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Utilization of volcanic ashes for the production of geopolymers cured at ambient temperature

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ABSTRACT

Two types of volcanic ash were characterized (chemical and mineralogical compositions, amorphous phase composition, particle size distribution and specific surface area) and then used as raw materials for the synthesis of geopolymer cements cured at ambient temperature (24 ± 3 °C). The synthesized products were characterized by determination of setting time, 28-day compressive strength, X-ray diffraction and Fourier Transform Infrared Spectroscopy. The mineralogical composition, the amorphous phase composition, the particle size distribution, the specific surface area of the volcanic ashes as well as the molar ratios of Na_2O/Al_2O_3 of the synthesized products and of SiO_2/Na_2O of the alkaline solutions were the main parameters which influenced the synthesis of geopolymers with attractive characteristics at ambient curing temperature. The volcanic ash sample whose mineralogical composition contained anhydrite, low content of free CaO, low specific surface area (2.3 m^2/g) and synthesized products with Na_2O/Al_2O_3 molar ratios between 1.23 and 1.44 led to long setting time (test samples could be handled easily only after 14 days at ambient atmosphere of the laboratory) and low 28-day compressive strength (9–19 MPa) geopolymers. Moreover, its products swelled and presented cracks resulting from the formation of ettringite. The volcanic ash sample with large ($Al_2O_3 + SiO_2$)% wt of amorphous phase, high specific surface area (15.7 m^2/g) and synthesized products with Na_2O/Al_2O_3 molar ratios between 1.04 and 1.31 led to more effective geopolymers: setting time was between 490 and 180 min and 28-day compressive strength between 23 and 50 MPa at ambient curing temperature (24 ± 3 °C).

1. Introduction

Geopolymers are prepared by alkaline activation of aluminosilicate materials such as calcined clays, certain industrial wastes and natural minerals or a mixture of such materials cured at ambient or slightly higher temperature [1]. The geopolymerization process depends on many parameters including chemical and mineralogical compositions, particle size distribution and specific surface area of the raw material, curing temperature, composition of alkaline solution, etc. Geopolymers are attractive for building materials, hazardous waste encapsulation, high strength, low shrinkage, low carbon emission, acid resistance, etc. [1,2].

The raw materials commonly used to synthesize geopolymers are fly ash, metakaolin, or certain industrial wastes [3–7]. Volcanic ashes are almost aluminosilicate-type whose percentage in Al_2O_3 and SiO_2 can allow their utilization in the synthesis of geopolymers [8]. An increasing number of researchers have been using volcanic ash as raw materials for the synthesis of geopolymers [9,10]. So far, geopolymer pastes synthesized from volcanic ash have either exhibited long setting time [11] or have not yet led to cement pastes with compressive strength greater than 44 MPa when cured between 20 and 40 °C [9,10]. This might result from chemical and mineralogical compositions of the used raw materials. The "Cameroon line" oriented N30°E is made up of volcanic units and volcano-plutonic complexes of tertiary age [12] whose activities have generated vast deposits of volcanic ash [13]. In Cameroon, little amount of this raw material is used as additive for the production of Portland cement, for improvement of the quality of untarred roads or as aggregate for concrete. The possibility of utilizing volcanic ash for the synthesis of effective geopolymers cured at ambient temperature could be of economic importance for countries with huge deposits of this raw material.

In this study, volcanic ashes were collected from two Cameroonian localities (Djoungo and Galim) and used in their natural state for the synthesis of geopolymers cured at ambient temperature (24 ± 3 °C). For this purpose, the volcanic ashes were characterized by determining their chemical and mineralogical compositions, chemical composition of amorphous phase, particle size distribution and specific surface area. Five alkaline solutions with SiO_2/Na_2O molar ratios between 0.7 and 1.4 were prepared and used as activators for the synthesis of geopolymer cement pastes. The products were characterized by determination of setting time and 28-day compressive strength. Hardened geopolymer cement

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paste powders were also submitted to X-ray diffraction and Fourier Transform Infrared analysis.

2. Materials and experimental methods

2.1. Materials

The volcanic ashes originate from Djoungo and Galim, two localities along the "Cameroon line" [12]. They are vesicular particles of low density and various forms and they are referenced as Z_D (volcanic ash from Djoungo) and Z_G (volcanic ash from Galim). Z_D is dark-red and partially used for improvement of the quality of untarred roads or as additive for the production of Portland cement. Z_G particles are black and the local population commonly uses them as aggregate for concrete. The collected samples were washed with distilled water, dried at 105 °C for 72 h, ground and then sieved to 80 μm. A brick-red powder was obtained from Z_D and a grey powder from Z_G.

The alkaline solution was a mixture of an aqueous solution of sodium hydroxide (12 M) and sodium silicate with bulk density of 1400 kg/m^3. The sodium hydroxide solution was obtained by dissolving sodium hydroxide pellets of 99% purity in distilled water. The sodium silicate solution was made up of 28.7% SiO_2, 8.9% Na_2O and 62.4% H_2O by weight. In order to determine the most convenient alkaline activating solution for effective geopolymers, five mixtures with SiO_2/Na_2O molar ratios of 0.7, 0.9, 1.1, 1.3 and 1.4 were prepared. Before use, the alkaline activating solutions were stored at ambient temperature (24 ± 3 °C) for at least 24 h.

2.2. Experimental methods

The chemical analyses were carried out by Inductive Coupled Plasma-Atomic Emission Spectrometry. The crystalline phases were determined by X-ray diffraction using a Philip PW 3050/60 diffractometer, operating by reflexion of $K_{\alpha 1}$ radiation of Cu. The particle size distribution was performed using a courter LS 130 and the specific surface area by BET method using a Micromeritics Flowsorb ASAP 202. The amorphous phase content was determined by a method performed by Segalen [14]. Determination of the chemical composition of amorphous phase consisted of subtracting the content of dried unreacted matter via Segalen's method from the bulk composition. Cement pastes were obtained by mixing powder of volcanic ashes and alkaline activating solution for 10 min in a Hobart mixer (*M&O, modèle N50-G*). In order to get mixtures of good workability, the liquid/solid mass ratios were 0.49 (products from Z_G) and 0.37 (products from Z_D). Since the alkaline solutions were of SiO_2/Na_2O molar ratios 0.7, 0.9, 1.1, 1.3 and 1.4, the cement pastes produced were labeled as Z_{D7}, Z_{D9}, Z_{D11}, Z_{D13} and Z_{D14} (Z_D-based geopolymers) and Z_{G7}, Z_{G9}, Z_{G11}, Z_{G13} and Z_{G14} (Z_G-based geopolymers). Fresh pastes were molded in cylindrical PVC moulds (diameter 31 mm; height 62 mm). Once molded, they were vibrated for 10 min on an electrical vibrating table (*M & O, type 202, N°106*) to remove entrapped air bubbles, then covered with a thin film of polyethylene to avoid water evaporation and later on cured at ambient temperature (24 ± 3 °C) for 24 h before demolding (except the Z_D-based geopolymers which needed more days for consolidation). Setting time was measured using the Vicat apparatus according to the EN 196-3 standard. Compressive strength was determined on hardened geopolymer cement cylinder paste samples aged of 28 days using an electro-hydraulic press (*M & O, type 11.50, N°21*) at an average rate of 3 mm/min. Fourier Transform Infrared Spectroscopy was performed on powders of Z_D and Z_G and 28-day geopolymers with the aid of a Bruker Alpha-p, operating in absorbance mode.

Table 1
Chemical composition of the volcanic ashes (LOI = loss on ignition). Z_D: volcanic ash from Djoungo region and Z_G: volcanic ash from Galim region.

Oxides (%)	Z_D	Z_G
SiO_2	44.04	41.36
Al_2O_3	15.26	15.41
Fe_2O_3	12.77	12.88
TiO_2	2.87	3.04
MnO	0.17	0.2
MgO	7.00	6.45
CaO	9.29	7.88
K_2O	1.35	0.90
Na_2O	5.64	2.22
SO_3	0.01	Trace
P_2O_5	0.53	0.48
Cr_2O_3	0.02	0.03
LOI	1.1	9.31
SiO_2/Al_2O_3 (molar ratio)	4.90	4.55
(SiO_2 + Al_2O_3)	59.30	56.77
Total	100.04	100.10

Table 2
Chemical composition of amorphous phase of the volcanic ashes, % wt.

Volcanic ashes	Z_D	Z_G
Al_2O_3	5.31	11.15
SiO_2	15.33	25.80
CaO	3.23	5.11
Other oxides + organic matter	10.93	22.74
Total	34.8	64.8

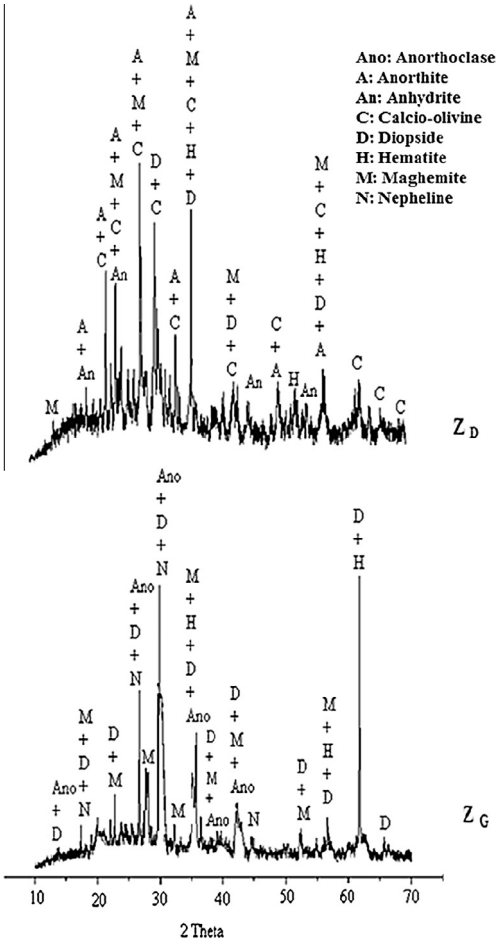


Fig. 1. X-ray diffractograms of the volcanic ashes.

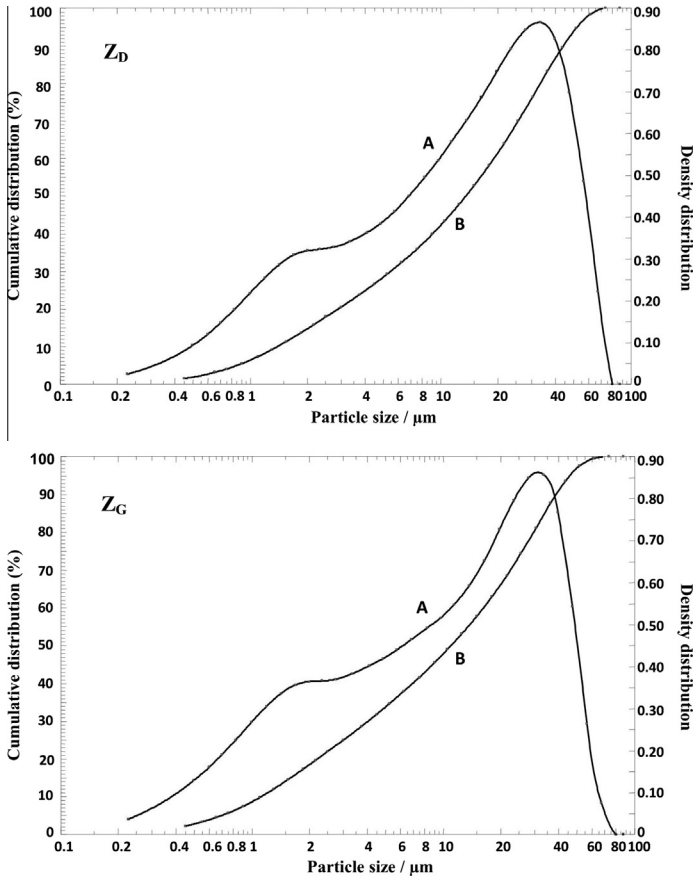


Fig. 2. Particle size distribution of the volcanic ashes (A: density distribution; B: cumulative distribution).

3. Results and discussion

3.1. Characteristics of volcanic ashes

The chemical composition of the volcanic ashes is summarized in Table 1. The SiO_2/Al_2O_3 molar ratio is 4.90 for Z_D and 4.55 for Z_G and the sum ($SiO_2 + Al_2O_3$) is 59.4% wt for Z_D and 56.8% wt for Z_G. These values are among the basic ingredients of raw materials used for geopolymer synthesis and are contained in the intervals reported by Davidovits [3] and Palomo et al. [4]. Calcium oxide content has been reported to have a significant influence on the properties of geopolymer cements [15,16] and is 9.29% wt for Z_D and 7.88% wt for Z_G. Loss on ignition is 1.1% wt (Z_D) and 9.3% wt (Z_G) which may likely indicate presence of organic matter in Z_G. The chemical composition and amount of amorphous phase of Z_D and Z_G are given in Table 2. It is evident that presence of amorphous phase with great content of ($SiO_2 + Al_2O_3$)% wt in an aluminosilicate is beneficial for the synthesis of geopolymers [15,17,18]. Thus, Z_G-based geopolymers could be more effective than those of Z_D-based geopolymers. On the other hand, the greater content of free CaO in Z_G than in Z_D may allow lower setting time in the Z_G-based geopolymers [18]. By the contrast, the low content of free CaO indicates that calcium belongs mostly to the composition of Z_D crystalline phases (Tables 1 and 2). In addition to maghemite, hematite and diopside that are common minerals to these volcanic ashes, Z_D also contains calcio-olivine, anorthite and anhydrite and Z_G anorthoclase and nepheline (Fig. 1). The particle size distribution ranges from 0.23 to 80 μm, the mode is around 35 μm (Z_D) and 32 μm (Z_G) and the average particle size (d_{50}) is 13.01 and 10.68 μm respectively for Z_D and Z_G (Fig. 2). The specific surface area is 2.3 m^2/g for Z_D and 15.7 m^2/g for Z_G. These results indicate

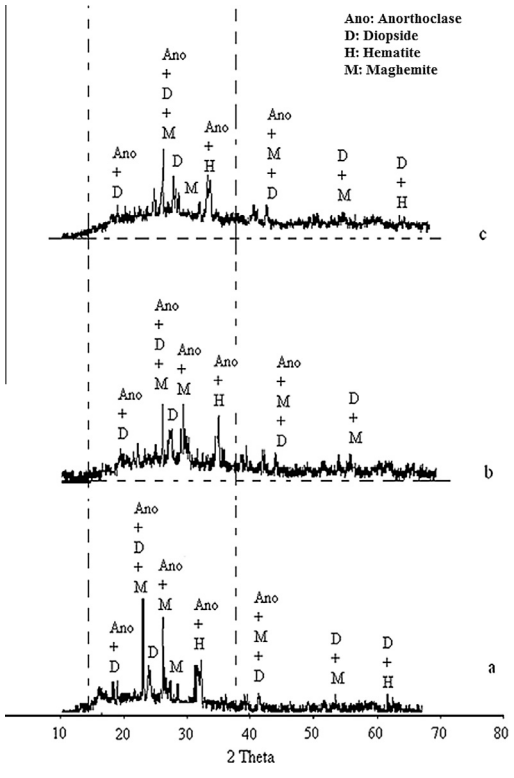


Fig. 3. X-ray diffractograms of the Z_G-based geopolymers ($a = Z_{G7}$; $b = Z_{G11}$; $c = Z_{G14}$).

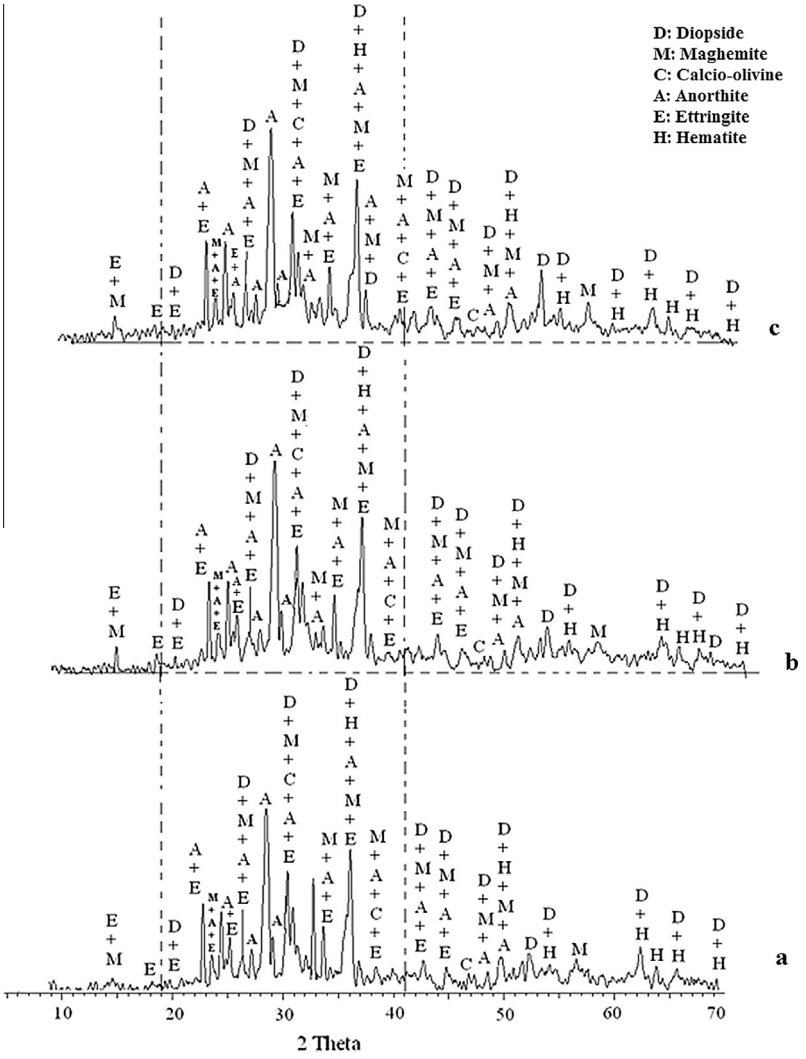


Fig. 4. X-ray diffractograms of the Z_D-based geopolymers ($a = Z_{D7}$; $b = Z_{D11}$; $c = Z_{D14}$).

good correlation between the particle size distribution and the specific surface area, i.e. the finer the particles, the greater the specific surface area. The greater specific surface area exhibited by Z_G by comparison with Z_D may result from presence of more porous microstructure of Z_G particles. Particle size distribution is one of the important physical parameters impacting on geopolymer synthesis of aluminosilicates as well as on resulting products since a significant part of reaction occurs at the particle–liquid interface [19]. Thus for a given aluminosilicate raw material, the smaller and the more porous microstructure particles, the greater the specific surface area and the more it is activated in alkaline medium. All these results suggested that the Z_G-based geopolymers could be more effective than the Z_D-based geopolymers.

3.2. Characteristics of geopolymers

3.2.1. X-ray diffraction

Figs. 3 and 4 show the X-ray diffraction patterns of the geopolymers. Except anhydrite and nepheline, all the crystalline phases observed are the same as those present in Z_D and Z_G (Figs. 1, 3 and 4). The presence of ettringite ($6CaO{\cdot}Al_2O_3{\cdot}3SO_3{\cdot}31H_2O$) in the Z_D-based geopolymers originates from the chemical reaction between anhydrite ($CaSO_4$) and $[Al(OH)_4]^-$ complex resulting from dissolution of the raw material in alkaline medium [20]. The absence of nepheline in the Z_G-based geopolymers indicates that it has reacted. In fact, nepheline is an aluminosilicate. The halo peak between 18° and 40° is characteristic of amorphous phase of geopolymers. However the area under each halo peak of the diffractograms increases with the SiO_2/Na_2O molar ratio of the alkaline solutions (Figs. 3 and 4). It seems as if there exists a correlation between this area and the amount of amorphous phase contained in the geopolymers. In fact, an increase of the SiO_2/Na_2O molar ratio of the alkaline solution promotes the polycondensation stage of the geopolymer synthesis [21].

3.2.2. FTIR spectra

Figs. 5 and 6 show the FTIR results of the volcanic ashes and the geopolymers. The spectra of Z_D and Z_G (Figs. 5a and 6a) exhibit two main absorption domains and those of the geopolymers three (Figs. 5b–d and 6b–d). The first absorption band is a sharp peak at 2360 cm^{-1} and concerns the geopolymers. This peak is characteristic of stretching vibrations of –OH under strong hydrogen bonding [21], which gives evidence of adsorbed water in the geopolymers. The second domain is made up of two absorption bands, one at 1650 cm^{-1} and the other at 1540 cm^{-1}. Both indicate the H–O–H bending vibrations of water molecules [22]. The third domain contains an important absorption band around 950–1000 cm^{-1} and is attributed to asymmetric stretching vibrations of Si–O–Si [23]. The intensity of this peak is high enough for volcanic ashes but is obviously reduced with the increasing of SiO_2/Na_2O molar ratio of alkaline solution as regards the geopolymers (Figs. 5b–d and 6b–d). This result seemed to indicate the rareness of Si–O–Si

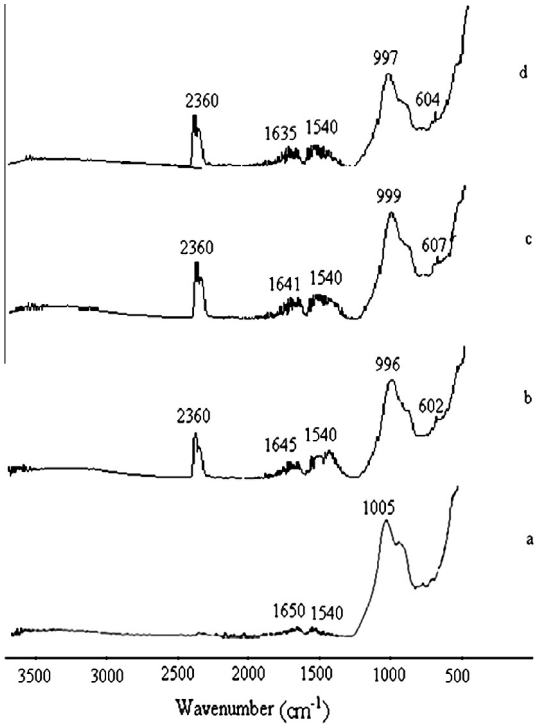


Fig. 5. IR spectra of the Z_G-based geopolymers ($a = Z_G$; $b = Z_{G7}$; $c = Z_{G11}$; $d = Z_{G14}$).

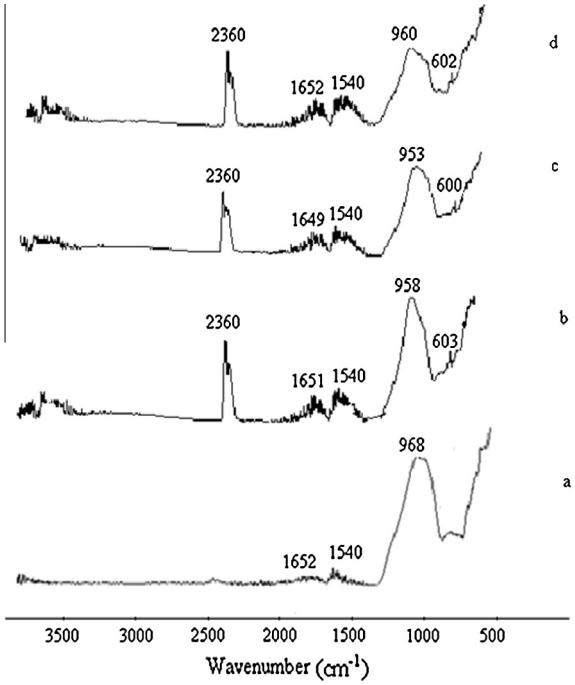


Fig. 6. IR spectra of the Z_D-based geopolymers ($a = Z_D$; $b = Z_{D7}$; $c = Z_{D11}$; $d = Z_{D14}$).

groups when the synthesis of geopolymers is on. The weak peak around 600 cm^{-1} is ascribed to the symmetric stretching vibrations of Si–O–Al and Si–O–Si groups of the geopolymers [24].

3.2.3. Setting time

The Z_D-based geopolymers could be handled easily only after 14 days at ambient curing temperature (24 ± 3 °C). As a result of this, their setting time was not taken into account in Fig. 7. Setting time is generally influenced by parameters such as chemical composition and particle size distribution of raw material used for the synthesis of geopolymers [1]. Thus, long setting time of the Z_D-based geopolymer pastes could result from both low specific surface area (2.3 m^2/g) and low content of free CaO (3.23% wt) of amorphous phase of Z_D particles. In fact, the lower the specific surface area and content of free CaO of an aluminosilicate, the higher the setting time of geopolymer pastes [17,18,25]. Elsewhere, setting time of the Z_G-based geopolymer pastes was between 490 and 180 min, and it decreased with the increase of SiO_2/Na_2O molar ratio of alkaline solutions. This result was in accordance with specific surface area (15.7 m^2/g) and content of free CaO (5.11% wt) of Z_G particles along with the result obtained by Cheng and Chiu [26] who showed that the lower the SiO_2/Na_2O molar ratio, the higher the setting time.

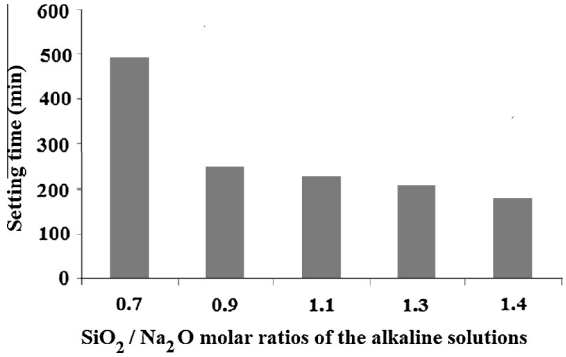
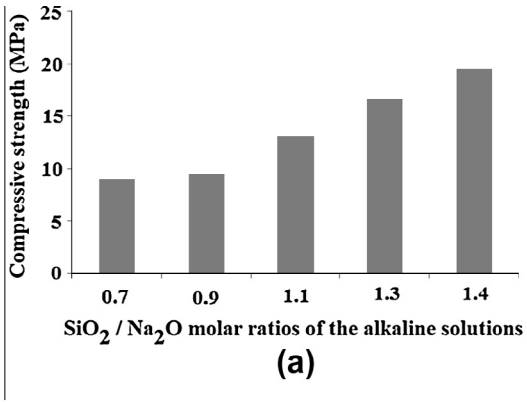


Fig. 7. Setting time of the Z_G-based geopolymers.



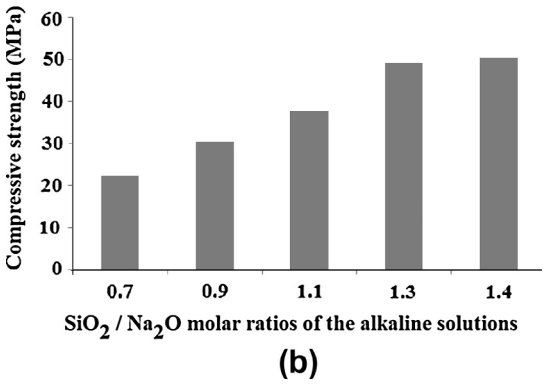


Fig. 8. Compressive strength of the Z_D-based geopolymers (a) and the Z_G-based geopolymers (b).

3.2.4. Compressive strength

Fig. 8 shows the 28-day compressive strength of the geopolymers cured at ambient temperature (24 ± 3 °C) as a function of

Table 3
Al_2O_3/SiO_2 and Na_2O/Al_2O_3 molar ratios of geopolymers.

Geopolymers	Z_{D7}	Z_{D9}	Z_{D11}	Z_{D13}	Z_{D14}	Z_{G7}	Z_{G9}	Z_{G11}	Z_{G13}	Z_{G14}
SiO_2/Al_2O_3	5.43	5.55	5.64	5.70	5.75	5.26	5.41	5.52	5.60	5.66
Na_2O/Al_2O_3	1.44	1.36	1.30	1.28	1.23	1.31	1.20	1.13	1.08	1.04

SiO_2/Na_2O molar ratio of the alkaline solutions. Compressive strength increased with increase of SiO_2/Na_2O molar ratio of the alkaline solutions. It is well known that increase of SiO_2/Na_2O molar ratio of alkaline activating solution results in acceleration of geopolymer synthesis and causes the reaction to proceed to a higher extent [26]. In addition, better strength properties are reported for geopolymers with SiO_2/Al_2O_3 molar ratios in the range of 3.0–3.8 and Na_2O/Al_2O_3 molar ratios of about 1 [27,28]. From the previous reports, it appeared that the synthesized geopolymers fell down with regard to their SiO_2/Al_2O_3 molar ratios but the sample Z_{G14} as regards its Na_2O/Al_2O_3 molar ratio (Table 3). However, it is interesting to mention that the Z_G-based geopolymers exhibited higher 28-day compressive strength values (23–50 MPa) than the Z_D-based geopolymers (9–19 MPa) at ambient curing temperature (24 ± 3 °C). Although silica and alumina are among the main precursors for the geopolymer reaction, there are other characteristics of raw materials and of synthesized products that play significant roles to get high compressive strength values. Among them are the composition of amorphous phase (Table 2), the average particle size (13.01 μm for Z_D and 10.68 μm for Z_G), the specific surface area (2.3 m^2/g for Z_D and 15.7 m^2/g for Z_G) as well as the Na_2O/Al_2O_3 molar ratios of the geopolymers (Table 3). In fact, during the reaction between volcanic ash and alkaline solutions, the higher the content of $(Al_2O_3 + SiO_2)$% wt of amorphous phase, the higher the amount of $[SiO(OH)_3]^-$ and $[Al(OH)_4]^-$ intermediate complexes released at the stage of dissolution of the aluminosilicate [19]. This allows the formation of a large amount of geopolymer binder that leads to high 28-day compressive strength values. The particle size also affects geopolymerization reaction: the finer the particle size the greater the specific surface area and the more reactive the raw material [19]. The low 28-day compressive strength values of the Z_D-based geopolymers might also be attributed to the swelling and the presence of cracks which appeared in the products few days after demoulding. These cracks resulted from presence of ettringite in the geopolymers [22,29]. In fact, presence of ettringite is attributed to the chemical reaction between anhydrite (Fig. 1) and $[Al(OH)_4]^-$ intermediate complex formed during dissolution of volcanic ashes in alkaline medium. Greater compressive strength values may be expected from volcanic ash-based geopolymers via slight increase of curing temperature [9].

4. Conclusion

This study investigated the utilization of two types of volcanic ash to produce geopolymers cured at ambient temperature. The properties of the synthesized products were dependent on certain characteristics of the raw materials. The volcanic ash sample with low specific surface area and low content of free CaO led to geopolymers with long setting time. On the other hand, the formation of ettringite generated swelled products with cracks which led to low compressive strength (9–19 MPa). The volcanic ash sample with great specific surface area and important $(Al_2O_3 + SiO_2)$% wt of amorphous phase gave geopolymers with compressive strength between 23 and 50 MPa. Although the SiO_2/Al_2O_3 molar ratios of the investigated geopolymers were greater than those reported by literature to give better properties, it was possible to get Na_2O/Al_2O_3 molar ratios giving attractive geopolymers. Anyway, volcanic ash has the potential to be raw material for geopolymer synthesis.

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