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Pozzolanic Reactivity Analysis of Kaolinite Clays from the Ketou Region in Benin

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Abstract

This study evaluates the possibility of using metakaolin produced from kaolinite clays from the villages of adjouzoumè and Adakplamè to substitute part of the Portland cement. The two clays are ground in a corn mill and passed through an 80-micron sieve before being calcined in a kiln at a speed of 10˚C/min up to 700˚C. Once 700˚C has been reached, this temperature is maintained for one hour. Reactivity is analyzed in accordance with ASTM C618. Mechanical tests were carried out on 40 × 40 × 160 mm^3 specimens to determine compressive strength. Chemical analysis was used to estimate the levels of the main oxides. The results showed that Adjozoumè and Adakplamè metakaolins have a strength activity index of 97.5% and 96.26% respectively. The main oxide contents were 94.5% and 95% respectively. In the sense of this standard, these metakaolins are reactive.

Keywords

Kaolinite Clays, Benin, Portland Cement, Metakaolins, Pozzolanic Reactivity

1. Introduction

Numerous studies have highlighted the need to reduce carbon dioxide (CO_2) emissions associated with the production of Portland cement ([1]-[5]). These emissions are estimated at around 7% to 8% of global GHG emissions [6]. This situation has stimulated a great deal of scientific research into alternative cementitious materials in all countries.

Interesting results have focused in particular on the use of metakaolin, which is obtained by controlled heat treatment of pure or impure kaolinitic clays [7]-[9]. Metakaolin improves strength, and substituting 10% to 25% of cement does not result in a significant loss of performance. Partial substitution of cement by metakaolin reduces CO_2 emissions by up to 25%, depending on the substitution rate [10]. It also reduces energy consumption during firing and overall cost in certain regions where clays are abundant and locally available [11]. Replacing between 10% and 30% of Portland cement with metakaolin improves long-term mechanical strength (beyond 28 days), and increases the durability of concrete against chloride penetration, carbonation and sulfate attack [12]. Metakaolin reacts rapidly with calcium hydroxide formed during hydration of Portland cement, forming secondary C-S-H phases that improve mechanical and physicochemical properties. However, reactivity is highly dependent on the mineralogical nature of the source clay, the calcination process (temperature, duration) and the fineness of the final product [13]-[16].

Benin has several kaolinitic clay deposits in the villages of Adjozoume and Adakplame in the Ketou region. It is therefore essential to determine their pozzolanic potential in order to determine whether they can be used as a substitute for Portland cement in concretes and mortars [17].

The aim is to determine the pozzolanic activity index and the main oxide composition of the metakaolins produced from these two clays.

2. Materials and Methods

2.1. Materials

- ***Kaolinitic clays***

The kaolinitic clays come from two villages in the Ketou region. These are the villages of Adakplamè and Adjozoumè. They are sampled and ground in a corn mill. The material passing through the 80-micron sieve is recovered for testing.

- ***Cement***

The cement used for our tests comes from the company "Nouvelle Cimenterie du Benin" SA (NOCIBE SA) and is of type CEM II/B-LL 32.5R with a specific density of 3.2 t/m^3 and complies with standard NF EN 197-1.

- ***Sand***

The sand used is locally available natural sand. Its characteristics are presented in **Table 1**.

Table 1. Characteristics of the sand used.

Characteristics	Value
Fineness modulus	1.81
Uniformity coefficient	4.07
Curvature coefficient	1.04
Sand equivalent (NF EN 933-8)	ES sight: 76 ES piston: 78
Bulk density	1468 kg/m^3
Absolute density	2697 kg/m^3

These characteristics show that the sand has a spread granulometry as Cu = 4.07 > 2. Its granularity is tight as Cc = 1.04. The sand equivalent value shows that the sand used is a clean sand with a low percentage of clay fines. It can therefore be used to produce good quality mortar or concrete.

2.2. Methods

The two kaolinitic clays are ground in a corn mill and the 80-micron passing are collected for calcination. An initial cycle of manual grinding was carried out to gradually break down the material. After each grinding cycle, the product obtained was sieved through a 1mm mesh sieve. The fractions that passed through the 1mm sieve were then subjected to a second, more intensive grinding using a mechanical mill of the "corn mill" type. This grinding was also carried out cyclically. After each pass through the mill, the ground product was sieved using an 80μm mesh sieve. The particles passing through this fine sieve were retained as the final product of this stage. This processing step is necessary as fineness plays an important role in reactivity. The passing are then calcined in a laboratory furnace. The furnace is programmed to a ramp rate of 10˚C·min^{-1} up to 700˚C. The temperature rise is monitored by the furnace's built-in PID controller. The ramp time to reach 700˚C is approximately 70 minutes. Once 700˚C is reached, the temperature is maintained constant for 60 minutes (soak time) to complete dehydroxylation and ensure homogeneous transformation throughout the entire powder volume. At the end of the soak, the furnace is shut down and the sample cools naturally in situ, with the door closed, to avoid thermal shocks that could induce micro-cracks in the agglomerates formed or alter the texture. Once the temperature has dropped below ≈100˚C, the crucibles are removed and the sample is left at room temperature until it has cooled completely. A 2-theta X-ray diffraction (XRD) test is used to analyze the mineralogy and principal oxides of the calcined clays.

Three types of cement mortar were formulated: control mortars with no cement replacement and mortars with 20% and 25% cement replaced by the metakaolin produced. The ratio of water to cement (W/C) used follows the recommendation of ASTM C311. A ratio sand to cement (S/C) of 2.75 is used.

Test specimens measuring 4 × 4 × 16 cm^3 are produced after mixing sand, cement, water, and/or metakaolin in a mortar mixer. The molds are then placed in the impact table and vibrated in accordance with ASTM C311. After demolding, the test specimens are stored in water until the crushing date. The 4 × 4 × 16 test specimens are first crushed in three-point bending, and the half-cubes are then crushed in compression (**Photo 1**).

The strength activity index is calculated in accordance with ASTM C618 as the ratio of the 28-day compressive strength of the specimen with 20% and 25% cement replacement by MK to that of the control specimen. When this index cumulatively exceeds 75% (in the case of 20% replacement) and the main oxide content exceeds 70%, the replacement material is pozzolanic [18].

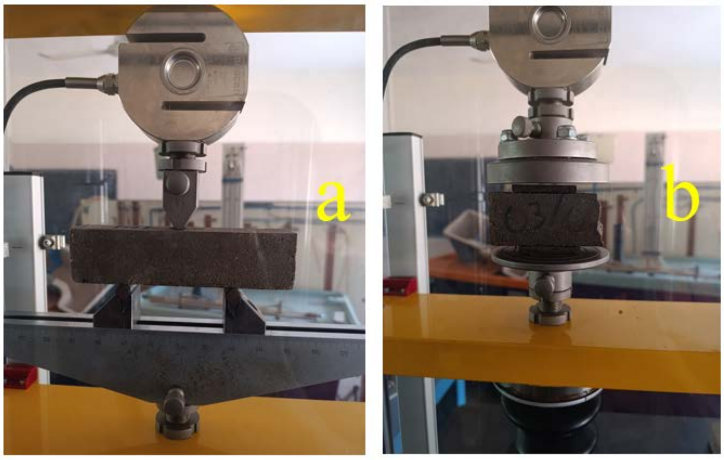

Photo 1. (a) three-point bending crushing; (b) compression crushing.

3. Results and Discussions

3.1. Mineralogical and Chemical Analysis

X-ray diffraction was used to identify the different minerals present in the two metakaolins produced. The diffractograms of the raw clays and metakaolins show that the peaks typical of kaolinite (12.3˚ for example) are absent (**Figures 1-4**). This indicates the disappearance of kaolinite crystalline structures, with the formation of amorphous phase (metakaolinite) increasing the indication of good calcination. Mineralogical analysis shows that these two metakaolins are composed of quartz (SiO_2), metakaolinites ($Al_2Si_2O_7$), kaolinite residues [$Al_2Si_2O_5(OH)_4$)] and amorphous impurities.

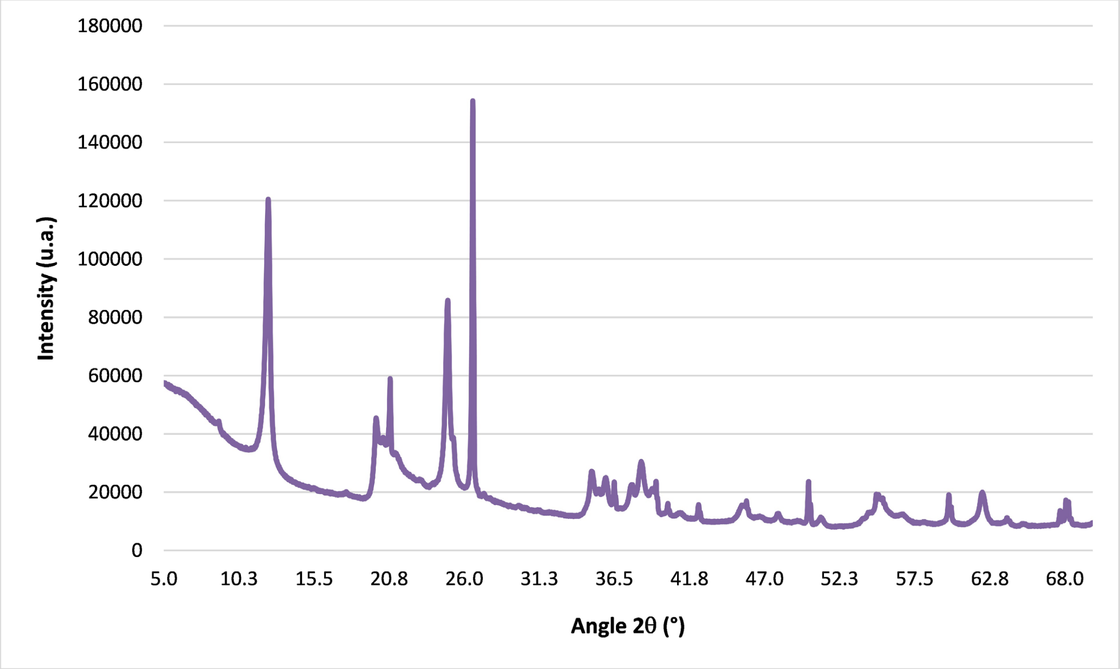

Figure 1. Adjozoumè raw kaolin diffractograms.

Chemical analysis was used to estimate the proportion of main oxides in the two metakaolins. The results are presented in the following **Table 2**. This chemical analysis reveals that metakaolin from Adjozoumè and Adakplamè respectively have a main oxide content (silica, alumina, iron oxide) of 94.5% and 95%, far exceeding the minimum required value. This means they fully comply with the first requirement of ASTM C618. These rates are higher than those of metakaolins produced by other researchers, [19]-[21].

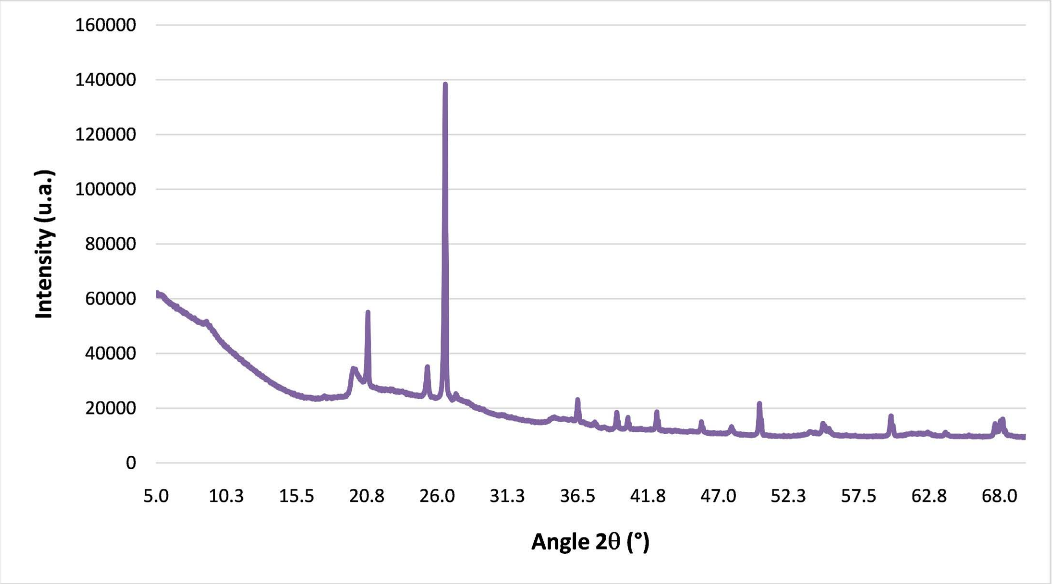

Figure 2. Calcined adjozoumè kaolin diffractograms.

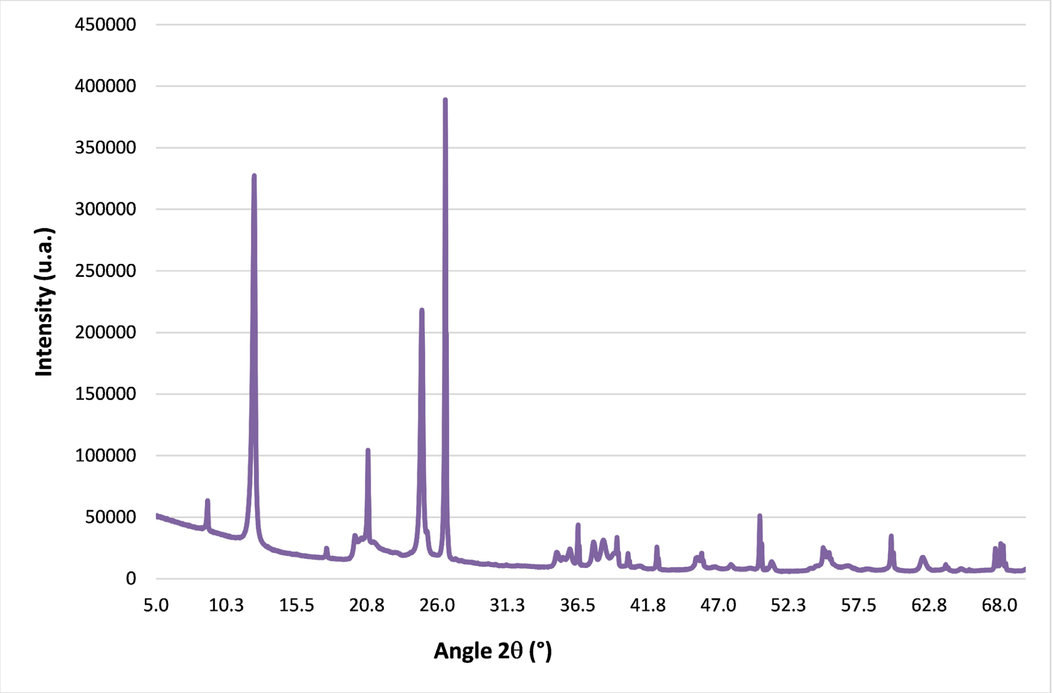

Figure 3. Crude Adakplamè kaolin diffractograms.

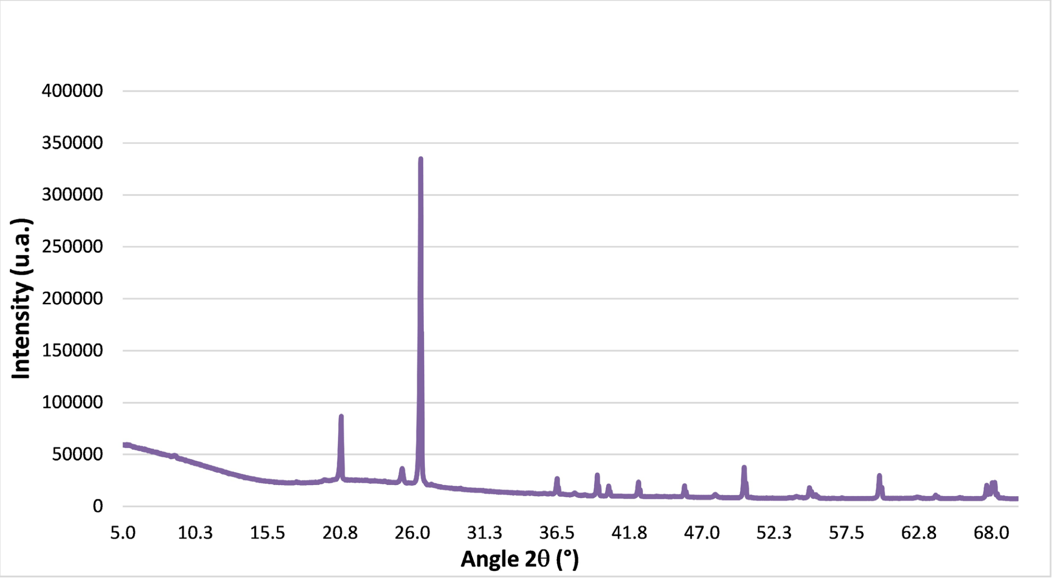

Figure 4. Calcined adakplamè kaolin diffractograms.

Table 2. Chemical composition of calcined kaolins.

		Proportion (%)	
Oxides	Formula	Adjozoumè (MK1)	Adakplamè (MK2)
Silica	SiO_2	55.0	57
Alumina	Al_2O_3	37.0	36
Iron oxide	Fe_2O_3	2.5	2
Potash	K_2O	1.0	1.0
Soda	Na_2O	0.5	0.5
Lime	CaO	0.3	0.5
Magnesia	MgO	0.2	0.5

3.2. Activity Index by Strength

Compressive strengths are measured on specimens at various times. The results obtained for control specimens (MT), specimens containing adjozoumè metakaolin (MK1) and those containing adakplamè metakaolin (MK2) are shown in the graphs in **Figure 5** and **Figure 6**.

The results show a continuous increase in compressive strength for all specimens. This demonstrates a pozzolanic reaction between Portland cement and metakaolin [22]. The evolution of compressive strength shows a good consumption of Portlandite to form additional silicate phases. The calcination temperature as well as the fineness due to the sieving with an 80 micron sieve contributed to the development of the pozzolanic reaction as indicated in the works of Geu [23] and Morh [24].

The activity indices per strength of the metakaolins produced from Adjozoumè and Adakplamè clays are therefore 97.5% and 96.26% respectively. These values are well above those required by ASTM C618. These values are also higher than the values obtained for metakaolins studied in the work of other researchers [5] [20] [21].

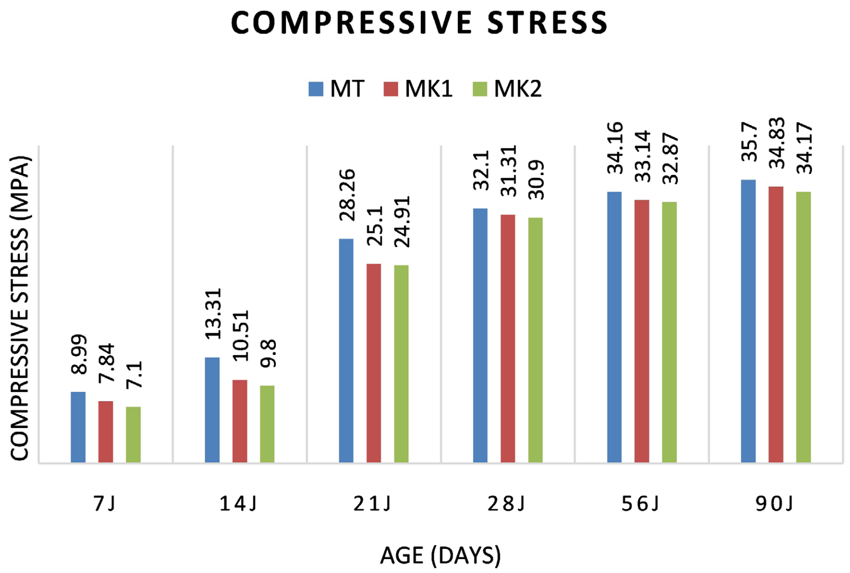

Figure 5. Compressive strength of mortars with 25% of substitution.

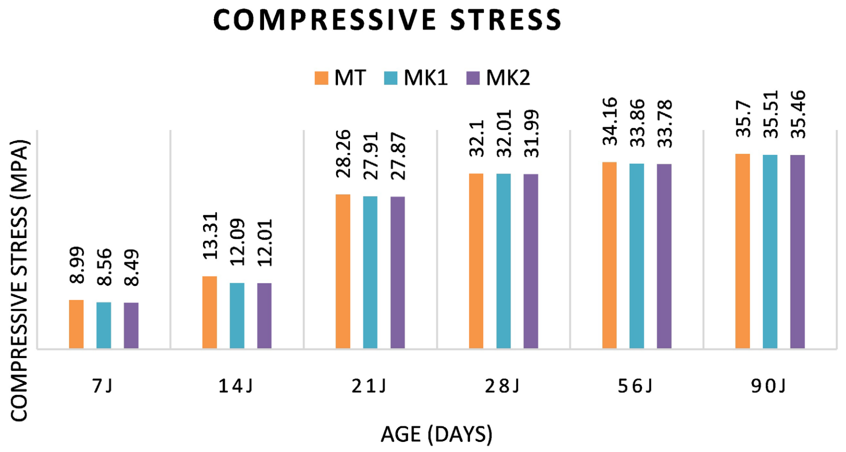

Figure 6. Compressive strength of mortars with 20% of substitution.

The drop in resistance when going from a substitution rate of 20% to 25% could be explained by a deficit of portlandite resulting from the hydration of the cement.

4. Conclusion

This study highlighted the pozzolanic potential of metakaolins produced from impure kaolins from the villages of Adjozoumè and Adakplamè in the Ketou region, by estimating their chemical compositions and determining pozzolanic activity indices. Chemical analysis showed that the two metakaolins produced have main oxide contents (SiO_2 + Al_2O_3 + Fe_2O_3) of 94.5% and 95%. Pozzolanic activity indices are 97.5% and 96.26% respectively. These values are well above those required by ASTM C618 demonstrating that these metakaolins are reactive and can substitute Portland cement. It should be noted that the substitution rate of cement by metakaolin used to measure pozzolanic activity indices is 25%. These metakaolins could therefore help produce mortars or concretes with higher performance than Portland cement mortars and concretes alone, provided the substitution rate remains below 25%. Metakaolins are produced by calcining kaolinitic clays in a laboratory furnace set at a rate of 10˚C per minute until a temperature of 700˚C is reached. Once this temperature has been reached, it is maintained for one hour. This method favors the reduction of calcination energy and is favorable for kaolinitic clays.

Conflicts of Interest

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

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