

# Characterisation of cementitious composite materials based on calcined clay activated by silicate from rice husk ash and Ingessil silicate - activator: sodium hydroxide (NaOH)

Ludovic Ivan Ntom Nkotto<sup>1,2</sup>, Gaëlle Dounbissi Kamgang<sup>2</sup> Lionel Pierre Nkengue<sup>3</sup>, Judicaël Sandjong Kanda<sup>4</sup>, Jordan Valdès Sontia Metekong<sup>2</sup>

<sup>1,2</sup>Laboratoire en Engineering Civil et Mécanique, Ecole Nationale Supérieure Polytechnique de Yaoundé, BP 8390, Yaoundé, Université de Yaoundé I, CAMEROUN

<sup>2</sup>Laboratoire d'Analyses des Matériaux, Mission de Promotion des Matériaux Locaux (MIPROMALO), BP 2393, Yaoundé, CAMEROUN

<sup>3</sup>Laboratoire de Mécanique et Système Complexe, Faculté des Sciences de Yaoundé, BP 337, Yaoundé, Université de Yaoundé I, CAMEROUN

<sup>4</sup>Laboratoire de Recherchesur les Risques Naturels, Institut Nationale de la Cartographie (INC), BP 157, Yaoundé, CAMEROUN

\*Corresponding Author: e-mail: ludovicivan.li@gmail.com, Tel (+237)693005228  
ORCID iDs: <http://orcid.org/0000-0002-8768-7751> (Ntom Nkotto)

## Abstract

The ever-increasing cost of cement and its production of harmful carbone dioxide (CO<sub>2</sub>) has led development actors to continuously search for less environmentally polluting and low-cost binders. Hence the interest in the valorisation of agricultural resources through the production of different binders such as silicate for the elaboration of geopolymers at low manufacturing temperatures (<100 °C). In this study, Ingessil industrial silicate and rice husk ash silicate were used as pozzolan raw materials to prepare geopolymers with sodium hydroxide as activator at a concentration of 12 M. Two types of samples were developed: geopolymer mortars based on Ingessil silicate and geopolymer concretes based on Ingessil industrial silicate and rice husk ash silicate. Sand and gravel were used as fine and coarse aggregates for the production of geopolymer concretes. They were represented 80% of the mass of the geopolymer concretes according to the standards of elaboration. The porosity values obtained were of the order of 30%, i.e. water absorption values of about 18%, apparent specific weight of about 5% and bulk density of 1.7%. The mechanical values of BGAC-RHA samples are higher than those of BGAC-I. The physico-chemical properties of geopolymer mortars and concretes were similar.

**Keywords:** Clay, rice husk ash, geopolymer binder, composite materials, physico-chemical properties

DOI: <http://dx.doi.org/10.4314/ijest.v15i2.3>

Cite this article as:

Ntom Nkotto L.I., Dounbissi Kamgang G., Nkengue L.P., Sandjong Kanda J., Sontia Metekong J.V. 2023. Characterisation of cementitious composite materials based on calcined clay activated by silicate from rice husk ash and Ingessil silicate - activator: sodium hydroxide (NaOH). *International Journal of Engineering, Science and Technology*, Vol. 15, No. 2, pp. 23-35. doi: 10.4314/ijest.v15i2.3

Received: February 20, 2023; Accepted: March 1, 2023; Final acceptance in revised form: March 7, 2023

## 1. Introduction

Cement is a material that plays a major role in the construction of buildings, roads, dams, bridges and other infrastructure. The construction of these infrastructures requires a significant amount of Ordinary Portland Cement (OPC). OPC is the most important source of cement for these projects. This OPC is known to produce excellent concretes and mortars with very good mechanical properties (25-35 MPa at 28 days) depending on their applications and thus allow for easy large- and small-scale use (Farhan,

Sheikh, and Hadi 2019; Bernal et al. 2011; Lee and Lee 2013). Despite their good characteristics, OPC products are not stable in aggressive environments such as salty or acidic environments (Bernal et al. 2011; Lee and Lee 2013; Singh et al. 2015). Moreover, these cements are energy intensive and account for about 6-7% of global CO<sub>2</sub> emissions, hence the implementation of new CO<sub>2</sub> emission reduction policies following the COP 21 (Conference of Parties)(Ademe et al. 2021). The stakes are therefore very high for the United Nations (UN), which through a recent Intergovernmental Panel on Climate Change (IPCC) report aims to reduce the rate of CO<sub>2</sub> emissions by 50% by 2030 in order to maintain the climate threshold between 1.5 and 2% (IPCC, 2022). In addition to this climate problem, Africa, and Cameroon in particular, faces several other difficulties related to the production and use of OPC. These problems are mainly linked to the import of clinker and are manifested by recurrent shortages of cement and even by the high cost of this material, which is so essential for the implementation of major structural projects and for improving housing. In about ten years, the demand for OPC will double (Baumert and Melchior, 2015), hence the need to think about an alternative to OPC is imminent.

To date, several studies have been carried out to modify the composition of OPC through the use of various admixtures such as silica fume, CaCO<sub>3</sub>, metakaolin, rice husk ash, fly ash, nanomaterials and slag, in order to improve its performance under extreme conditions (Wardhono et al. 2015; Kubba et al. 2018; Cassagnabere et al. 2012; Shi, Jiménez, and Palomo 2011). These research results have provided the beginning of a solution for the partial or total substitution of OPC in the formulation of composite materials. They have led to the synthesis of geopolymer binders that are environmentally friendly and have similar and/or better properties than those based on OPC.

Geopolymers, introduced by Joseph Davidovits (Habert et al. 2011), are inorganic aluminosilicate materials obtained by activation at room temperature of a low-calcium aluminosilicate source by an alkali hydroxide solution (Saliha 2017). In the last decades, they have become the subject of great interest for researchers due to their good mechanical properties and low CO<sub>2</sub> emission rate (Rangan 2010). Several recent studies have shown that geopolymeric binders can be synthesised by alkaline activation of aluminosilicates obtained from industrial wastes such as fly ash (Djobo et al. 2014), metakaolins resulting from the calcination of clay between 550 and 750°C (Sontia et al. 2021; Sontia et al. 2022; Rahier et al. 1996; Elimbi, Tchakoute, and Njopwouo 2011). Another activity has also been developed in natural minerals such as the use of volcanic ash at ambient or fired temperature (around 100°C) or laterite.

This study involves the formulation of two optimal content composite materials. One based on clay and silicate from rice husk ash, the other based on clay and Ingessil industrial silicate. It is of economic interest through the promotion of silicate based on husk ash, which is local and therefore less expensive than industrial silicate - a scientific interest through the determination of the optimal formulation of the composite and its mechanical properties. An environmental interest in the valorisation of silicate from rice husk waste and the creation of a material with a low level of pollutants and which can be recycled.

The objective of this work is to study the properties of geopolymeric products obtained on the one hand by alkaline activation with clay ground and calcined at 750°C with a mixture of Ingessil sodium silicate and a sodium hydroxide solution, and on the other hand with clay, ground and calcined at 750°C with a mixture of sodium silicate from Ndop rice husk ash and a sodium hydroxide solution. These two samples were each formulated with a liquid/solid ratio of 0.75 (Sontia et al. 2022; Kaze et al. 2020).

This work also aims to carry out a comparative study between the industrial silicate Ingessil and the local silicate made from Ndop rice husk ash while looking for a local geopolymer having good mechanical properties and likely to be useful in the field of construction, roads and engineering structures. The physico-chemical properties were evaluated with respect to bulk density, water absorption, porosity, flexural and compressive strengths. These properties of geopolymer concretes were compared to those made from solution industrial silicate Ingessil and the local silicate made from Ndop rice husk ash. The obtained results were interpreted correlating the mix design and end properties.

## **2. Materials and Methods**

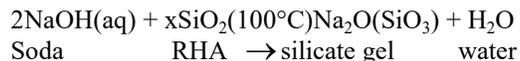
### **2.1. Materials**

The sand used in this study is of local origin commonly called Sanaga sand. It is an alluvial sand from the locality of Ebebdá, which is much sought after for its good properties in the construction field. This sand is extracted either by dredging the bed of the active watercourse or by diving by local residents (NtomNkotto et al. 2021). Tests such as: particle size analysis and sand equivalent were carried out on the said material to ensure its cleanliness and the quantity of fines present in the sand. The clay used in this experiment was taken from the locality of Nkooza in the city of Yaoundé in the Centre Region of Cameroon. This input to the geopolymer formulation was steamed at a temperature of 105°C for 24 hours, then ground and sieved to 80 µm. This clay was subjected to mineralogical analysis. After mineralogical study, the clay sample was placed in the oven for calcination at a temperature of 750 °C and then stored under ambient conditions before the geopolymer synthesis. Rice husk ash and commercial sodium silicate solution used in this study are collected in Ndop (South-West-Cameroon Region) and Ingessil s.r.l. (Verona, Italy). Granites are the aggregates used in this study and were collected in Yaoundé (Centre Region Cameroon). They were dried and sieved to obtain a grain size between 5 and 15 mm.

## 2.2. Geopolymer mortar and concrete formulations

### 2.2.1. Synthesis of activator solutions

The rice husk ash (RHA)/NaOH activator was prepared by adding a 6M NaOH solution to the as-collected rice husk ash (RHA) at a solid/liquid ratio consistent with the research work of Venyite et al. (Venyite et al. 2021). The mixture was then heated in a silver cookware at 100°C for 2 hours and the final product obtained was a dark grey viscous gel. The main reactive constituent of RHA is assumed to be silica which constitutes about 98% of RHA (Kamseu et al. 2017). The equation below represents the reaction of silicate gel formation from NaOH and silica (Myllyam et al. 2017).



The NaOH (sodium hydroxide, 8M) was prepared from solid pellets, 98.99wt%, from Sigma Aldrich, Italy. The Na<sub>2</sub>SiO<sub>3</sub> (sodium silicate) is an industrial product from Ingessil srl, Verona, Italy having SiO<sub>2</sub> of 26.6wt% and Na<sub>2</sub>O of 8.86wt% and ~60wt.% of loss of ignition. The spectroscopy of this material has been studied in more detail in the work by Tene et al. (Tene et al. 2015).

### 2.2.2. Geopolymer mortar formulation

The geopolymer mortars were formulated with Ingessil industrial silicates and those obtained from rice husk ash. We formulated our geopolymer on the basis of a liquid/solid ratio of 0.75. For this, we mixed 438g of calcined clay with 328.5ml of the alkaline solution in a 5L dish. In order to homogenise the composite, we mixed the geopolymer elements with an electric mixer ELE MOTAR MIXER for 5 minutes. The synthesized geopolymer was introduced into cylindrical moulds of dimensions 40×80 mm.

In order to see how our geopolymer reacts with other elements, we formulated a mortar by adding to the previous composition 1314 g of coarse Sanaga sand. This quantity of sand was defined according to the principles of mortar formulation with a ratio of sand mass to calcined clay mass of 3. The geopolymer plus coarse sand mixture was homogenised using the ELE MOTAR MIXER for 5 minutes. The resulting geopolymer mortar was moulded in prismatic moulds of dimensions 40×40×160 mm. These geopolymer mortars, made from Ingessil industrial silicate, were named MGAC-I.

All samples obtained were cured at room temperature for 28 days to assess their 28-day mechanical performance and physical properties. The test identified for this purpose were: water absorption and porosity, apparent specific gravity, apparent density for the determination of physical properties and the 28-day compression test each to highlight the mechanical properties.



**Fig.1** Geopolymer mortar samples

**2.2.3. Formulation of geopolymer concretes**

The geopolymer concretes were processed as follows: 442g of calcined clay and 333g of alkaline solution in a 5-litre dish. This mixture was made at a liquid/solid ratio of 0.75. The total mass of aggregates (sand + 5/15 gravel) to be added was defined as 80% of the geopolymer mass according to the standards for geopolymer concrete (Rangan 2010). After mixing the geopolymer for 5 minutes, 620g of aggregates were introduced into the geopolymer and the mixture was synthesised again in the mixer for 5 minutes. The geopolymer concretes from the Ingessil industrial silicate and the Ndop rice husk ash were named BGAC- I and BGAC-RHA respectively. These concretes were moulded in prismatic moulds of dimensions 40×40×160 mm. The samples obtained were kept at room temperature in order to identify the conditions of implementation on a construction site. After 14 days of curing, these samples were demoulded in order to evaluate their mechanical performance and physical properties in a wet area. The tests identified for this purpose were: water absorption and porosity, apparent specific gravity, apparent density for the determination of physical properties and compression and flexural tests at 14 days each to highlight the mechanical properties.

**2.3. Characterisation methods**

The grain size analysis was carried out to determine the grain size class of the material. It aims to determine the dimensions of the grains of the material studied and the percentages of grains of each size. Its implementation conditions are collected in the NF P 18-560 standard (NF P 18-560 1990). This sand has been classified according to its minimum and maximum grain size according to the standard (Mbessa, 2005). Thus, for an appropriate field of use listed in Table 1, the dimensions of the sand were identified.

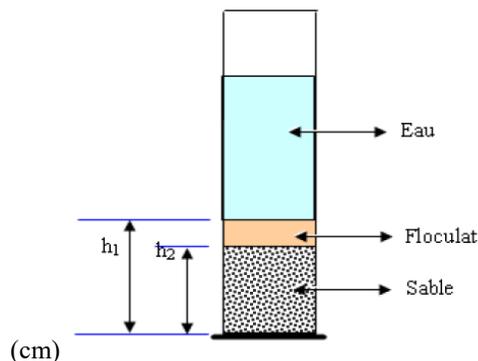
**Table 1.** Aggregate classification (Mbessa, 2005)

Designations	Pebbles			Gravel and chippings			Sands			Fines/Flour /Fillers
	Coarse	Medium	Small	Coarse	Medium	Small	Coarse	Medium	Small	
Dimensions	50-80 mm	31.5-50 mm	20-31.5 mm	12.5-20 mm	8-12.5 mm	5-8 mm	1.25-5 mm	0.315-1.25 mm	0.08-0.315 mm	<0.08 mm
Uses	Large concrete (dam...) Stonework Jackets - drainage - roads			Normal concrete Sleeves – drainage			Concrete - plaster - mortar - screed - prefabrication			Concrete

The sand equivalent was carried out to determine the cleanliness of our sand, i.e. the proportion of clay contained in the sand. This is because a high proportion of very fine particles ( $d < 0.08\text{mm}$ ) can lead to their flocculation in the presence of water, thus limiting the cohesion of the composite material. This would have a direct impact on the mechanical and physical characteristics of the material. The procedure for carrying out this test is described in standard NF P 18-598 (NF P 18-598 1991). In this study, this test was carried out wet in order not to lose any fine elements. This is because a small amount of fines also allows for workability. At the end of this manipulation, the Sand Equivalent (SE) is deduced by the formula (1)

$$SE(\%) = \frac{h_2}{h_1} \times 100 \tag{1}$$

Where  $h_1$  is the height of clean sand + fines (cm) and  $h_2$  is the height of clean sand only



**Fig.2** Schematic representation of the sand equivalent test.

Thus, the nature of our sand was identified according to **Table 2**

**Table 2.** Recommended values for sand equivalent by G.DREUX (Mbengue and Abdoul 2006)

ES on sight	ES on piston	Nature and quality of the sand
ES < 65 %	ES < 60 %	Clayey sand: risk of shrinkage or swelling Sand to be rejected for quality concrete
65 % < ES < 75 %	60 % < ES < 70 %	Slightly clayey sand of acceptable cleanliness for standard quality concrete when the shrinkage has no significant effect on the quality of the concrete
75 % < ES < 85 %	70 % < ES < 80 %	Clean sand with a low proportion of fine clay content perfectly suitable for quality concrete
ES > 85 %	ES > 80 %	Very clean sand. The almost total absence of clay fines may lead to a plasticity defect in the concrete which must be compensated for by increasing the water content.

The XRD was performed following the protocol of the Laboratoire de Géologie et Environnement Sédimentaire (AGES) of the University of Liège. The data were recorded with a Bruker D8-Advance diffractometer using the  $K\alpha_1$  radiation of copper ( $\lambda = 1.5418 \text{ \AA}$ ). The measurement speed of the goniometer is 10s/step, rotational speed  $2^\circ 2\theta/\text{min}$ , inducing an analysis time of several minutes to cover an angular range from  $2\theta=2$  to  $70^\circ$  for non-oriented powders and  $2\theta = 2$  to  $30^\circ$  for oriented slides. The acceleration voltage is 40KV, the current 30mA. It is connected to a computer for automatic data recording. The diffractograms obtained are analysed with the help of a software used for the identification of the crystalline phases DIFFRAC Plus Release 2000-EVA 6.0 and the data supplied by the PDF files (Powder Diffraction File). The chemical composition of the calcined clays was carried out by Bruker S8 Tiger X-ray fluorescence spectrometer. Prior to analysis, each sample powder was mixed with lithium borate salt and vitrified in a Pt crucible at  $1060^\circ\text{C}$ . The loss on ignition at  $1050^\circ\text{C}$  was considered to make the calculation of the final chemical compositions. Water absorption, density and porosity were determined in accordance with the procedures in ASTM C 373 (ASTM 1999). The flexural strength test was carried out on the  $40\times 40\times 160$  mm samples using a material characterisation apparatus with a three-point bending device. The handling conditions are described in EN 12390-5 (NF EN 12390, 2003) and the bending strength (in MPa) is obtained from the equation (2).

$$\sigma_{\max} = \pm \frac{3}{2} \times \left( \frac{F \times L}{b \times h^2} \right) \quad (2)$$

Where  $F$  denotes the load applied at the centre (N),  $L$ : the length between supports (mm),  $b$ : the width of the specimen (mm) and:  $h$  the height of the specimen (mm).

In order to determine the compressive strength of our composite materials as described in EN 12390-3 (NF EN 12390, 2003), the half-prisms of the previously used  $40\times 40\times 160$  mm flexural specimens were reused (Kubba et al. 2018). The specimens were subjected to simple compression until crushing using a compression test press and the compressive strength is calculated according to the equation(3):

$$R_c = \frac{F}{S} \quad (3)$$

With  $R_c$  being the compressive strength or breaking stress in MPa,  $F$  the compressive force at break in  $N$  and  $S$  the area of force application in  $\text{mm}^2$ .

### 3. Results and discussion

#### 3.1. Characteristics of geopolymer raw materials

##### 3.1.1. Sand grain size

The results obtained from the granulometric analysis of the alluvial sand from Ebebda in Figure 3 show 7% of coarse particles, 68% of medium particles and 25% of fine particles. This sand is suitable for concrete and mortar because of the proportion of medium and coarse particles that are suitable for the strength of the matrix and the proportion of fine particles that are suitable for cohesion (acting as a binder for the material). (Billhout 1994)

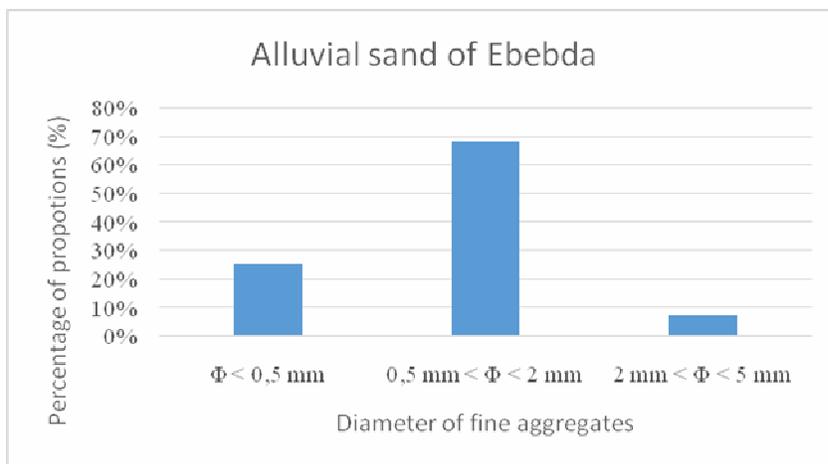


Fig. 3 Grain size of the alluvial sand of Ebebda

### 3.1.2. Sand equivalent

The alluvial sand used during this work has a sand equivalent value of 94.5% because the values of  $h_1$  and  $h_2$  are 16.50 cm and 15.60 cm respectively. This sand is intended to be clean in accordance with the French standard NF P18-598, which prescribes a clean sand for a value of  $>80\%$ , thus indicating the almost total absence of fines. It is therefore ideal for the use of concrete, including high-performance concrete. Indeed, the properties of the aggregate affect the durability and performance of the concrete, so fine aggregate is an essential component of concrete and cement mortar (Hudson 1997).

### 3.1.3. Mineralogical composition of clay

The analysis range is from  $2^\circ$  to  $70^\circ$  with a step size of  $0.02^\circ$  and an acquisition time of 2s. The crystalline phases present in the material are identified by comparison with the PDF (Powder Diffraction Files) standards of the ICDD (International Center for Diffraction Data):

- Kaolinite 7,16 Å ; 3,57 Å ; 2,56 Å ; 1,48 Å (PDF 14 – 164) ;
- Illite 4,44 Å ; 3,34 Å ; 1,98 Å (PDF 26 – 911) ;
- Quartz 4,25 Å ; 3,34 Å ; 2,45 Å ; 2,28 Å ; 2,23 Å ; 2,12 Å ; 1,98 Å ; 1,81 Å ; 1,65 Å ; 1,54 Å ; 1,45 Å ; 1,38 Å 1,37 Å (PDF 46 – 1045);
- Calcite 2,28 Å ; 1,91 Å (PDF 5 – 586);
- Muscovite 3,33 Å ; 2,56 Å ; 2,12 Å ; 1,65 Å (PDF 21 – 993) ;
- Hematite 1,48 Å ; 1,45 Å (PDF 33 – 664).

Chemical compositions of the various minerals, including: kaolinite ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ),

illite ( $\text{K,H}_3\text{O})(\text{Al,Mg,Fe})_2(\text{SiAl})_4\text{O}_{10}[(\text{OH})_2,(\text{H}_2\text{O})]$ ), quartz ( $\text{SiO}_2$ ), calcite ( $\text{CaCO}_3$ ), muscovite ( $\text{KA}_2\text{Si}_3\text{AlO}_{10}(\text{OH})_2$ ) and hematite ( $\alpha - \text{Fe}_2\text{O}_3$ ). These results obtained are similar to the work of researchers Sontia et al. (Sontia et al. 2022), Kaze et al. (Kaze et al. 2020), Nana et al. (Nana et al. 2020) and Tchakouté et al. (Tchakouté et al. 2020) who worked with clayey-lateritic, clay, halloysite and kaolin for the formulation of geopolymer binders. Kaolinite present will be transformed into metakaolinite during calcination at  $750^\circ\text{C}$  and thus contribute to the formation of the amorphous phase necessary for geopolymer synthesis as reported by others on different clays used as solid precursors (Sontia et al. 2022, Kaze et al. 2020, Nana et al. 2020, Tchakouté et al. 2020). The strong presence of iron and aluminium in the formulation of our geopolymers reveals that our clay material is predominantly illite.

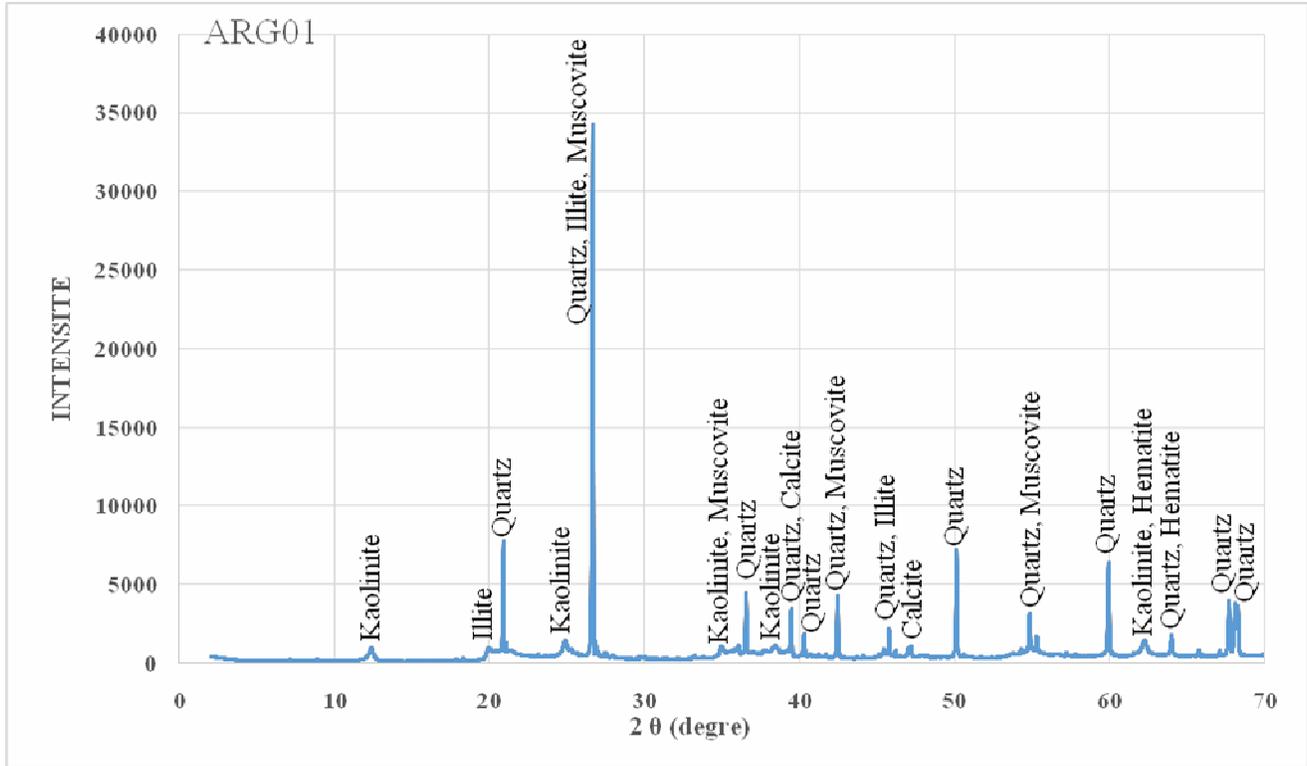


Fig. 4 Mineralogical composition of clay

3.1.4. Chemical composition of clay

The result of the X-ray fluorescence giving the base oxides of the Nkolbisson clay (ARG01) is contained in the following Table 3.

Table 3. Chemical composition of clay

Sample	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	PaF	Total
ARG01	65,14	17,63	4,59	0,06	0,23	0,05	0,69	0,15	0,18	10,09	98,81

This clay has a high silica and aluminium content, but a low calcium content. This justifies the fact that this material reacts easily with the alkaline solution.

3.2. Characteristics of composite materials: geopolymers

3.2.1. Geopolymer mortars: MGAC-I

3.2.1.1. Water absorption and porosity of geopolymer mortars

The results for water absorption and porosity are given in Tables 4 and 5 respectively. The average values are 12.16% and 23% respectively. Our geopolymers show a low water absorption rate compared to the work of Bignozzi et al. (Bignozzi et al. 2014) who compounded geopolymers with grog and different amounts of sodium silicate solution in combination with a constant amount of 8M NaOH as activating agents. This difference could be explained by the mineralogical nature of the clay used or the Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> ratio and the sodium silicate content used. This absorption rate also remains lower than the work of Beleuk et al (Beleuk et al. 2017). This author carried out similar work to ours, with the exception of the use of grog in that work. Thus, the mineralogical nature of our base materials contributed to the differentiation of our results.

Table 4 . Water absorption rate of geopolymer mortars based on Ingessil industrial silicate

Samples	Dry mass (g)	Saturated mass (g)	Water absorption(%)
MGAC1	86.68	97.99	13.05
MGAC2	82.57	91.87	11.26
MGAC			12.16

Porosity is one of the most important physical characteristics of the material and has a direct influence on the mechanical properties of the material. The nature and size of the pores allows the assessment of other properties such as: density, thermal conductivity, strength, water absorption capacity and durability. Thus, control of porosity, including its amount, distribution and average pore size, is always one of the main objectives of material design. (Hudson 1997)

**Table 5.** Porosity of geopolymer mortars based on the industrial silicate Ingessil

Samples	Dry mass (g)	Saturated mass (g)	Suspended mass (g)	Volume of the sample (cm <sup>3</sup> )	Porosity (%)
MGAC1	86.68	97.99	51.4	46.59	24.28
MGAC2	82.57	91.87	49.05	42.82	21.72
MGAC					22.99

### 3.2.1.2. Apparent specific weight and bulk density of geopolymer mortars

It provides information on the water content of our materials and the ability of a material to absorb and retain liquid water. Our material has an average bulk specific gravity of 8.27 which is shown in **Table 6**.

**Table 6.** Apparent specific gravity of geopolymer mortars based on the industrial silicate Ingessil

Samples	Dry mass (g)	Saturated mass (g)	Apparent specific weight (g/cm <sup>3</sup> )
MGAC1	86.68	97.99	7.66
MGAC2	82.57	91.87	8.88
MGAC			8.27

The average bulk density obtained is 1.89 as shown in **Table 7**, this value is within the scope of the work of Bignozzi et al. (Bignozzi et al. 2014).

**Table 7.** Bulk density of geopolymer mortars based on the industrial silicate Ingessil

Samples	Dry mass (g)	Saturated mass (g)	Suspended mass (g)	Volume of the sample (cm <sup>3</sup> )	Bulk density (g/cm <sup>3</sup> )
MGAC1	86.68	97.99	51.4	46.59	1.86
MGAC2	82.57	91.87	49.05	42.82	1.93
MGAC					1.89

### 3.2.1.4. Compressive strength of geopolymer mortars

**Table 8** shows the results of the compressive strength of geopolymer mortars. The geopolymer mortars used had an average compressive strength of 5.21 MPa. This value remains above that prescribed by the NF EN 772-1 standard for the use of lightweight concretes suitable for masonry elements such as breeze blocks and hoardings (NtomNkotto et al 2021). Furthermore, the average strength value obtained of 5.21 MPa is similar to the maximum value of Zeynab et al (Zeynab et al. 2015), i.e. 5.27 MPa with the same Na<sub>2</sub>SiO<sub>3</sub>/NaOH ratio of 2.5. However, the silicate used in the work of Zeynab et al. (Zeynab et al. 2015) is the one based on RHA rice husk ash. These physical and mechanical characteristics are similar to the work of Beleuk et al (Beleuk et al 2017).

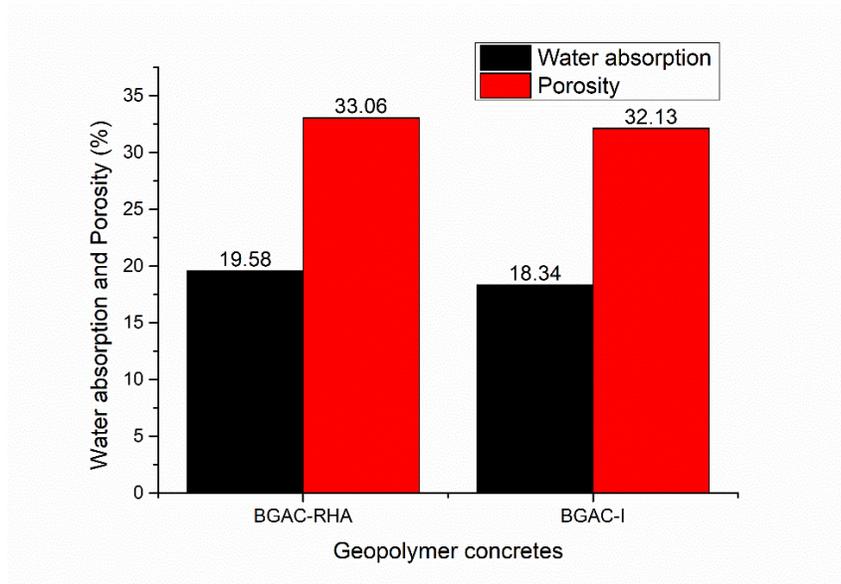
**Table 8.** Compressive strength of geopolymer mortars based on the industrial silicate Ingessil

Samples	Surface (cm <sup>2</sup> )	Applied force (kN)	Longitudinal Compressive Strength (MPa)
MGAC 1	16	6	3.75
MGAC 2	16	7	4.375
MGAC 3	16	12	7.5
MGAC			5.21

### 3.2.2. Geopolymer concretes: BGAC-I and BGAC-RHA

#### 3.2.2.1. Water absorption and porosity of geopolymer concretes

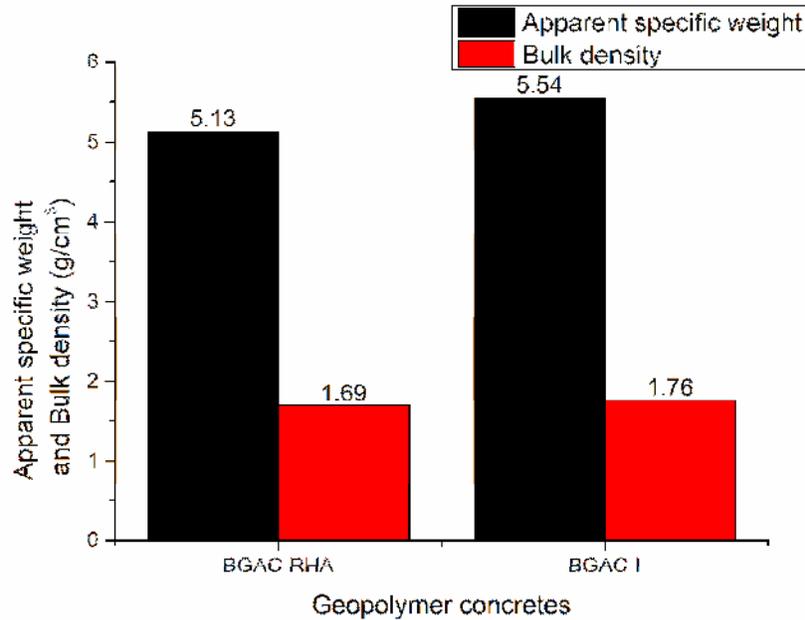
Water absorption and porosity of geopolymer concretes cured at room temperature for 28 days are shown in **Fig 5**. The water absorption values of the BGAC-RHA and BGAC-I samples were 19.58 % and 18.34 % respectively. While the porosity of the BGAC-RHA and BGAC-I samples were 33.06 % and 32.13 % respectively. The results obtained are similar for both the rice husk ash activator solution and the Ingessil solution. But the values of BGAC-RHA samples are higher than those of BGAC-I. Furthermore, the porosity values are approximately double those of the water absorption.



**Fig. 5** Water absorption and porosity of geopolymer concretes at 28 days

#### 3.2.2.2. Apparent specific weight and bulk density of geopolymer concretes

Apparent specific weight and bulk density of geopolymer concretes cured at room temperature for 28 days are shown in **Fig 6**. The apparent specific weight values of the BGAC-RHA and BGAC-I samples were  $5.13\text{g/cm}^3$  and  $5.54\text{g/cm}^3$  respectively. While the bulk density of the BGAC-RHA and BGAC-I samples were  $1.69\text{g/cm}^3$  and  $1.76\text{g/cm}^3$  respectively. The results obtained are similar for both the rice husk ash activator solution and the Ingessil solution. But the values of BGAC-I samples are greater than those of BGAC-RHA. But the values of BGAC-I samples are higher than those of BGAC-RHA. Furthermore, the bulk density values are approximately one quarter those of the apparent specific weight. This is due to the fact that geopolymer concretes are porous.



**Fig. 6** Apparent specific weight and Bulk density of geopolymer concretes at 28 days

This is the weight per unit volume of the concrete without its interstitial voids. The results below are therefore in agreement with those given by the porosity, since BGAC-RHA is more porous than BGAC-I, the weight of a volume of concrete without gaps will therefore be greater in the latter. The difference between the average specific weight and bulk density is significant for both types of concrete. However, it is much greater in BGAC-I. This shows that BGAC-I contains more interstitial voids than BGAC-RHA (Cassagnabere et al 2013). The compactness rate will therefore be higher in BGAC-RHA. All these concretes have an average bulk density of less than 2.30. This is due to the high value of the liquid/solid ratio (0.75): a better density would have been obtained if a much smaller ratio had been used. However, these concretes can be used as thermal insulators because of their low average bulk density.

### 3.2.2.3. Flexural and compressive strengths of geopolymer concretes

Flexural and compressive strength of geopolymer concretes cured at room temperature for 14 days are shown in **Fig 7**. The flexural values of the BGAC-RHA and BGAC-I samples were 3.13 and 2.25 MPa respectively. While those of the compressive of the BGAC-RHA and BGAC-I samples were 14.06 and 10.56 MPa respectively. The results obtained are similar for both the rice husk ash activator solution and the Ingessil solution. But the mechanical values of BGAC-RHA samples are higher than those of BGAC-I. This is due to the fact that the activating solution from rice husk ash is richer in pure silica than the industrial solution from Ingessil. Thus, the said solution will contribute to the multiplication of Si-O-H bonds in contact with the metakaolinite, hence the good mechanical properties.

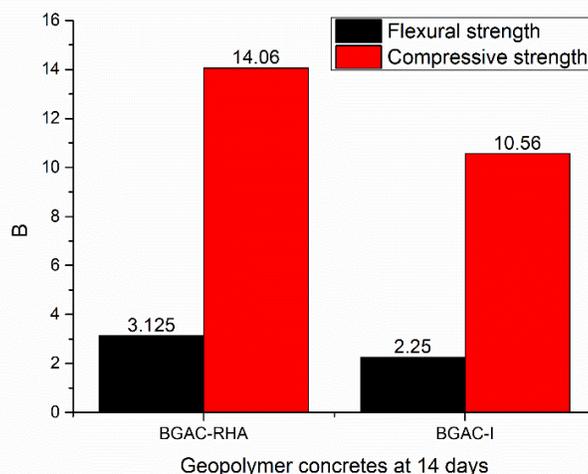


Fig. 7 Flexural and compressive strengths of geopolymer concretes at 14 days

#### 4. Conclusion

The main objective of this project was to investigate the properties physico-chemical of geopolymer concretes based on calcined clay at 750 °C in comparison using the rice husk ash activator solution and the Ingessil solution. Two formulations were considered in this project, named BGAC-RHA (rice husk ash activator solution) and BGAC-I (Ingessil solution). The flexural and compressive strengths were determined on the concrete samples at 14 days. From these results, the following conclusions were drawn:

- The water absorption, porosity, apparent specific weight and bulk density values of BGAC-I samples are higher than those of BGAC-RHA;
- The mechanical values of BGAC-RHA samples are higher than those of BGAC-I.

Based on the conclusions drawn from the results, the rice husk ash activator solution can be used for the formulation of geopolymer materials.

#### Acknowledgments

The authors would like to thank the authorities of MIPROMALO for their supervision during this work.

#### References

- Ademe. E. Mari, S. Sourisseau, A. Bouxin, C. Borde, G. Daill, S. Padilla, T. Gourdon. 2021. Plan de Transition Sectoriel de l'industriecimentière en France: Premiers résultatstechnico-économiques. Rapport de synthèse. 20 pages.
- ASTM. 1999. ASTM C373-14 Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products, Astm C373-88.
- Baumert K. and Melchior B., 2015. The practice of archipelagic states: a study of studies. *Ocean Development & International Law*, Vol. 46, No. 1, pp 60-80, <https://doi.org/10.1080/00908320.2015.995970>.
- Baumert K.A., Herzog T., Pershing J. 2015. Navigating the numbers greenhouse gas data and international climate policy. *World Resources Institute*. Part I, pp.17-19.
- Beleuk, L.M., Mohamed, H., Kamseu, E., Billong, N. and Melo, U.C. 2017. Properties of geopolymers made from fired clay bricks wastes and rice husk ash (RHA)- sodium hydroxide (naoh) activator. *Materials Sciences and Applications*. Vol. 8, pp537-552. <https://doi.org/10.4236/msa.2017.87037>
- Bernal, S. A., R. Mejía, D. Gutiérrez, A. L. Pedraza, J. L. Provis, E. D. Rodriguez, and S. Delvasto. 2011. Effect of binder content on the performance of alkali-activated slag concretes. *Cement and Concrete Research*, Vol. 41, No. 1, pp. 1–8. <https://doi.org/10.1016/j.cemconres.2010.08.017>.
- Bignozzi M.C., O. Fusco, A. Fregni, L. Guardigli and R. Gulli. 2014. Ceramic waste as new precursors for geopolymerization, *Advances in Science and Technology*, Vol. 92, pp26-31, doi:10.4028/www.scientific.net/AST.92.26
- Billhout H. 1994. Béton de sable:caractéristique set pratiques d'utilisation, Projet National de Recherche de Développement SABLOCRETE, Université de Paris, Presse de l'Ecole des Ponts et Chaussées, 121p.

- Cassagnabere, F, P Diederich, M Mouret, G Escadeillas, and M Lachemi. 2012. "Impact of metakaolin characteristics on the rheological properties of mortar in the fresh state, *Cement and concrete composites*, Vol. 37, pp. 95-107. <https://doi.org/10.1016/j.cemconcomp.2012.12.001>.
- Djobo, J. N.Y., L. N. Tchadjié, H. K. Tchakoute, B. B.D. Kenne, A. Elimbi, and D. Njopwouo. 2014. Synthesis of Geopolymer Composites from a Mixture of Volcanic Scoria and Metakaolin. *Journal of Asian Ceramic Societies*, Vol. 2, No. 4, pp. 387–98. <https://doi.org/10.1016/j.jascer.2014.08.003>.
- Elimbi, A., H. K. Tchakoute, and D. Njopwouo. 2011. Effects of calcination temperature of kaolinite clays on the properties of geopolymer cements. *Construction and Building Materials*, Vol. 25, No. 6, pp. 2805–2812. <https://doi.org/10.1016/j.conbuildmat.2010.12.055>.
- Emdadi Z., Asim N., Yarmo A., Shamsudin R. and Alghoul M.A. 2015. The effect of mix composition on the water absorption, density and compressive strength of rice husk based geopolymers. *5th International Conference on Environment Science and Engineering*, Vol. 83 of IPCBEE. pp. 92-96. <https://doi.org/10.7763/IPCBEE.2015.V83.15>.
- Farhan, N.A, Sheikh M.N. and Hadi M.N.S. 2019. Investigation of engineering properties of normal and high strength fly ash based geopolymer and alkali-activated slag concrete compared to ordinary portland cement concrete. *Construction and Building Materials*, Vol. 196, pp. 26–42. <https://doi.org/10.1016/j.conbuildmat.2018.11.083>.
- Foret R., Guigou-Carter C., Chéné J. 2010. Paramètres des matériaux poreux influant sur la performance acoustique des systèmes du bâtiment. 10ème Congrès Français d'Acoustique, Lyon, pp.12-16.
- Grouped experts Intergouvernemental sur l'Evolution du Climat (GIEC). 2022. Communiqué presse du giec, Secrétariat du GIEC c/o OMM, 7 bis, Avenue de la Paix, C.P: 2300, CH-1211 Genève 2, Suisse, Courriel IPCC-Sec@wmo.int ,2022, 6p. [www.ipcc.ch](http://www.ipcc.ch)
- Habert, G, J B Espinose, De Lacaillerie, and N Roussel. 2011. An environmental evaluation of geopolymer based concrete production: reviewing current research trends. *Journal of Cleaner Production*, Vol. 19, No. 11, pp.1229–1238. <https://doi.org/10.1016/j.jclepro.2011.03.012>.
- Hudson B. P. 1997. Manufactured sand for Concrete. *The Indian Concrete Journal*, pp. 237 -240
- Kamseu, E, Moungam L.M.B., Cannio M., Billong N., Chaysuwan D., Melo U.C. and Leonelli C.. 2017. Substitution of sodium silicate with rice husk ash-naoh solution in metakaolin based geopolymer cement concerning reduction in global warming. *Journal of Cleaner Production*, Vol. 142, No. 4, pp. 3050-3060. <https://doi.org/10.1016/j.jclepro.2016.10.164>
- Kaze C.R., Alomayri T., Hasan A., Tome S., Lecomte-Nana G.L., Nemaleu J.G.D., Tchakoute H.K., Kamseu E., Melo U.C., Rahier H. 2020. Reaction kinetics and rheological behaviour of meta-halloysite based geopolymer cured at room temperature: effect of thermal activation on physicochemical and microstructural properties. *Applied Clay Science*, Vol. 196, 105773, <https://doi.org/10.1016/j.clay.2020.105773>.
- Kubba, Z., Fahim G., Rahman A., and Sam M.. 2018. Case Studies in construction materials impact of curing temperatures and alkaline activators on compressive strength and porosity of ternary blended geopolymer mortars. *Case Studies in Construction Materials* 9: e00205. <https://doi.org/10.1016/j.cscm.2018.e00205>.
- Lee, N K, and H K Lee 2013. Setting and mechanical properties of alkali-activated fly ash/slag concrete manufactured at room temperature. *Construction and Building Materials*, Vol. 47, pp. 1201–1209. <https://doi.org/10.1016/j.conbuildmat.2013.05.107>.
- Metekong J.V.S., Kaze C.R., Adesina A., Nemaleu J.G.D., Djobo J.N.Y., Lemougna P.N., Thamer A., Elie K., Chinje M.U., Tatiétsé T.T., 2022. Influence of thermal activation and silica modulus on the properties of clayey-lateritic based geopolymer binders cured at room temperature, *Silicon*, Vol. 14, pp7399–7416, <https://doi.org/10.1007/s12633-021-01566-7>.
- Metekong J.V.S., Kaze C.R., Deutou J.G., Venyite P., Nana A., Kamseu E., Melo U.C., Tatiétsé T.T., 2021, Evaluation of performances of volcanic-ash-laterite based blended geopolymer concretes: Mechanical properties and durability. *Journal of Building Engineering*, Vol. 34, 101935, <https://doi.org/10.1016/j.jobte.2020.101935>.
- Mbengue, S., and M. Abdoul. 2006. Etude Comparative Des Résultats in Situ En Vue de La Maitrise de La Qualité : Application Au Béton de Densité Normale, Projet de Fin d'étude, Département de Génie Civil Centre de Thiès, 115p.
- Mbessa, M. 2005. Traité de Génie Civil, Vol. 1 : Matériaux, Département de Génie Civil de l'ENSP de Yaoundé I, 172p.
- Myllyam L, Mohamed H, Kamseu E, Billong N. 2017. Properties of geopolymers made from fired clay bricks wastes and rice husk ash (RHA)-sodium hydroxide (NaOH) activator. pp. 537–552. <https://doi.org/10.4236/msa.2017.87037>
- Nana A., Alomayri T.S., Venyite P., Kaze R.C., Assaedi H.S., Nobouassia C.B., Sontia J.V.M., Ngouné J., Kamseu E. and Leonelli C., 2020. Mechanical properties and microstructure of a metakaolin-based inorganic polymer mortar reinforced with quartz sand, *Silicon*, <https://doi.org/10.1007/s12633-020-00816-4>.
- NF EN 12390, 2003, Essais pour béton durci," AFNOR sagaweb.
- NF P 18-560. 1990. Granulat-Analyse Granulométrique.
- NF P 18-598. 1991. Granulat-Equivalent de Sable.
- Ntom Nkotto L.I., Tchoungang Kouonang S., Sofack J.C., Ndigui B. et Likiby B. 2021. Caractérisation Des Agglomérés Produits Avec Les Agrégats de Rivière et de Carrière, Centre-Cameroun. *Afrique Science*, Vol. 18, No. 2, pp 108–123.
- Rahier, H., B. Van Mele, M. Biesemans, J. Wastiels, and X. Wu. 1996. Low-temperature synthesized aluminosilicate glasses. *Journal of Materials Science*, Vol. 31, No. 1, pp71–79. <https://doi.org/10.1007/bf00355129>.
- Rangan, B. V. 2010. Fly ash-based geopolymer concrete. *Proceedings of the International, Workshop on Geopolymer Cement and Concrete*, pp. 68–106. <https://doi.org/10.1007/s10853-006-0523-8>.

- Saliha, A. 2017. Synthèse, caractérisation, microstructure et durabilité des géopolymères à base de cendres volantes. PhD Thesis, University of Mohammed V, Faculty of Sciences, Rabat, 178p.
- Shi, C., Jiménez A.F., and Palomo A.. 2011. New cements for the 21st century: The pursuit of an alternative to portland cement, *Cement and Concrete Research*, Vol. 41, No. 7, pp. 750–63. <https://doi.org/10.1016/j.cemconres.2011.03.016>.
- Singh, B., G. Ishwarya, M. Gupta, and S. K. Bhattacharyya. 2015. Geopolymer concrete: A review of some recent developments, *Construction and Building Materials*, Vol. 85, pp. 78–90. <https://doi.org/10.1016/j.conbuildmat.2015.03.036>.
- Tchakouté H.K., Melele S.J.K., Djamen A.T., Kaze C.R., Kamseu E., Nansu C.N.P., Leonelli C., Rüscher C.H., 2020. Microstructural and mechanical properties of poly(sialate-siloxo) networks obtained using metakaolins from kaolin and halloysite as aluminosilicate sources: A comparative study, *Applied Clay Science*, Vol. 186, doi: 10.1016/j.clay.2020.105448.
- Tenefongang R.T. Pemndje P.N. Lemougna U. Chinje Melo C.P. Nansu B. Nait-Ali E. Kamseu C. Leonelli. 2015. “Cleaner Production of the lightweight insulating composites: Microstructure, pore network and thermal conductivity. *Energy And Buildings*, S0378-7788(15)30194-8. <http://dx.doi.org/doi:10.1016/j.enbuild.2015.08.009>, Reference: ENB 6072
- Venyite, P., Makone E.C., Kaze R.C., Nana A., Nemaleu J.G.D., Kamseu E., Melo U.C., and Leonelli C., 2021. Effect of combined metakaolin and basalt powder additions to laterite-based geopolymers activated by rice husk ash (RHA)/NaOH solution. *Silicon*. Vol. 14, pp. 1643–1662. <https://doi.org/10.1007/s12633-021-00950-7>.
- Wardhono A., Law D.W., Strano A. 2015. The strength of alkali-activated slag/fly ash mortar blends at ambient temperature, *Procedia Engineering*, Vol. 125, pp. 650–656. <https://doi.org/10.1016/j.proeng.2015.11.095>.

### Bibliographical notes

**Ing. Ludovic Ivan Ntom Nkotto**, Design engineer in civil and urban engineering. He is currently working in the research institute MIPROMALO (Mission de Promotion des Matériaux Locaux) as a research officer in the Department of Design and Construction (D.D.C.). He is also a thesis student at the ENSP (Ecole Nationale Supérieure de Polytechnique de Yaoundé I).

**Architect Gaëlle Dounbissi Kamgang**, works as a research associate at MIPROMALO. In addition, he is doing research work at the University of Douala in the Master II cycle.

**Ing. Lionel Pierre Nkengue**, engineer of public works design, he is also carry out research work in doctoral cycle option physics at the University of Yaounde I

**Ing. Judicaël Sandjong Kanda**, is an environmental engineer, specialising in environmental sanitation and restoration, and he is doing thesis work at the University of Yaounde. In addition, he works as a research assistant at the Natural Hazards Laboratory of the National Institute of Cartography (N.I.C.)

**Ing. Jordan Valdès Sontia Metekong**, Civil and Urban Engineering Design Engineer. He is currently working in the MIPROMALO (Mission de Promotion des Matériaux Locaux) Research Institute as a research officer at the Department of Design and Construction (D.D.C.).