

Comparative Study on Geopolymer Binders Based on Two Alkaline Solutions (NaOH and KOH)

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Abstract

This study specifically investigated the influence of the composition of aluminosilicate material *i.e.* the substitution of metakaolin by rice husk ash and the nature of alkaline activators (Na⁺/K⁺) on mineralogical, structural, physical and mechanical properties of geopolymer binders. This influence was evaluated based on X-ray diffraction (XRD), Fourier Transform InfraRed spectroscopy (FTIR) and Scanning Electron Microscope (SEM analyses, apparent density, water accessible porosity, compressive strength and thermal properties. Two types of geopolymer binder were synthesized according to the type of alkali activator used, the NaOH-based geopolymer and the KOH-based geopolymer. The results of characterization performed after 14 days of curing of geopolymer samples showed that the activation of the aluminosilicate powder using alkaline solution led to change in their microstructure. The highest compressive strength was obtained with the NaOH-based geopolymer.

Keywords

Geopolymer, NaOH, KOH, Metakaolin, Rice Husk Ash, Compressive Strength

1. Introduction

Burkina Faso has huge reserves of clay materials which have known little local

valorization, only limited to artisanal applications such as pottery and building materials [1]. Additionally, value addition to these materials can potentially be achieved through their geopolymers yielding geopolymers binder for stabilization of compressed earth bricks (CEBs) [2]. This approach not only improves the performance of CEBs but also limit the environmental damages linked to the production of the commonly used cementitious binders such as cement and lime [3]. The geopolymer, being more environmentally friendly and cost-effective than the cementitious binder [4], would contribute to catering for affordable and decent housing to the needy majority of rural population in Burkina Faso.

The geopolymer binder is obtained by geosynthesis process through activation of amorphous aluminosilicate materials with alkaline solution of sodium or potassium hydroxide (Na^+/K^+). Previous studies have shown that the highest level of dissolution of alumina (Al_2O_3) and silica (SiO_2) precursor is achieved in the presence of Na^+ rather than K^+ cations. On the other hand, the degree of gel formation is more important in potassium-based rather than sodium-based geopolymer [5]. Therefore, the nature of the alkaline solution appears to play fundamental role in the geopolymer synthesis and can influence the properties of the final materials.

Xu and Deventer [6] claimed that KOH-based geopolymer has higher compressive strength compared with NaOH-based geopolymer. However, Palomo *et al.* [7] additionally showed that NaOH-based geopolymer can yield higher compressive strength than KOH-based geopolymer depending on the variations in curing temperature and time or alkali activator/aluminosilicate ratio. Duxson *et al.* [8] concluded that the Si/Al ratio is an important parameter for studying the effect of alkali activators on the compressive strength of the geopolymer. Activation of aluminosilicates can be accelerated or improved by applying moderate heat treatment. Nevertheless, hardening at elevated temperatures (above 100°C) promotes the appearance of cracks and can negatively affect the properties of the geopolymer binder [9].

Metakaolin and rice husk ash were used as the main source of aluminosilicate material for the synthesis of geopolymer as in previous studies [10] [11]. In the present study, two alkaline solutions (NaOH/KOH) were additionally used as activator to investigate the influence of their nature on the properties of geopolymer binders. This study specifically focuses on the mineralogical, physico-mechanical and thermal properties of the geopolymer binders. It also aims to provide better understanding of the type of alkaline solution to be used for the geopolymersization of compressed earth bricks.

2. Materials and Experimental Methods

2.1. Materials

Two powders (Figure 1), aluminosilicates and siliceous respectively sourced from metakaolin and rice husk ash, were used as basic materials for the formulation of geopolymers binders. Metakaolin (MK) is obtained by heat treatment of local kaolin (K) at 700°C. The rice husk ash (RHA) was obtained by 3 hours'

mineralization at 550°C of carbon residue resulting from rice husk gasification. Mineralogical, chemical and physical characterizations of these materials showed their amorphous nature and potential for synthesis of geopolymers [10].

The solutions of sodium and potassium hydroxide concentrated at 12 M were used for the activation of these two powders. They were obtained by dissolving pellets of NaOH and KOH (99% purity and provided by COPROCHIM company) in distilled water.

2.2. Experimental Methods

2.2.1. Samples Preparation

The formulation of the geopolymers binders using NaOH and KOH activators was made according to the description made in the previous study on the characterization of NaOH-based geopolymers [10]. The geopolymers paste is obtained by mixing the alkaline solution with powders.

Mass ratios (alkaline solution/powder) of 0.7 and 0.8 were used for the formulation of the pastes using KOH and NaOH activators, respectively. The two mass ratios (0.7 and 0.8) were adopted with regard to the difference in the densities of the two alkaline solutions in order to obtain the similar consistency of the paste. Homogenization of the blends was achieved using HOBART blender for 10 minutes. The paste obtained is used to make prismatic test pieces ($4 \times 4 \times 16$ cm 3). A total of six formulations of geopolymers samples were synthesized:

- AN: aluminosilicate powders (100% metakaolin) + NaOH.
- BN: aluminosilicate powders (95% metakaolin and 5% rice husk ash) + NaOH.
- CN: aluminosilicate powders (90% metakaolin and 10% rice husk ash) + NaOH.
- AK: aluminosilicate powders (100% metakaolin) + KOH.
- BK: aluminosilicate powders (95% metakaolin and 5% rice husk ash) + KOH.
- CK: aluminosilicate powders (90% metakaolin and 10% rice husk ash) + KOH.

The different test pieces were then cured for 14 days, including 7 days at room temperature in the laboratory ($30^\circ\text{C} \pm 5^\circ\text{C}$) and 7 days at 60°C with an oven. After the curing, they underwent different characterizations (mineralogical, physical, mechanical and thermal) in order to highlight the influence of the nature of the alkaline solution and the substitution rate of metakaolin by the rice husk ash on the geopolymers binders.

2.2.2. Mineralogical and Chemical Characterization

X-ray diffractometry (XRD—Siemens D5000 diffractometer, operating with Co K α , $k = 1.789$ Å, acquisition: 0.04° -(4° - 70°) and 2s per step), Fourier transform infrared spectrometry (FTIR—Bruker Alpha-p IR spectrophotometer, operating in absorbance mode, acquisition: $600 - 4000$ cm $^{-1}$ with 4 cm $^{-1}$ of resolution under 12 scans, sample mass: 50 mg, data processing: SPECTRUM software), Thermogravimetric and differential thermal analyses (TG/DTA—Setaram Setsys Evolution

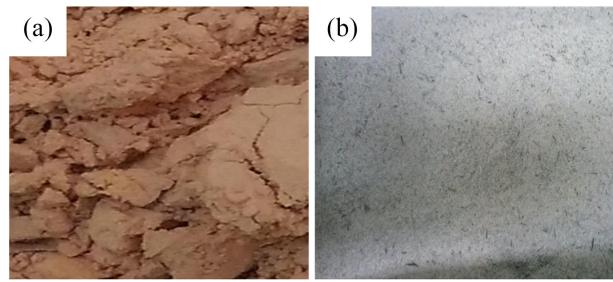


Figure 1. Aluminosilicates and siliceous powders—(a) meta-kaolin, (b) rice husk ash.

device up to 1050°C (heating rate 10°C/min) under dry air (100 mL/min), sample mass: 50 mg) and microscopic observation (SEM—Zeiss Supra 55VP) were performed on geopolymers binders based on NaOH and KOH. These analyses aimed at understanding the role of alkalis on the geopolymersization process in order to choose the alkaline solution most appropriate for the activation of aluminosilicate materials used in this study.

2.2.3. Physical Mechanical and Thermal Properties

The following characterization was performed to evaluate the influence of the nature of the alkaline solution on geopolymers binders:

- ✓ Water accessible porosity and apparent density (ISO-5017) are determined, in Equations (1) and (2), respectively, from the measurements of 3 different weight (in grams) of the geopolymers sample: weight in water (M_w), weight in air (M_o) and dry weight of the sample (M_d).

$$\varepsilon = 100 \times \frac{M_o - M_d}{M_o - M_w} \quad (1)$$

$$\rho_d = \frac{M_d}{M_o - M_w} \times \rho_w \quad (2)$$

where:

ρ_d is the apparent density of the geopolymers samples;

ρ_w is the density of water;

ε is the water accessible porosity of geopolymers samples (%).

- ✓ The compressive strength was determined by applying Equation (3). The breaking force is determined using hydraulic press (ETI-Proeti) which has load cell capacity of 300 kN, at loading rate of 0.25 kN/s.

$$R_c = 10 \times \frac{F_r}{S} \quad (3)$$

where:

R_c : compressive strength in MPa;

F_r : breaking force of the sample in kN;

S : surface area of the tested samples in cm².

- ✓ Thermal conductivity is the only thermal property measured on geopolymers binder samples. It was measured using hot wire method. This method con-

sists in applying a constant power to an electric wire immersed in the cylindrical sample (4 cm diameter considered to be infinite). The desired thermal parameter is then deduced from the mathematical descriptions.

3. Results and Discussion

3.1. Mineralogical and Chemical Characterization

3.1.1. Mineralogical Characterization of Geopolymer Binders by XRD

Figure 2 shows the diffractograms of the geopolymers based on NaOH (AN, BN and CN) and KOH (AK, BK and CK). It reveals the formation of zeolitic products whose nature and degree of crystallinity depend on the nature of the cation (Na^+ or K^+) from the alkaline solution used during the synthesis and also on the composition of the aluminosilicates powders.

The diffractograms of the NaOH-based geopolymers in **Figure 2(a)** show the formation of crystalline zeolitic products such as zeolite A, faujasite and hydrosodalite. The formation of these minerals was also influenced by the addition of rice husk ash during geopolymer synthesis. Thus, zeolite A appears only on samples which do not contain rice husk ash (AN) while the crystallinity of faujasite intensifies with the addition of rice husk ash (BN and CN). The hydrosodalite, unlike faujasite, has crystallinity peaks that gradually disappear as function of the addition of rice husk ash.

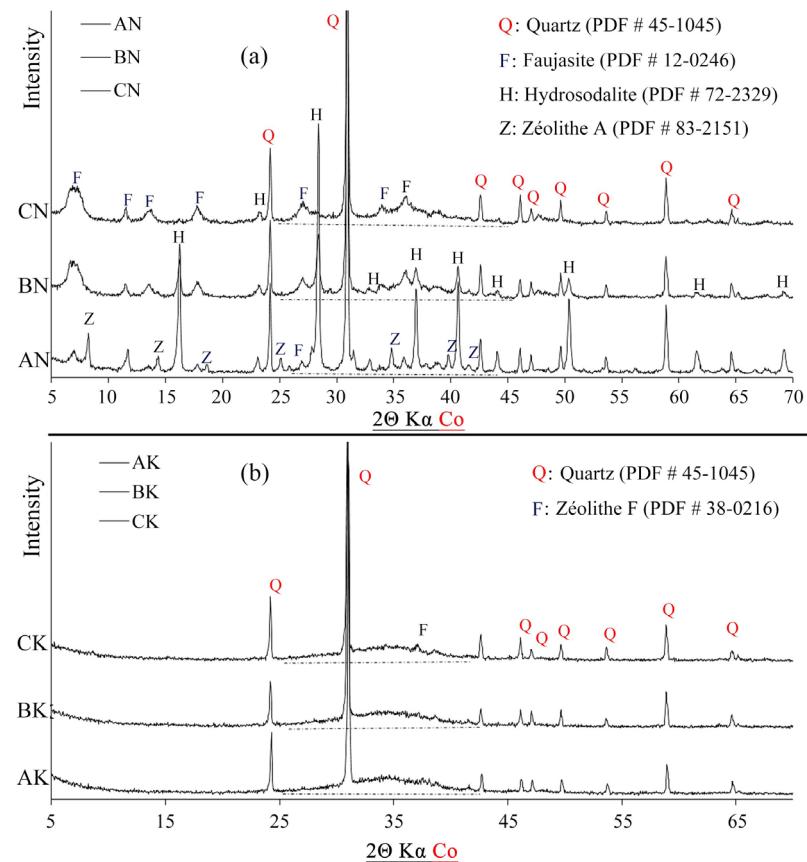


Figure 2. XRD diffractograms of geopolymer binder based on NaOH (a) and KOH (b).

The diffractograms of the KOH-based geopolymers in **Figure 2(b)** show less crystalline peaks compared to the diffractograms of the NaOH-based geopolymers. In addition to quartz reflection, zeolite F is the only mineral crystallized in these geopolymer samples, showing very low level of intensity. The presence of zeolite F seems to be favored by the addition of the rice husk ash, the characteristic peak of the zeolite F appears more visible on the sample CK (containing 10% of rice husk ash).

In addition, halo peaks ranging from 25° to 45° 2θ are observed on all geopolymer samples. They are more pronounced on samples containing rice husk ash (BN and CN). This reveals the amorphous character within these samples. The halos characteristics of the amorphous phases were previously observed on the diffractograms of metakaolin and rice husk ash from 15° to 40° and 15° to 45° 2θ, respectively [10]. They tend to slightly move to higher angles on the diffractograms of geopolymers based on KOH and NaOH: 22° to 43° and 22° to 45° 2θ, respectively. This shows the partial dissolution of the amorphous phase from the raw materials and the formation of new amorphous phase within geopolymer materials [12]. The NaOH-based geopolymers presented higher crystallinity than those based on KOH. This could influence the mechanical and physical properties of these geopolymer binders.

3.1.2. Mineralogical Characterization of Geopolymer Binders by FTIR

The infrared (IR) spectra of geopolymers based on NaOH and KOH is shown on **Figure 3(a)** and **Figure 3(b)**, respectively. The bands around 3365 - 3255 cm⁻¹ and 1635 or 1646 cm⁻¹ respectively express the deformation due to bonds vibrations of O-H and H-O-H of the water molecules [13]. The bands at 1439 or 1384 cm⁻¹ are attributed to the deformation O-C-O bond vibrations, which suggest the presence of sodium or potassium carbonate. The formation of these carbonates can be explained by the atmospheric carbonation of alkaline activator [14].

The main band detected at 955 cm⁻¹ is attributed to the Si-O-M⁺ (M = K or Na) bonds that characterize the zeolites [15] [16]. The zeolithic products were identified on XRDs diffractograms of the different geopolymers (**Figure 2**). The fundamental difference between NaOH and KOH-based geopolymers lies between the bands in the range of 800 to 600 cm⁻¹. On the NaOH-based samples (**Figure 3(a)**), four bands (734, 704, 681 and 663 cm⁻¹) are identified which are respectively associated with Si-O-Al, Si-O-Si and Si-O bonds. These bands appear only on the spectrum of the sodium compounds so they seemingly mark the presence of zeolites as was previously identified on the XRDs of these compounds. In addition, their intensity decreases with silica content (BN and CN) which is associated with the formation of the geopolymer gel. On the spectra of the KOH-based geopolymers (**Figure 3(b)**), a single band around 675 cm⁻¹ is identified and is associated with the Si-O bonds of quartz [17]. Contrary to the infrared spectra of NaOH-based geopolymers, the intensity of this band remains the same on the three samples (AK, BK and CK). This is in agreement with the

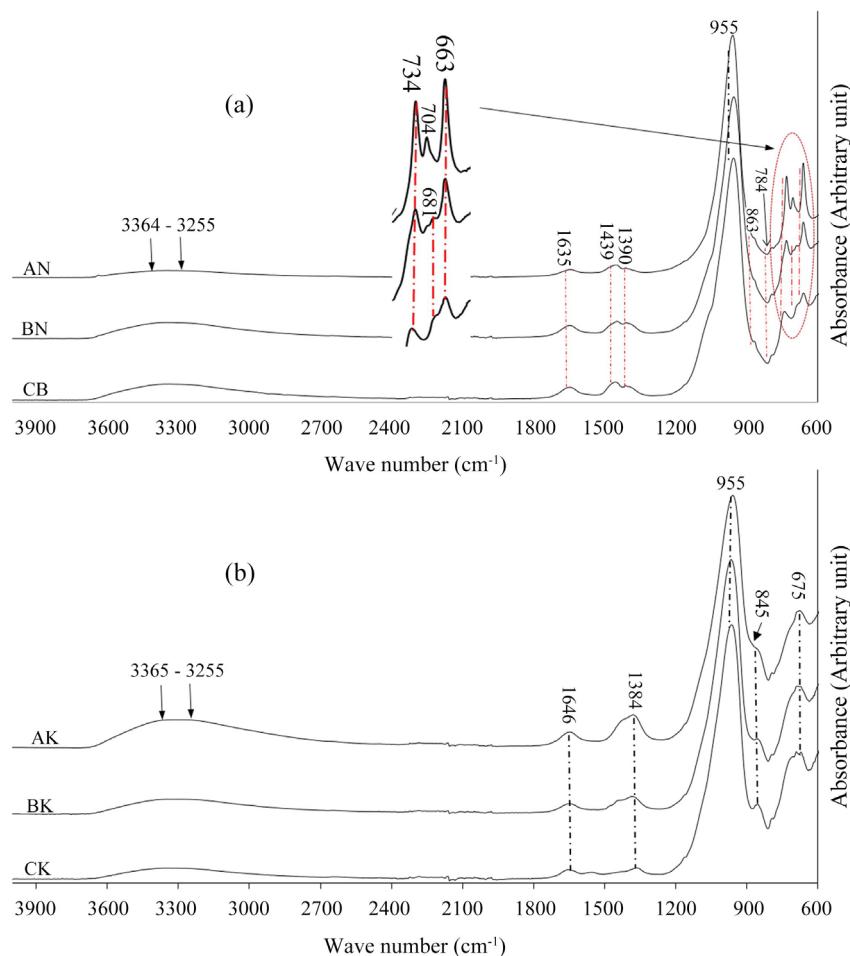


Figure 3. IRTF of the samples: (a) NaOH-Based and (b) KOH-based geopolymer.

diffractograms of samples AK, BK and CK which are substantially identical with the only exception of the zeolitic peak visibly appearing on the CK sample (**Figure 2(b)**).

3.1.3. Thermal Analyses (TG/DTA) Geopolymer Binders

The thermal analyzes (TG and DTA) of the various geopolymer compositions are presented in **Figure 4**. Thermogravimetric analyses recorded the similar mass losses for NaOH-based compounds (only 4.5% difference) for all silica content (**Figure 4(a)**). For KOH-based compounds, the increase of the silica content increased the loss of mass, recording 16.5% of difference in mass loss (**Figure 4(b)**).

The thermo-differential analyses recorded endothermic reactions at 170°C and 380°C for NaOH-based compounds (**Figure 4(a)**), reflecting the evaporation of bonded water. Their intensities increase at higher silica contents. The endothermic reaction at 575°C corresponds to the transformation of quartz ($\alpha \rightarrow \beta$) [18]. The endothermic reactions at higher temperatures (683°C and 750°C) correspond to zeolite or geopolymer constituents [19]. The exothermic reactions at 706°C, 822°C and 862°C can be associated with the recrystallizations

of zeolites or geopolymers [20]. The endothermic reaction at 890°C can be related to the decomposition of the formed carbonate as was observed in FTIR of the NaOH-based compounds (**Figure 3(a)**).

For the KOH-based compounds (**Figure 4(b)**), the dehydration reactions at 170°C and 402°C are well marked for sample BK. These compounds record similar endothermic and exothermic reactions as for the NaOH compounds. It is noteworthy that the temperatures at which exothermic reactions took place lowered from sample AK to CK (858°C → 820°C and 729°C → 673°C). As in the previous case, the reaction at 890°C can be related to the decomposition of K_2CO_3 .

3.2. Physical, Mechanical and Thermal Characterization

3.2.1. Physical Characterization of Geopolymer Samples

Figure 5 shows the evolution of water accessible porosity and its relationship with apparent density of geopolymer binders. The water accessible porosity of all samples varies from 34% to 42%, whereas their apparent density varies from 1.2 to 1.3. These properties evolved differently in NaOH-based from KOH-based compounds depending on their compositions.

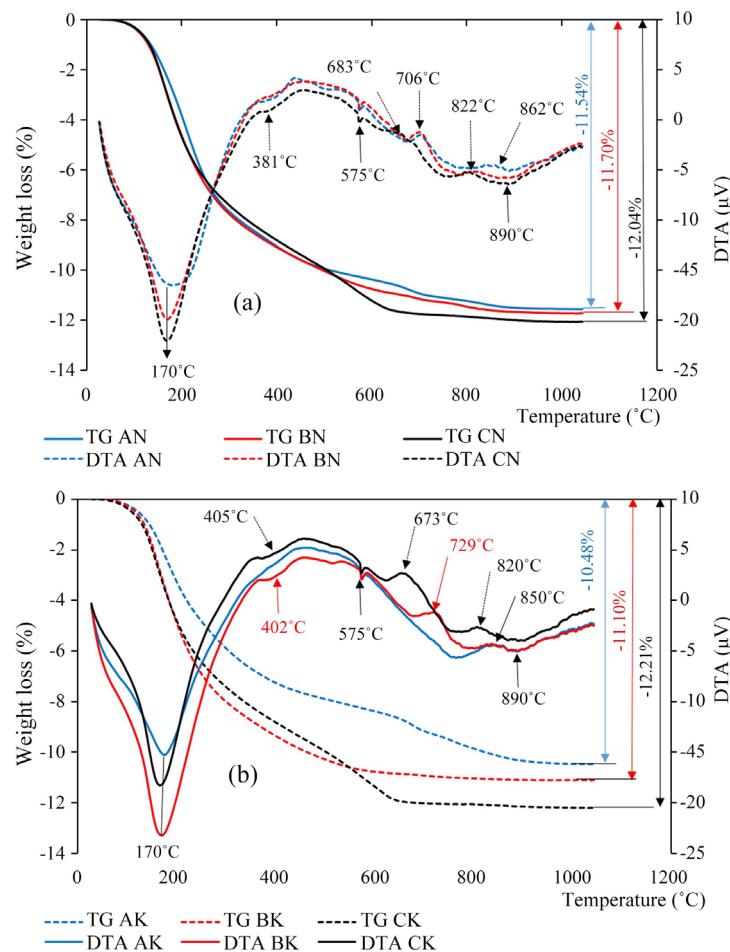


Figure 4. Thermal analysis of geopolymers samples based on NaOH (a) and KOH (b).

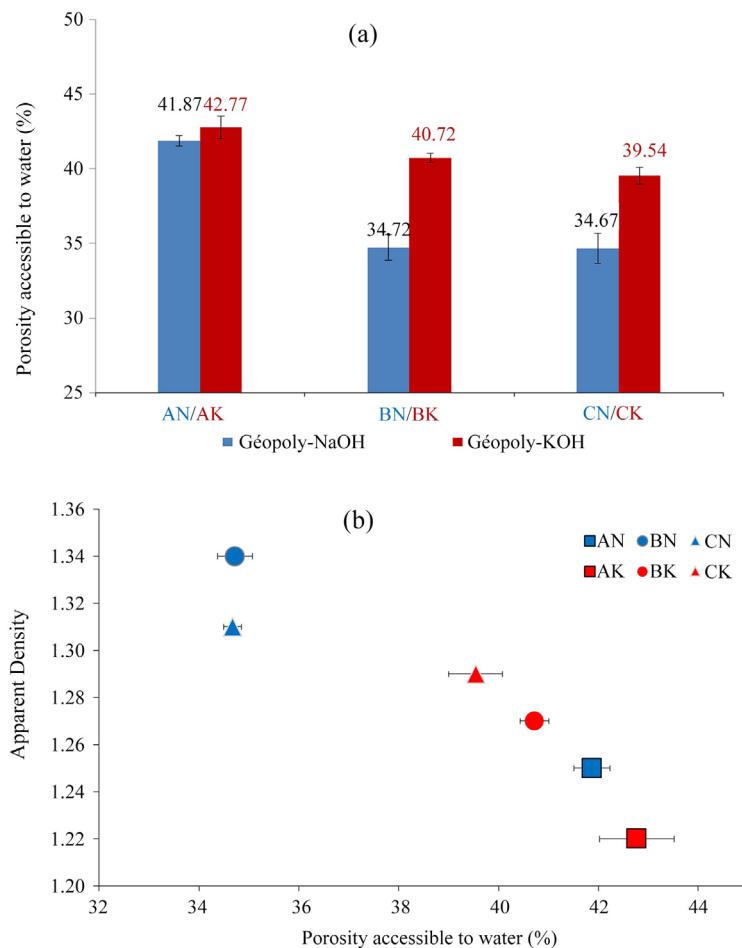


Figure 5. Porosity accessible to water (a) and apparent density (b).

For KOH-based compounds, the correlation between porosity and density is clearly highlighted. The porosity decreases as the density increases which is related to their silica content (**Figure 5(b)**). These variations can be explained by the poor crystalline character of these geopolymers, their high sensitivity to the evaporation of water and the contraction of their network.

The NaOH-based compounds always recorded higher density and lower porosity than their equivalent KOH-based compounds. These differences are essentially related to the crystallized nature of geopolymers consisting mainly of zeolites. Small decrease in density of CN compound can be explained by its well-developed geopolymer phase.

To better understand the influence of the type of alkaline solution on the porosity of the geopolymer binders, microscopic observations were performed on geopolymer samples CN and CK. The micrographs taken at two different scales (10 µm and 1 µm) reveal higher porosity for the KOH-based samples compared to those based on NaOH (**Figure 6**). This difference in porosity within the two samples is distinctively remarkable on the micrographs at 1 µm scale. This agrees with the results of water accessible porosity shown in **Figure 5**. Moreover, these micrographs reveal a dense homogeneous structure and some silica grains which

are respectively identified as geopolymer gel and probably quartz from the metakaolin.

3.2.2. Mechanical Characterization of Geopolymer Samples

The compressive strengths of the NaOH and KOH-based geopolymers are shown in **Figure 7**. The NaOH-based geopolymers show higher compressive strengths than those of the KOH-based samples. This result can be related to the lower porosity of the NaOH-based geopolymer binders as shown in **Figure 5** and their higher crystallinity. Similar behavior of the mechanical properties of geopolymers was previously reported for those based on NaOH compared to those based on KOH [8] [21]. Xu and Deventer [6] argued that better performance in compression of samples based on NaOH against those based on KOH can be attributed to the high degree of dissolution of the aluminosilicates in the presence of the sodium hydroxide solution. According to their study, the small ionic radius of Na^+ compared to that of K^+ favors the reaction of ionic pairs with smaller silicate oligomers, thus improving the bond between particles [6]. Therefore, the compressive strength of geopolymers strongly depends on the nature of the alkali hydroxide used as activator.

Moreover, the compressive strength of the geopolymer binders was improved with the addition of rice husk ash to metakaolin. The strength respectively reached the highest value at 5% and 10% for NaOH-based and KOH-based samples. This reveals the effect of silica (rice husk ash) content regardless of the type of alkaline solution. The increase of the mechanical strength is quasi-linear for the geopolymers based on KOH (8.3 - 11.9 - 15.2 MPa). For the NaOH-based geopolymers, it is noted that this increase in compressive strength has a maximum value for the BN (5% rice husk ash) sample (25.2 MPa).

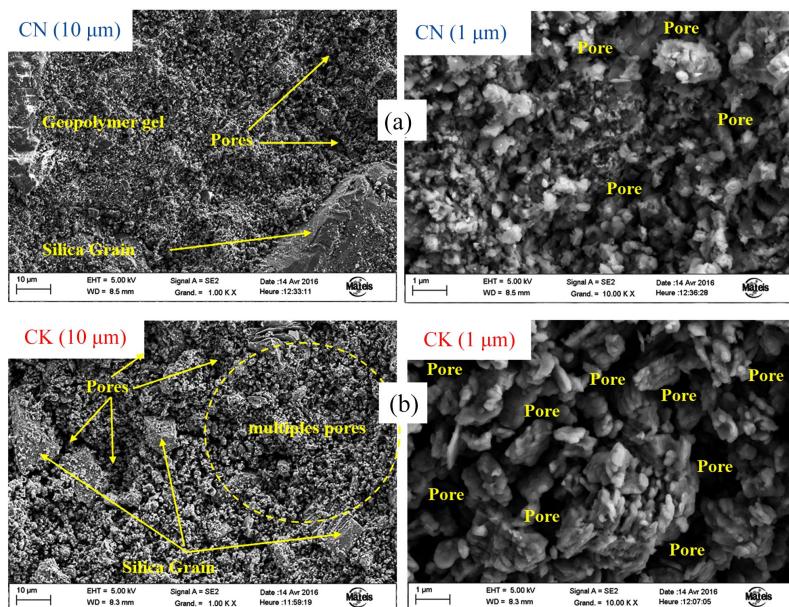


Figure 6. Micrographs of geopolymer samples CN (a) and CK (b) at two different scales (10 μm and 1 μm).

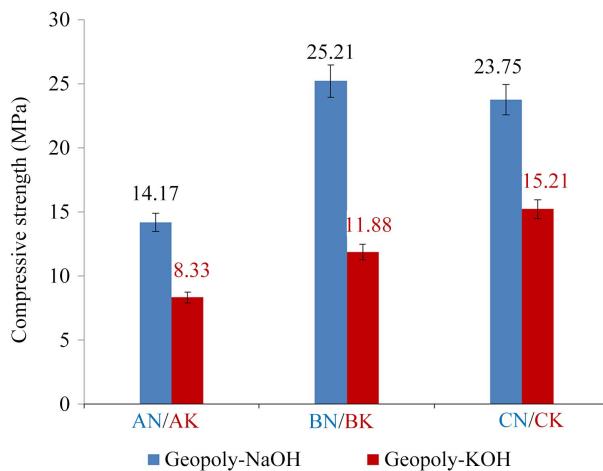


Figure 7. Compressive strength of geopolymer samples.

The addition of the rice husk ash to metakaolin increased the amount of amorphous silica in the aluminosilicate complex mixture. This possibly promoted the release of silicate $[\text{SiO}(\text{OH})_3]$ species during the dissolution stage induced by alkaline hydrolysis. The concentration (12 M) of the two types of alkaline solutions was relatively high and resulted in the formation of gel consisting of large three-dimensional network of aluminosilicate. This was responsible for the bond between particles of the formed products, allowing them to withstand the elevated stresses during the compressive test.

3.2.3. Thermal Characterization of Geopolymer Samples

The thermal conductivity of the different geopolymer formulations is shown in **Figure 8**. NaOH-based geopolymers have thermal conductivity varying from 0.31 to 0.44 W/m.K, while the values for those based on KOH, which recorded the lowest densities, range from 0.22 to 0.28 W/mK. The thermal conductivity of potassium is known to be approximately half that of sodium [22]. This may have partly contributed in achieving low thermal conductivity of KOH-based geopolymers. Additionally, **Figure 8(b)** shows the correlation between the thermal conductivity and the water-accessible porosity of the geopolymer samples. Samples with higher porosity tend to have lower density which also may have contributed to reducing their thermal conductivity. Feng *et al.* [23] indeed reported that the higher the porosity, the lower the thermal conductivity. Moreover, the thermal conductivity is influenced by the addition of the rice husk ash. **Figure 8(a)** shows that geopolymer binders containing rice husk ash have higher thermal conductivity. This is due to their relatively denser matrix. The densification is associated with higher $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio resulting in better polycondensation reaction in these geopolymer binders [24].

4. Conclusions

The results obtained in this study highlight the influence of the nature of alkaline solution (NaOH or KOH) used as activator of the aluminosilicate complex

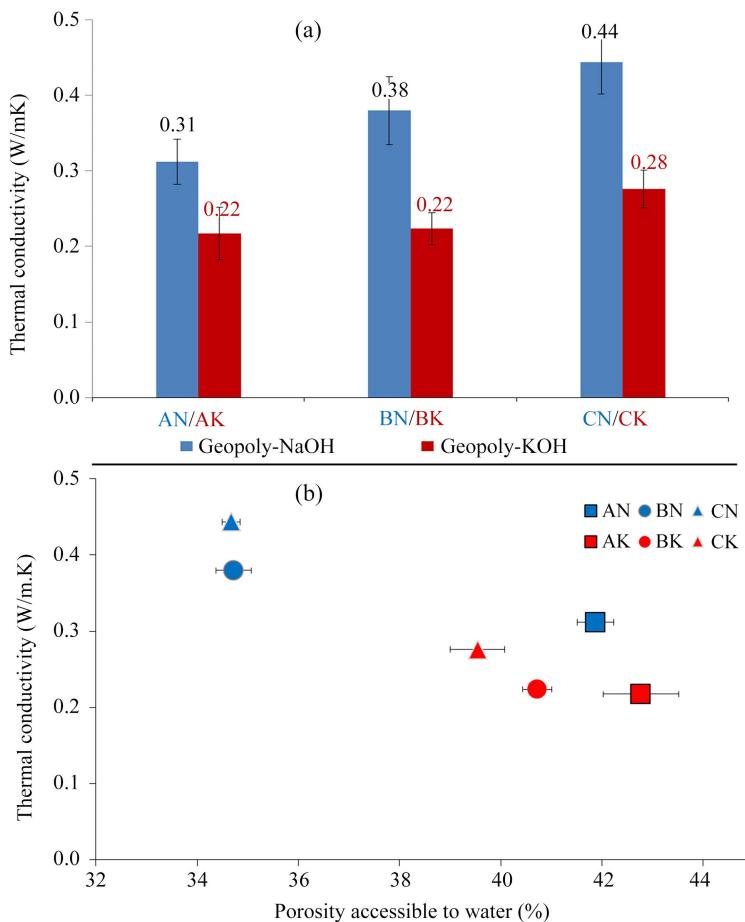


Figure 8. Thermal Conductivity and water accessible porosity of geopolymers samples.

(metakaolin with or without rice husk ash) on the various properties of the geopolymer binders. The XRD mineralogical analyses of geopolymer binders mainly show the formation of zeolite minerals (especially on NaOH-based samples) and the presence of amorphous phases (more pronounced on KOH-based samples). The presence of these zeolite products has been confirmed by FTIR.

The type of alkali solution used for activation of the aluminosilicate complex also significantly affects the physico-mechanical and thermal properties of geopolymer samples. NaOH-based samples yield the highest compressive strength, the lowest water-accessible porosity, and the highest apparent density. KOH-based geopolymers recorded the least thermal conductivity, which can be related to their higher porosity.

The addition of rice husk ash also improved the physical and mechanical properties but increased the thermal conductivity of geopolymer samples. Although the thermal conductivity of NaOH-based materials is higher than that of KOH based materials, it still remains lower than that of cementitious materials.

Given the results presented in this study and considering higher cost of the potassium hydroxide (approximately twice the cost of the sodium hydroxide), sodium hydroxide turns out to be more appropriate for the geopolymerization of compressed earth bricks.

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Conflicts of Interest

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

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