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ISSN - 2277 - 0593  
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Journal of Natural  
Sciences, Engineer-  
ing and Technology

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## EVALUATION OF A CLAY MINERAL DEPOSIT IN ABEOKUTA, SOUTH-WEST NIGERIA

\*<sup>1</sup>O.S. OLOKODE, <sup>1</sup>P.O AIYEDUN, <sup>1</sup>S.I. KUYE, <sup>1</sup>N.O ADEKUNLE  
AND <sup>2</sup>W.E. LEE

\*<sup>1</sup>Mechanical Engineering Department, College of Engineering,  
University of Agriculture, Abeokuta.

<sup>2</sup>Materials Department, Imperial College, London, SW7.AZ.

\*Corresponding author: engr\_olokode@yahoo.com Tel:+2348169677160

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### ABSTRACT

Clay, in regard to its origin is unique. The kaolin of the mine of Ajebo has a hydrothermal geological formation. It presents itself a fragile and crumbly material, of whitish colour. Therefore, the objective of this paper was to evaluate the clay mineral of Ajebo deposit, its microstructure using scanning electron microscopy, X-ray diffraction and thermal analysis (DSC/TG). The results showed that Ajebo clay belongs to the kaolin group (Kaolinite, nacrite, dickite, and halloysite). Elementary analysis showed the mineral to be composed mainly of aluminium, oxygen and silicon, with a low content of iron. The dehydrated state of the mineral was confirmed by XRD analysis; the basal spacing reflections indicate a sharp peak at 7Å of d001 and the absence of a peak at 10Å, which is indicative of hydrated kaolin. The SEM/SE studies indicated that Ajebo clay mineral particles are smaller than 10µm, forming agglomerates.

**Keywords:** Kaolin, Evaluation, Thermogravimetric, X-ray Diffraction, Differential scanning calorimetric, Ajebo.

### INTRODUCTION

The exploitation of the white clay by the Chinese in the industry of ceramics goes back to the first millennium (Grim, 1968). Various layers of this ore were thereafter discovered. Four types of minerals were distinguished: Kaolinite most abundant, the dickite (1888), nacrite (1807), halloysite (1826) which belongs to the kaolin group. These minerals of the group kaolin have mainly three origins (Caillere *et al.*, 1982) by heritage where the inherited minerals are obtained by deterioration and transport, also by degradation major transformations (Hurst and Kunkle, 1985) These are 1.1 phyllosilicates, characterised by a  $Al_2Si_2O_5$

$(OH)_4$ . Kaolinite is interesting as a mineral precursor due to its unique asymmetry layered structure with  $(SiO)_6$  macroning on one side and aluminium on the other side. This asymmetry creates large cohesive energy (Bailey, 1988a).

Their chemical composition is almost uniform 46.54% of  $SiO_2$ , 39.50% of  $Al_2O_3$ , 13.96% of  $H_2O$ ; corresponding to the general formula  $Al_2Si_2O_5(OH)_4.2H_2O$ . Hoffman *et al.* (1934), Ross and Kerr (1934) established its chemical composition for hydrated form  $Al_2Si_2O_5(OH)_4.2H_2O$  and the basal distance d001 of this ore increases by 7Å. To differentiate these two situations, the

metahalloysite term was introduced Mehmel, (1935) for the dehydrated form and by using the halloysite term for the hydrated form (Churchman *et al.*, 1972). This nomenclature was accepted by Keller and John (1976), Brindley (1980). In this present research, our investigation will concern the chemical composition in order to evaluate the concentrations out of silica, alumina, water and the elements such as: potassium, sodium, iron, phosphorus and manganese. The thermal gravimetric analysis makes it possible to characterise the type of clay (Caillere *et al.*, 1982). X ray diffraction which is a means of identification of crystallographic texture is studied; the inductively coupled plasma (ICP). Chemical composition by wt% of constituent oxides present. Scanning electron microscopy (SEM) is also put at contribution to know the morphology of the kaolin.

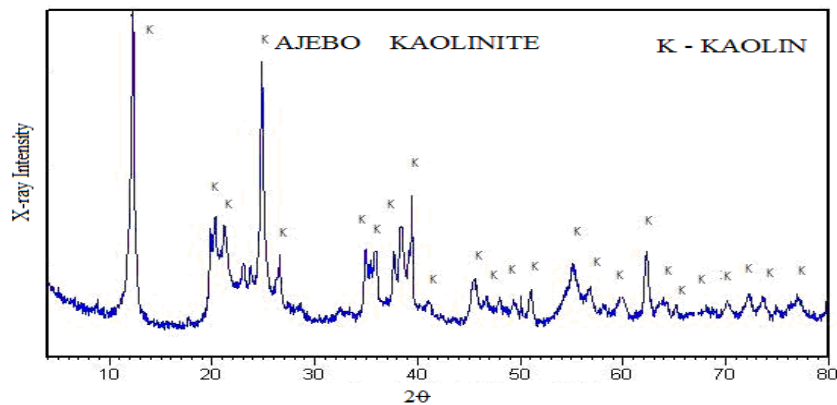
## MATERIALS AND METHODS

The kaolin used as clay in this study was obtained from Ajebo in Abeokuta, south-west Nigeria. The type of kaolin is much rarer than other clay minerals (Papke, 1971). Details about physiochemical properties were determined by inductively coupled plasma (ICP), X-ray diffraction, thermogravimetric and scanning electron microscopy. The chemical compositions of the kaolin was found to be as presented Table 1.

X-ray diffraction analysis (PW 1700, Phillips instrument automated powder diffractometer using  $CuK_{\alpha}$  radiation at 40Kv/40mA from  $5^{\circ} < 2\theta < 85^{\circ}$ ) with steps 0.04 at the counting time of 2 seconds was used to determine the mineral present in Ajebo as kaolin, at peaks  $12.31^{\circ} 2\theta$  (7.19Å), and  $24.85^{\circ} 2\theta$  (3.58 Å) respectively.

**Table 1: Chemical Composition of Ajebo clay deposit from Abeokuta, South-West, Nigeria**

Raw Materials	Content
SiO <sub>2</sub>	46.40
TiO <sub>2</sub>	1.69
Al <sub>2</sub> O <sub>3</sub>	34.00
Fe <sub>2</sub> O <sub>3</sub>	2.49
MnO	0.00
MgO	0.04
CaO	0.02
Na <sub>2</sub> O	0.03
K <sub>2</sub> O	0.08
P <sub>2</sub> O <sub>5</sub>	0.04
LOI	17.70

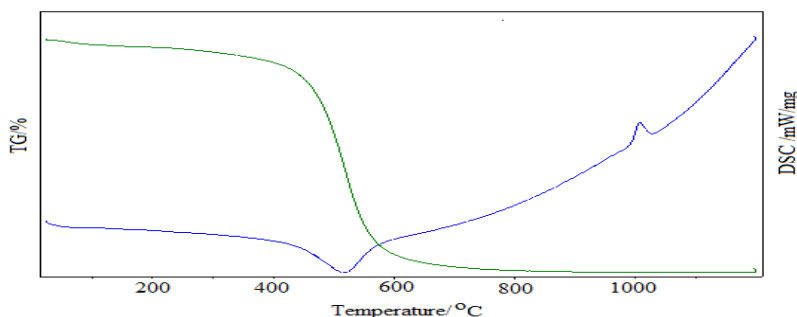


**Figure 1: - X-ray diffractometer traces of the untreated Ajebo raw clay**

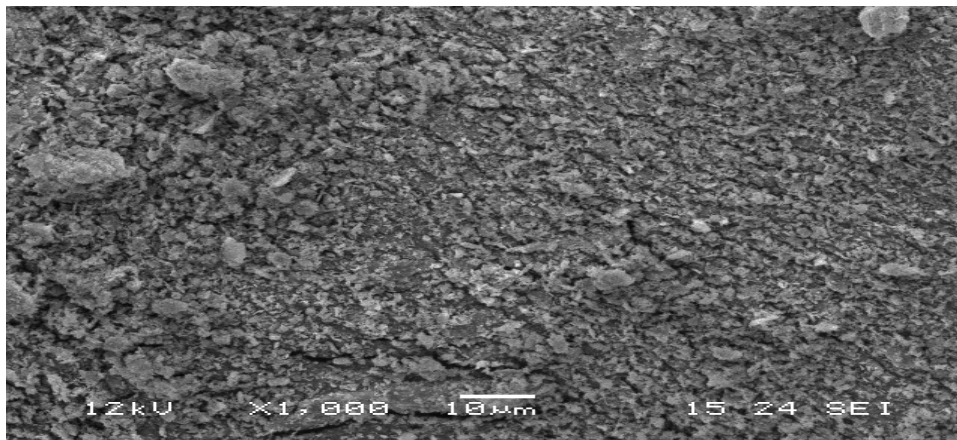
Thermogravimetric and differential scanning calorimetric analysis (TG/DSC) using Netzsch. STA 449 at a heating rate of 10°C/min. up till 1200°C was used. The endothermic event at ~530°C is due to the organic burn off, and probably consists of hygroscopic water release. This event was followed by a wt % loss in the clay sample, as observed in the TG/DSC trace. The weight loss was due to removal of adsorbed water at ~100 °C. Organic matter burn-off may be responsible for the upward slope on the thermogravimetric analysis (TGA) trace. At 800-993°C, the crystallisation of a spinel structure  $Al_2O_3$  was formed, followed by the release of free silica, which assist in the

liquid formation leading to pronounced shrinkage ~2.5% of the clay body temperature. Exothermic reaction clearly observable from ~993-1044°C. This reaction may account for the beginning of the growth of crystallisation of mullite or  $Al_2O_3$  spinel.

The microstructure of the fired materials was analysed by scanning electron microscope, (SEM-JSM 5000) equipped with an energy dispersion spectroscopy (EDS).



**Fig 2: Thermogravimetric analysis/Differential scanning calorimetric (TG/DSC) of Ajebo raw clay in Abeokuta, Ogun-State**



**Fig. 3: Overall morphology of Ajebo clay deposit in Nigeria**

## RESULTS AND DISCUSSION

The overall morphology of the Ajebo clay deposit showed that all particles are smaller than 10  $\mu\text{m}$ , forming agglomerates is presented in Figure 3.

The loss on ignition corresponds to water vapour from dehydroxylation reactions in the clay minerals, carbonate decomposition into  $\text{CO}_2$  and oxides as well as burning out of organic matter. From the chemical formula, a pure kaolinite clay sample could lose 13.9wt% of its mass on ignition due to dehydroxylation with water release. In the case of Ajebo sample clay it has 17.7wt% loss on ignition of which is 3.8wt% higher than 13.9wt%. This is due to the fact that the Ajebo clay was not treated (either by sedimentation or deflocculated). Ajebo clay is rich in alumina and silica as in Table 1 whereas manganese oxide is absent. Its appearance is whitish/greyish in colour which appears to be pure kaolin mineral. The XRD analyses clearly showed kaolin as the main clay minerals present in Ajebo clay as shown in Figure 1.

The thermal gravimetric analysis/Differential scanning calorimetric (TGA/

DSC) trace of the untreated Ajebo clay is shown in Figure 2. The endothermic event at  $\sim 530^\circ\text{C}$  is due to the organic burn off, and probably consists of hygroscopic water release. This event was followed by a wt % loss in the clay sample, as observed in the TG/DSC trace. The weight loss was due to removal of adsorbed water at  $\sim 100^\circ\text{C}$ . Organic matter burn-off may be responsible for the upward slope on the thermogravimetric analysis (TGA) trace.

At  $800\text{-}993^\circ\text{C}$ , the crystallisation of a spinel structure  $\text{Al}_2\text{O}_3$  was formed, followed by the release of free silica, which assist in the liquid formation leading to pronounced shrinkage  $\sim 2.5\%$  of the clay body temperature.

Exothermic reaction clearly observable from  $\sim 993\text{-}1044^\circ\text{C}$ . This reaction may account for the beginning of the growth of crystallisation of mullite or  $\text{Al}_2\text{O}_3$  spinel.

## CONCLUSION

The research employs the inductively coupled plasma analytical technique (ICP) for the composition and structure of the constituents of the clay sample. The chemical composition of the sample gave percentages

of silica and of aluminium respectively 46.4 and 34.0%, the loss on ignition is 17.7%. This values approach those found in literature.

The curve of TGA presents asymmetric endothermic peak at 530°C, characteristic of metahalloysite. The TGA confirmed the proceeding results with two losses; the first is due to the removal of molecular water adsorbed on the external surface and the second is attributed to the loss of dehydroxylation step of kaolin.

X-ray diffraction revealed principal peak at  $12.31^\circ 2\theta$  (7.19 Å) and a peak at  $24.85^\circ 2\theta$  (3.58 Å). The peak (d001) of maximum asymmetry gives a measurement of the degree of the crystalline disorder of this clay. SEM show kaolin used is largely micro tubular and relatively uniform with occasional aggregates. This result confirmed the presence of kaolin mineral from Abeokuta, South West, Nigeria.

### ACKNOWLEDGEMENTS

This research was supported by Commonwealth scholarship programme 2006/07 at Imperial College, London. The authors are gratefully acknowledge to Materials Department for providing the equipment used. The authors thank to Mr. Sweeney Richard and Dr. Mahmood of Materials Department for their experimental assistance to Dr. Olokode O.S.

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(Manuscript received: 28th June, 2010; accepted:, 6th December, 2010).