part* geopolymer systems are investigated employing XRD, FTIR spectroscopy, SEM/EDS and compressive strength measurements. Volcanic ash based *one-part* geopolymers are synthesized at ambient conditions with varying molar Si/Al and Na/Al ratios ranging between regions of 2.5–4.5 and 1.1–5.1, respectively. Volcanic ash is used without any thermal activation procedure. Geopolymer structures consist of x-ray amorphous glassy networks together with crystalline components (aluminosilicate and iron oxide phases) embedded in the amorphous matrices. The position of the main absorption band in the FTIR spectra indicating asymmetric stretching vibrations of Si–O–T bonds (T: Si, Al) in the geopolymeric framework constantly shifts to lower frequencies with increasing molar Si/Al and Na/Al ratios. These changes are coupled with systematic decreases in the intensities of the FTIR bands corresponding to Si–OH and carbonates. Concomitantly, the corresponding compressive strength values of *one-part* geopolymers ascend from 13.89 MPa to 19.6 MPa as the SEM morphologies of the specimens display denser, smoother and more homogenized matrices.

1. Introduction

Geopolymers are alkali activated amorphous aluminosilicate materials. There are three main reaction steps in geopolymerization: hydrolysis, oligomerization and polycondensation. When the alkali activator attacks to the surface of aluminosilicate raw material, hydrolysis reactions start and dissolution of the aluminosilicate raw material takes place. This step is followed by the formation of oligomers. Finally, these oligomers form 3-D aluminosilicate network by means of polycondensation reactions [1–3]. The structural and mechanical properties of the final product are affected by chemistry and mineralogy of the raw material, type and concentration of the alkali activator and curing conditions [4–6]. The type and concentration of the alkali activator, more specifically the available alkalinity in the environment is critical for reactions because both the dissolution of aluminosilicates and polycondensation of oligomers are controlled by the activator [6,7]. The traditional geopolymers synthesized by dissolving alkali activator in water before mixing with solid aluminosilicate precursor are referred as *two-part* geopolymers. This synthesis procedure has some disadvantages such as handling of corrosive and viscous alkaline solutions in commercial scale and scaling up problems [8]. Therefore, researchers started to focus on alternative geopolymers referred as *one-part* geopolymers that would be more convenient for commercial usage. These

geopolymers are synthesized based on “*just add water*” concept which is similar to the utilization of conventional Portland cement. In this method, water is added to a solid mixture of aluminosilicate raw materials, alkali silicates and/or alkali hydroxides [3]. The studies in this growing area report that mechanical performance of *one-part* geopolymers is comparable to the *two-part* geopolymers [8]; however, it is suggested that *one-part* geopolymer precursors should be researched in detail in terms of their mineralogy and reaction mechanisms [6]. The authors also emphasize that highly alkaline environment (pH ~ 11) is required for the dissolution of aluminosilicate raw materials and this is easily achieved with alkaline solutions in *two-part* geopolymers; on the other hand, the environment reaches slowly to higher alkalinity in *one-part* mixes. Therefore, a possible way to overcome this problem in *one-part* geopolymers is selecting an aluminosilicate raw material which has high amount of easily leachable alkali [4,6]. In addition to alkalinity, particle size of the raw material is reported to play a key role in controlling the dissolution rates of Al and Si species in the solution that directly affects the final properties of the geopolymers [9].

Aluminosilicates form approximately %60 of the earth's crust and are very abundant in the earth crust [8]. Volcanic ash is one of these aluminosilicate materials that can be utilized in the field of construction materials. Recently, the possibilities for evaluating volcanic products as recycled materials as binders in partial replacement of cement and as

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recycled aggregates in mortar and concrete production are investigated [10]. It was reported that employing volcanic pyroclastic deposits as aggregates is feasible while their utilization in cement production is not practicable based on their poor pozzolanic behavior. On the other hand, it should be noted that volcanic ash would be advantageous in geopolymerization since it does not require any thermal activation as 1:1 clays do; hence, this property provides low energy consumption and economic gain [11]. Besides, since volcanic ashes are formed and deposited on the surface during volcanic activities, surface mining also provides economic gain when compared to the other types of mining [12]. Nevertheless, there are only a few studies on geopolymerization of volcanic ash in the literature and no research about volcanic ash based *one-part* geopolymers. Studies focusing on volcanic ash based geopolymers point out low reactivity of volcanic ash in geopolymerization [13]. In order to address this issue, various pre-treatment procedures are proposed to enhance reactivity of volcanic ash including calcination [14], alkali fusion [15] and mechanical activation [16] processes. Calcination process is an energy-intensive procedure requiring temperatures between 700 and 900 °C. Alkali fusion process includes calcination of volcanic ash in the presence of NaOH at a relatively lower temperature (~500 °C). This procedure is considered to be limited because of the requirement of additional aluminosilicate sources in order to consume excess sodium hydroxide present in the system. Mechanical activation of volcanic ash is reported to increase the amount of amorphous phase as well as the number of reactive centers in the raw material and thus seems to be more effective than calcination procedures [16]. When evaluated together, all of these procedures require additional energy requirements and/or cause extra complexities in large-scale utilization of volcanic ash in geopolymerization technology. In this context, the focus of this study is i) to employ volcanic ash without any thermal pretreatment procedure in synthesizing *one-part* geopolymers that are more convenient for industrial applications, and ii) to investigate the relations between structural characteristics and mechanical properties of these geopolymers. A set of geopolymer specimens is prepared with varying amounts of Si/Al and Na/Al ratios in the regions of 2.5–4 and 1.1–5.1, respectively. These specimens are characterized by X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) Spectroscopy and Scanning Electron Microscopy (SEM). Compressive strength measurements are performed in order to complement characterization studies and to determine structure–performance relationships in this system.

2. Experimental

Volcanic ash used in this study is supplied by MK İnşaat from Manisa, Turkey. Chemical composition of volcanic ash is obtained using XRF spectroscopy and is given in Table 1. Volcanic ash is milled and sieved to 100 µm. Table 2 summarizes synthesis parameters employed for obtaining volcanic ash based *one-part* geopolymers. The specimens are named based on their molar Si/Al ratios. Volcanic ash particles are mixed with anhydrous sodium metasilicate particles for about 3 min to assure good blending of these solids and a set of solid mixtures with

Table 1
Chemical composition of (wt%) volcanic ash.

Chemical Composition	Percentage (wt%)
SiO ₂	45.7
Al ₂ O ₃	18.3
Fe ₂ O ₃	11.6
CaO	8.6
K ₂ O	5.2
Na ₂ O	3.9
MgO	2.6
TiO ₂	2.3
L.O.I	1.9

Table 2

Mixture design parameters used in the synthesis of volcanic ash based *one-part* geopolymers. Geopolymers are named using their molar Si/Al ratios. For instance, VAGP2.5 refers to volcanic ash based *one-part* geopolymer with molar Si/Al ratio of 2.5.

Geopolymer	Molar Si/Al ratio	Molar Na/Al ratio	Water/Solid ratio (g/g)
VAGP2.5	2.5	1.1	0.3
VAGP3.0	3.0	2.1	0.3
VAGP3.5	3.5	3.1	0.3
VAGP4.0	4.0	4.1	0.3
VAGP4.5	4.5	5.1	0.3

molar Si/Al and Na/Al ratios stated in Table 2 are obtained. Then, these solid mixtures are directly mixed with water using a water/solid (g/g) ratio of 0.3. The mixtures are stirred for about 20 min at 400 rpm in a low shear stirrer (Heidolph, RZR 2041) and poured into prismatic 4cmX4cmX16cm steel molds. Geopolymer specimens are cured at 60 °C for ten days under atmospheric conditions before structural characterization and performance measurements. Compressive strength measurements are performed on cubic 4cmX4cmX4cm specimens that are derived from prisms at the end of the curing period.

X-ray Diffraction (XRD) measurements are performed using a Bruker D8 Discover X-Ray Diffraction system with a Cu Kα₁ radiation source operating with a voltage and current of 40 kV and 40 mA, respectively, and scanning with a step size of 0.01260° for 2θ from 10° to 90°. ICDD PDF-4 2016 database was used for identification of phases. Fourier Transform Infrared (FTIR) spectra of the specimens are collected with Attenuated Total Reflection (ATR) technique using a Perkin Elmer One Spectrometer to detect 650–2000 cm⁻¹ spectral region. Morphological characteristics of the geopolymers in this study are investigated using Scanning Electron Microscopy (SEM). FEI-Philips XL30 ESEM-FEG with SE/BSE detector and EDAX EDS Analysis System were used on powdered samples for morphology and elemental analysis, respectively. Images were obtained under ultravacuum with an accelerating voltage of 15 kV and working distance of 9.5 – 11.2 mm. Compressive strength measurements are performed with a servo-hydraulic test machine (model: MTS) with capacity of 100 kN and loading rate 0.01 mm/s. The compressive strength measurements are run in duplicates and average values are reported.

3. Results and discussions

The XRD pattern of the volcanic ash used in this study is presented in Fig. 1. It displays a broad feature (halo) between 2θ region of 15–35° demonstrating that volcanic ash predominantly consists of an amorphous structure. The crystalline phases detected in volcanic ash are augite [(Mg_{0.73}Fe_{0.23}Al_{0.02}Ti_{0.02})(Ca_{0.83}Na_{0.02}Mg_{0.04}Fe_{0.11})(Si_{1.92}Al_{0.08}O₆)] (PDF: 01–073–8536), anorthite [CaAl₂Si₂O₈] (PDF: 00–041–1486), albite [NaAlSi₃O₈] (PDF: 04–007–5466), diopside [(Ca_{0.99}Fe_{0.01})(Mg_{0.83}Fe_{0.17})Fe_{0.02}(Si₂O₆)] (PDF: 01–077–5197), hematite [Fe₂O₃] (PDF:

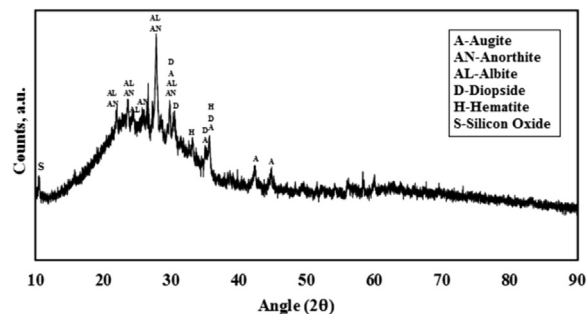


Fig. 1. XRD pattern of volcanic ash.

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