Journal of Geoscience and Environment Protection, 2019, 7, 195-209
https://www.scirp.org/journal/gep
ISSN Online: 2327-4344
ISSN Print: 2327-4336

Characterization of Pyroclastic Deposit from Three Different Areas within Foumbot Region (West-Cameroon): Comparative Studies of Their Effects as Pozzolanic Materials in Mortars and Cement Manufacture

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How to cite this paper: Mounjouohou, M. A., Moundi, A., Ntieche, B., Dawai, D., & Michel, F. (2019). Characterization of Pyroclastic Deposit from Three Different Areas within Foumbot Region (West-Cameroon): Comparative Studies of Their Effects as Pozzolanic Materials in Mortars and Cement Manufacture. *Journal of Geoscience and Environment Protection, 7,* 195-209.
https://doi.org/10.4236/gep.2019.711014

Received: September 30, 2019
Accepted: November 25, 2019
Published: November 28, 2019

Abstract

Pyroclastics which are known natural pozzolanic materials due to amorphous contents, are present in several areas of the Mbepit Massif in West Cameroon. In this work natural pozzolan from three zones namely Pouoloum, Njimbouot and Nkouonja were characterized. A comparative study was then developed to attest the effect of these pyroclastics as partial substitution in portland cement. The mixtures were made at different pozzolanic proportions (00%, 10%, 15%, 25% and 35%) of substitution of the cement. The compressive and flexibility strength was carried out at 7, 28 and 90 days on mortar specimens (4 × 4 × 16 cm^3). The results revealed apozzolanicactivity index of 81.99 %, 83.47% and 74.54% respectively for rock sample from Pouoloum (PCB), sample from Njimbouot (PCC) and sample from Nkouonja (PCN). After 90 days, for a substitution rate of 25% compressive strength are respectively 55.69 MPa, 60.4 MPa, 53.34 MPa for PCB, PCC and PCN. According to the American Society for Testing and Materials (ASTM) C618 classification, the pyroclastics are in accordance with all the criteria to be classified as pozzolan. Independent of the substitution rate, the mechanical performance increases with age in PCB, PCC and PCN. However PCC is most reactive than PCB and PCN. This may be due to the amorphous reactive content in this material and can be linked to the eruptive dynamisms which were more explosive in some areas than in others. The amorphous content is 32.01%, 36.99%, 24.84% for PCB, PCC and PCN respectively. These results also prove that Natural Pozzolan is interesting in the manufacture of composite cement CEM II, CEM IV in accordance with EN197-1 or can be added in mortar for buildings and sustainable environmental management.

Keywords

Pyroclastics, Pozzolanic Activity, Compressive Strength, Composite Cement, Natural Pozzolan, Mbepit Massif

1. Introduction

Pozzolans are a broad class of siliceous or siliceous and aluminous materials which, in themselves, possess little or no cementitious value but which will, in finely divided form and in the presence of water, react chemically with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties (Mehta, 1987). The quantification of the capacity of a pozzolan to react with calcium hydroxide and water is given by measuring its pozzolanic activity (Snellings et al., 2012). Pyroclastic rocks are blocks of solidified lava ejected during the eruption of a volcano and are classified as ashes, lapilli, bombs and blocks depending on their diameter (Heiken & Wohletz, 1985). The samples were collected in the three different areas because pozzolan coming from different areas may not necessarily react in the same manner. Also, there are some variations in grain size in the different pozzolans in these three areas.

The Mbepit massif in the Noun Division (**Figure 1**) contains huge volcanic deposits of ash (diameter < 2 mm) and lapilli (2 mm > diameter > 64 mm), exploitable as pozzolanic materials, for mixtures in mortars and manufactures of cements. The pozzolan of Foumbot area comes from the volcanic activities. Those activities have resulted in materials with different textures and structures according to the specific condition of the emission of lava and eruptive styles. Studies reveal that pozzolanic activity of materials from different areas may not

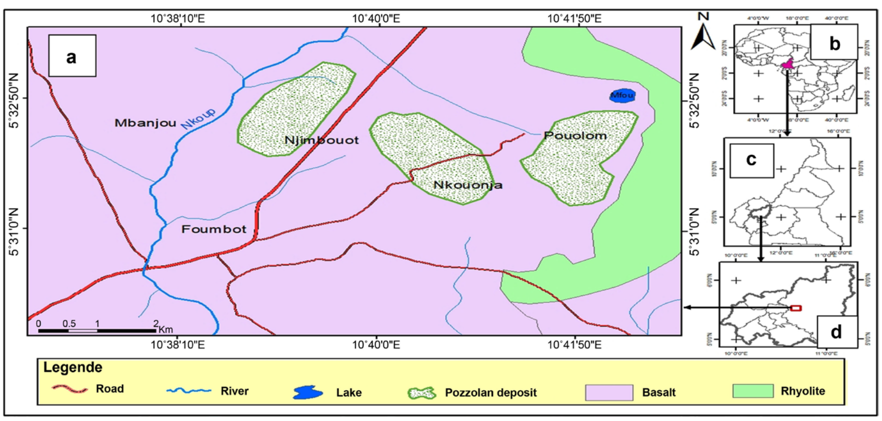


Figure 1. Geological map (a) showing the location of Cameroon (b); the location of Noun Division (c) and location of the study area (d).

be the same and some parameters influencing the pozzolanic reactions include the nature of the active phases and their proportions in SiO_2 content. The pozzolan of Foumbot area in the Noun Division comes from several episodic volcanic activities and dynamisms (effusive and explosive). These phenomenons prevent the total crystallization of minerals and promote the formation of amorphous and glasses, responsible for pozzolanic activity when they are combined with calcium hydroxide ($Ca(OH)_2$).

The natural pozzolans used as binder helps to protect the environment by reducing the consumption of portland cements. This also aids in reducing environmental pollution due to the manufacture of portland cement by partial substitution of clinker in portland cement with pozzolanic materials such as natural pozzolan (Mokhtaria et al., 2009), metakaolin, industrial waste such as silica fume, fly ash (Ayrinhac, 2005; Hosseini et al., 2011), blast furnace slag (Elke, 2012) and coal bottom ash (Cheriaf et al., 1999; Haldun & Mine, 2007). The pozzolanic materials will contribute to the improvement of the mechanical properties of concretes due to the development of pozzolanic activity and the formation of second-generation calcium silicate hydrates CSH II (Cheriaf et al., 1999; Haldun & Mine, 2007). The manufacture of portland cement produces very large quantities of CO_2 during the decarbonation of $CaCO_3$. The cement industry emits 5% to 7% of the global CO_2 rate emitted into the atmosphere, with 0.9 tons of CO_2 emitted during the manufacture of one ton of cement (Emad et al., 2013). The responsiveness of a pozzolanic material depends on several parameters and its reactivity varies from one pozzolan to another. This work will help 1) appreciate the mechanical resistance of mortars containing natural pozzolan from the Foumbot area and 2) to compare the pozzolanicity of pyroclastic material from three different sites (**Figure 1**) and 3) to appreciate their use in cement manufacture.

2. Materials and Methods

In this area, the basement of recent volcanic lavas is made up of gneiss and granite. The volcanic rocks are made up of basalts, rhyolites and colossal quantities of pyroclastic materials deposited in different areas (Tchokona, 2010).

2.1. Materials

Natural pozzolans were collected in three different areas, namely Pouoloum, Njimbouot and Nkouonja and the samples pseudo named as PCB, PCC and PCN respectively (**Figure 2**). The pozzolanic effect depends on the fineness of the particles (Isaia et al., 2003) suggesting grinding as the most appropriate treatment method in the study of natural pozzolan. The duration of grinding and different sieving aided approximatively the same granulometry in the three pozzolans after grinding. Moreover, the parameters influencing the pozzolanic reactions are the nature of the active phases and their proportions, the SiO_2 content, the lime/pozzolan ratio in the mixture, the duration of the cure, the fineness of the

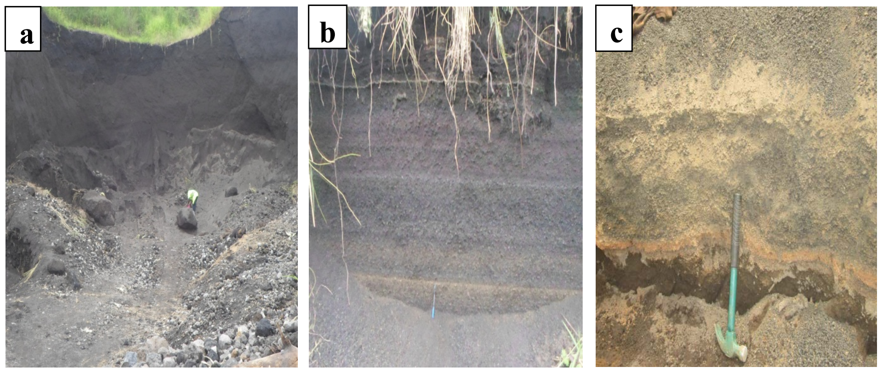


Figure 2. Natural pozzolan deposit in (a) Pouoloum site, (b) Njimbouot site and (c) Nkouonja site sampled for analysis.

pozzolan, the ratio W/C of the mixture, the temperature, and the optimization of pozzolanicity (Shi, 2001). The mechanical performances are obtained due to the development of the pozzolanic activity. The sieving granulometry and the laser granulometry permitted to know the particle size distribution of the pozzolan particles both in the natural state and after grinding. The density measurement of pozzolan was made using the Pycnometer MultiVolume 1305 with gas, Brunauer-Emmett-Teller (BET) and the specific surface area using Micromeritics Instrument Corp. X-ray fluorescence reveals the composition of the major element composition of the pozzolan. Mineralogical phases content was done using X rays diffraction (XRD) method. The Keyser test permitted the estimation of the vitreous phase by the chemical method. This method consists in observing the dissolution kinetics of the amorphous sillico-aluminates in a 1% hydrofluoric acid solution in order to estimate the amorphous rate.

According to the American Society for Testing and Materials (ASTM) C618 (Standard ASTM standard, C618, 2008), a material is pozzolanic if:

- its chemical composition verified that $SiO_2 + Al_2O_3 + Fe_2O_3 > 70\%$;
- its glass content is such that $(SiO_2 - CaO) > 34$;
- its activity index for a substitution rate of 25%, I_{25} at 28 days of cure verified that $67\% < I_{25} < 100\%$.

$$Ip = (Rp/R_0) \times 100$$

R_0 = compressive strength of the control mortar with 100% cement;

p = 25% of pozzolanic material;

I = pozzolanic activity index;

R_p = compressive strength of mortar with p% of pozzolan replacement by weight in cement.

2.2. Methods

2.2.1. Mixtures and Tests

Mixtures were made using water, sand, portland cement 52.5 with different mass substitution rate of cement by natural pozzolans. The mortars were molded into bars ($4 \times 4 \times 16\ cm^3$). They were demolded after 24 hours and then kept under water in favourable laboratory conditions. The compressive and flexural strength tests were carried out at 7, 28, and 90 days according to the European Standard methods of testing cement determination of strength (EN 196-1) (Standard EN 196-1, 2006).

2.2.2. Estimation of the Vitreous Phase by the Keyser Method

The standard Keyser method was done using 1% hydrofluoric acid solution for the dissolution of the amorphous silico-aluminates. 1g of pozzolanic material was introduced into 200 ml of 1% hydrofluoric acid solution and the solution was kept for 40 minutes and then filtered on an ashless filter paper. The residue was oven-dried at 105˚C for 24 hours. The amorphous phase of the material was given by the formula below:

$$\%\ \text{amorphous} = (m_1 - m_2)/m_1$$

m_1 = mass of material before dissolution;

m_2 = mass of the material after dissolution.

3. Results and Discussions

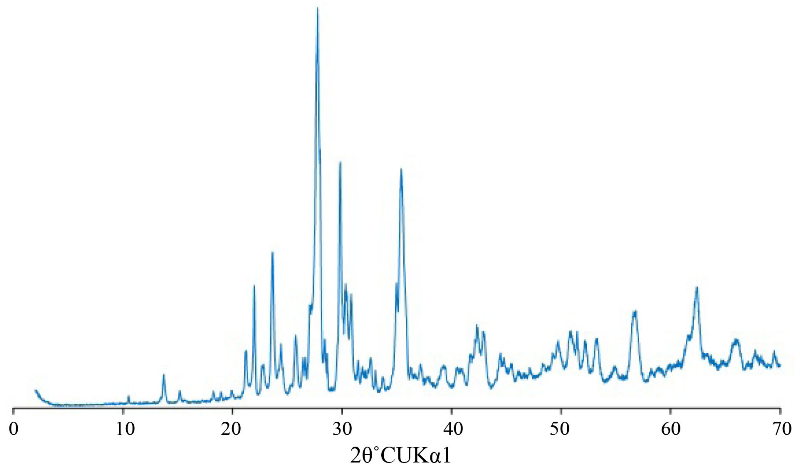
According to **Table 1**, the pozzolans of Foumbot area are basic in nature with SiO_2 concentrations less than 50.88%. These natural pozzolan contains iron, oxide, and aluminum oxide with a calcium oxide content less than 8.37%. The loss on ignition (LOI) of the different samples is below 3 testifying the freshness of the pozzolanic materials. The K_2O/Na_2O is below 1 showing that the pozolanic materials are mostly sodic. PCC sample has less content of CaO. The XRD results (**Figures 3(a)-(c)**) show that all those pozzolans contain an amorphous phase. Pozzolan of our study area contains plagioclases, feldspars and pyroxenes and very low quartz content. This may testify the basic nature of the pyroclastics. The cement used is rich in CaO and SiO_2 (**Table 2**).

Table 1. XRF analysis of PCB, PCC, PCN.

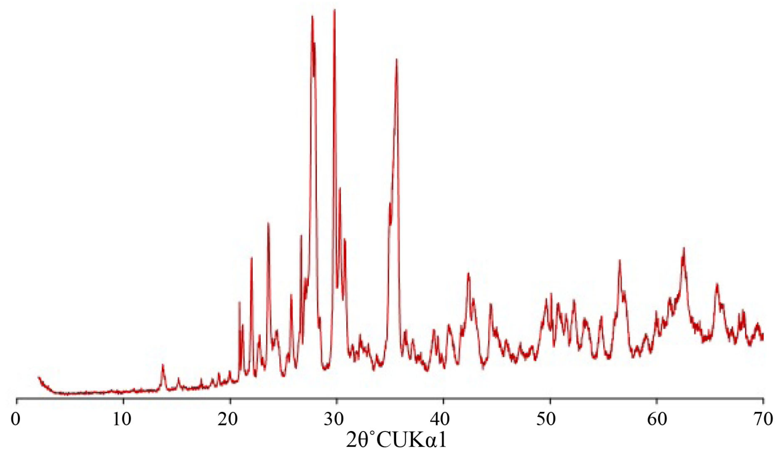
Chemical composition	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	LOI	Sum
Pozzolan (PCB)	50.88	2.51	15.25	11.52	0.2	4.63	7.97	3.35	2.96	0.57	0.4	100.24
Pozzolan (PCC)	46.17	2.57	15.12	12.03	0.21	4.14	7.51	3.35	2.08	0.63	2.79	96.59
Pozzolan (PCN)	49.78	2.74	15.42	12.56	0.2	4.87	8.37	3.44	2.26	0.6	0.4	100.64

Table 2. Chemical composition of cement.

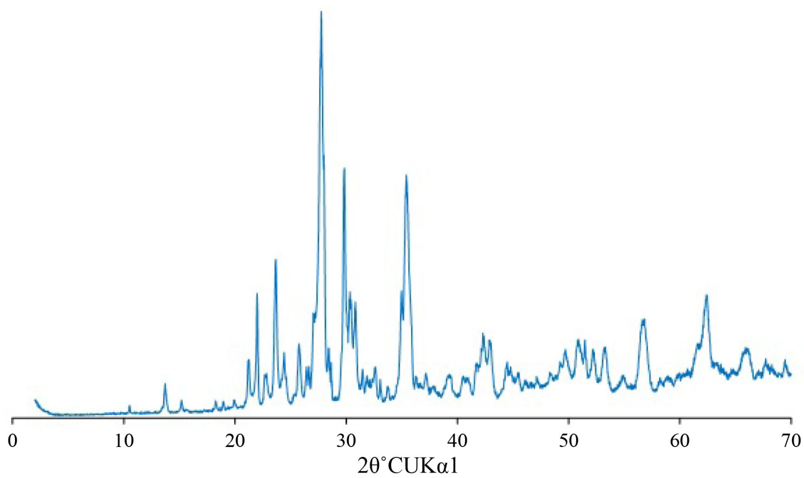
Chemical and mineralogy	CaO	SiO_2	Al_2O_3	Fe_2O_3	C_2A	SO_2	Insoluble residue	LOI	Chlorures	Chrome (VI)$^+$	Na_2Oeq^{++}	Sulfures
average composition	63.4	20.4	4.8	3.4	7.2	3.2	0.6	1.6	0.08	0.00012	0.74	-
Density	3100 Kg/m^3											



(a)



(b)



(c)

Figure 3. (a) X-ray fluorescence (XRD) of sample PCB; (b) XRD of sample PCN; (c) XRD of sample PCC.

3.1. Granulometric Results

The granulometric results show that the natural pyroclastics are mostly made up of volcanic ashes i.e. less than 2 mm (Figure 4) and after grinding the average sizes were dominated by 10 µm (Figure 5).

3.2. Specific Surface Area

The specific surface area of the pozolans is about 2.5 m^2/g for the three samples after grinding.

3.3. Keyser Results

The obtained results show that the amorphous phase is higher in PCC with 36.99% than PCB and PCN (Table 3(a)). These amorphous phases are responsible for the pozzolanic activities.

3.4. Mixtures

The table summarizes the various proportion of pozzolan that were successively utilized in the mixtures (Tables 3(b)-(d)).

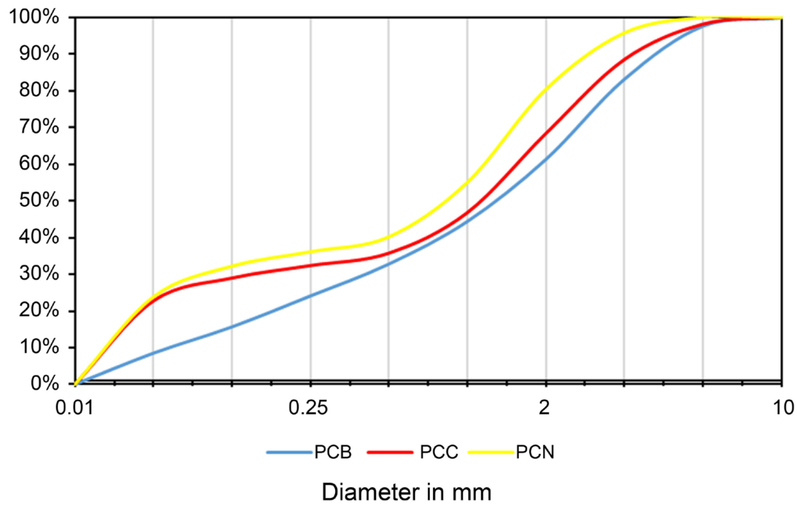


Figure 4. Particle size distribution of natural pozzolan.

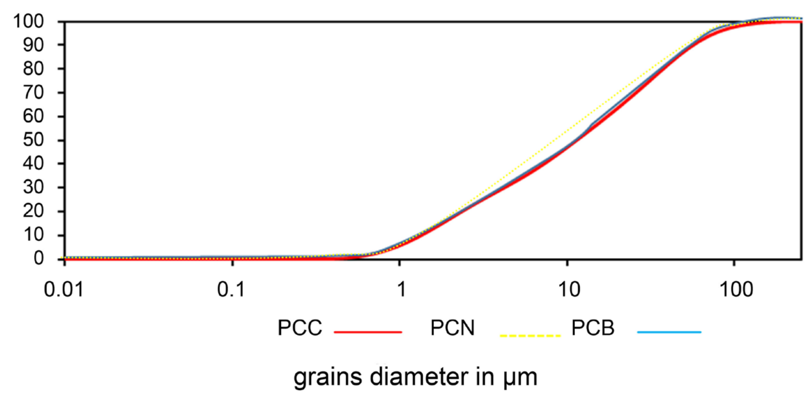


Figure 5. Particle size distribution of grinding pozzolan.

Table 3. (a) Results from Keyser test; (b) Mixture proportion for PCB; (c) Mixture Proportion for PCC; (d) Mixture proportion for PCN.

(a)

	PCB	PCC	PCN
Values	$m_1 = 1.0176$ $m_2 = 0.6919$	$m_1 = 1.0033$ $m_2 = 0.6322$	$m_1 = 1.0195$ $m_2 = 0.7663$
Amorphous phases	32.01%	36.99%	24.84%

(b)

Mixture proportions	Pozzolan rate	Quantity (g)	Substitution cement	Quantity (g)	w/c	Sand (g)	water (ml)
PCB-00	0%	0	100%	450	0.5	1350	225
PCB-10	10%	45	90%	405	0.5	1350	225
PCB-15	15%	67.5	85%	382.5	0.5	1350	225
PCB-25	25%	90	80%	360	0.5	1350	225
PCB-35	35%	112.5	75%	337.5	0.5	1350	225

(c)

Mixture proportions	Pozzolan rate	Quantity (g)	Substitution cement	Quantity (g)	w/c	Sand (g)	water (ml)
PCC-00	0%	0	100%	450	0.5	1350	225
PCC-10	10%	45	90%	405	0.5	1350	225
PCC-15	15%	67.5	85%	382.5	0.5	1350	225
PCC-25	25%	90	80%	360	0.5	1350	225
PCC-35	35%	112.5	75%	337.5	0.5	1350	225

(d)

Mixture proportions	Pozzolan rate	Quantity (g)	Substitution cement	Quantity (g)	w/c	Sand (g)	water (ml)
PCN-00	0%	0	100%	450	0.5	1350	225
PCN-10	10%	45	90%	405	0.5	1350	225
PCN-15	15%	67.5	85%	382.5	0.5	1350	225
PCN-25	25%	90	80%	360	0.5	1350	225
PCN-35	35%	112.5	75%	337.5	0.5	1350	225

Table 4. Compressive strengh results.

Substitution rate of pozzolan (%)	Compressive strengh (Mpa)								
	7 days			28 days			90 days		
	PCB	PCC	PCN	PCB	PCC	PCN	PCB	PCC	PCN
0	52.02	52.02	52.02	65.05	65.05	65.05	67.02	67.02	67.02
10	49.8	50.1	47.8	59.03	63.2	57.06	62.94	66.03	59.8
15	48.09	49.5	44.9	56.94	58.5	52.43	58.96	62.83	56.06
25	39.05	43.6	37.05	53.34	54.3	48.49	55.69	60.4	53.34
35	36.6	40.5	35.08	44.59	50.96	42.6	50.15	52.3	46.31

Table 5. Flexural strength development results.

Substitution rate of pozzolan	Flexural strengh (Mpa)								
	7 days			28 days			90 days		
	PCB	PCC	PCN	PCB	PCC	PCN	PCB	PCC	PCN
0	8.8	8.8	8.8	10.08	10.08	10.08	10.98	10.98	10.98
10	7.58	8.6	7.1	8.98	9.56	8.55	9.25	10.01	8.9
15	7.01	8.1	6.7	8.02	9.22	7.91	9.02	9.99	8.2
25	6.5	7.41	6.3	7.38	8.64	7.08	8.69	9.56	7.89
35	6.01	6.9	6.1	7.09	8.32	6.96	8.12	9.09	7.11

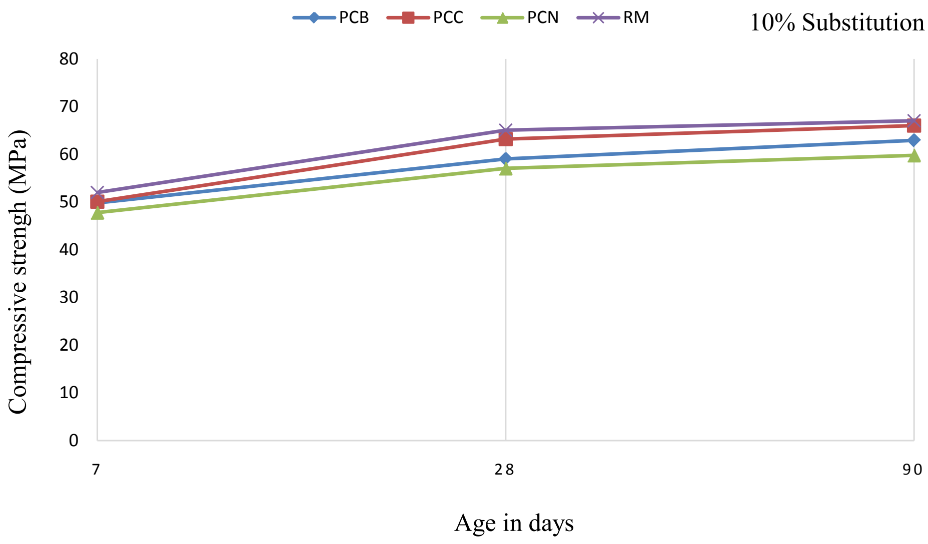
The results in Figures 6(a)-(d) and Figures 7(a)-(d) show that the compressive and flexural strength decrease with the increase of the substitution rate of pozzolan. The compressive and the flexural strength are low at 7 and 28 days in mortar containing pozzolan. But at old age (90 days) the mortar having 10% of pozzolan has greater compressive values 62.94 Mpa, 66.03 Mpa, 59.8 Mpa respectively for PCB, PCC and PCN, closer to the reference mortar which has a value of 67.02 MPa at the same age. This is because at a young age (before 28 days) many pozzolan ash particles remained unattacked by calcium hydroxides as it is the case in coal bottom ash particle and in rice husk ash (Hwang et al., 2011). Mortars made from pozzolan, despite their poor performance at a young age tend to catch up with time the reference mortar at 90 days (Table 4 and Table 5).

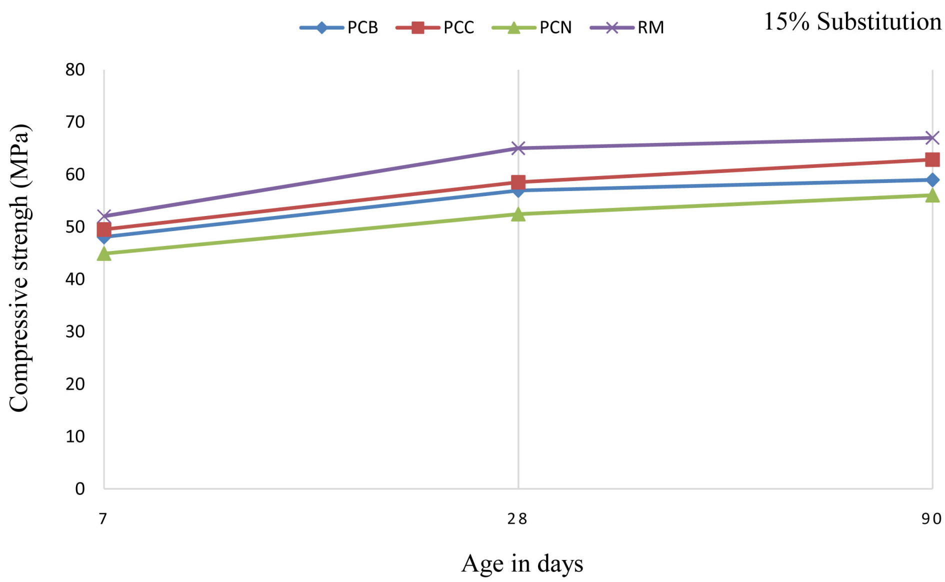
3.5. Pozzolanic Index Activity

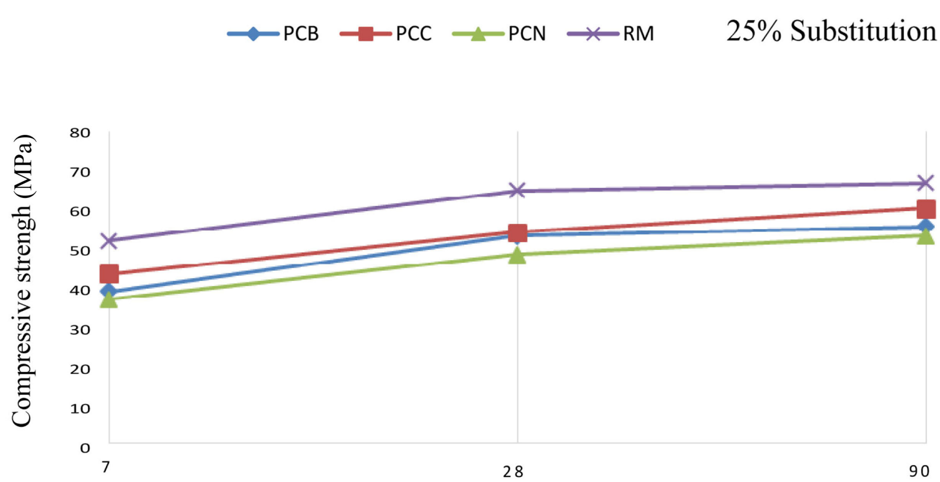
The results reveal that for the three study pyroclastics, the pozzolanic activity index is greater or equal to 74.54% (Table 6).

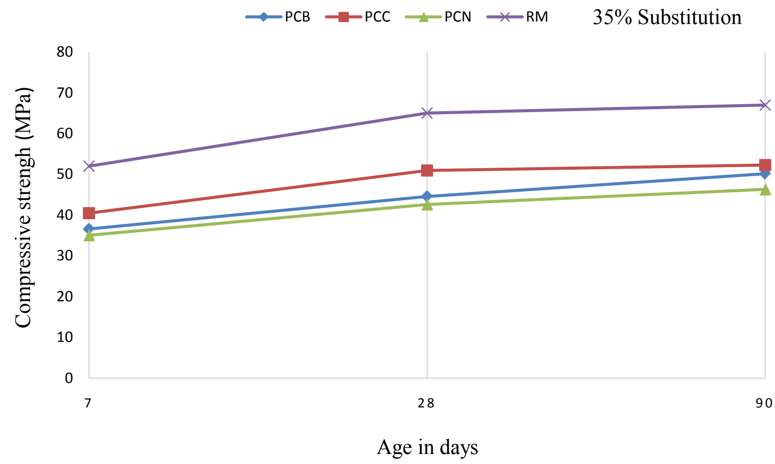
According to Figure 8, the activity index values decrease with the increase of pozzolan content. The 03 pozzolans PCB, PCC and PCN activity indexes are 82%, 83.47% and 74.54% respectively. This shows that PCC is the most reactive pozzolan followed by PCB and PCN.

According to ASTM Standard C 618 standard the pyroclastic materials (volcanic ashes and lapilli) of the study area are pozzolans. The results show that at a young age (28 days), the compressive strength decreases with the increase of the pozzolan substitution rate. This would be due to the low power consumption of lime $Ca(OH)_2$ by pozzolan at this age. At 10% of substitution rate, PCC sample compressive strength is 66.03 MPa closer to the reference moctar value of 67.02 MPa at 90 days compared to PCB (62.94 MPa) and PCN (59.8 MPa) (Table 7). Up to 35% of pozzolan substitution rate, the value of PCC (52.3 MPa) is greater than those of PCB (50.15) and PCN (46.31) at 90 days. No matter the substitution rates, the compressive strength of PCC, PCB, PCN increase with age. This is due to the pozzolanic activity of those pyroclastic materials which allow them to consume lime $Ca(OH)_2$ and CH and densify voids and pores in the mortar. This justifies the increase of compressive strength with age of the mortars. The hydration products fill the capillary pores and increase the resistance by refining these capillary pores and by transforming the large crystals of CH into CSH.


PCB
PCC
PCN
RM
10% Substitution
Compressive strengh (MPa)
Age in days
(a)

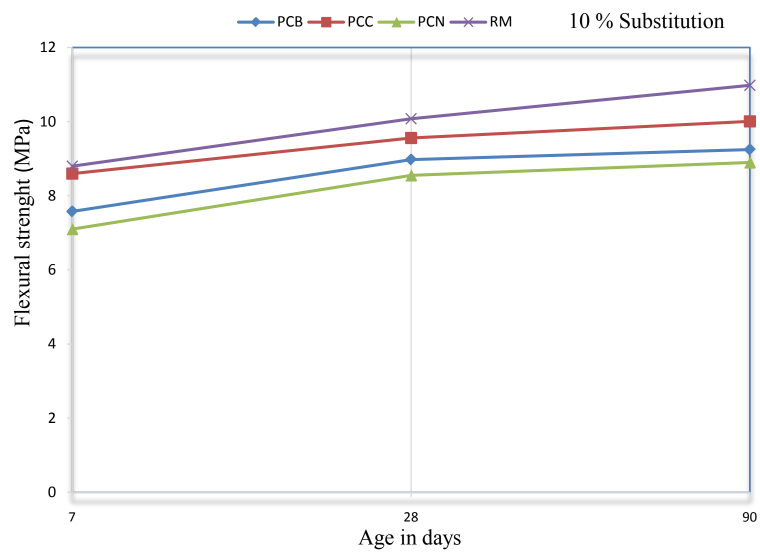

PCB
PCC
PCN
RM
15% Substitution
Compressive strength (MPa)
Age in days
(b)


PCB
PCC
PCN
RM
25% Substitution
Compressive strength (MPa)
(c)

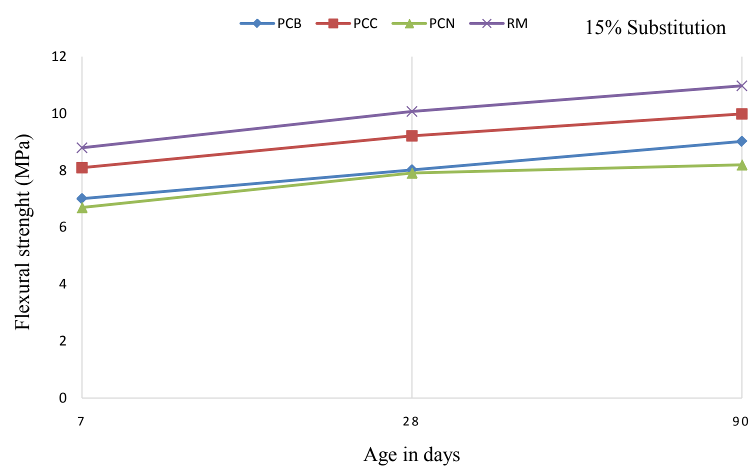


(d)

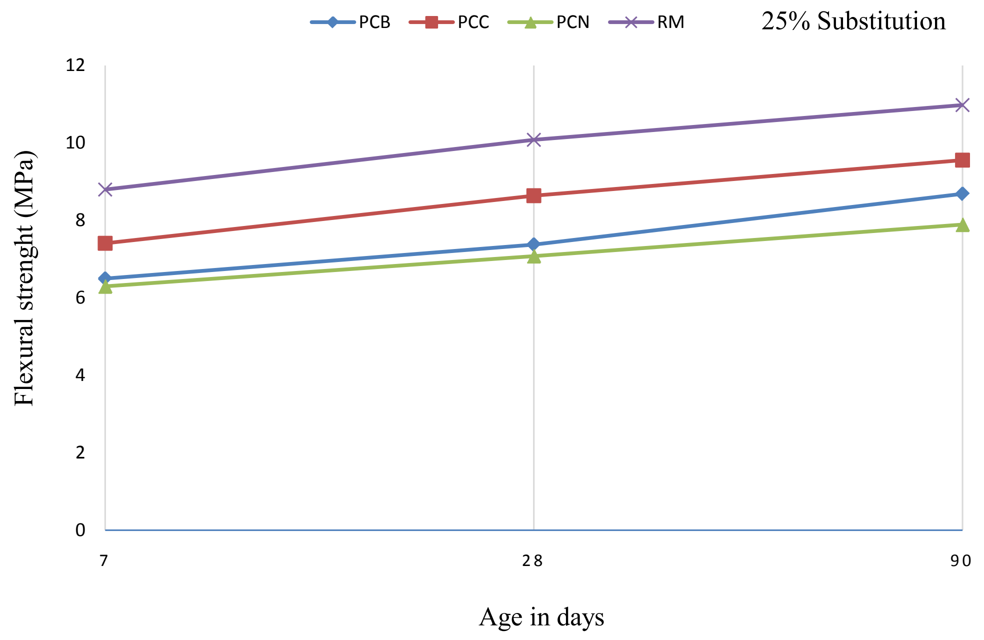
Figure 6. (a) Compressive strength development at 10% substitution rate; (b) compressive strength development at 15% substitution rate; (c) compressive strength development at 25% substitution rate; (d) compressive strength development at 35% substitution rate.



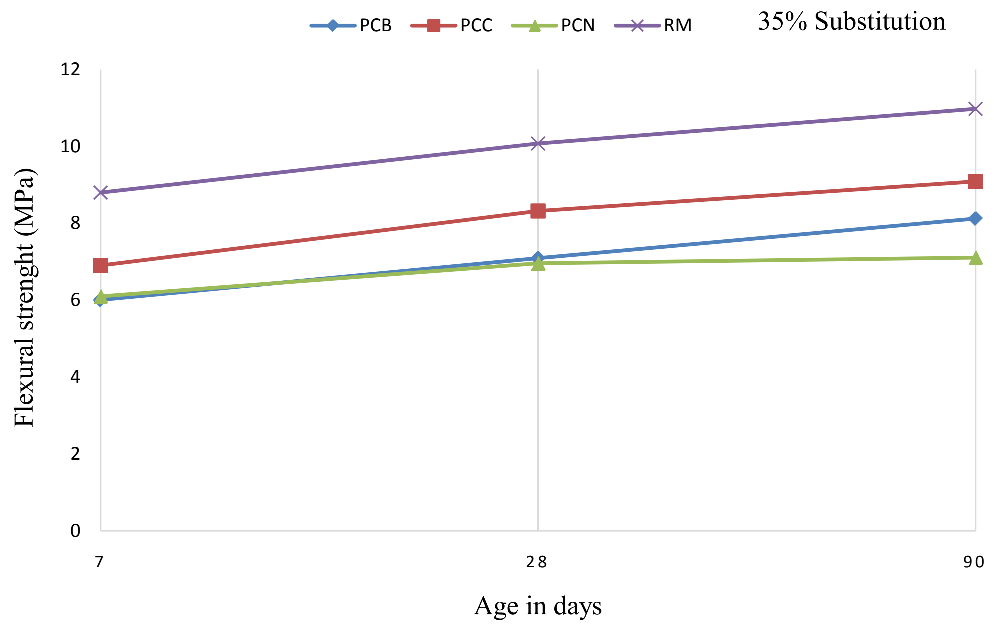
(a)



(b)



(c)



(d)

Figure 7. (a) Effect of pozzolan on flexural strength at 10% substitution rate; (b) effect of pozzolan on flexural strength at 15% substitution rate; (c) effect of pozzolan on flexural strength at 25% substitution rate; (d) effect of pozzolan on flexural strength at 35% substitution rate.

Table 6. Activity index development.

Mixture N°	Substitution rate	Compressive strength at 28 days	Reactivity index
PCB	25%	53.34	81.99%
PCC	25%	54.3	83.47%
PCN	25%	48.49	74.54%

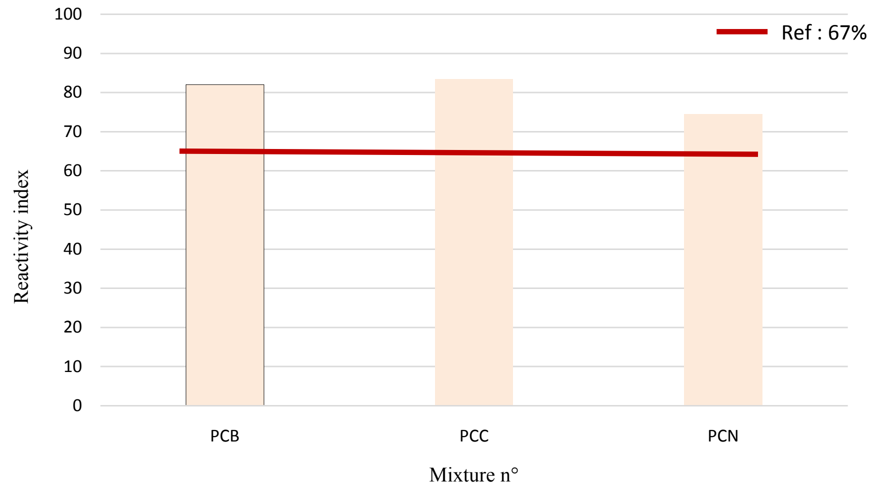


Figure 8. Activity index of pozzolan.

Table 7. ASTM parameters of pozzolan samples.

Parameter	PCB	PCC	PCN
$Al_2O_3 + SiO_2 + Fe_2O_3$	77.65	73.32	77.76
$SiO_2 - CaO$	42.91	38.66	41.41
IR	81.99%	83.47%	74.54%

$$SiO_2 + CH + H_20 \rightarrow Cx'SHy' + (2 - x)\ CH$$

The results also show that the pozzolans of the area are interesting to manufacture composite cement CEM II and CEM IV A in accordance with EN197-1 (Standard NF EN 197-1, 2001) or to add it in mortar for buildings and sustainable environmental management. Using a CEM I 52.5 with 10% substitution by pozzolans, the compressive strength values are 59.03 MPa, 63.2 MPa, 57.06 MPa for PCB, PCC and PCN respectively at 28 days. This result shows that it is possible to manufacture composite cement CEMII using PCB, PCC and PCN pyroclastites as natural pozzolan. At 15% substitution, PCB (56.94 MPa) and PCC (58.5 MPa) fulfill the criteria to manufacture a composite cement CEM II but not PCN (52.43 MPa). At 25% substitution rate, it is possible to use PCB and PCC to manufacture pozzolanic cement CEM IV A with regard to the values of 53.34 MPa and 54.3 MPa repectectively obtained for PCB and PCC at 28 days. At this age, PCN has a value of 48.49 which is under 52.5 MPa. However, it may be possible to use PCN in order to manufacture a cement CEM IV 42.5 or 32.5 from a CEM I 52.5.

4. Conclusion

The results of the study reveal that the three pyroclastic materials are pozzolans according to the American ASTM C 618 classification, with the sum of major elements $Al_2O_3 + SiO_2 + Fe_2O_3 > 70\%$, the activity index at 28 days $I_{25} > 67\%$ and the difference of major elements ($SiO_2 - CaO$) > 34. At 90 days, mortars having

10% of pozzolan have better compressive and flexural strengths closer to the reference mortar. The results reveal that it is possible to manufacture composite cements CEMII and CEMIV using the Foumbot pyroclastics as natural pozzolan and that mortar mixtures with pozzolan will portray good mechanical properties at 90 days. Pozzolan PCC from Njimbouot is more reactive than the others and is expected to be best suited for cement manufacture. Composite cements manufactured from these rocks are environmentally sustainable as they will emit low CO_2 and is highly economically viable as the process requires less energy compare to Portland cement. The low production cost and the environment-friendly nature of the composite cement provide an affordable option for the construction industry.

Acknowledgements

We thank the "Académie de Recherche et d'Enseignement Supérieur (ARES)" grants program that financed my travel, tests and analyses at the construction and building materials laboratory of the University of Liège, Belgium.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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