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Research Paper

THERMAL BEHAVIOR OF CLAY FROM AKOKO SOUTH-WESTERN NIGERIA

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The aim of this work is to determine the suitability of Akoko clay for use in the production of cement. In this light, the clay fractions from four locations were used. The clay fractions were characterized thermally and using other spectroscopic tools. The obtained amorphous materials were also subjected to X-ray diffraction, Raman spectroscopy and Transmission electron microscopy analysis. From the TGA curve, all clay samples from different locations fall within the standard for commercial kaolinite clay. The analysis generally showed that the metakaolin from Akoko clay is highly ordered thermally stable and viable for commercial use.

Keywords: Clay, Akoko, Thermal analysis, Cement

INTRODUCTION

Akoko is a large Northeast Yoruba settlement in Yorubaland, the area spans from Ondo state to Edo state in southwest Nigeria. Akoko takes a large percentage of the local governments in Ondo state.Akoko comprises about 40 towns, predominantly situated in rocky areas of Ondo state. The rocky terrain nevertheless, may have helped the region to become a melting pot of sorts with different cultures coming from the north, eastern and southern Yoruba towns and beyond. Akoko became one of the few Yoruba clans with no distinctive local dialect of their own.Ajowa for example as the prince and jewel of Akoko land speaks eight distinct dialects. Major Akoko settlements include Ikare, Oka, Oba, Ikun, Arigidi, Ogbagi, Okeagbe, Ikaram, Ibaram, Iyani, Akungba, Erusu, Ajowa, Akunu, Gedegede, Isua, Auga, Ikakumo, Supare, Epinmi, Ipe, Ifira, Ise, Iboropa, Irun, Afin, Igashi, Sosan, Ipesi, Etioro, Ayegunle and Oyin. In addition to this group, there are several other autonomous communities (Akoko, 2015).

One of the pre-occupation of some settlers in this region is poultry, owning to the large deposits of kaolinite clay scattered all over the region.Kaolinites have been found to be of immense application in alum and alumina production, ceramics, zeolite catalysts, adsorbents for heavy metals and catalysis (Ahmed, 1986; Aderemi, 2000, 2001; Edomwonyi,

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2009, 2010) and as geopolymers. Most of these processes involve the thermal treatment of the kaolinite which leads to a transformation of its structures with the removal of the bonded water.

In 1970s Davidovits stated that, geopolymers are a new class of three dimensional silico-aluminate materials (Cioffi, 2003; Davidovits, 1988). Products arising from geopolymer reactions have certain advantages: high mechanical properties, resistance to high temperature and chemical agents, low shrinkage, etc. (Yunsheng, 2010). The synthesis of geopolymers can be carried out by using the sol-gel method. The raw materials commonly used are those rich in alumino-silicate such as natural pozzolan, fly ash, blast furnace slag, and calcined kaolinite clays.

Metakaolin is essentially an anhydrous alumino-silicate obtained from the calcination of kaolinite clays. Due to its disordered structure, it possesses a huge reactive potential when it is activated in alkali solution (Davidovitts, 1988; 1999; Deepak, 1997; Palomo, 1999; Granizo, 2002) or calcium hydroxide in presence of water (Murat, 1983; Murat and Camel, 1983; Ambroise, 1985). Changes in chemical or physical properties of material as a function of temperature in a controlled atmosphere may be determined by thermal analysis (Nadiye, 1975) which covers changes in energy, weight, crystal dimension and evolved volatiles. This enables clays to be classified according to their heating pattern.

The objective of this work is to determine the thermal behavior of claysamples (raw and calcined-clay) collected from different location in Akoko land. For this study, Thermogravimetric Analysis (TGA) is used.

The samples were all subjected to thermal analysis in air and in inert conditions.

Materials and Experimental Methods

The clay samples were collected from different locations in Akoko land. To make laboratory-sized samples, the lumps were mixed and crushed together so that a representative sample can be used for analysis. The crashed clay was mixed with de-ionised water and stirred with a stirrer for three hr .The suspension was left to settle for 48 h (two days). The slurry was blunged and screened through a 75 µm mesh sieve and then allowed to settle.The water was siphoned off and the clay was dried for further use.

After the treatment above, the powdered clay was calcined at 800°C for 3 h in an electric furnace. During the calcination the structure of kaolinite clay was degraded and two molecules of waters released.

$$Si_2O_5(OH)_4Al_2 \rightarrow Al_2Si_2O_7 + 2H_2O$$

The two treated samples resulting from the processes above (from different locations) were subjected to thermal analysis.

Thermo-gravimetric-mass spectroscopy (TG-MS)

TGA is a technique in which the weight of a given sample is monitored continuously as a function of time and/or temperature, while under flowing air or nitrogen. TGA was used to probe the decomposition route of the sample and the thermal stability of the clay. The heating rate was set at 283 K/min from 298 to 1273 K in air stream (100ml/min).

RESULTS AND DISCUSSION

TGA measures the amount of weight change of a material, either as a function of increasing

Table 1: TGA Data for Clay and Calcined Clay

CODE	AIR	ARGON
C1	Step -5.2830 %	Step -2.7936 %
	-1.5860mg	-1.0440mg
	Step -0.5193 %	Step -0.7414%
	-0.1559mg	-0.2771mg
	Step -5.3622 %	Step -5.1931 %
	-1.6097mg	-1.9407mg
C2	Step -1.5863%	Step -0.8989 %
	-0.5032mg	-0.2955mg
		Step -0.1812 %
		-5.96E-02
R1	Step -3.1622%	Step -3.0206%
	-0.8788mg	-1.0246mg
	Step -1.2629%	Step -1.2298%
	-0.3510mg	-0.4171mg
	Step -8.7944%	Step -8.8874%
	-2.4440mg	-3.0146mg
R2	Step -0.7047%	Step -0.4275 %
	-0.2116mg	-0.1404mg
S1	Step -5.7784 %	Step -5.4734 %
	-1.8907 mg	-1.5030 mg
	Step -1.3001 %	Step -0.8725 %
	-0.4254 mg	-0.2396 mg
	Step -6.6965 %	Step -6.5474 %
	-2.1911 mg	-1.7979 mg
S2	Step -0.9285 %	Step -0.6013 %
	-0.2915 mg	-0.1638 mg
	Step -0.1799 %	Step -0.1101 %
	-56.4565e-03 mg	-30.0026x03 mg
	Step -0.3404 %	

Table 1 (Cont.)

CODE	AIR	ARGON
	-0.1069 mg	
	Step -0.5146 %	
	-0.1615 mg	
M1	Step -4.8560 %	Step -4.7669 %
	-1.5709 mg	-1.7814 mg
	Step -0.9664 %	Step -0.8216 %
	-0.3126 mg	-0.3070 mg
	Step -6.5204 %	Step -6.1855 %
	-2.1093 mg	-2.3115 mg
M2	Step -1.3154 %	Step -0.5374 %
	-0.5217 mg	-0.2170 mg
		Step -0.1189 %
		-48.0003x03 mg

Note: C1, R1, S1 and M1 are treated clay from various locations in Akoko not disclosed because I do not have the permission of the communities to do so.C2.R2; S2 and M2 are the calcined clay from different locations.

temperature, or isothermally as a function of time, in an atmosphere of nitrogen, helium, air, other gas, or in vacuum.

Table 2: Percentage (%) Decomposition of Clay and Calcined Clay

Sample Code	Air	Argon
C1	11.1645	8.7257
C2	1.5863	1.0801
R1	13.2195	13.1378
R2	0.7047	0.4275
S1	13.7750	12.8933
S2	1.9634	0.7114
M1	12.3428	11.7740
M2	1.3154	0.6563

Figure 1: Laser Raman Spectrum for Clay

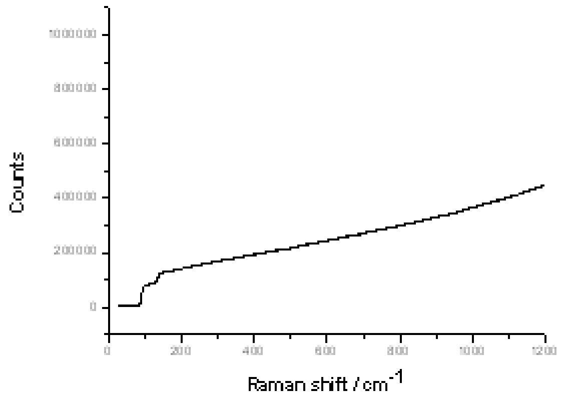


Figure 2: Laser Raman Spectrum for Calcined Clay

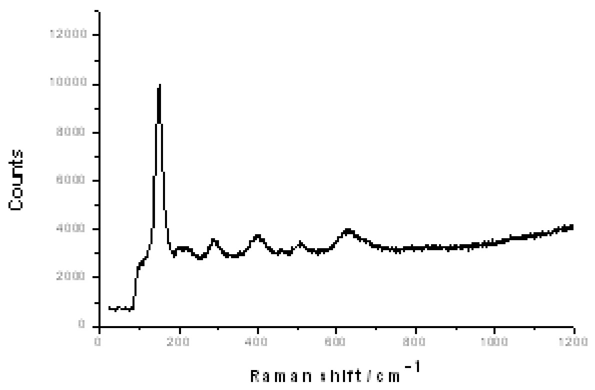
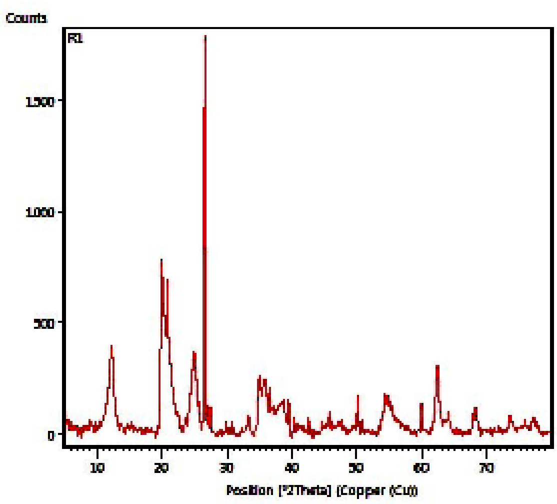
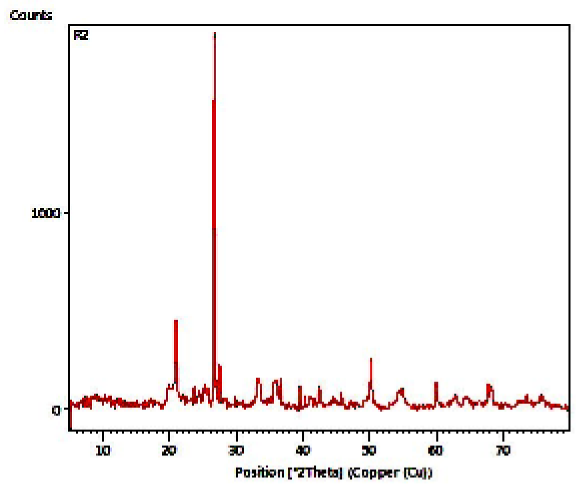


Figure 3: X-ray Diffraction Pattern for Clay



The TG curves of the claysC1-M1 and C2-M2 have the same appearance and exhibit the thermal behaviour of clays (Mackenzie, 1957; Bouaziz, 1972). From the thermogravimetric curve, the samples exhibits different decompositional loss

Figure 4: X-ray Diffraction Pattern for Calcined Clay



summarized in Table 2. Generally, the curves shows peaks between 30-100^{0}C and between 300-500^{o}C normally associated with loss of surface water and dehydroxylation of the samples respectively except for all calcined clay samples under air environment.

The X-ray diffractions of powders from clay show the presence of a high broad 2θ (18-25^o) peak band. The xrd of the calcined clay powder do not show peak but the presence of a halo peak band with intensity lower than the previously observed for uncalcined clay.All the X-ray diffractions recorded also show the presence of the characteristic peaks of quartz and feldspar in the samples.

Figures 5 and 6 show the Transmission electron microscopic image of the raw and calcined clay. On the micrographs of calcined clay,the phases are trapped in a whitish phase far more than the uncalcined clay. The reactivity

Figure 5: Transmission Electron Microscopic Image of Clay

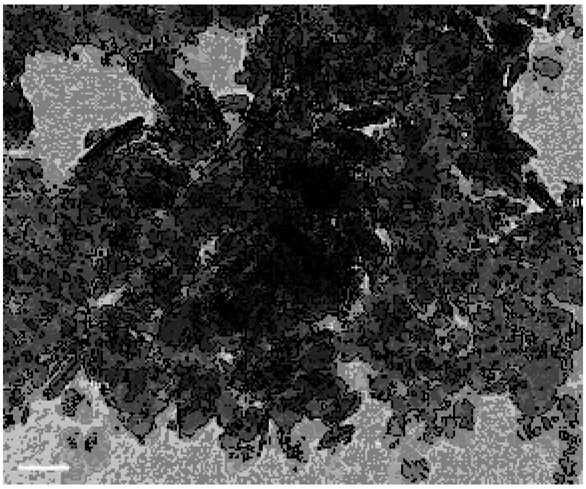
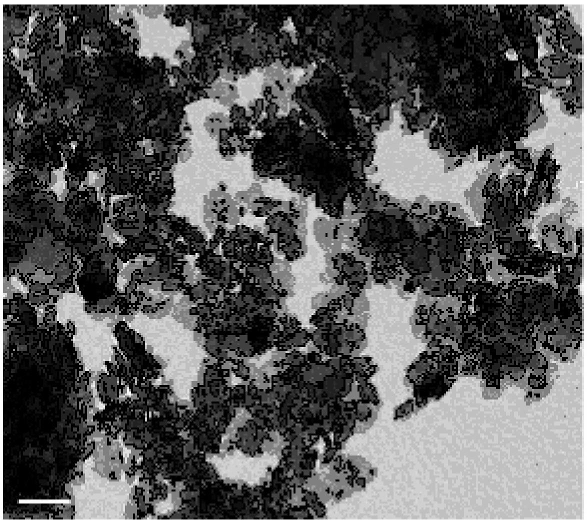


Figure 6: Transmission Electron Microscopic Image of Calcined Clay



of the material is also known to be enhanced as it converts the crystalline clay to the reactive amorphous material which can be easily dealuminated by inorganic acids such as hydrochloric acid, Nitric acid or sulfuric acid to yield aluminium sulfate which can be used as an additives in cement.

CONCLUSION

In this research work, samples were collected from different part of Akoko land, calcined and subjected to thermal treatment in vacuum and inert environment. From our findings, decompositional loss of water occur more in air than under inert condition. Also, the calcined clay from all locations showed little loss of water. The determination of other characteristics such as Raman, x-ray diffraction and Transmission electron microscopy reviles that the calcined clay is made up of metakaolinite which is an alumino-silicate material good for making quality cement.

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