Available online at www.sciencedirect.com

SciVerse ScienceDirect

Ceramics International 39 (2013) 269–276

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Synthesis of geopolymers from volcanic ash via the alkaline fusion method: Effect of Al_2O_3/Na_2O molar ratio of soda–volcanic ash

H. Kouamo Tchakoute[a,*], A. Elimbi[a,*], B.B. Diffo Kenne[a], J.A. Mbey[a,b], D. Njopwouo[a]

[a]*Laboratoire de Physico chimie des Matériaux Minéraux. Université de Yaoundé I. Faculté des Sciences, Département de Chimie Inorganique, B.P. 812 Yaoundé, Cameroon*

[b]*Laboratoire Environnement et Minéralurgie, UMR-Université de Lorraine, 15 Avenue, du Chamois, B.P. 40, F-54501 Vand?uvre-lès-Nancy Cedex, France*

Received 23 April 2012; received in revised form 30 May 2012; accepted 7 June 2012
Available online 18 June 2012

Abstract

The alkaline fusion method was used to enhance the reactivity of volcanic ash for geopolymer synthesis. To that end, different mixtures of fused soda–volcanic ash (fused volcanic ash) were used to assess reactivity for geopolymer synthesis. The amount of amorphous phase was determined both in the volcanic ash and the fused volcanic ash and X-ray diffraction analysis was used to evaluate effect of the alkaline fusion method. Different geopolymer mortars were prepared by alkaline activation of mixtures of powders of fused volcanic ash and metakaolin and river sand using sodium silicate as activator. Metakaolin was considered as consumer of excess of alkali contained in the fused volcanic ash. The geopolymer mortars were characterized by determination of setting time, linear shrinkage, compressive strength and scanning electron microscopy. The amount of amorphous phase and excess of fused soda content of the fused volcanic ash depended on molar ratio of Al_2O_3/Na_2O and played a key role for geopolymer synthesis. The most convenient Al_2O_3/Na_2O molar ratio of fused volcanic ash to produce effective geopolymer mortars ranged between 0.13 and 0.18. This study showed that volcanic ash can be used successfully as an alternative raw material for production of geopolymers via alkaline activation of fused volcanic ash.

Keywords: Alkaline fusion method; Fused volcanic ash; Geopolymer synthesis; Compressive strength

1. Introduction

Geopolymers were introduced by Davidovits in the early 1970s. They are a class of X-ray amorphous materials largely synthesized by reaction between aluminosilicate and highly concentrated alkaline solution at ambient or slightly elevated temperature [1]. The network consists of SiO_4 and AlO_4 tetrahedra linked alternately by sharing all the oxygen atoms. The Al^{3+} ion in four-fold coordination demands the presence of cations such as K^+, Na^+, Li^+ or Ca^{2+} in the framework to balance the negative charge. According to Davidovits [1], the empirical chemical formula of geopolymers also known as poly(sialates) is as follows: $M_n\{-(SiO_2)_z-AlO_2\}_n \cdot wH_2O$ where M is a cation, n the degree of polycondensation, w the number of water molecules and z equals to 1, 2 or 3. Compared to conventional ordinary Portland cement, geopolymer cements possess some advantages such as higher mechanical strength, excellent chemical resistance, fire resistance, low thermal conductivity, low shrinkage, etc. [2,3]. Previous studies showed that geopolymers synthesized using fly ash, blast furnace slag or metakaolinite as aluminosilicates led to products with compressive strength higher than those synthesized using minerals such as kaolinites, albite, stilbite, etc. [4].

Volcanic ash is made up of low density vesicular fragments with chemical composition varying from batch to batch [5]. Those fragments contain variable amount of amorphous phase which can react with portlandite generated by cement hydration to yield hydrated calcium silicates and aluminates with low solubility and good cementitious properties [6,7]. Recent studies reported volcanic ash to be a potential candidate for the synthesis

*Corresponding authors. Tel.: +237 77979617/+237 77612623
E-mail addresses: htchak@yahoo.fr (H. Kouamo Tchakoute), aelimbi2002@yahoo.fr (A. Elimbi).

0272-8842/$36.00 © 2012 Elsevier Ltd and Techna Group S.r.l. All rights reserved.
http://dx.doi.org/10.1016/j.ceramint.2012.06.021

of geopolymers [5,8]. Large volcanic ash deposits exist along the "Cameroon line" oriented N30°E, specially around the Mount Cameroon, the Mount Manengouba, the Mount Bamboutos, the Mount Galim, the Tombel plain, the Noun plain, the Kumba plain, the region of Lake Nyos, the Adamaoua plateau, etc. [9]. Most of these volcanic ash deposits are readily accessible and have advantage of being low cost mined. In Cameroon little amount of volcanic ash is used as additive for the production of Portland cement, improvement of quality of untarred roads or as aggregate for concrete. Utilization of volcanic ash as aluminosilicate for production of effective geopolymers could be of great economic impact in countries with large deposits.

A previous study showed that geopolymer cements synthesized using volcanic ash as aluminosilicate and curing between 20 and 40 °C exhibited long setting time, high enough linear shrinkage and low compressive strength [10]. This could result from low amorphous phase content of such materials [11]. Because volcanic ash is generally composed of substantial amount of silica and alumina [5,8], it is convenient to take it for testing in the synthesis of geopolymers via alkaline fusion method which is a conventional process for chemical analysis. This latter is meant to decompose materials containing silicon and/or aluminum [12–16]. This method is generally used in the synthesis of zeolites [12,13]. Shigemoto et al. [12] tested the alkaline fusion of a mixture of soda–fly ash to get silicate and aluminate sodium salts.

The present study investigated the feasibility of using volcanic ash as raw material for the synthesis of geopolymers via alkaline activation of fused volcanic ash. The effect of Al_2O_3/Na_2O molar ratio in the mixtures of soda–volcanic ash was evaluated because the degree of alkaline fusion process may determine properties of fused volcanic ash. The amount of amorphous phase was determined both in the volcanic ash and the fused volcanic ash. Different geopolymer mortars were prepared by alkaline activation of mixtures of powders of fused volcanic ash, metakaolin and river sand using sodium silicate as an activator. Metakaolin known as a good source of Al_2O_3 and SiO_2 [17] was used as additional aluminosilicate to consume excess alkali contained in the fused volcanic ash. The geopolymer mortar samples were characterized by determination of setting time, linear shrinkage, compressive strength and scanning electron microscopy.

2. Experimental procedure

2.1. Materials

Both the volcanic ash denoted as Z_G and the kaolin denoted as MY which were used in this study were collected respectively from the Mount Galim (Department of Bamboutos, West Region of Cameroon) and Mayoum (Department of Noun, West Region of Cameroon). A previous study [18] reported that the main components of the kaolin are kaolinite (79% wt), quartz (8% wt) and illite (6% wt). The kaolin particles passing through 80 μm mesh were calcined at 700 °C at a programmable electric furnace (*Nabertherm*, Model LH 60/14). It was heated for 4 h at a heating rate of 5 °C/ min to get a highly reactive metakaolin (MK) [19]. Particle size distribution of powders of Z_G and MK were determined using a laser diffraction granulometer (*Sympatec*, equipped with the HELOS optical system and the WINDOX software for data acquisition). The particle size distribution curves are shown in Fig.1 and the average particle size (d_{50}) was 10.68 μm for Z_G and 9.95 μm for MK. The specific surface area of powders of Z_G and MK were determined using the Brunauer–Emmett–Teller (BET) method. It was performed via nitrogen adsorption with an automatic homemade apparatus at Laboratoire Environnement et Minéralurgie (EML, UMR-Université de Lorraine). The results were 15.7 m^2/g for Z_G and 20.5 m^2/g for MK. The chemical analyses of Z_G and MK were carried out by ICP–AES (Inductive Coupled Plasma–Atomic Emission Spectrometry) and the results were displayed in Table 1. The mineralogical compositions of MY, MK and Z_G were determined using a *Bruker D8 advance* diffractometer, operating on the cobalt kα radiation. Comparison between the XRD patterns of MY and MK (Fig. 2) showed clearly the disappearance of the kaolinite peaks and the appearance of a hump at 2θ between 18° and 40° which indicated the formation of an amorphous phase in the MK powder. The crystalline phases detected in MK were quartz (SiO_2), hematite (Fe_2O_3) and illite ($KAl_2(AlSi_3O_{10})\cdot(OH)_2$) (Fig. 2). In addition anorthoclase ($(Na,K)_2O\cdot Al_2O_3\cdot 6SiO_2$), diopside ($CaO\cdot MgO\cdot 2SiO_2$), hematite, maghemite ($Fe_2O_3$), muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$), nepheline ($Na_2O\cdot Al_2O_3\cdot 2\ SiO_2$) and a minor

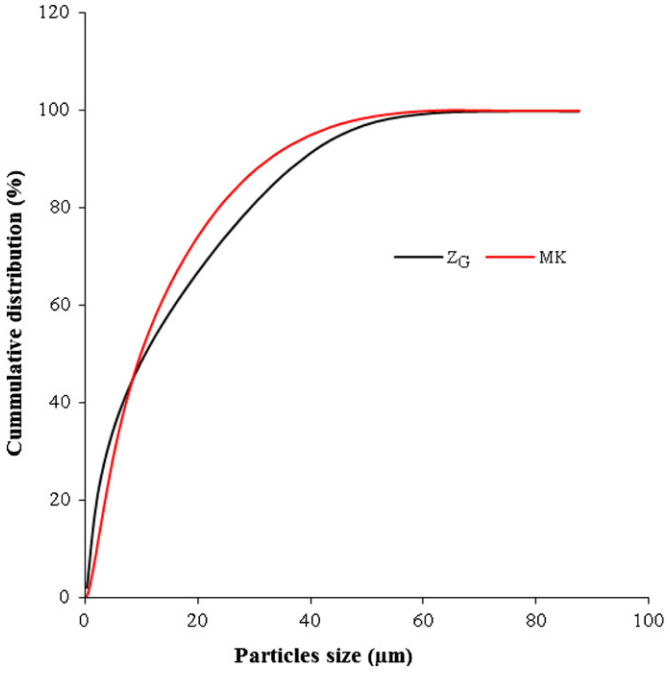

Fig. 1. Particle size distribution of the volcanic ash (Z_G) and metakaolin (MK).

Table 1
Chemical composition of volcanic ash (Z_G) and metakaolin (MK). LOI: loss on ignition.

Element as oxide	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	MnO	MgO	CaO	K_2O	Na_2O	P_2O_5	Cr_2O_3	LOI	SiO_2/Al_2O_3	Total
Z_G	41.36	15.41	12.88	3.04	0.2	6.45	7.88	0.90	2.22	0.48	0.03	9.31	4.55	100.10
MK	48.31	40.48	2.62	4.45	0.03	0.36	0.04	1.30	0.15	0.02	0.02	2.43	1.42	100

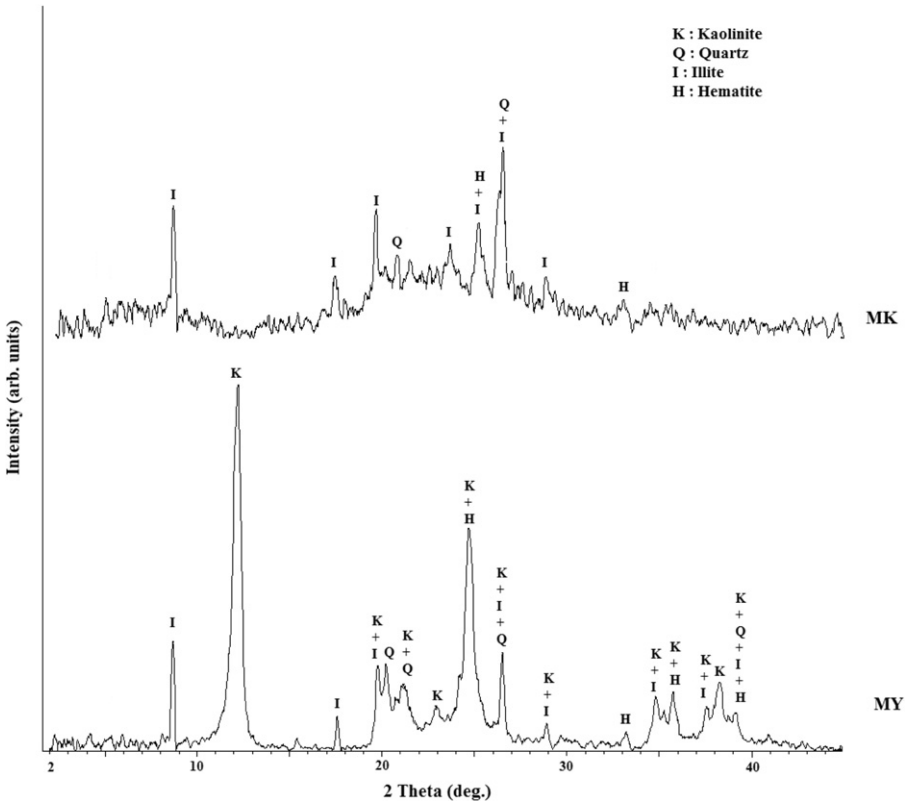

Fig. 2. XRD patterns of kaolin (MY) and metakaolin (MK).

amount of amorphous phase (Fig. 3) were found in the powders of Z_G. The aggregate used was a collected river sand with major particles passing through a 500 μm mesh sieve. The chemical composition of sodium silicate was 28.7% SiO_2, 8.9% Na_2O with the modulus 3.22. The soda was composed of pellets of sodium hydroxide of 99% purity.

2.2. Synthesis and characterization of the geopolymers

Different mixtures of soda–volcanic ash powders with Al_2O_3/Na_2O molar ratios given in Table 2 were thoroughly mixed followed by calcination in a programmable electric furnace (*Carbolite, Serial No.*11/92/1736. *Type BOF* 11/13) at 550 °C during 1 h at a heating rate of 5 °C/min. The resulted products labeled as Z_1, Z_3, Z_5, Z_7, Z_9, Z_{11}, Z_{13} and Z_{15} were ground in a mortar and sieved to get a 80 μm mesh passing powders of fused volcanic ash. Both the volcanic ash and the fused volcanic ash powders were submitted to XRD analysis to assess effectiveness of alkaline fusion process. On the other hand, a batch of geopolymer mortars was prepared between powders of the fused volcanic ash (70% wt), metakaolin (30% wt) and river sand (aggregate) using sodium silicate as alkaline activator. A binder to aggregate ratio was kept at 1:1.5 throughout the study. The assembly was mixed in a Hobart mixer (*M & O*, *model N50− G*) for 20 min. In order to get good workability, liquid/solid mass ratio of 0.60 was used. The slurry mortars were used for determination of setting time and molding of two species of cylindrical samples (diameter: 31 mm; height: 62 mm and diameter: 10 mm, height: 20 mm). Once molded, the cylinders were vibrated for 5 min on an electric vibrating table (*M & O, type 202, N° 106*) to remove entrapped air bubbles. During their hardening, the cylinder samples were covered with a thin film of polyethylene to avoid water evaporation and then kept for 24 h at the ambient atmosphere of the laboratory before demolding. Because the mixtures of soda–volcanic ash were at Al_2O_3/Na_2O molar ratios given in Table 2, the resulting geopolymers were labeled as Z'_1, Z'_3, Z'_5, Z'_7, Z'_9, Z'_{11}, Z'_{13} and Z'_{15}. Depending on duration of stay (1, 7, 14, 21 or 28 days) of the geopolymers at the ambient temperature of the laboratory (24 ± 3 °C), linear shrinkage was determined on cylinder samples with 31 mm height thanks to a calliper.

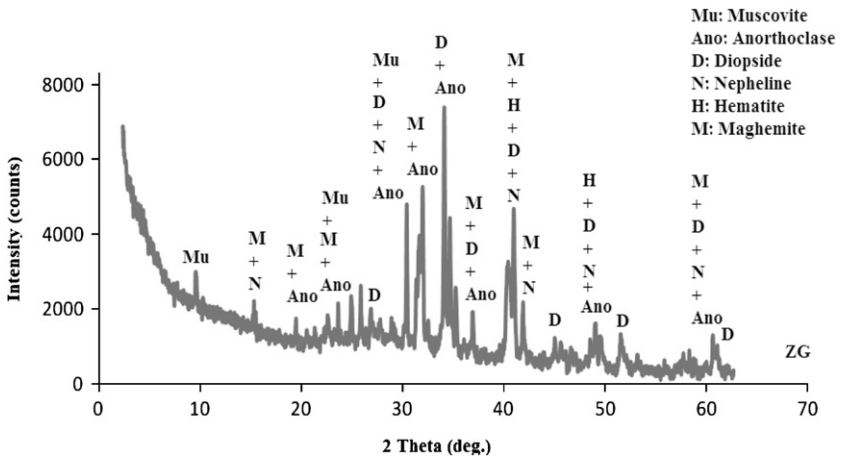

Fig. 3. XRD patterns of volcanic ash (Z_G).

Table 2
Molar ratios Al_2O_3/Na_2O and amount of amorphous phase of volcanic ash and all fused volcanic ashes.

Samples	Z_G	Z_1	Z_3	Z_5	Z_7	Z_9	Z_{11}	Z_{13}	Z_{15}
Al_2O_3/Na_2O (molar ratios)	/	0.77	0.29	0.18	0.13	0.10	0.08	0.07	0.06
Amount of amorphous phase (%)	29.0	6.7	34.3	51.0	76.0	73.7	67.3	60.3	60.1

Compressive strength was determined on cylinder samples with 31 mm height and aged 28 days using an electro-hydraulic press (*M & O, type* 11.50, No. 21) at an average rate of 3 mm/min. The geopolymer cylinder samples with 20 mm height were meant for SEM analysis using a *Hitachi S−4800* scanning electron microscope. Setting time was measured using Vicat apparatus according to the EN 196-3 standard.

In order to determine the amount of amorphous phase contained both in the volcanic ash and the fused volcanic ash, the method performed by Chindaprasirt and Rattanasak [20] was used. Thus, 3 g of powder was treated with 30 mL of sodium hydroxide (8 M) at 60 °C for 1 h. The sample was cooled to room temperature and filtered through pre-weighed membrane-filter. The insoluble residue was washed to neutral pH, dried at 105 °C for 24 h and weighed. The amount of amorphous phase was determined as the weight of powder material minus the residue.

3. Results and discussion

3.1. Characterization of fused volcanic ash

3.1.1. X-ray diffraction

The XRD patterns of the fused volcanic ash were presented in Fig. 4. All the minerals initially contained in the volcanic ash remained but at different scales (variation of heights of their peaks on the diffractograms) except muscovite which was totally consumed in the samples of Z_3–Z_{15} (Figs. 3 and 4b–h). This could mean that either these minerals were partially dissolved in fused soda or they had reacted with soda. The case of muscovite was particular: it is an aluminosilicate which may have reacted with soda through geopolymer synthesis or was dissolved in fused soda. The presence of muscovite on the diffractogram of the sample Z_1 may express an insufficient amount of soda to allow complete reaction or total dissolution. Sodium hydroxide was not observed on all the diffractograms which meant that either it had reacted or its excess was amorphous. Two groups of XRD patterns were observed: the group with broad hump at 2θ between 30° and 45° (Fig. 4b–f) and the group with small hump at 2θ between 30° and 45° (Fig. 4a, g and h). Generally hump at 2θ between 30° and 40° on diffractograms of aluminosilicates expresses the presence of amorphous phase [8,17]. It could consequently be concluded that effectiveness of getting fused volcanic ash with abundant amorphous phase was reached by using mixtures of soda–volcanic ash with Al_2O_3/Na_2O molar ratios between 0.29 and 0.06 (Table 2). The XRD patterns of the samples Z_{13} and Z_{15} revealed the presence of thermonatrite ($Na_2CO_3 \cdot H_2O$) and sodium carbonate (Na_2CO_3) (Fig. 4g and h). These crystalline phases resulted from chemical reaction between excess of fused soda and carbon dioxide in the presence of water at ambient air.

3.1.2. Amount of amorphous phase

Although the XRD patterns could be used to express qualitatively the variation of amorphous phase content of both the volcanic ash and the fused volcanic ash (i.e. extent of the hump at 2θ between 30 and 45° in Figs. 3 and 4),

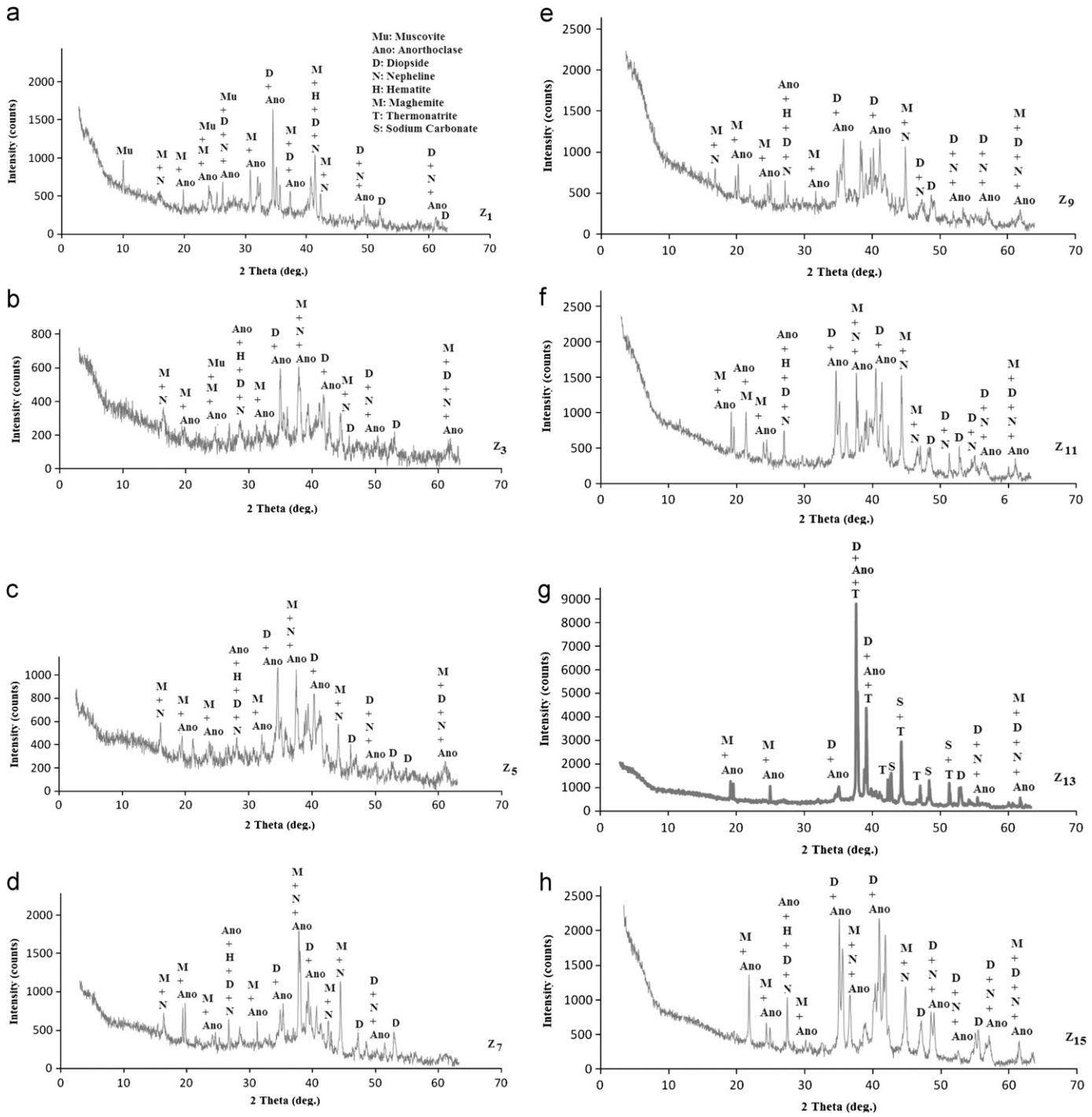

Fig. 4. XRD patterns of all fused volcanic ash.

they did not reveal how readily this phase was available. Therefore the alkaline fusion method was used to quantitatively determine the amount of amorphous phase content of the fused volcanic ash and the results were given in Table 2. Except the sample Z_1, the amorphous phase content of the fused volcanic ash increased with decreasing of Al_2O_3/Na_2O molar ratio as from 34.3% (sample Z_3) to 76.0% (sample Z_7) and dropped as from 73.7% (sample Z_9) to 60.1% (sample Z_{15}). It could be concluded that effectiveness of the alkaline fusion method to bring about increasing amount of amorphous phase in the fused volcanic ash was limited by the molar ratio of Al_2O_3/Na_2O. Using mixture of soda–volcanic ash with Al_2O_3/Na_2O molar ratio between 0.10 and 0.06 decreased the amount of amorphous phase. For the case of the sample Z_1, the amount of amorphous phase was drastically lower than the one contained in the volcanic ash. A clear explanation of this difference could be attributable to the

formation of other phases instead of thinking of usual analytical errors.

3.2. Characterization of the geopolymers

3.2.1. Setting time

Except for the sample Z'_1, setting time of the geopolymer mortars ranged between 15 and 25 min as illustrated in Fig. 5. The cylinder samples of Z'_1 could be easily handled only after 25 days of stay at ambient temperature of the laboratory (24 ± 3 °C). This result was close to that obtained with geopolymers of the volcanic ash [10] which showed inefficiency in testing the fused volcanic ash sample Z_1 (Al_2O_3/Na_2O molar ratio of 0.77) to get lower setting time. As it was presented in Fig. 5, setting time depended on Al_2O_3/Na_2O molar ratio of the fused volcanic ash. It decreased between 25 min (sample Z'_3) to 15 min (sample Z'_7) and increased between 16 min (sample Z'_9) to 20 min (sample Z'_{15}). These results correlated with the variation of amount of amorphous phase content of the fused volcanic ash (Table 2 and Fig. 5). The obtained results could mean that the fused volcanic ash samples of Z_9–Z_{15} contained excess of fused soda. This excess of fused soda could be likely responsible to the increase of setting time. In any way, setting time of the geopolymer mortars from the fused volcanic ash was generally lower than the one obtained from metakaolinite at ambient temperature [19,21,22].

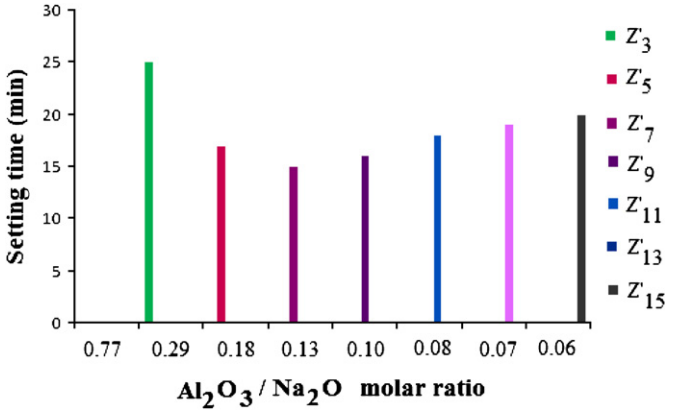

Fig. 5. Setting time of geopolymer mortars.

3.2.2. Linear shrinkage

Linear shrinkage as a function of the age of hardened geopolymer mortars was determined at ambient temperature of the laboratory (24 ± 3 °C) and the results presented in Fig. 6. According to the age of the geopolymer mortars, linear shrinkage decreased for the batch of $Z'_3 - Z'_7$ and increased for the batch of Z'_9–Z'_{15}. Linear shrinkage originates from capillary tensions within the gel framework during geopolymer synthesis [23]. Thus, high shrinkage may be an indication of insufficient geopolymer synthesis. According to the results in Fig. 6, increase of reactivity of geopolymer synthesis was observed with the samples of Z'_3–Z'_7. In spite of important amorphous phase content of the fused volcanic ash samples of Z_9–Z_{15} (Table 2), the geopolymer mortars (Z'_9–Z'_{15}) exhibited low reactivity. Such behavior was attributed to the excess of fused soda. Thus it could be concluded that the mixtures of soda–volcanic ash with Al_2O_3/Na_2O molar ratio between 0.18 and 0.13 were the most recommended ratios to get effective fused volcanic ash for geopolymer synthesis. Excess of fused soda hindered the geopolymer synthesis process of fused volcanic ash.

3.2.3. Compressive strength

The 28 days compressive strength of geopolymer mortars as a function of Al_2O_3/Na_2O molar ratio of the fused volcanic ash was determined at ambient temperature of the laboratory (24 ± 3 °C) and the results presented in Fig. 7. It appeared that the 28 days compressive strength increased between 15.3 MPa (sample Z'_1) and 41.5 MPa (sample Z'_7) and dropped between 33.5 MPa (sample Z'_9) and 14.4 MPa (sample Z'_{15}). This behavior was in agreement with the

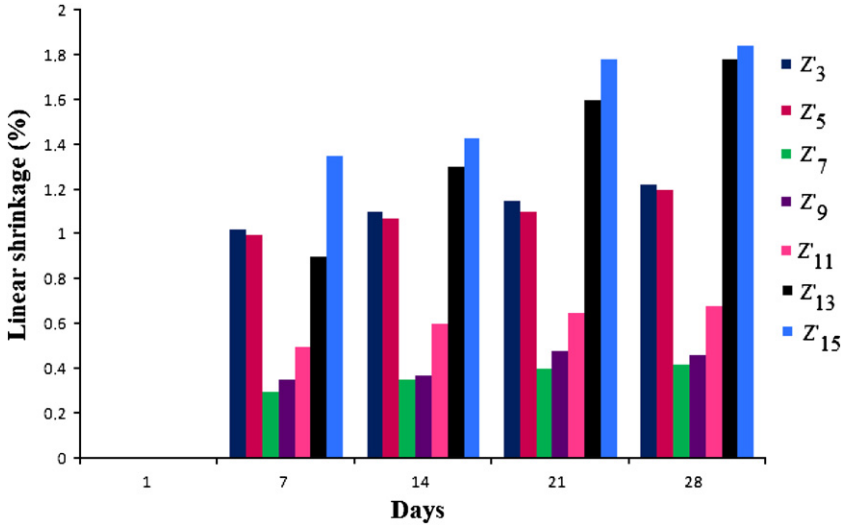

Fig. 6. Linear shrinkage of geopolymer mortars.

results of Table 2. Hence the batch of the fused volcanic ash with increasing amount of amorphous phase (the samples Z_1–Z_7) contained low amount of excess of fused soda and generated geopolymer mortars with increasing 28 days compressive strength. Excess of fused soda hindered geopolymer synthesis process. Since the amount of amorphous phase and fused soda contents were the important parameters that governed effectiveness of geopolymer synthesis, it became clear that the fused volcanic ash with Al_2O_3/Na_2O molar ratio between 0.10 and 0.06 contained low amount of geopolymer phase (Fig. 8) which may weaken likely their compressive strength.

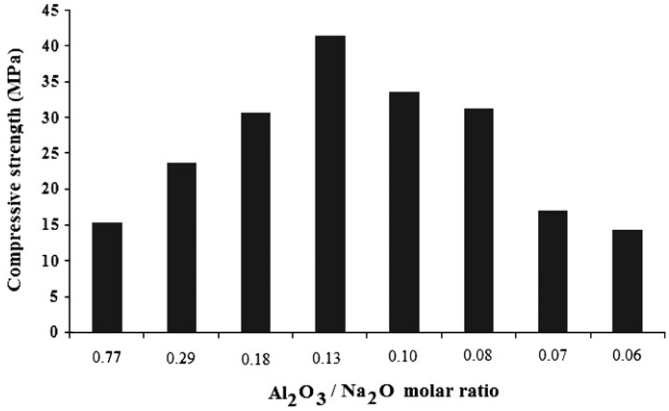

Fig. 7. Compressive strength of geopolymer mortars.

3.2.4. Microstructure

The SEM micrographs of the samples Z'_1, Z'_7, Z'_9 and Z'_{15} of geopolymer mortars were presented in Fig. 8. There were two main types of phases which composed the samples. The first type was a fairly compact matter and was made up of geopolymer phase imbedding aggregate particles. The second type was composed of heaps of aggregate poorly consolidated because of lack of enough geopolymer phase. From one micrograph to another, the microstructure exhibited differences. The micrograph of Z'_1 was composed mainly of heaps of aggregate poorly bonded and few domains of geopolymer phase which indicated poorly consolidated geopolymer mortar. It was interesting to observe abundant amount of geopolymer phase on the micrograph of the sample Z'_7 with few heaps of aggregate but some cracks appeared. The micrograph of the sample Z'_9 was composed of both geopolymer phase and heaps of aggregate associated with few pores. By the contrast, the micrograph of the sample Z'_{15} was composed mainly of a very small domains of geopolymer phase together with particles of aggregate and pores, all scattered through the material. As it could be seen, there was less consolidation of the samples Z'_1 and Z'_{15} which explained their low 28 days compressive strength (Fig. 7). These results were in accordance with Table 2. In fact, geopolymers synthesized from the fused volcanic ash with great content of amorphous phase led to the products with abundant amount of geopolymer phase (the samples Z'_7

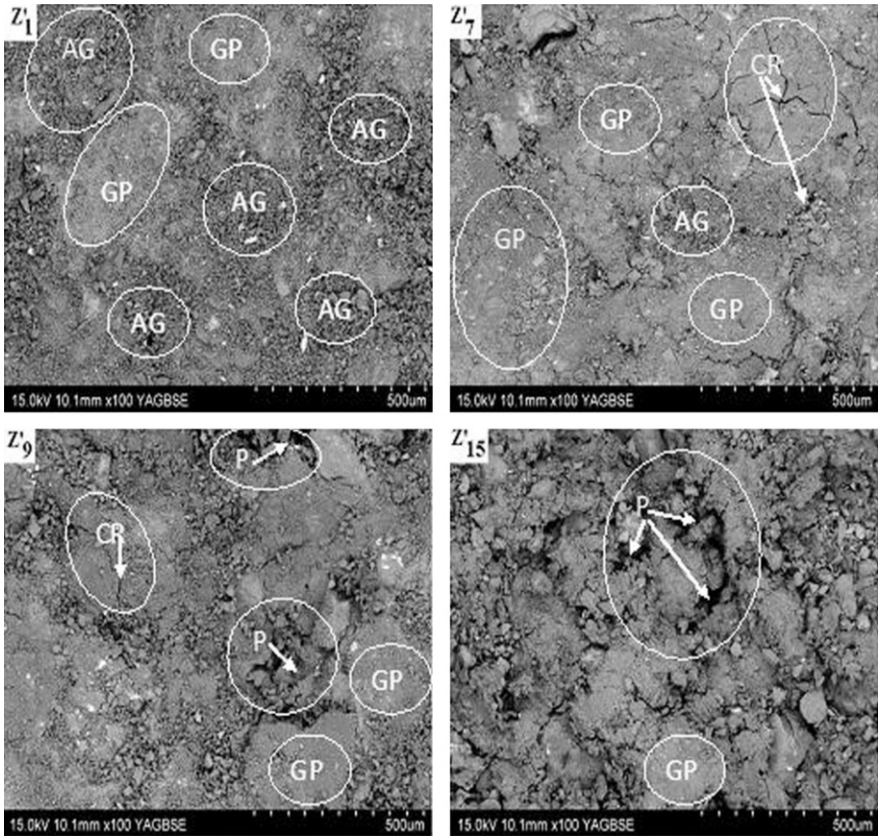

Fig. 8. SEM micrographs of geopolymer mortars (GP: geopolymers phase, P: pores, CR: cracks).

and Z'_9). Since the geopolymer mortar of Z'_7 was synthesized from the fused volcanic ash Z_7 with amorphous phase content greater and excess of fused soda lower than that of the sample Z_9, it led to the 28 days compressive strength greater than the sample Z'_9 (Fig. 7).

4. Conclusion

From the results of the investigation, it could be concluded that volcanic ash via the alkaline fusion method has the potential to be a raw material for geopolymer synthesis. Fused mixtures of soda–volcanic ash (fused volcanic ash) with Al_2O_3/Na_2O molar ratios between 0.29 and 0.06 generated great amount of amorphous phase which was an important parameter for geopolymer synthesis. Reactivity of geopolymer synthesis increased with increasing amount of amorphous phase content of fused volcanic ash, with Al_2O_3/Na_2O molar ratio ranging between 0.29 and 0.13. Fused volcanic ash with Al_2O_3/Na_2O molar ratio greater than 0.13 contained excess of fused soda which hindered geopolymer synthesis process. Geopolymer mortars synthesized from fused volcanic ash exhibited a low setting time (15–25 min). Using fused volcanic ash with Al_2O_3/Na_2O molar ratio between 0.29 and 0.13 led to geopolymer mortars with increasing compressive strength. Conversely, geopolymer mortars synthesized from fused volcanic with Al_2O_3/Na_2O molar ratio greater than 0.13 led to the decrease of compressive strength. In any way, using fused volcanic ash for making geopolymers led to lower setting time and greater compressive strength than using volcanic ash.

References

[1] J. Davidovits, Geopolymers—inorganic polymeric new materials, Journal of Thermal Analysis and Calorimetry 37 (1991) 1633–1656.
[2] K. Komnitas, D. Zaharaki, Geopolymerisation: a review and prospects for the minerals industry, Minerals Engineering 20 (2007) 1261–1277.
[3] J. Temuujin, R.P. Williams, A. van Riessen, Effect of mechanical activation of fly ash on the properties of geopolymer cured at ambient temperature, Journal of Materials Processing Technology 209 (2009) 5276–5280.
[4] H. Xu, J.S.J. van Deventer, Geopolymerization of multiple minerals, Minerals Engineering 15 (2002) 1131–1139.
[5] D. Bondar, C.J. Lynsdale, N.B. Milestone, N. Hassani, A.A. Ramezanianpour, Effect of adding mineral additives to alkali-activated natural pozzolan paste, Construction and Building Materials 25 (2011) 2906–2910.
[6] S. Martinez-Ramirez, M.T. Blanco-Valera, I. Erena, M. Gener, Pozzolanic reactivity of zeolitic rocks from two different cuban deposits: characterization of reaction products, Applied Clay Science 32 (2006) 40–52.
[7] M.T. Blanco Varela, S. Martinez-Ramirez, I. Erena, M. Gener, P. Carmona, Characterization and pozzolanicity of zeolitic rocks from two cuban deposits, Applied Clay Science 33 (2006) 149–159.
[8] H.K. Tchakoute, A. Elimbi, J.A. Mbey, S.C.J. Ngally, D. Njopwouo, The effect of adding alumina-oxide to metakaolin and volcanic ash on geopolymer products: a comparative study, Construction and Building Materials 35 (2012) 960–969.
[9] C. Leonelli, E. Kamseu, D.N. Boccaccini, U.C. Melo, A. Rizzuti, N. Billong, P. Miselli, Volcanic ash as alternative raw materials for traditional vitrified ceramic products, Advances in Applied Ceramics 103 (2007) 1–7.
[10] H.K. Tchakoute, A. Elimbi, E. Yanne, C.N. Djangang, Utilization of volcanic ash for the production of geopolymers cured at ambient temperature., Cement and Concrete Composites, Submitted for Publication.
[11] E.I. Diaz, E.N. Allouche, S. Eklund, Factors affecting the suitability of fly ash as source material for geopolymers, Fuel 89 (2010) 992–996.
[12] N. Shigemoto, H. Hayashi, K. Miyaura, Selective formation of Na–X zeolite from coal fly ash by fusion with sodium hydroxide prior to hydrothermal reaction, Journal of Materials Science 28 (1993) 4781–4786.
[13] W.F. Hillebrand, G.E.F. Lundell, H.A. Bright, J.I. Hoffman, in: Applied Inorganic Analysis, 2nd Ed., Wiley, New York, 1962 p. 836.
[14] H. Xu, L. Qin, L. Shen, M. Zhang, J. Zhai, Low-reactive circulating bed combustion (CFBC) fly ashes as source material for geopolymer synthesis, Waste Management 30 (2010) 57–62.
[15] H.L. Chang, W.H. Shih, A general method for the conversion of fly ash into zeolites as ion exchangers for cesium, Industrial and Engineering Chemistry Research 37 (1998) 71–78.
[16] F. Li, Q. Li, J. Zhai, G. Sheng, Effect of zeolitization of CFBC fly ash on immobilization of Cu^{2+}, Pb^{2+} and Cr^{3+}, Industrial and Engineering Chemistry Research 46 (2007) 7087–7095.
[17] P. De Silva, K. Sagoe-Crenstil, V. Sirivivatnanon, Kinetics of geopolymerization: role of Al_2O_3 and SiO_2, Cement and Concrete Research 37 (2007) 512–518.
[18] D. Njoya, Minéralogie, propriétés physiques et mécaniques des céramiques des argiles de Mayouom (Cameroun). Thèse de Doct 3eme cycle. Yaoundé, Université de Yaoundé I, 2005.
[19] A. Elimbi, H.K. Tchakoute, D. Njopwouo, Effects of calcination temperature of kaolinite clays on the properties of geopolymer cements, Construction and Building Materials 25 (2011) 2805–2812.
[20] P. Chindaprasirt, U. Rattanasak, Utilization of blended fluidized bed combustion (FBC) ash and pulverized coal combustion (PCC) fly ash in geopolymer, Waste Management 30 (2010) 667–672.
[21] D. Hardjito, C.C. Cheak, C.H. Lee, Ing Strength and setting times of low calcium fly ash-based geopolymer mortar, Modern Applied Science 2 (2008) 3–11.
[22] S. Jumrat, B. Chatveera, P. Rattanadecho, Dielectric properties and temperatures profiles of fly ash-based geopolymer mortar, International Communications in Heat and Mass Transfer 38 (2011) 242–248.
[23] C.J. Brinker, G.W. Scherrer, Sol Science. The Physics and Chemistry of Sol–gel Processing, Academic Press, San Diego, 1990.