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Thermal Treatment of Kaolinite: Effects of Calcination Method on Chemical and Mineralogical Properties

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Abstract: The aim of this research work is to analyze the possibility of heat-treating kaolinite clays under economic production conditions. It presents the results of heat treatment of kaolinite clays from Adjozoumè in the Kétou region of the Republic of Benin. Three heat treatment methods were analyzed. The first consisted in calcining the clay in a ceramic enclosure placed on a charcoal hearth, where the temperature was controlled and limited to 600 °C for 3 h. The second technique consists of calcining the clay in a laboratory kiln. The temperature is set at a rate of 10 °C per minute up to 700 °C. Once this temperature has been reached, it is maintained for 1 h. Finally, the last technique involves re-treating the clay from technique 1 in the laboratory oven at 700 °C for 1 h. Each of the samples resulting from these three techniques underwent XRD (X-ray diffraction) testing at an angle of 2θ. Both chemical and mineralogical compositions were analyzed. The results showed that all three samples had a major oxide content higher than that required by ASTM C618 (92.5%, 94.5% and 96% respectively). However, the clay processed in the furnace had a residual kaolinite content of 10%, suggesting incomplete calcination. Technique 3 seems to be the best, since it ensures complete dehydroylation of the kaolinite and has the best metakaolinite content. The first technique remains valid for the production of metakaolinite for pozzolanic use, but its performance is not as good.

Key words: Clay, metakaolinite, heat-treating, pozzolanic, Benin, Ketou.

1. Introduction

The need to build cost-effective structures, combined with the urgent need to protect the environment, has led those involved in building and public works to look for alternative materials to those more expensive and less environmentally friendly. The use of metakaolins (heat-treated kaolinite clays) has demonstrated their potential as a partial substitute for clinker or directly for portland cement in concrete. Several calcination techniques for these clays have been proposed in the literature. These include the traditional method of calcination in kilns [1, 2] and new methods known as flash calcination [3-6]. These calcination techniques have been analyzed in works and the strengths and weaknesses of each method [7, 8]. However, access to flash calcination machines or rotary kilns remains problematic in Benin, especially given the need to keep production costs as low as possible. The work of Ayadji et al. [9] and Soglo et al. [10] has shown that charcoal kilns can be used to calcinate biomass in a quest to transform it into pozzolan. In this study, we analyze the feasibility of thermal treatment of kaolinite clay using an optimized charcoal kiln and compare its performance with other techniques.

2. Method

2.1 Materials

The material used in this work is kaolinite clay. It comes from the village of Adjouzoumè in the commune

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of Kétou in the Republic of Benin. To obtain a representative average sample, we took several samples at different points in the quarry. These samples were mixed to obtain the sample for laboratory testing.

2.2 Sample Preparation

Once in the laboratory, the samples are ground by hand and then in a corn mill. The ground material is then sieved using an 80-micron diameter sieve. Only the passings are used for heat treatment (calcination). The different transformation phases are shown in Fig. 2.

2.3 Calcination Methods

In order to analyze the impact of calcination technique on the mineralogical and chemical properties of metakaolins, we used three calcination techniques:

Technique1: kaolinite clay is calcined in a ceramic chamber on a temperature-controlled charcoal hearth. Calcination lasts 3 h.

Technique 2: crushed kaolinite clay is calcined in a laboratory furnace. The temperature is set at a rate of 10 °C per minute up to 700 °C. Once this temperature has been reached, it is maintained for 1 h.

Technique 3: the material obtained after calcination with technique 1 is sent to the laboratory furnace for further heat treatment at 700 °C for 1 h.

Calcination products cool slowly in air. The samples obtained from calcination using these three techniques are designated T1, T2 and T3 respectively. The raw kaolinite clay sample tested is designated T0.

2.4 Tests Carried Out

Visual observation is made of the materials treated with each technique. Clays calcined using each of the three techniques are subjected to XRD (X-ray diffraction) testing to identify mineralogical phases. From the results, an estimation was made to determine the mineralogical and chemical compositions of the calcined clays. The rate of dehydroxylation was also measured. XRD testing was also carried out on the ground or calcined clay to observe these parameters in the raw state.

3. Results and Discussion

3.1 Visual Appearance

The three samples T1, T2 and T3 have the same visual appearance. Indeed, calcined kaolinite clays, shown in Fig. 3, have a reddish color, indicating the possible presence of iron oxide.

3.2 Mineralogical Composition

The difractogram of raw kaolinite clay is shown in Fig. 1.

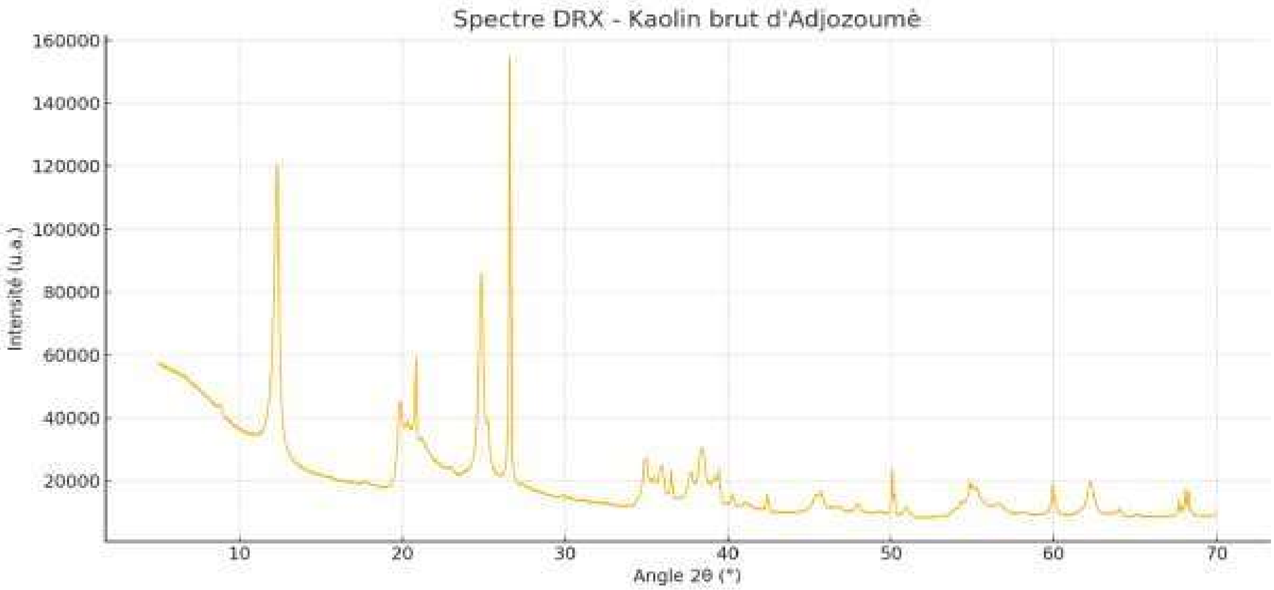

Fig. 1 Diffractogram of raw kaolin.

Fig. 2 Grinding of kaolinite clay.

Fig. 3 Visual appearance of calcined clay.

Table 1 Mineralogical composition T0 sample.

Mineral	Chemical formula	Estimated proportion (%)
Kaolinite	$Al_2Si_2O_5(OH)_4$	50
Quartz	SiO_2	45
Muscovite/illite	$KAl_2(AlSi_3O_{10})(OH)_2$	3
Hematite/goethite	Fe_2O_3/FeOOH	1
Feldspars (albite, orthoclase)	$NaAlSi_3O_8$/$KAlSi_3O_8$	1

Several intense peaks can be observed, notably around 12°, 20°, 26° and 36 °C, typical of kaolin clays. The mineralogical analysis of Adjozoumè raw kaolin derived from this diffractogram is presented in Table 1.

The raw kaolin sample from Adjozoumè is predominantly composed of kaolinite and quartz, with traces of micas (illite/muscovite). This composition is typical of well-leached lateritic clays from southern Benin. The absence of smectites or montmorillonite indicates non-swelling behavior. The kaolinite present in the material is highly crystalline, as demonstrated by the net basal peak at 12.30°. The material is marginally a clay. The net peak at 26.6° shows that the quartz observed is inert with free grains or inclusions. A few traces of ferric oxides with reddish kaolin staining are observed. Traces of feldspars (albite, orthoclase) between 20 and 25 °C are detectable, but of little significance. This composition of kaolin shows that it is suitable for ceramic uses, but also as a source of alumina and silica for pozzolanic formulations after transformation into metakaolinite through calcination.

The diffractogram of the sample calcined using technique 1 is shown in Fig. 4.

The mineralogical analysis of the Adjozoumè kaolinite clay calcined under the conditions of technique 1 is presented in Table 2.

The diffractogram of the sample calcined using technique 1 shows peaks similar to those of raw kaolin but does not possess the same characteristics. A strong peak at 26.6° shows the presence of unchanged quartz with a stable structure. A visible amorphous halo of dehydroxylated kaolinite shows the presence of amorphous metakolinite. Peaks at around 12° and 20° show the presence of residual kaolinite following incomplete or heterogeneous calcination. A few amorphous impurities consisting of vitrified inclusions and partially disorganized oxides are also present.

The diffractogram of the sample calcined using technique 2 is shown in Fig. 5.

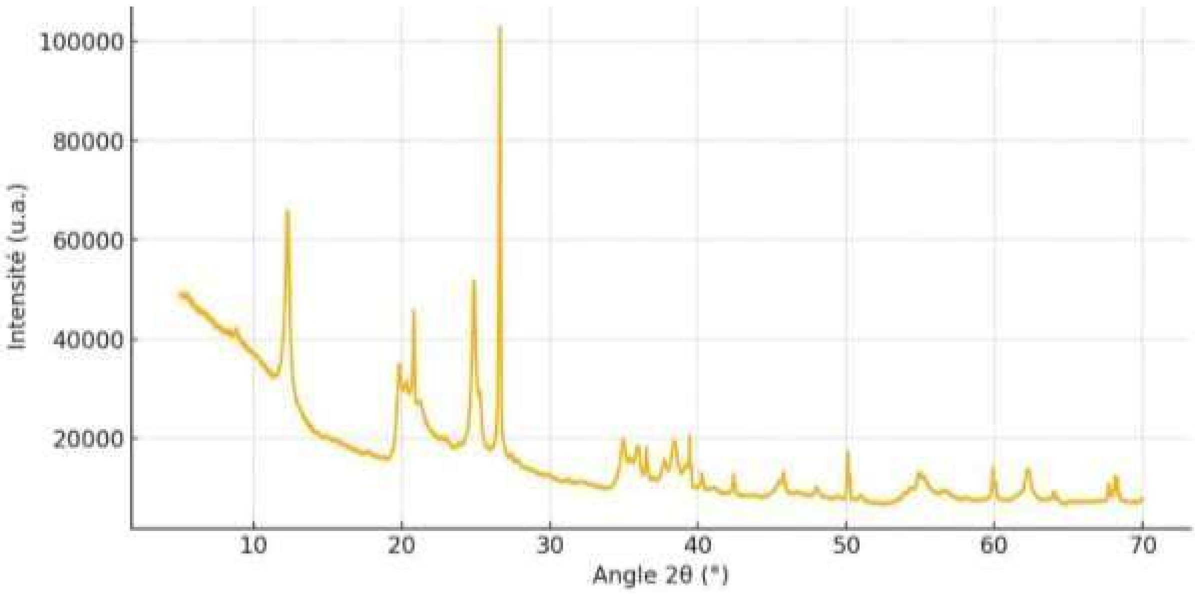

Fig. 4 Diffractogram of sample T1.

Table 2 Mineralogical composition T1 sample.

Phase or mineral	Chemical formula	Estimated proportion (%)
Quartz	SiO_2	40
Amorphous metakaolinite	$Al_2Si_2O_7$ (non-crystalline)	45
Residual kaolinite	$Al_2Si_2O_5(OH)_4$	10
Amorphous impurities/oxides	Fe_2O_3, molten traces	5

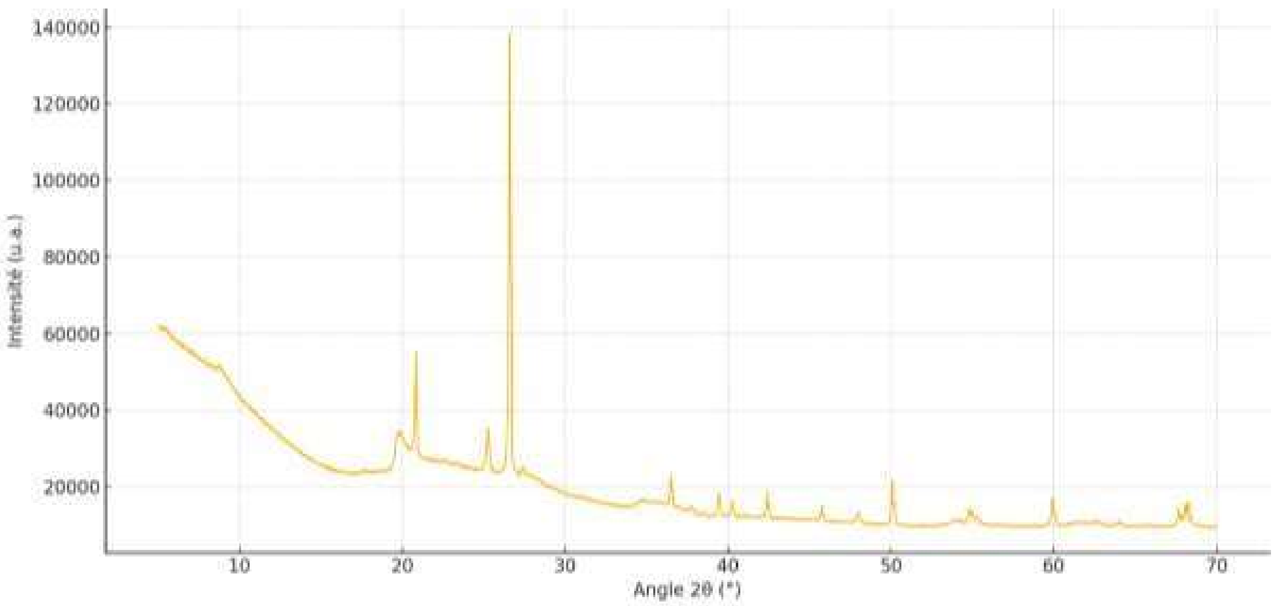

Fig. 5 Diffractogram of sample T2.

This XRD of Adjozoumè kaolin calcined using technique 2 shows:

- a clear reduction in kaolinite peaks, particularly at 12.2°,
- persistence of quartz (26.6°),
- a more diffuse profile, indicating amorphous transformation (metakaolinite formation).

Mineralogical analysis yielded Table 3:

Table 3 Mineralogical composition sample T2.

Mineral or phase	Chemical formula	Estimated proportion (%)
Quartz	SiO_2	45
Amorphous metakaolinite	$Al_2Si_2O_7$ (non-crystalline)	50
Residual kaolinite	$Al_2Si_2O_5(OH)_4$	3
Miscellaneous amorphous impurities	Fe_2O_3, vitreous flux	2

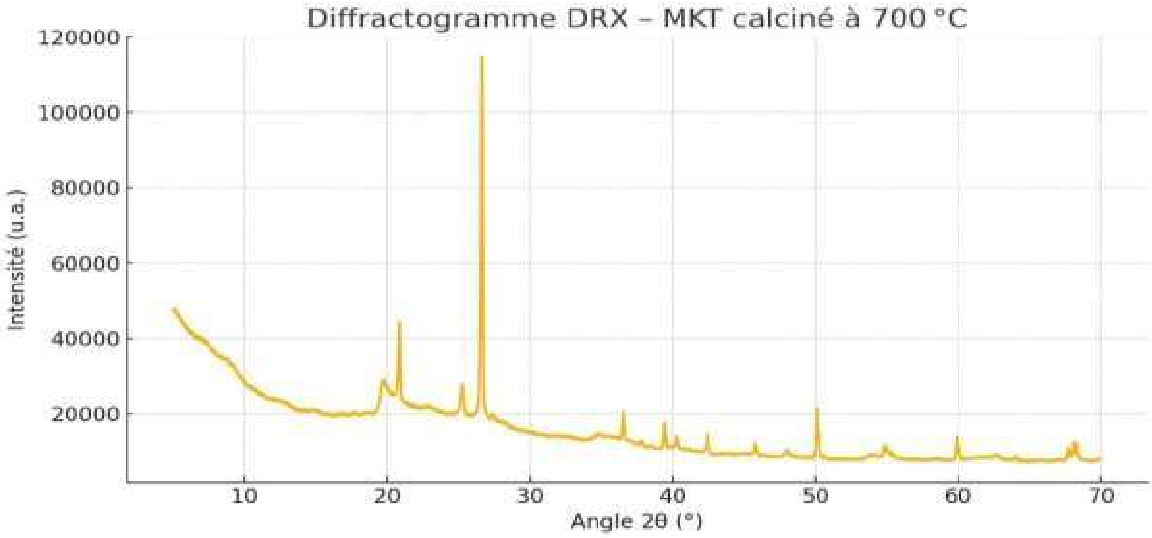

Fig. 6 Diffractogram of sample T3.

Table 4 Mineralogical composition sample T3.

Mineral phase	Chemical formula	Fixed proportion (%)
Amorphous metakaolinite	$Al_2Si_2O_7$	54
Quartz	SiO_2	44
Residual kaolinite	$Al_2Si_2O_5(OH)_4$	1
Other (amorphous oxides)	Fe_2O_3, trace feldspars	1

Sample T2 shows a diffuse halo between 5° and 6.3° showing an amorphous formation from kaolinite. However, a few traces can be seen at 20.8° showing residual kaolinite. Dehydroylation is partial, showing almost complete calcination. A clear peak at 26.6° shows the presence of quartz in the preserved inert crystalline phase. Traces of iron oxides or unidentifiable inclusions are observed.

The diffractogram of sample T3 is shown in Fig. 6 and its mineralogical analysis is presented in Table 4.

This processed sample shows a dominant amorphous phase resulting from the complete dehydroxylation of kaolinite. This amorphous phase is 4% higher than that observed in the sample calcined with technique 3. A weak residual peak at 20.8° shows the presence of residual kaolinite in this sample. However, the residual kaolinite content is 3 times lower than in sample T2. This sample also contains vitrified impurities composed of non-detectable oxides.

3.3 Chemical Composition of Metakaolin

Estimated chemical compositions of the three samples T1, T2 and T3 are presented in Table 5.

These compositions show that the three heat-treated samples are essentially composed of silica and alumina oxides. These oxides are derived from reactive metakolinite and inert quartz. The presence of iron oxide (approx. 2.5%) in all three samples justifies the reddish color observed visually. The zero level of structural water shows the complete dehydroxylation of samples T2 and T3. Dehydrohylation is partial, but has reached a value of 94.7%.

Table 5 Chemical composition of samples T1, T2 and T3.

Oxide	Formula	Fixed proportion (%)		
		Technique 1	Technique 2	Technique 3
Silica	SiO_2	54.0	55.0	56.0
Alumina	Al_2O_3	36.0	37.0	38.0
Iron oxide	Fe_2O_3	2.5	2.5	2.0
Potash	K_2O	1.0	1.0	1.0
Soda	Na_2O	0.5	0.5	0.5
Lime	CaO	0.4	0.3	0.3
Magnesia	MgO	0.3	0.2	0.2
Structural water	-	5.3	0.0	0.0

According to ASTM C618 [11], all three samples are pozzolanic from a chemical point of view, as the sum of the main oxides is well above the required 70%. These samples are also much better than the metakaolins from kaolinite clays presented in the work of Abdul Razack et al. [12]. These compositions are also much better than those presented in the work of Refs. [13-15].

We note that:

- The raw kaolin from adjozoumè is highly crystalline, and shows no pozzolanic character.
- Sample T1, obtained by calcination on a charcoal hearth for 3 h, shows a disorganized structure, but still retains residual traces of kaolinite (peak at ~12.3° visible), suggesting incomplete or heterogeneous calcination.
- When calcined at 700 °C in a kiln (sample T2), adjozoumè kaolin shows clear structural disorganization, with disappearance of the basal kaolinite peak (12.2°), indicating the formation of amorphous metakaolinite.
- Sample T3 is the most transformed: the kaolinite has completely disappeared, an amorphous halo has developed, and the structure is largely disordered, confirming the complete formation of highly reactive metakaolinite**.**

4. Conclusion

The following conclusions can be drawn from this study:

- The Adjozoumè kaolinite clay is an impure kaolin containing quartz and iron minerals.
- Calcining this kaolin in a ceramic chamber on a charcoal hearth for 3 h at a temperature of 600 °C transforms 90% of the kaolinite into metakaolinite suitable for pozzolanic use. The presence of 10% residual kaolinite is due to heterogeneous calcination.
- Calcining this clay in a 700 °C kiln under the conditions described above improves the transformation of kaolinite into metakaolinite (from 10% residual kaolinite to 3%).
- Technique 3 seems to be the best, enabling complete dehydroxylation with the transformation of residual kaolinite from technique 1, from 10% to 1%.
- In all cases, the quantities of main oxides did not vary greatly. A variation of 2% between T1 and T3 is observed.
- Calcination of kaolinite clay would be interesting in an economical production context, provided the calcination time is increased (to 5 h, for example), but its performance will remain less good.

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