

# Characterisation of the Nigerian Kankara Kaolinite Clay Particulates for Automobile Friction Lining Material Development

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## Abstract

The exact batch composition of given automobile brake pads are closely guarded secrets from their various manufacturers. Ceramics have remained some of the recent friction lining materials used in brake pad material development within the last two decades. Nigerian Kankara kaolinite clay was characteristically investigated for its availability and suitability as particulated reinforcement filler; heat and wear resistance in this researchwork. Recently, high efficient brake pads has been found; but with the expensive carbon fibers and other carbon silicon carbide composites. Today all these compounds are still highly expensive materials for commercial vehicles, except for sport racing and other very special cars. Functions of carbon fibers were therefore substituted with clay for examinations and expectations in this research. Alternatively, Asbestos is also considered as cheap materials in terms of production cost and thermal efficiency. Legally, it is still commercially accepted that automobile brake pad could contain asbestos. However clay being healthy, cheaper, and abundantly available, environmentally friendly for production, and capable of performing all the required functions of friction living material is scientifically investigated in this researchwork. This study is to develop an automobile brake pad without harshness, noise, vibration and with long life pad wear for vehicle users.

**Keywords:** Clay; Sinterability, Functions; Brake Pad; Development.

## INTRODUCTION

Mostly, various Nigerian clays have been investigated as explained by Aderiye, (2005). But most of these viable commercial clay deposits such as Kankara, Giro, Ray Field, Sabon-Gida, Isan and Ise-Ekiti, Warran, and Onibode to mention few as reported have been examined by Ahmed, (1986). The scope of investigations of these clays have not gone beyond exploration and exploitation. Or atimes, the characterization of these clay materials have been studied. (Aderibigbe, (1984). Also some of the Nigerian clay depositional locations with their various chemical compositions and refractoriness were presented in the works of Aniyi, (1985) and Shittu, (1998) respectively. Many researchers did not reveal the industrial uses of these clay deposits after their chemical physical and mineralogical compositions. Majority of these Nigerian clays have only been identified as kaolin ; but without detailed scientific analyses and its commercial uses. Yet many of the deposits have been leased out or eventually sold out cheaply either for building purposes; mining activities or for road constructions(Ahmed, 1986).

Asbestos was widely used in pads for its heat resistance. But due to health and environmental risks, it has been fairly replaced now. Alternatively, it is perfectly legal for brake pads to contain asbestos so that the brake pad material could still be inexpensive instead of using most expensive but better performing carbon fibers and other reinforced silicon carbide composites (RSCC). Generally, all these ceramic composites last longer than semi-metallic pads. However, ceramic brake pads are made up from ceramic fibers, filler materials and binding agents. Surprisingly, ceramic pads are not made from the traditional method of making fine china food plates or tea cups. The latest or newer ceramic brake pads are made of exotic materials like ceramic fibers and other composite plastics. Moreso, ceramic brake pads should not be confused with those made of carbon fiber and associates. They are extremely expensive for the current scope of this research.

However, clay is healthy, cheaper, available and environmentally friendly. Therefore, its compounds could be cheaply employed as friction lining material if researched upon. Ceramic materials and compounds come in varieties of heating refractories and insulating materials. The study of Kankara kaolinite clay sinterability should indicate whether it could be used as a particulated reinforcement filler material in an automobile brake pad development. Hypothetically, is the Kankara clay capable of performing all the five principal functions required for automobile brake pad? Characteristically, Nigerian Kankara clay would be therefore tested as a filler material, binding material, abrasive material, increase braking performance; reduce wear resistance and harshness for vehicle rotors and drums.

## MATERIALS AND METHOD.

### NIGERIAN KANKARA KAOLINITE CLAY PURIFICATION

Theoretically, Nigerian clay material if obtained directly from the deposits requires purification treatment

somehow. Basically, this is to separate the unwanted impurities from the required clay. Sedimentation process is commonly used in such purification process. (Wills, 1992). Both the aims and objectives are directed at subtracting certain unwanted minerals from the needed mineral specifically on the particle basis. Fundamentally, no two minerals are likely to have exactly the same size distribution. (Bridges, 1979). Therefore, sedimentation principle could be effectively applied for separating clay mineral from unwanted sand, quartz and silt as fine clay fractions are required as particulate for automotive friction lining composite material. However, wet sieving method is another effective alternative process for clay mineral purification. Wet sieving technically could separate various unwanted impurities on a size or grain particle basis also. In this research work, wet sieving method is preferred to the sedimentation technique because it is cheaper ; economical and much faster to accomplished for experimental projects and research works.

In this research, exploration of the Nigerian Kankara clay deposit was made. Clay samples were exploited from the deposit at eight cardinal areas. Thereafter, the clay samples were beneficiated with 45micron sieve. The purification process was done to chiefly remove organic materials, and to obtain very fine clay sample free from contamination for further processing as particulate filler material for friction lining material (Figures 1 and 2) respectively.

### **CHEMICAL COMPOSITION ANALYSES OF NIGERIAN KANKARA KAOLINITE CLAY**

Chemical analyses of Kankara clay samples were carried out at the analytical computerized laboratory centre of energy research and training Zaria, Nigeria. The instrument used at the centre is mini pal energy dispersive x-ray spectrometer. It is designed for detection and measurement of elements for characterization chemical properties of Kankara clay samples. The result of the tests conducted is presented in comparison with other foreign clays (Table 1). Principally, the comparison analyses of Kankara clay with foreign clays show that Kankara clay and other foreign ones could be employed as a raw material for automobile friction lining material development. Asides, some commercial brake pad materials, with other locally found materials were also tested, analyzed and eventually found employable as brakepad materials. Chemically, there is a strong relationship between these tested materials and the tested commercial brakepad presented in (Table 2). Metallic oxides found in the brakepads are also found in the locally found materials. The only different is in quantities which could be adjusted at batch calculation and composition.

There are lots of empirical works on Nigerian clays that have been intensively investigated. But their scopes and core research activities are on clay exploration and characterization. (Robertson, 1978; Adigwe, 1983; Oyinloye, 1997).

Aside the location, (Table 3) shows chemical composition and refractoriness of Nigerian clays as reported by researchers such as (Aniyi 1985, Ahmed, 1986 and Aderibigbe, 1984). However, Literature works are very scanty on the uses of Kankara clay as Kaolinite clay for friction lining material as particulated filler. This research work also reported the estimates and mineralogical compositions of Kankara clay with other notable Nigeria refractory clays. (Tables 4 and 5) respectively.

### **PLASTICITY TEST ANALYSES OF NIGERIAN KANKARA KAOLINITE CLAY.**

Clay plasticity test is necessary for the binding ability of such material within a batch composition. Characteristically, if Kankara clay is to be employed as particulated filler in the automobile brakepad production; its plasticity was examined with Atterberg plasticity index tests (API) out of other plasticity measurements that Include Astbury, Moore, Pfefferkorn and Absolute Water Miscibility Tests. In this research work plasticity of Kankara clay material was taken as its ability to be shaped under applied stress or pressure; and to retain the formed shape thereafter.

Since plasticity is associated with very small particles of colloidal dimensions. Shape of the clay particles are therefore a reasonable factor to be considered. Heckroodt, (1994), explained that clay particles in a plastic mass carry negative charges with a film of absorbed water molecules bonded by electrostatic forces. But, two types of water are present within the particles of clay. These are “bound” water in the films and “free” water in excess that required to form the films.

Available surface area of clay particles is also very significant. Scientifically, most platy fibrous particles possess greater surface areas than spheres or cubes. All these explanations eventually make clay platy particles conducive to a high specific surface that favours plasticity index for the Nigerian Kankara clay. Adigwe, (1983) presented Atterberg plasticity index for the Nigerian refractory clays at 25% minimum for industrial works.(figure 2) While Worrall, (1975) reported the Atterberg plasticity indices for various Kaolinites and Montmorillonites. Empirical evidences have shown 37% is for ca-kaolinite; 26% for Na-kaolinite; 101% for Ca-montmorillonite and 251% for Na-montmorillonite. Analytically, since majority of Kankara Clay consists (these two aforementioned) mineral clays of high plasticity percentage. Therefore. Figure 2 presented in the research confirmed that Kankara clay is highly plastic and could be used as a binder and particulate filler material within a composite for automobile brakepad.

## RESULTS AND DISCUSSION.

### BRAKE PAD BATCH COMPOSITION AND NIGERIAN KANKARA KAOLINITE CLAY PURIFICATION.

Apart from the purification process carried out by removing the unwanted impurities of organic materials and other foreign products. 45micron sieve used in Figure 2 as a size “cut” was made to have qualitative kaolinite clay. Above 45micron clay particles were rejected with the unwanted materials. Although uneconomically but qualitatively, Kaolin clay less than 45micron obtained was further processed; and significantly free from quartz and silica sand for better quality. Montmorillonite is much finer particle clay material than kaolinite. While bentonite clay material is predominated by montmorillonite clay mineral (Green, 1998; Wattachman, 1998). Therefore, less than 95% of clay was obtained in this research work for the particle friction lining material in the automotive brakepad composite development consisted of kaolin, bentonite and montmorillonite clay minerals principally. Methodologically, this was due to 45micron sieve used for the Kankara clay beneficiation process (Aderiye, 2005).

Undoubtedly, 45micron sieve was employed to assess the Kankara clay plasticity, cohesion with other Polyvinyl chloride composite material as particulates and inbuilt sinterability if eventually all used composite materials are fired. The particle size distribution of Kankara clay was a varied clay contamination with other associative impurities (Table 6). Significantly, the research performed in this project shows that the percentages of fine grain particles below 45micron determined the cohesion and sinterability of Kankara clay. Therefore, the higher the 45 micron grain particle size the more the clay (Figure 2). While the friction coarse (0.5-0.05mm) grain particles of mostly quartz and silica sand described the nature of the contaminating impurities. Technically, the 45micron sieve used has removed all the impurities and other fraction coarser grain particles within the clay majority. This makes Kankara clay useful as particulate material for brake pad development as compared with other chemical oxides and minerals in table 1.

### SINTERABILITY OF NIGERIAN KANKARA KAOLINITE CLAY

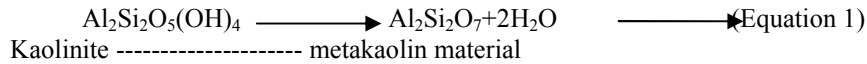
No manufacturer would ever reveal the details of brake pad materials. The exact composition of any given brakepad is a closely guarded secret. However, heat is generated when a moving speedy car applies brake and stop. Such evolution of heat is due to conversion from kinetic energy to potential energy. Therefore a refractory clay could be beneficial; it will be necessary to know the exact percentage of clay and the clay type (mineralogical composition) that can absorb heat generated through the braking system (Nieh, 1990). Therefore, the refractoriness and sinterability of clay to be used in the brake pad batch composition is necessary to be known. There is one important point that every brakepad buyer should commit to memory; the best brakepads to obtain are the pads that are designed for the automobile system. A balanced system is made up of brake pads, calipers drum or rotor (Toyota, 2013). Generally, brakepads materials are made up of a batch of five types of composite materials. The exact percentage of each of these remains is a guided trade secret of the manufacturer.

Secretly or technically, all the required raw or composite materials are usually manipulated to produce the designed qualitative brake pad for a profitable venture by a given pad manufacturer. Due to stiff competition among the brake pad manufacturers both the quantity and quality of the raw materials used for the brake pad production would not be stated in a given literature (Wright, 1990). Wisely, no manufacturer will want to be out of fierce or intensive global competition. Whereas, local brake pad makers are now facing foreign competition even in their home Market. Therefore, a research could be conducted to obtain the best characteristic properties from various commercial brake pads; and other suggestive automobile friction lining. Technically, this could provide the idea use in such brake pad materials. This is the statement of this research problem. Aimly to produce a reasonable brake pad at lowest cost for high profit with the Kankara clay particulates within polyvinyl chloride matrix.

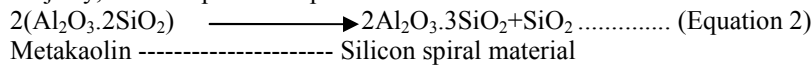
The term sinterability of Kankara clay encompasses both the drying and firing behavior of the clay. The characteristic properties mentioned above were applied to know the shock resistance, heat absorption, and improved performance of a brakepad. Although clay generally, posses the ability to shrink in volume on drying and firing as the particles come close together (Green,1998). Technically, this ability would enable Kankara clay to be made into various parts for various technical components with the required densities and strengths (figure 3). Normally, vehicles with brakepads that are made up of unfired clay within its batch composition that are speeding either on pool of rain water or snowfall would somehow absorb water. Such clay could be swelled up; after which it would be dried up; the clay should be expected to shrink definitely. Characteristically wear, and shock resistance of such brakepad will be very low for commercial activities and it will make such pad production unviable.

Alternatively, if fired clay is made of brake pad, little or no water will be absorbed because of low porosity of such clay (Table 7). Therefore clay plasticity contains large volume of water which is directly responsible for both the dried and fired clay shrinkages (Mccolin, 1990). Mineralogically, bentonite and montmorillonite clays are highly plastic, the use of 45micron sieve definitely increased the clay plasticity

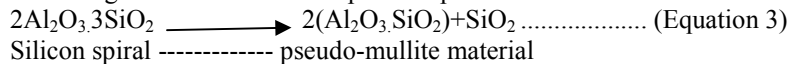
through the water within the Kankara kaolinitic clay (Singer 1963). Experimentally, during the clay firing, absorbed water was removed at early stage of firing temperature of 100<sup>0</sup>C. Thereafter, the Kankara kaolinite decomposed around 500<sup>0</sup>C temperature. Thereby, hydroxyl groups of water within the clay were lost (Table 6). Many researchers, scientists, and material technologists are called the product formed metakaolin. (Ragone, 1995). Significantly, the process of vitrification can be simply explained in these equations ( 1 to 3) as stated below.



Further heating of metakaolin material to 900<sup>0</sup>C temperature was resulted into crystalline compounds. Majorly, Silicon spinel was present.



Additional heating on silicon spiral material to higher temperatures of about 1100<sup>0</sup>C made the material to undergo further reactions to produce pseudomullite.



Intensive heating on pseudo mullite material was finally disintegrated to cristobalite and mullite at about 1400<sup>0</sup>C temperature.

Due to these aforementioned three equations, there were significant changes in volume of the ceramic brake pad made, mainly because of the chemical changes that occurred during firing from 100<sup>0</sup>C to 1400<sup>0</sup>C temperature. These changes were irregular due to contraction and expansion of rapid chemical changes during the chemical reactions. (Figure 4). The ions reacted with silica at about 1000<sup>0</sup>C temperature to form a viscous liquid phase and lowered the melting point of the refractory binary system. In this research work the liquid glassy phase principally acted as binder when fired up to 1000-1400<sup>0</sup>C temperature. This unhomogeneous complex glassy phase is also responsible for the vitrification process and strength of brake clay pad which on cooling at 37<sup>0</sup>C did not crystalline but solidifies to form ‘glassceramic’ brake pad mullite, cristobalite, complex glass and some unchanged quartz (Table6) Characteristically, firing shrinkage, bulk density and water absorption are some of the significant indices of Nigerian Kankara kaolinite clay that could be used for the ceramic brake pad material development as presented in (Table 7). Further research work will be made on polychloride (PVC) matrix as a binder with the kankara kaolinitic clay particulates as presented on (table8).

## CONCLUSION

Summarily, exploration of the Nigerian Kankara Kaolinte clay deposit was made. Clay samples were exploited from the deposit from 8 metres downward at eight different cardinal areas. Thereafter, the clay samples were beneficiated with 45micron sieve. The purification process was done to chiefly remove both the unwanted coarse silica sand and other unwanted organic materials. Significantly, samples free from contaminated were obtained for further processing as particulate filler material for the friction lining material development.

In summary, based on the results obtained and discussion of Kankara clay investigated, Kankara clay had more than 95% of its fine particles between the range of a unit microns to 75microns. It has lowest coarse and unwanted impurities. The beneficiated clay was more plastic than the raw clay. While the range of kaolin could be found between 70-90% even at 1200<sup>0</sup>C temperature. Research results eventually obtained from all the experiments There is high percentage of aluminum oxide and silicon dioxide. These refractory oxides could be technically employed as friction lining material for very high thermal characteristics properties; medical reasons; environmental friendliness and economical considerations in automobile disc brake pad production.

Factually, with the test conducted, the Nigerian Kankara clay was found to be kaolinitic in nature, but with some Impurities of montmorillonites, mica and quartz. The kaolinite content of the beneficiated clay was higher than the Kankara raw clay. It has about 96% kaolinite. If fired to 1400<sup>0</sup>C temperature, the maximum firing shrinkage was 10% with an average density of 1.5gramm/cm<sup>3</sup>. Some of the fired Kankara castable batches were strong enough to meet automobile brake pad modulus of rupture (MOR) requirements. It has value of less than 8.00MN/m<sup>2</sup>. While the thermal shock resistance of the Kankara castable fired at 1400<sup>0</sup>C temperature for 12hours.

Other refractory characteristic properties such as strength, density and porosity increased with an increased firing temperature. Even with very high temperature; refractory under load test showed that the Nigerian Kankara clay could be used for special engineering and other furnace applications at operating temperatures of about 1400<sup>0</sup>C temperature.

Based on obtained results of this research work further investigation could still be made on the Nigerian Kankara clay with other materials like carbon composite various proportions. There are also other Nigerian kaolinitic clays that required detailed examinations for friction lining materials and automobile vehicle parts development, (Aigbodion, 2010). Therefore intensive investigations should be carried out to identify these

refractory clays with their characteristics properties and usefulness to the Nigerian manufacturing industries.

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**Table 1:** Comparative analyzed of some foreign clays with Nigerian Kankara kaolinite clay from various researchers.

Chemical oxides and minerals	Researcher Name	Singer and singer (1963)					Ahmed (1986)	Aderibigbe (1984)	Aderiye (2005)	Robertson (1985)	Current research work	Aniyi (1985)
	Mineral	Cornish China Clay	Zettlitz Kaoline	Kemmilt Kaoline	North Carolina Kaoline	Sergent China Clay	Kankara	Kankara	Kankara	Kankara	Kankara	Kankara
	Country	British	Czecho-slovakia	Germany	U.S.A	U.S.A	Nigeria	Nigeria	Nigeria	Nigeria	Nigeria	Nigeria
SiO <sub>2</sub>		45.45	46.09	58.30	48.46	44.90	44.47	44.50	54.8	48.46	54.8	44.50
TiO <sub>2</sub>		0.34			0.06	1.32			0.046	0.45	0.046	
Al <sub>2</sub> O <sub>3</sub>		38.33	39.28	29.31	36.53	38.90	36.08	38.64	42.8	34.49	42.8	36.50
Fe <sub>2</sub> O <sub>3</sub>		0.55	0.76	0.87	0.15	0.40			0.409	1.17	0.409	
MgO		0.12		Trace	0.03	0.10				0.95	-	
CaO		0.06	0.15	0.51	0.13	0.06		1.30	0.35	0.31	0.35	
Na <sub>2</sub> O		0.43	0.03		0.21	0.22				0.19	-	
K <sub>2</sub> O		2.22	0.12	1.26	1.34	0.20			0.961	0.33	0.961	
LOI		12.44	13.58	10.62	13.03	14.20	13.91	16.70		13.19	-	14.05
Clay substance			98.3	54.9								
Potash (mica)												
Soda (mica)			1.2	43.8								
Quartz			0.4	1.3								
Feldspar												

**Sources:** Aderiye, 2005; Ahmed, 1986; Aderibigbe, 1984, Singer and Singer, 1963; Robertson, 1978; Aniyi, 1985.

**Table 2**

Comparative chemical analyses of suggestive automotive friction lining materials (periwinkle shell, coconut shell, Ground nut shell, Kaolin clay, waste glass and some commercial brake pads)

	Kaolin clay	Waste glass	PWS	CNS	GNS	Benz B/Pad	Toyota B/Pad	Peugeot B/Pad	Honda B/Pad
RUO <sub>2</sub>	0.409	-	-	-	-	-	0.4	-	-
Yb <sub>2</sub> O <sub>3</sub>	0.026	-	-	-	-	-	-	0.01	-
Au	0.019	-	1.30	-	-	-	-	-	-
MgO	0.95	3.8	1.54	0.96	1.50	0.93	7.02	15.07	-
Y <sub>2</sub> O <sub>3</sub>	0.026	-	-	0.71	0.40	-	-	-	-
Na <sub>2</sub> O	0.19	12.8	0.10	0.16	0.10	0.02	-	1.0	-
Al <sub>2</sub> O <sub>3</sub>	42.8	1.4	-	-	1.68	3.30	0.22	2.82	3.6
S <sub>1</sub> O <sub>2</sub>	54.8	72.8	0.09	6.14	11.48	12.98	2.05	17.16	11.7
P <sub>2</sub> O <sub>5</sub>	-	-	-	3030	2.01	-	-	-	-
S <sub>0</sub> 3	-	0.3	0.30	-	2.31	11.4	1.80	14.10	7.85
K <sub>2</sub> O	0.961	0.8	0.20	7.28	24.50	0.55	0.15	0.97	0.75
C <sub>a</sub> O	0.35	8.2	96.09	3.80	15.20	1.14	47.90	1.56	6.34
πO <sub>2</sub>	-	-	0.04	0.95	1.80	1.20	0.10	1.60	-
Cr <sub>2</sub> O <sub>3</sub>	0.019	-	0.003	0.51	0.30	-	0.76	-	-
M <sub>n</sub> O	0.011	-	0.06	-	0.63	0.17	0.14	-	-
Fe <sub>2</sub> O <sub>3</sub>	0.409	0.1	0.79	12.71	11.01	36.46	5.28	15.30	44
N <sub>1</sub> O	-	-	-	0.20	0.18	0.04	0.20	0.07	-
CuO	0.059	-	0.09	2.9	0.40	0.16	0.09	0.12	-
ZnO	0.059	-	-	-	2.00	0.23	0.17	0.29	-
SrO	-	-	0.76	-	0.28	1.11	0.20	0.7	-
As <sub>2</sub> O <sub>3</sub>	-	-	-	0.6	0.05	-	-	-	-

Aderiye, 2012.

**Table 3** Chemical Composition and Refractoriness of some Nigeria Clays.

Clay	Location (State)	Al <sub>2</sub> O <sub>3</sub> (%)	SiO <sub>2</sub> (%)	Fe <sub>2</sub> O <sub>3</sub>	CaO + MgO (%)	L.O.I (%)	Refractoriness (P.C.E)	
							Cone No.	Equiv. temp (°C)
Alkaleri	Bauchi (BA)	25.43	54.30	1.05	1.00	15.73	33.5	1740
Onibode	Abeokuta (OG)	39.30	42.30	Trace	1.05	14.20	>34	>1760
Oshiele	“	28.30	53.40	1.35	0.88	15.00	33	1730
Orun	“	34.55	50.50	2.05	1.10	15.50	33	1730
Ozubulu	Nnnewi (AN)	19.31	58.30	1.55	1.25	14.16	32	1710
Enugu	Enugu (AN)	22.71	55.00	2.42	1.95	16.35	29	1650
Nsu	Okigwe (IM)	30.22	50.60	1.90	1.08	10.54	32	1710
Okpekepe	Auchi (EDO)	24.30	53.20	1.45	1.30	16.86	29	1650
Kankara	Kankara (KST)	38.64	44.50	Nil	1.30	16.70	34	1760
Giro	Giro (SO)	38.72	41.26	2.10	1.48	14.00	34	1760
Warram*	Warram (PL)	37.13	43.54	1.15	0.58	14.20	34	1760
Sabon Gida	Jos (PL)	25.88	25.32	13.10	3.20	18.36	27	1610
Maja-Hota +	Majahota (PL)	-	-	-	-	-	-	-
Nafuta +	Nafuta (PL)	-	-	-	-	-	-	-

Sources: (Aderibigbe and Chukwuogo) (1984); (Ahmed, (1986);

\* Average values for two differently located samples. (Adigwe,(1983)

\* Data not available but vast reserves of white kaolin were reported.

**Table 4** Reserve estimates of some Nigerian clays

Clay	Type	Reserve (Tonnes)	Estimate	Average Clay Content (%)	Current Lease Holder
Onibode	2 <sup>0</sup> Kaolin	4,500,000		NA	National steel Council
Kankara	1 <sup>0</sup> - Kaolin	891, 000		90	Kasha state Govt.
Majahota	“	850,000		78	Bisichi Tin Company
Warram	“	NA		88	Kaduna Prospectors Limited.
Nafuta	“	190,000		50	Kaduna Prospectors Limited

\*

Sources: Aderibigbe and Chukwuogo, (1984); Ahmed (1986) and Nigerian Geological Survey of Nigeria, 1986

Note: Na = Note Available

**Table 5** Chemical and Mineralogical Composition of Kankara Clay at 10 micron.

Chemical Analysis			Mineralogical Analysis (Rational Analysis)		
Parameter	Whole	<10µm	Parameter	Whole	<10µm
SiO <sub>2</sub>	48.46	54.27	Kaolinite	75.0	30.6
Al <sub>2</sub> O <sub>3</sub>	34.49	28.40	Allophan	9.0	40.7
Fe <sub>2</sub> O <sub>3</sub>	1.17	0.45	Free silica	5.6	16.9
MgO	0.95	0.56	feldspars	2.5	3.9
CaO	0.31	1.11	Saponite	3.6	5.2
Na <sub>2</sub> O	0.19	0.29	Muscovite	2.4	0.7
K <sub>2</sub> O	0.33	0.10	Gypsum	0.1	-
H <sub>2</sub> O(LOI)	13.19	14.60	Anatase	0.4	0.2
TiO <sub>2</sub>	0.45	0.19	Goethite	1.3	0.5
MnO	0.01	0.03	Calcite	-	1.1
So <sub>4</sub>	0.05	N.D			
<b>Total</b>	<b>100.00</b>			<b>100.00</b>	<b>99.8</b>

Sources: (Robertson Research, (1978), (Ahmed), (1986)

N.D. - Not detected

**Table 6** Chemical reactions and decomposition of the Nigerian kankara kaolinitic clay between 0°C to 1400°C temperature for automobile brake pad development.

TEMPERATURE (0°C)	KANKARA CLAY FIRING REMARKS
15 <sup>0</sup> to 250 <sup>0</sup> C	Remaining water (dampness due to humidity of atmosphere) was evaporated in the kiln
110 <sup>0</sup> to 250 <sup>0</sup> C	Plastic clays lost absorbed water (montmorillonate lost its nH <sub>2</sub> O)
250 <sup>0</sup> C to 650 <sup>0</sup> C	Kaolinite, Al <sub>2</sub> Si <sub>2</sub> O <sub>5</sub> (OH) <sub>4</sub>
500 <sup>0</sup> C to 650 <sup>0</sup> C	Kaolinite dehydrated; and free quartz present – changed
550 <sup>0</sup> C	Clay probably matakaolin (Al <sub>2</sub> O <sub>3</sub> .2SiO <sub>2</sub> ), with increase of pores and overall volume. Dry sintering began within also.
900 <sup>0</sup> C upwards	Recrystallization also began.
950 <sup>0</sup> C (at low temperature for 3 hour)	Sintering helped by liquid contraction. Vitrification began. Any mica present were dehydrated.
1000 <sup>0</sup> C upwards	Mullite began to appear.
1050 <sup>0</sup> C upwards	Mica decomposed to mullite and liquid.
1160 <sup>0</sup> C upwards (at rapid for 2 hours)	Cristoballite began to crystallize from the liberated silica, mullite increased in quantity and crystal size. Liquid increased in quantity also.
1200 <sup>0</sup> C to 1400 <sup>0</sup> C	Vitrification completed, Cristobalite and other free silica progressive dissolved in the liquid. All the alumina became mullite or joined the liquid phase.

TEMPERATURE (0°C)	Reactions of gas impurities within the brake pad developed
250 – 920 <sup>0</sup> C	S + O <sub>2</sub> → SO <sub>2</sub>
350	C + O <sub>2</sub> → CO <sub>2</sub>
350 – 450	FeS <sub>2</sub> + O <sub>2</sub> → FeS + SO <sub>2</sub>
400 – 900	MgCO <sub>3</sub> → MgO + CO <sub>2</sub>
500 – 800	4FeS + 7O <sub>2</sub> → 2Fe <sub>2</sub> CO <sub>3</sub> + 4SO <sub>2</sub>
560 – 775	Fe <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub> → Fe <sub>2</sub> O <sub>3</sub> + 3SO <sub>2</sub>
600 – 1050	CaCO <sub>3</sub> → CaO + CO <sub>2</sub>
800	4FeCO <sub>2</sub> → Fe <sub>2</sub> O <sub>3</sub> + O <sub>2</sub> + O <sub>2</sub> Fe <sub>2</sub> O <sub>2</sub> O <sub>3</sub> + 4CO <sub>2</sub>
1250 – 1400 <sup>0</sup> C	CaSO <sub>4</sub> → CaO + SO <sub>3</sub>

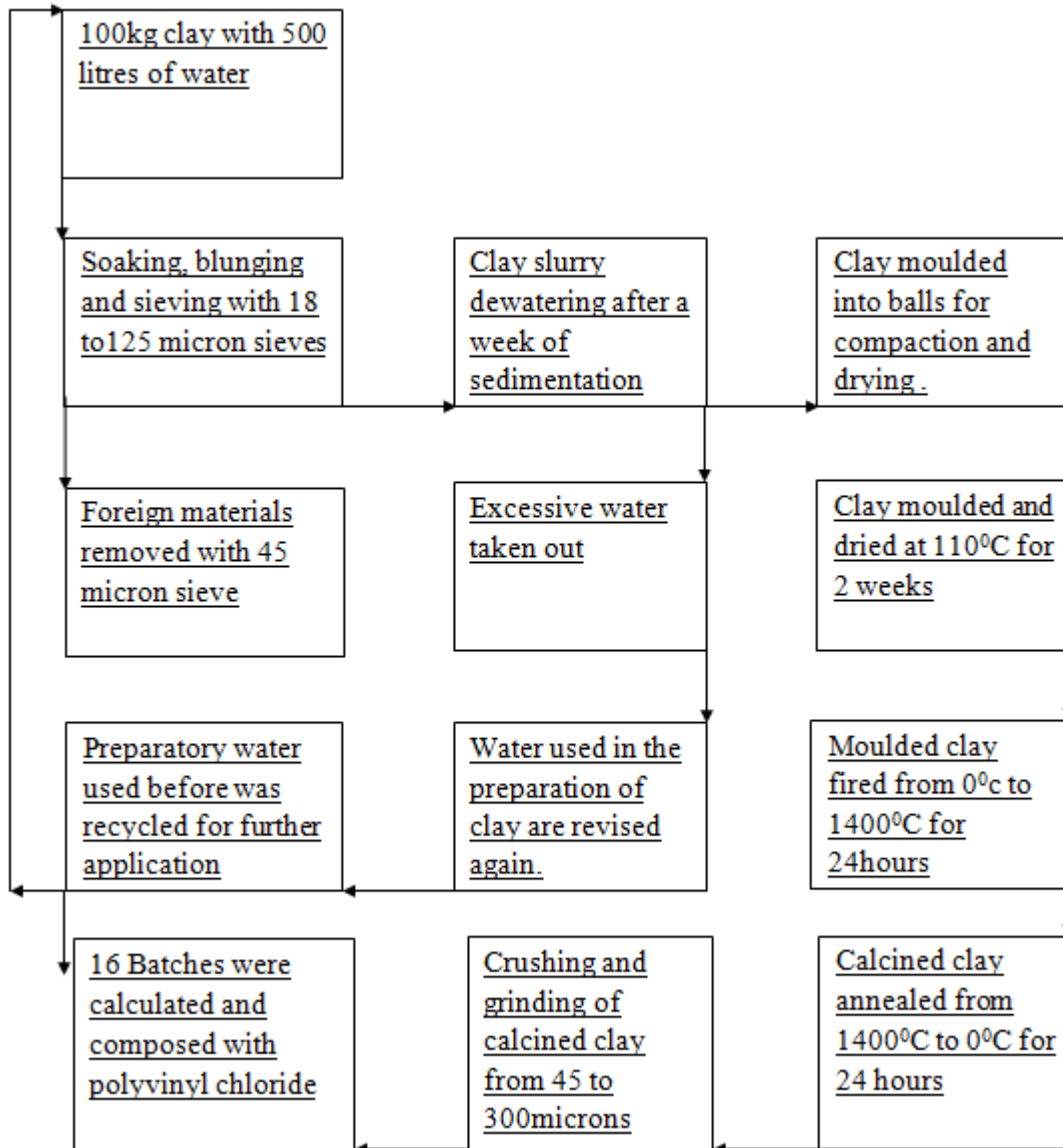
**Table 7** Qualitative control for Nigerian kankara clay for the automobile brake pad development.

No	Required Characteristic Properties	
1	Linear shrinkage % (Drying + firing)	10%
2	Apparent porosity $\frac{(WC-WA)}{(WC-WB)}$ %	10%
3	Bulk Density $\frac{(WA)}{(WC-WB)g/m^3}$	1.9gcm <sup>2</sup>
4	Crushing strength (KN/mm <sup>2</sup> ) (Comprehensive resistance)	100kg/cm <sup>2</sup>
5	Shapes	Good
6	Slag resistance (internal and surface areas)	No cracking, Good for acidic slag's and iron Oxide
7	Vitrification at 1300 <sup>0</sup> C (spalling resistance)	Good
8	Colouration	Light Brown
9	Alumina (25 – 45% for firebricks)	46% fair
10	Iron Content (less than 1%)	0.9% fair

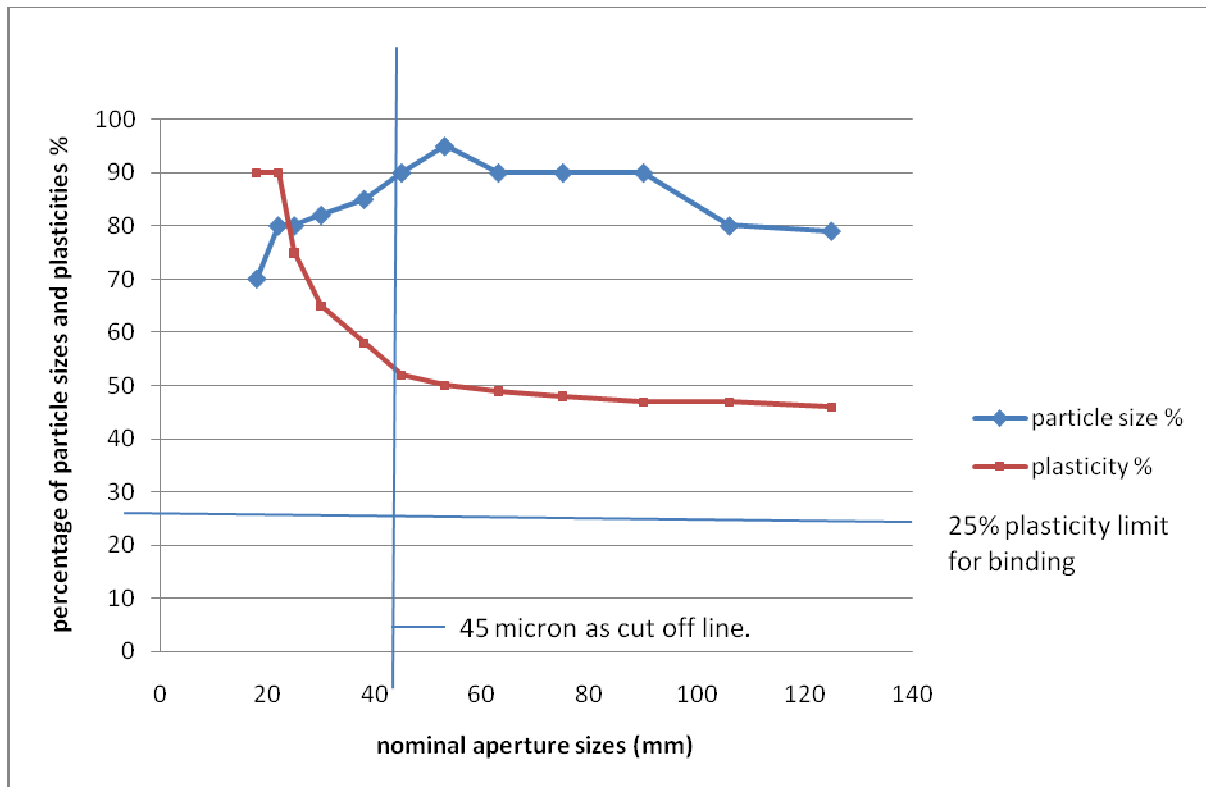


**Table 8** Standard batch calculation and composition of Nigerian Kankara clay used in the automobile brake pad development.

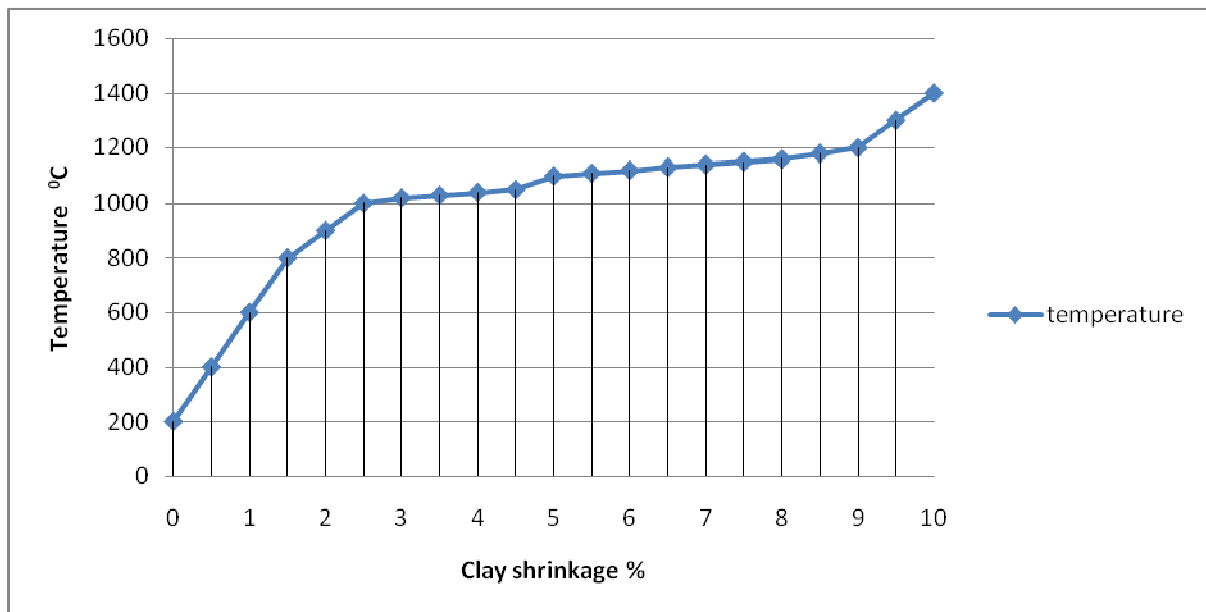
Size	Range (micron)	Mean (um)	Kankara clay at 1400 <sup>0</sup> C %										PVC% 30%
			100	90	80	70	60	50	40	30	20	10	
Very coarse	300 + 150 (um)	225	25	22.5	20	17.5	15	12.5	10	7.5	5	2.5	7.5
Coarse	150 + 75 (um)	113	25	22.5	20	17.5	15	12.5	10	7.5	5	2.5	7.5
Medium	75 + 45 (um)	60	25	22.5	20	17.5	15	12.5	10	7.5	5	2.5	7.5
Fine	45 + 0 (um)	23	25	22.5	20	17.5	15	12.5	10	7.5	5	2.5	7.5



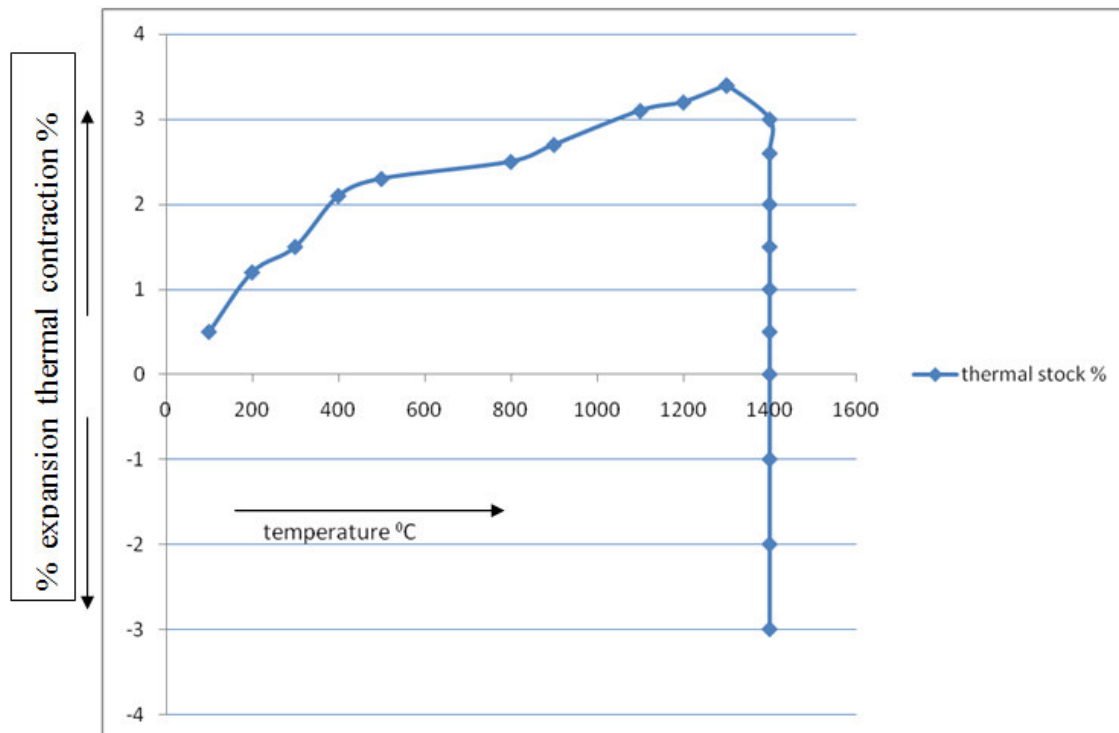
**Figure 1:** Flow chart for Nigerian kankara clay beneficiation process for the automobile brake pad development.



**Figure 2:** Benefication of kankara kaolinite clay using 45 micron sieve for particle size analyses and plasticity indices.



**Figure 3:** The kankara clay shrinkage is related with the clay plasticity.



**Figure 4:** Kankara clay thermal shock and refractoriness under load curve.

Note: Reasonable thermal shock resistance was obtained at 1400°C temperature within the range of 40 thermal cycles of test under load of 2kgf/cm<sup>2</sup> or 28PSI or 1.93 x 10<sup>5</sup> N/M<sup>2</sup> at 1400°C temperature.

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