



Rational utilization of volcanic ashes based on factors affecting their alkaline activation



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ABSTRACT

The present study evaluates the criteria that affect the characteristics of both volcanic ashes and their alkaline activated products, hence their rational utilization. To that end, five samples of volcanic ash were examined chemically, mineralogically and physically whereas certain characteristics of their alkaline activated products were determined. In order to elucidate the relationship between the characteristics of volcanic ashes and the products of their alkaline activation, mechanical strength, Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) of geopolymer specimens aged 14 days were examined. Volcanic ashes with large amount of amorphous phase (18.2–42.5% by mass) led to geopolymers with compressive strengths of 3.1–12.6 MPa. As for volcanic ashes with small amount of amorphous phase (10.2–12.5% by mass), alkaline activation at 80 °C led to geopolymers with compressive strength of 1.0–4.1 MPa. The results obtained showed that volcanic ashes could be used either for geopolymer synthesis or as filler. The classification depended mostly on the amount and the SiO₂/Al₂O₃ molar ratio of amorphous phase.

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1. Introduction

Volcanic ashes are natural waste materials consisting of particles of pulverized rocks that are formed during volcanic eruptions. These materials can be found in countries with past or present volcanism such as Colombia, the Democratic Republic of Congo, Indonesia, Italy, Russia, and the USA. In Cameroon, these materials are found along the “Cameroon Line”, oriented N30°E, especially at the foot of Mount Cameroon, Mount Manengouba, in the Tombel plain, around Foubot, the Kumba plain, the locality of Lake Nyos and the Region of Adamaoua [1]. Some of these materials are already being tested for alkaline activation [1–6] or used as filler in civil engineering [7]. They can be considered as natural pozzolan due to their high silica, alumina and iron oxide contents [8]. For geopolymer synthesis, parameters that define fundamental criteria during alkaline activation of aluminosilicates, such as particle size distribution, specific surface area, chemical and mineralogical compositions, amount and availability of silica and alumina in the amorphous phase must be examined [9–12]. Other parameters, such as curing temperature, age of geopolymers, type and composition of the alkaline solution are taken into account to optimize

the characteristics of synthesized products. Also, the properties of geopolymers can differ from one type of aluminosilicate to another due among other things to the amount and proportions of alumina and silica in the amorphous phase [9,13,14]. Chemical and mineralogical compositions of volcanic ashes are variable and are known to have low alkali reactivity [4]. This is related to the large amount of inert phases they contain and the presence of pyroclastic rocks among other things [15]. Some studies have already been done on alkaline activation of volcanic ashes [2–6]. However, understanding the criteria that govern their reactivity in alkaline medium in order to determine their utilization for either geopolymer synthesis or filler remains a challenge. Djobo et al. [16] increased the reactivity of volcanic ashes in an alkaline medium through mechanical activation, while Tchakoute et al. [17] performed their activation via the alkaline fusion method. Also, Djobo et al. [18] published a review article on the current state of the art and perspectives regarding volcanic ash-based geopolymers. To our knowledge, investigations on rational utilization of volcanic ashes for either geopolymer synthesis or filler via factors that affect their alkaline activation have not yet been undertaken. Based on alkaline activation of five samples of volcanic ash, the present study identifies certain criteria that affect their synthesized products in order to infer their utilization for either fabricating geopolymers or for use as filler in civil engineering. With that in mind, chemical and mineralogical compositions, fineness and specific surface area of particles, amount

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Table 1
Chemical analysis and specific surface area of volcanic ashes.

Oxide	Sample denomination				
	P1 (% mass)	P2 (% mass)	P3 (% mass)	P4 (% mass)	P5 (% mass)
SiO ₂	40.1	45.8	45.3	46.1	45.6
CaO	6.3	11.8	13.1	10.7	13.4
Al ₂ O ₃	16.3	13.8	12.4	14.0	12.6
Fe ₂ O ₃	13.5	13.5	12.9	13.1	13.3
K ₂ O	0.5	1.5	1.3	1.4	0.9
Na ₂ O	0.8	3.2	2.4	2.7	2.9
MgO	5.3	8.4	9.9	7.0	9.5
MnO ₂	0.2	0.2	0.2	0.2	0.2
TiO ₂	3.2	0.0	0.0	0.0	0.0
P ₂ O ₅	0.6	0.6	0.5	0.5	0.5
LOI	11.1	0.1	0.2	1.5	0.9
Total	97.9	98.9	98.2	97.2	99.8
Specific Surface area (m ² /g)	7319	2632	2647	3180	2614

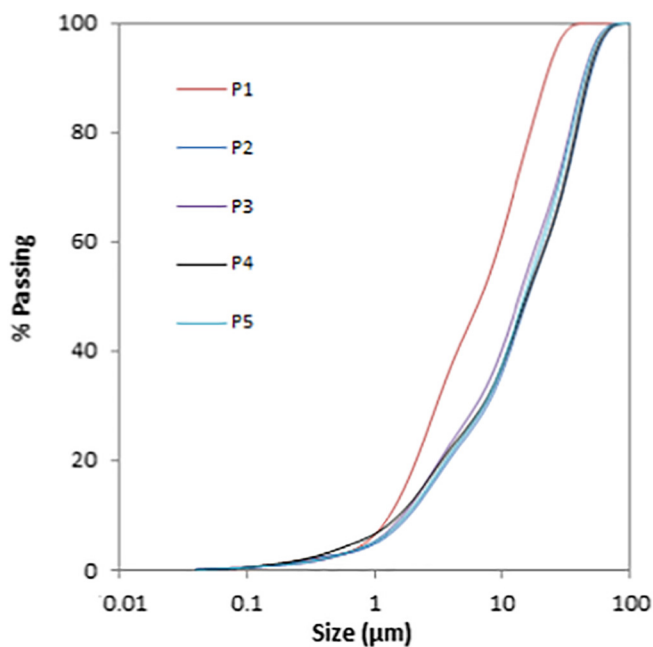


Fig. 1. Particle size distributions of volcanic ashes.

and SiO₂/Al₂O₃ molar ratio of amorphous phase of five samples of volcanic ash, compositions of alkaline solutions along with curing temperature during alkaline activation were the key investigated aspects in this study.

Table 2
Experimental program carried out on volcanic ash based geopolymers.

Mixture	Type	Sample	Fineness	Alkaline activator	Curing temperature (°C)	Curing time (days)
Mixture 1	Mortar	P1	F1	Na ₂ SiO ₃	25	1
Mixture 2	Mortar	P1	F1	NaOH	25	1
Mixture 3	Mortar	P1	F2	Na ₂ SiO ₃	25	1
Mixture 4	Mortar	P1	F2	NaOH	25	1
Mixtures 5	Pastes	P1 and P3	F1	Na ₂ SiO ₃	30	3
Mixture 6	Mortar	P2	F1	Na ₂ SiO ₃	80	4
Mixture 7	Mortar	P3	F1	Na ₂ SiO ₃	80	4
Mixture 8	Mortar	P4	F1	Na ₂ SiO ₃	80	4
Mixture 9	Mortar	P5	F1	Na ₂ SiO ₃	80	4
Mixture 10	Mortar	P2	F1	NaOH	80	1
Mixture 11	Mortar	P3	F1	NaOH	80	1
Mixture 12	Mortar	P4	F1	NaOH	80	1
Mixture 13	Mortar	P5	F1	NaOH	80	1

2. Materials and experimental methods

2.1. Materials

Five samples of volcanic ash denoted P1, P2, P3, P4 and P5 and whose chemical compositions and specific surface areas are given in Table 1, were collected from different deposits along the “Cameroon Line”. The collected materials were oven dried at 105 °C (Heraeus oven, type VT 5042 EK) and ground thanks to a planetary ball mill. The particles obtained were sieved at 71 µm (fineness F1: P1, P2, P3, P4 and P5) and at 41 µm (fineness F2: P1). Two types of alkaline solutions were used as activators. The first one was a commercial solution of sodium silicate whose composition (% mass) was as follows: 27.5 (SiO₂); 16.5 (Na₂O); 56 (H₂O). The second one was a solution of sodium hydroxide (12 M). The utilization of one or the other alkaline solution depended on the ease with which geopolymer reaction took place with a given sample of volcanic ash.

2.2. Experimental methods

The particle size distribution of volcanic ashes (Fig. 1) was obtained thanks to a Tristar 3020 particle size analyzer. The chemical compositions of the samples were determined by Inductively Coupled Plasma and Optical Emission Spectroscopy (ICP-OES) using a Perkin Elmer Spectrometer (Optima™ 7000 DV ICP-OES) equipped with a CCD sensor. The specific surface areas of powders were calculated from the Blaine model. XRD patterns obtained by powder method, were recorded from a diffractometer (D8 Advance, Bruker), using copper radiation (Cu Kα, λ = 1.5406 Å). A modified method based on French standard XP P18-594 [19,20] was used to determine the composition of amorphous phase of volcanic ash samples. It consisted of pouring a determined mass of volcanic ash powder into sodium hydroxide solution (8 M) contained in a flask, then heating the whole content at 80 °C in order to dissolve the amorphous phase [21]. This was followed by filtering and washing of the residue with acid chlorhydric (1 M) in order to dissolve the silica that had precipitated [22]. This allowed the determination of dissolved silica and alumina of the amorphous phase thanks to ICP-OES technique. Also, the insoluble residue was used to determine the amount of reactive phase. The pozzolanic activity of volcanic ash was determined thanks to DTG analysis of dried slips obtained from mixtures of Ca(OH)₂ and volcanic ash (mass ratio of 1:4) and which were denoted CH-P1, CH-P2, CH-P3, CH-P4 and CH-P5. When the latter mixtures were aged 7 and 28 days respectively, they were heated under argon atmosphere (NETZSCH STA 449T device) from 50 to 1000 °C at a rate of 10 °C/min. The pozzolanic activity of volcanic ash was determined according to Eq. (1) [23].

$$\% \text{Fixed Ca(OH)}_2 = \frac{\text{Ca(OH)}_{2i} - \text{Ca(OH)}_{2t}}{\text{Ca(OH)}_{2i}} * 100 \quad (1)$$

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