

Ceramic Raw Materials Development In Nigeria

P. S. A. Irabor, S. O. Jimoh, O.J. Omowumi

ABSTRACT: Raw materials development is critical to the success of the ceramic subsector of the nation's economic and industrial growth. The much taunted sing-song of Nigeria's abundant natural blessings in both metallic and non-metallic solid mineral raw materials resources in now staled news or history. The situation of raw materials development in Nigeria may be likened to the song by the reggae legend, Peter Tosh, who said, "many people want to go to heaven but non, want to die" just as many people, entrepreneurs, companies want to produce good quality ceramic products to create wealth but, none, want to prepare the raw materials. Consequently, this paper investigates, assesses and reviews the available local ceramic raw materials, identification, beneficiation techniques, and physical and exploitation development initiatives for the raw materials sector in Nigeria.

Keywords: Refining, calcinations, comminution, hydration, carbonation, bleaching, sedimentation, clarification, electro-magnetization and filter-pressing.

1. INTRODUCTION

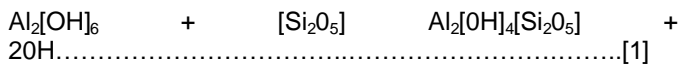
Naturally occurring clay is the oldest known ceramic material selected by the potter for its forming or plastic properties. It was also customary to add some other non-clay materials to modify the formulated product recipe. The appropriate addition of non-clay material such as crushed stone or sand would adjust the undesirable high contraction or shrinkage of the product mix and the use of unrefined clays or in combination of one or more granulated nonplastic materials in which clay mineral may exceed 20%. However, the last seventy years have seen an increasing interest in ceramic products made from highly refined material or synthetic compositions designed to offer special properties in new or fine ceramic products.^[1] The quantum development in the field of ceramic bestriding the traditional or conventional ceramic to such products that now find applications in high technology and engineering field as electronics, computers, optical communications, cutting tools, dies, wear resistant parts, seals, turbine blades, motor engine parts, medical implants, jet engine parts among others have been attributed to the advances made in materials science. The technology of ceramic manufacturing rests on appropriate measurement of the structural and chemical properties of the raw materials used in ceramic forming systems^[2]. With the established fact that the purity of materials has a profound influence on the high temperature properties of ceramics including strength, stress, rupture and oxidation resistance, the need for consistent raw material properties has continued to attract enormous research attention even in the developed nations^[3].

Clay-based ceramics are predominant among the wide range of ceramic products such as body recipes or formulas containing single or more clays mixed with modifiers like quartz and feldspar. The special properties of the clay minerals that allow the preparation of high solid fluid systems and plastic forming bodies are critical in the shaping of the ceramic product. The material refining and characterization concept has permitted the development of simple test procedures for use in mining and beneficiation control.^[4] Material characterization describe those features of the composition, structure and defects of the material that are significant for a particular preparation, study of properties that are sufficient for the reproduction of materials with the true characterization capturing the direct correlation between test results and properties. The many properties encountered in forming and firing a ceramic product have been found to be the consequences of the interaction of two or more of a limited list of fundamental characterizing features.^[5] The state of materials is so significant in ceramic production to such an extent that the rheology of clay-based forming systems can be altered adversely by very minor changes in subsieve particle-size distribution.^[6] In most fine ceramic products, colour is viewed critically for chinaware/tableware by controlling the effect of Fe₂O₃ and TiO₂ compared to their effect on sanitary ware or a material effect, where the presence of mica in sanitary ware slip-casting improves the casting rate and cast quality^[7]. In establishing standards for materials, ceramic manufacturers and raw materials suppliers usually work together in the developed economies to ensure the quality of materials but in the developing or underdeveloped nations, there are no reliable suppliers to depend upon and most often carries out field work, excavation and supply self for production.^[8] Consequent upon the Nigerian situation, a nation, that is known worldwide to be blessed naturally with both human and mineral resources abundantly, now sentenced to reliance on morbid import of all products of finished pottery and ceramics, the present work presents the modest efforts made in the area of raw materials characterization, beneficiation and refining technologies that have been adapted and developed to advance the science and manufacturing technology of ceramic materials and products in Nigeria.

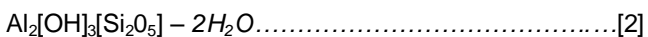
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BACKGROUND OF MATERIALS DEVELOPMENT

Ceramic raw materials has special character, though, like other materials, their properties are dictated by the types of atoms present, the type of bonding between the atoms and the way they are packed or arranged together in the atomic structure. The ceramic material atoms are held together by chemical bond and the two most common bonds for ceramic materials are covalent and ionic while that of iron is the metallic bond. In covalent and ionic types, the bonding is much stronger than that of metallic which is responsible for the brittle nature of ceramics and the ductile nature of metals. The ceramic materials character is in it having a wide range of properties that make them unique and possess a wide variety of potentials that make them suitable for a multitude of applications in all areas of modern day technologies and engineering. The typical and conventionally known ceramic clay is the Kaolinite. In most clay mineral lattices, two structural units are involved, one being the silica sheet, formed of tetrahedra which consist of one Si^{4+} surrounded by four oxygen ions. These tetrahedra are arranged to form a hexagonal network repeated to compose the Si_2O_5 . On the other hand, the second structural unit is the aluminium hydroxide or gibbsite sheet, consisting of octahedral in which an Al^{3+} ion is surrounded by six hydroxyl groups. These octahedral make up a sheet, due to the sharing of edges. The octahedral sheets condense with silica sheets to form important clays. The kaolinite, the major kaolin mineral has tabular particles made from units that results from the interaction of gibbsite and silica sheets thus:



Kaolinite crystals consist of large number of two-layer units held together by hydrogen bonds acting between OH groups of the gibbsite layer of one unit and oxygens of adjacent silica structural layers. In the case of halloysite, the unit layers are stacked along a and b axis in random fashion resulting in less hydrogen bonding allowing water penetration between layers thus forming a hydrated variety of kaolinite.^[9]



Halloysite can exist as spheres, tubular elongates, polygonal tubes, with kaolin occurring in a number of morphologies ranging from worms to sacks, irregular platelets, to crystals with particle morphology capable of having significant effects on ceramic forming systems.^[10]

GOVERNMENT AND PRIVATE SECTOR INITIATIVES

Records at the Geological Survey Department revealed that attempts to explore and exploit available solid mineral resource in Nigeria began as far back as 1903 and with the involvement of colonial personnel, several naturally deposited both metallic and non-metallic, solid minerals have been identified. From then till date Nigeria does not have a viable raw materials industry. However, there are now various government and private sector initiatives that

have failed to offer meaningful impact. Beginning from the Mining act of 1946 to the backward integration, import substitution and the structural adjustment programmes, there is now a long list of government initiatives and institutional policy frame-work aimed at the development of raw materials in Nigeria. Specifically, there are the old generation establishments or agencies such as the Geological Surveys of Nigeria, Kaduna, Nigerian Mining Corporation, Jos, Nigeria Steel Raw Materials Exploration Agency, Kaduna, Nigerian Coal Corporation, Enugu which had their mandates tied to specific industries of iron and steel and coal.^[11] The establishment of the Federal Institute of Industrial Research, Oshodi[FIIRO], Lagos in 1956 with the mandate to conduct applied research and development in food and agro-based processes, industrial raw materials, pulp and paper technologies including the upgrading of indigenous food process techniques to accelerate the pace of industrialization in Nigeria, brought great impetus to the hopes and aspirations for the Nation's rapid industrialization. In addition, came the establishment of the Project Development Agency [PRODA] Enugu about 1972; followed by the National Research Institute for Chemical Technology [NARICT], Zaria with equal mandate for materials and technological process development. In response to the raw materials crisis of the mid-1980, Government established the Raw Materials Research and Development Council [RMRDC], Abuja in 1987 with mandate to support materials research and promote same. Other such initiatives include Engineering Materials Institute, Akure, National Agency for Engineering Infrastructure [NASENI], Abuja. These include the various research activities spread across in the nation's higher institutions and the private sector initiatives in forms of investment in raw materials development that have largely been unsuccessful. Such private sector initiatives include the followings:

S/No	Company	Product	Remark
1.	Kavitex Kaolin processing co., Lagos	Kaolin	Failed
2.	Fakowsky Kaolin Industries, Jos	Kaolin	Failed
3.	Faskol Kaolin Plants, Lagos	Kaolin	Failed
4.	Freedom Development Co., Benin	Calcium Carbonate	Narrow/ construction
5.	Salami Orogo and Sons. Auchi	Calcium Carbonate	Failed
6.	Afric Mining Co. Ltd., Kaduna	P.O.P	Narrow
7.	Hydrates Lime Co. Auchi	Lime	Failed
8.	Brink Jones Ltd., Okitipupa	Kaolin	Failed
9.	Nigerian Mining Corp. Kaolin Plant	Kaolin	Failed
10.	MAICO Gylsum Refining Co. Lagos	P.O.P	Failed
11.	Delta Steel Limestone Processing Co., Warri	Calcium Carbonate	Failed
12.	Industrial Clays Ltd., Ukpok, Anambra	Kaolin	Failed

This list of failed raw material development industries is by no means exhaustive.

MAJOR MATERIALS PROCESSING AND DEVELOPMENT

MACHINERIES

(a) MATERIALS ANALYSIS AND CHARACTERIZATION:

S/No.	Name	Specification	Application	Source
1.	X-Ray Fluorescence analyzer [XRF]	Standard U300, 50kv, 1.7, ED XRF	Chemical analysis for constituent oxide composition	Europe
&	Instron Instrument	BS-161, 600N	Strength testing	Local Agent
3.	Microscope	Standard Scanning Electron [SEM] system	Microstructural analysis	Europe
4.	Sedigraph analyzer	Standard PSA unit	Particle size analysis	Europe
5.	Dialometer	DIL 402, 1600°C	Thermal expansion measurement	Europe

[b] MATERIALS PROCESSING AND REFINING:

S/No.	Name	Specifications	Application	Source
1.	Crusher/mills	Variable specs and sizes, 3 phases, 1 Okw.	Materials cumminution	Local agent
2.	Spiral classifier	Mild steel horizontal Isorm screw extruder 5kw	Materials separation slurry	Europe
3.	Magnetic/Vibratory	Electro-magnetic separator	Materials-model separator	Europe
4.	Rotary drier/kiln	Gear/belt driven cylindrical and rotating device	Drying or firing in calcinations of materials	Europe
5.	Reaction tank/vessels	Mild steel tanks	Blending/reaction	Local
6.	Filter press	Heavy duty press Hydraulic system	Dewatering	Europe

MATERIALS AND METHODS

A wide variety of raw materials are now used in the development, processing and manufacture of ceramic products. Identification, selection and beneficiation of the raw materials are significant. The most important and commonly used materials include the followings:-

S/No	Material	Chemical formula	Mol. Wt.
1.	Kaolin	$Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$	258.0
2.	Ball clay	$Al_2O_3 \cdot SiO_2$	
3.	Feldspar [Potash]	$K_2O \cdot Al_2O_3 \cdot 6SiO_2 \cdot H_2O$	556.0
4.	Silica/Quartz	SiO_2	60.0
5.	Fire clay	Al_2O_3/SiO_2	
6.	Limestone	$CaCO_3$	100.0
7.	Bentonite	$5Al_2O_3 \cdot 2MgO \cdot 4SiO_2$	
8.	Talc	$3MgO \cdot 4SiO_2 \cdot H_2O$	
9.	Barium Carbonate	$BaCO_3$	197.0
10.	Sodium Carbonate	$NaCO_3$	106.0
11.	Zinc Oxide	ZnO	81.0
12.	Zirconium Silicate	Z_2SiO_4	183.0
13.	Feldspar [Soda]	$Na_2O \cdot Al_2O_3 \cdot 6SiO_2$	524.0

14.	Borax	Na ₂ B ₄ O ₇	
15.	Kyanite		
16.	Gypsum	CaSO ₄ ·2H ₂ O	
17.	Fire clay	[Al _i .8Fe _o .i.MgO _o .i]o _j	
18.	Alumina	Al ₂ O ₃	102.0
19.	Lead Carbonate	2PbCO ₃ ·Pb[OH] ₂	775.6
20.	Clay [buff or Red		
21.	Dolomite	CaCO ₃ ·Mg·CO ₃	184.4

This level is beyond repeating the long list of available materials deposits and their location across the country as quite a number past works have reported same.^[11]

PROCEDURE

Ceramic raw materials development essentially entails a process of adding value because most clay materials such as the kaolinites occur in combination with impurities. The impurities should be removed or reduced to tolerable level to make them suitable for ceramic manufacturing. In material processing and development certain physical and or chemical processes are involved and specialized materials can be synthesized from basic raw material. In this work the following raw materials were processed, analysed and refined.

- (1) Kaolin
- (2) Limestone
- (3) Gypsum

The materials were analyzed using the x-ray fluorescence [XRF] technique. Physical and suitability tests were carried out on the materials according to the following specimen preparation.

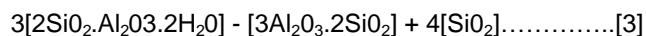
SPECIMEN PREPARATION

Approximately, 25kg of each material [Kaolin, Limestone and gypsum] were weighed out accurately. They were treated separately as each sample was crushed and hammer milled to reduce the lump size. They were screened through a 100 sieve mesh. The Kaolin was water-washed, screened and dried at 110°C. The specimens were analysed for their chemical constituents and other physical parameters.

REFINING PROCESS TECHNOLOGY

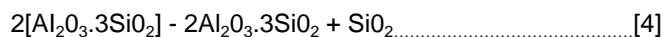
Ceramic raw material processing and refining is highly technical and requires both simple and advance facilities. Beside the water-wash techniques of refining the kaolin, the calcinations process is also used according to the following

expression:



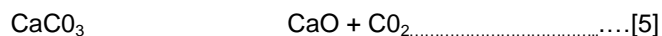
This is at the initial decomposition at about 500°C.

At 950°C the metakaolin condenses to the spinal structure thus:

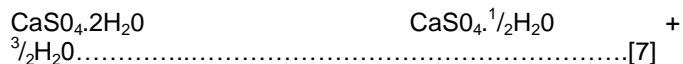


Most of the impurities such as TiO₂ Fe₂O₃ and other non colouring oxides should have been burnt off resulting in pure and white kaolin material. The calcination process was used for the limestone and gypsum based on their chemical reaction at elevated temperature.

CHEMICAL REACTIONS IN LIMESTONE PROCESSING



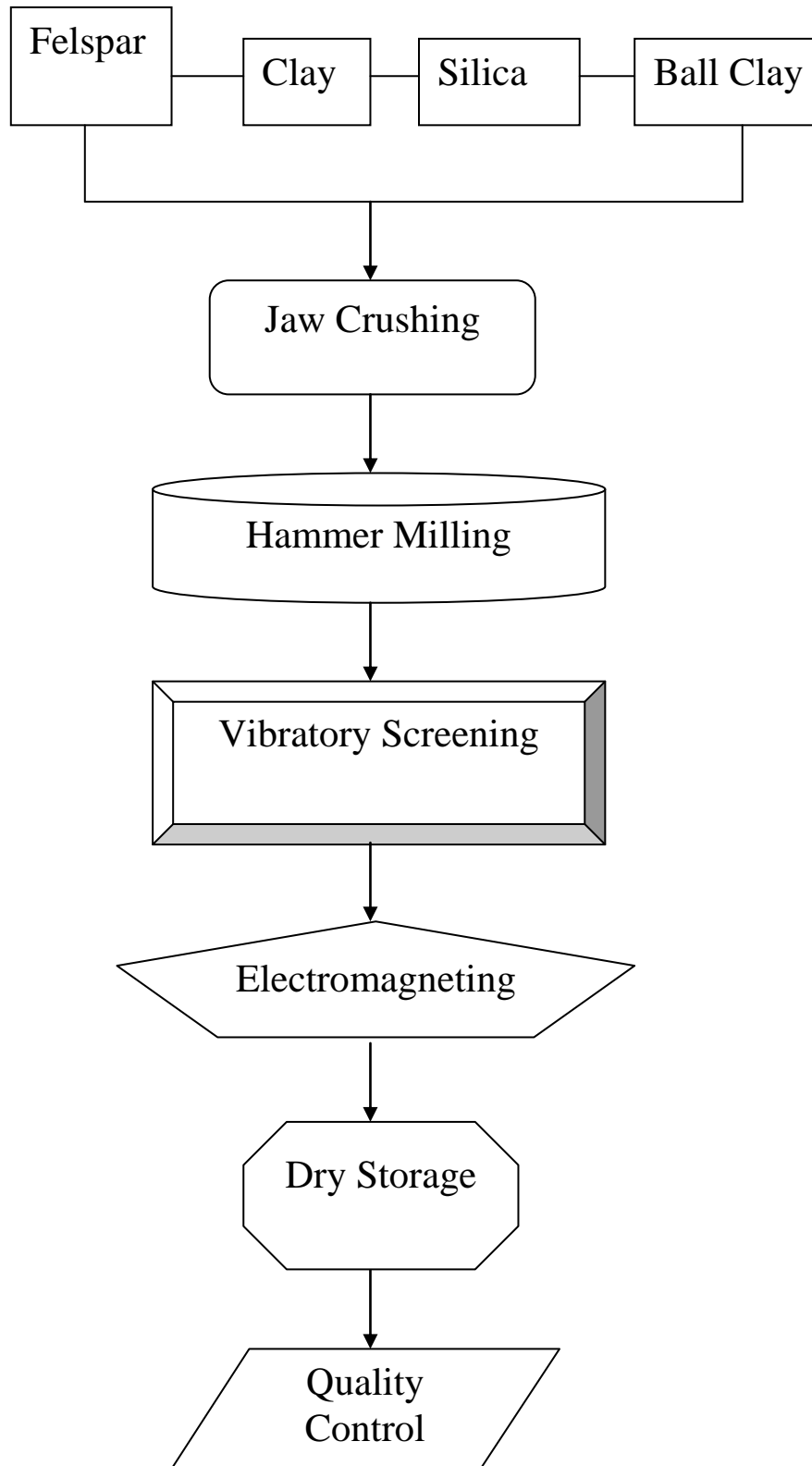
CHEMICAL REACTION IN GYPSUM PROCESSING

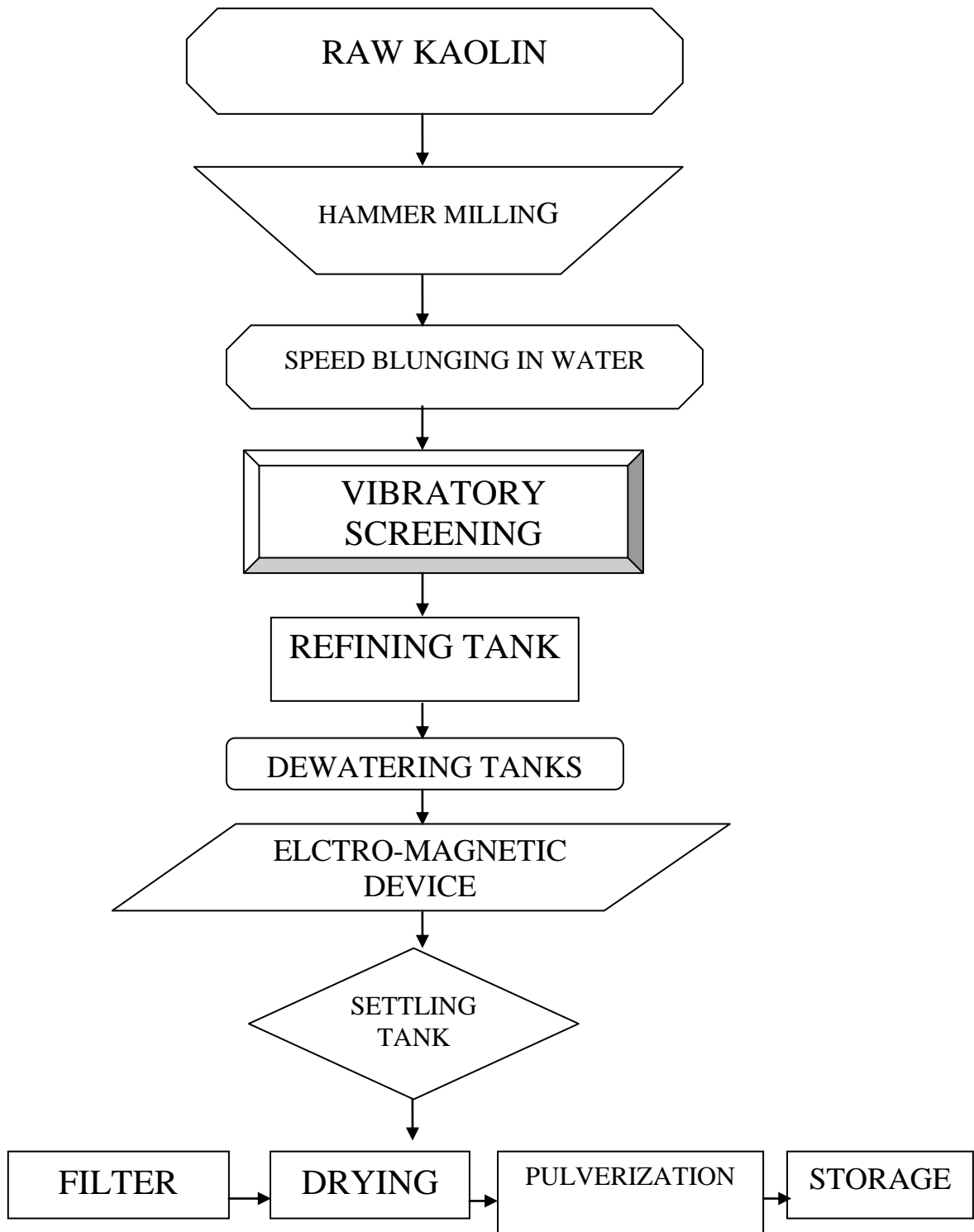


The all important plaster of paris[P.O.P] employed in the making of ceramic moulds and others are produced according to the above reactions. Recent advances in materials development has seen the use of processes such as carbothermic synthesis, gas phase synthesis, laser

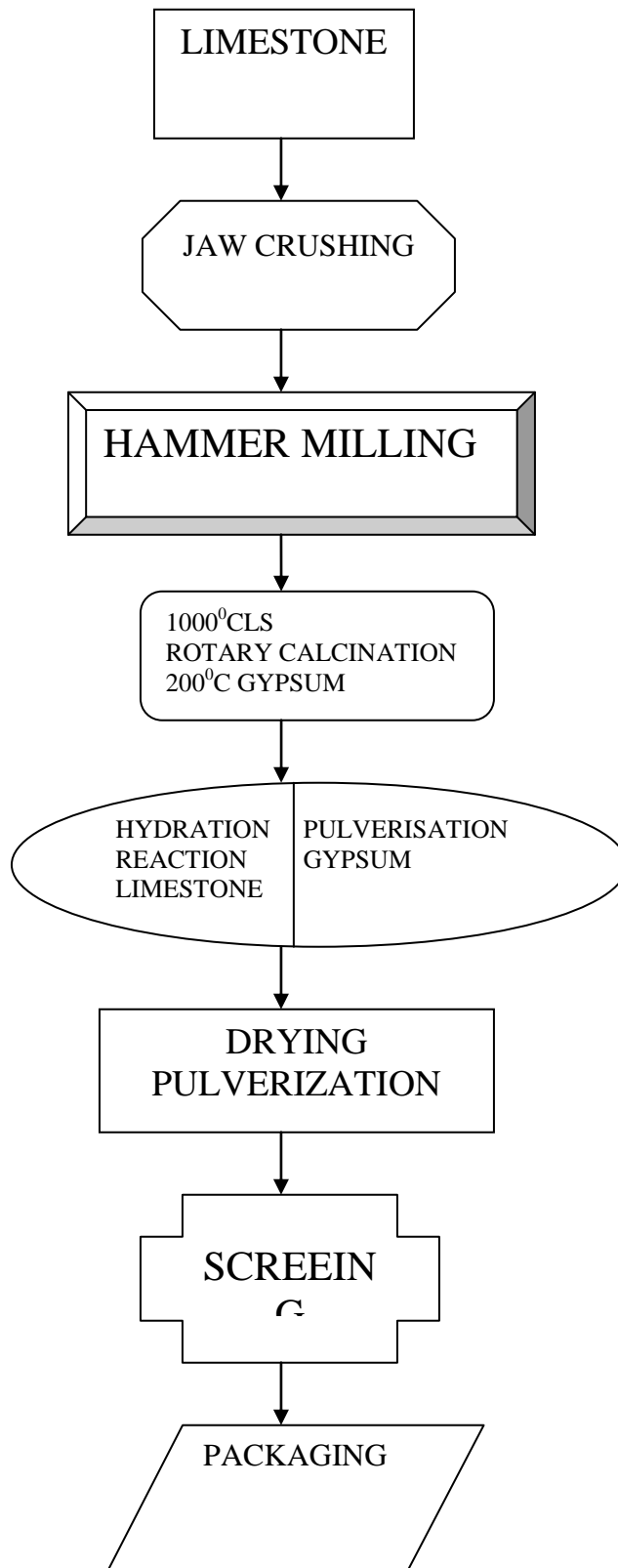
synthering, reaction bonding and nanotechnology. The basic material beneficiation of kaolin refining and the calcination process techniques are shown in flow-charts A - D.

(A) CERAMIC RAW MATERIAL BENEFICIATIONFLOW CHART

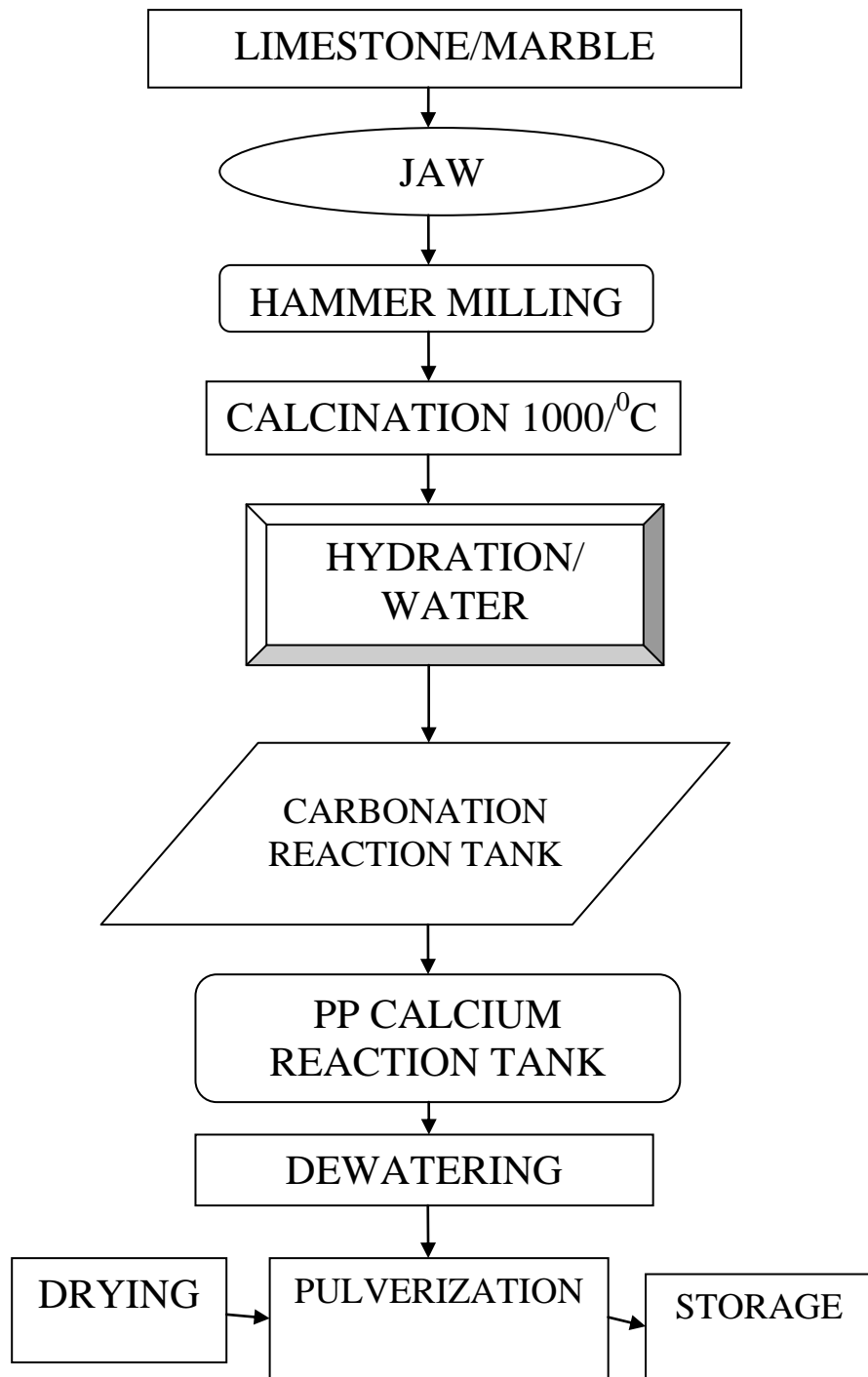


(B) KAOLIN PROCESSING AND REFINING FLOW CHART

[C] LIMESTONE/GYPSUM CALCINATION REFINING FLOWCHART



[D] LIMESTONE REFINING FOR CALCIUM CARBONATE PRODUCTION FLOW CHART



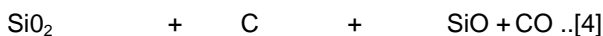
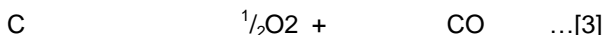
ADVANCE MATERIALS SYNTHESIS

Materials research and development has advanced beyond the basic beneficiation and process techniques to a more complex and sophisticated systems. One of such new innovations is the carbothermic reduction synthesis of high technology materials. The carbothermicreduction technique was used in the course of this work based on work by Acheson in 1893 when he used a mixture of silica sand and solid carbon to produce silicon carbide, SiC, at above 2000°C according to the following reactions:



Though the role of the gaseous species and the partial pressures were not understood then but this reaction formed the basis for the carbothermal reduction process technique. This novel technique is cheap and simple with possibilities for low temperature synthesis. The reduction reaction above could not adequately describe the SiC formation mechanism as it is known that SiO₂ first

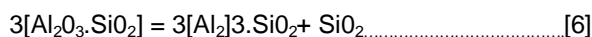
decomposes at high temperature to SiO and O₂ according to this equation:



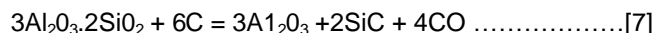
The SiO is then reduced by carbon thus:



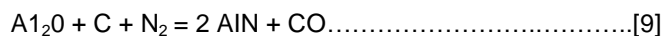
Other advanced materials have been produced using this technique. By using kaolin material, an aluminosilicate, as precursor in the carbothermal process, silicon carbide, silicon nitride or aluminium nitride can be produced accordingly:



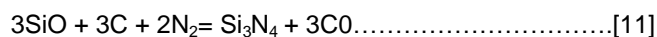
The silica formed above is cristobalite which is consumed by carbon to form silicon carbide in an overall reduction reaction thus:



Similarly, by using any selected special gas, AlN can be synthesized:



And to produce silicon nitride, Si₃N₄ the following reaction applies:



2. RESULTS AND DISCUSSION CHEMICAL ANALYSIS RESULTS

S/No	Material	SiO ₂	Al ₂ O ₃	Fe ₂ C>3	TiO ₂	CaO	Na ₂ O	K ₂ O	MgO	LOI
1.	Kaolin [Abeokuta]	45.90	35.10	2.40	1.95	0.05	0.06	0.71	0.06	12.80
2.	Kaolin [Auchi]	47.30	36.80	0.54	0.94	0.08	0.07	0.31	0.08	12.10
3.	Kaolin [Katsina]	46.90	38.60	0.68	0.05	0.06	0.01	0.29	0.07	13.40
4.	Kaolin [Jos]	47.30	37.30	0.95	0.48	0.13	0.01	0.62	0.26	13.0
5.	Limestone [Shagamu]	1.50	0.20	0.30	[80+]	45.40	-	-	0.60	38.30
6.	Limestone [Okene]	6.80	9.30	2.10	[85+]	48.10	-	-	5.50	41.8
7.	Limestone [Ukpilla]	3.25	1.10	0.20	[90+]	52.40	-	-	2.10	40.2
8.	Gypsum	0.68	-	0.05	SO ₂ 41.1	33.40	-	-	0.40	-
9.	Gypsum	0.50	-	0.8	SO ₂ 48.5	36.0	-	-	0.35	4.10
10.	Gypsum	1.20	-	0.2	SO ₂ 51.3	35.0	-	-	0.2	8.5

ANALYSIS OF THE PLASTER OF PARIS PRODUCT

S/No	Gypsum Plaster	CaO	SOi	MgO	Fe2O3	L.O.I	c/h ₂ o
1.	IGBOKOTO [POP]	29.70	43.50	0.30	0.21	7.80	9.80
2.	IBESHE [POP]	32.40	45.80	0.26	0.15	6.20	10..60
3.	POTISKUM [POP]	36.10	47.30	0.33	0.09	4.30	12.40

PHYSICAL PARAMETERS

S/No.	Gypsum Plaster	Density g/cm	Strength MOR N/mm ²	Colour Res. [120]	Sieve % Res. [120]	Initial Setting Time [sec]	Final Setting Time[mms]
1.	IGOKOTOR [POP]	2.59	3.76	80+	3.20	30-8.0	7 - 3 0
2.	IBESHE [POP]	2.60	2.98	85+	3.50	30 - 70	6 - 3 0
3.	POTISKUM [POP]	2.62	4.71	90+	3.17	30-60	5-35
4	Process Calcination Temperature / Time – 160 – 200°C/3-4 hours Water: Plaster ratio – 70: 30 Particle size - >75µm [70 – 90%/200mest.]						
5							
6							

FIG. 1 GRAPH OF IBESHE P.O.P

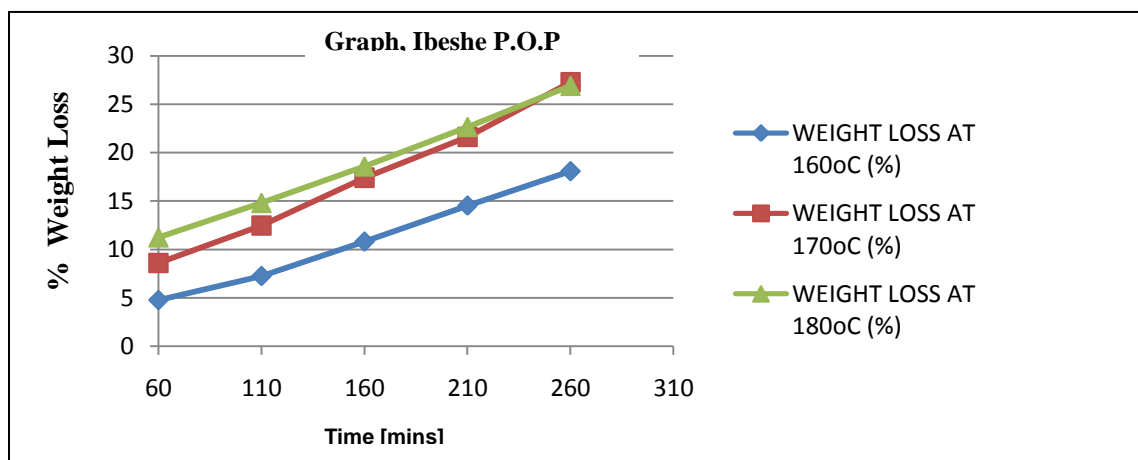


FIG. 2 GRAPH OF IGBOKOTOR P.O.P

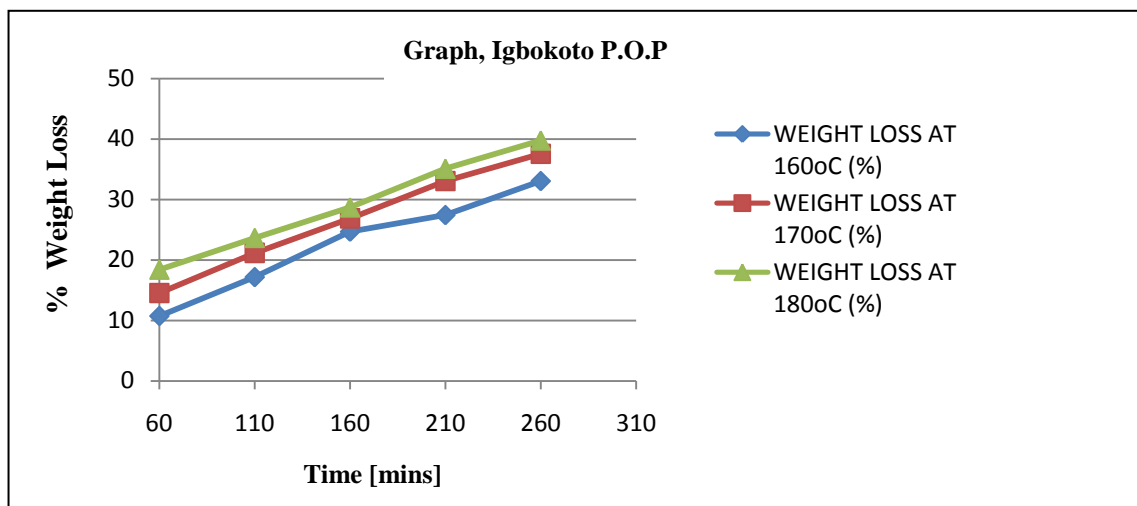
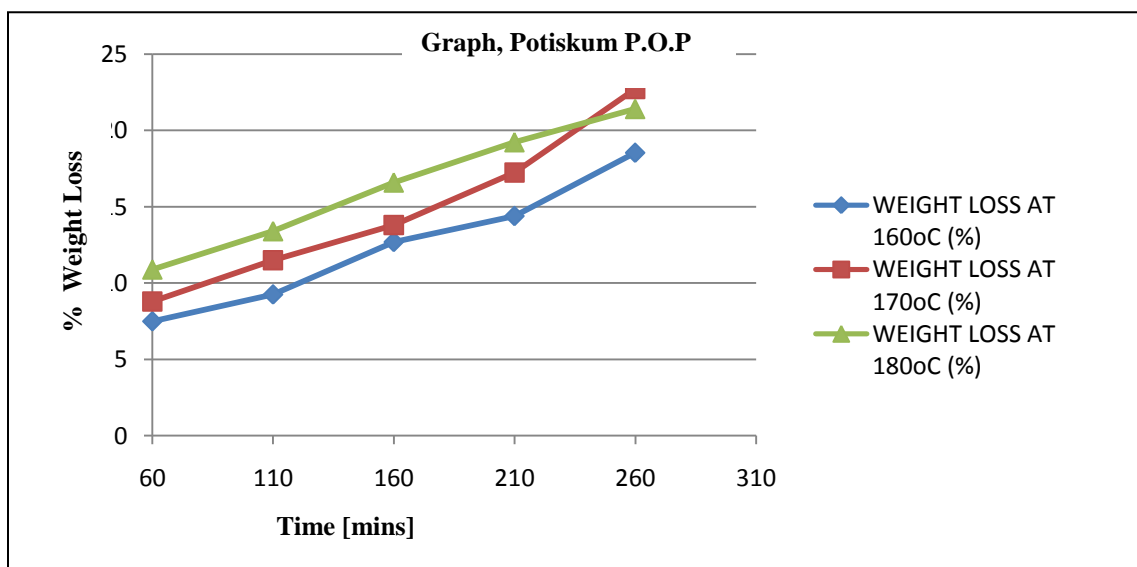


FIG. 2 GRAPH OF POTISKUM P.O.P



3. DISCUSSION

The results of the work reported in this paper have shown clearly that the quality of the materials investigated compares adequately with required standards. The chemical analysis results indicates the various levels of the constituents oxides in which it was convenient to know the degree of contaminating impurities and assist in determining the level of processing and beneficiation. The products obtained from the refining of kaolin, limestone and gypsum were of the highest quality, with the kaolin from Kankara in Katsina state, giving the best quality kaolin product with 98% brightness. While Ukpilla limestone produced the best quality calcium carbonate in terms of brightness, CaCO₃ content, while the Sagamu and Okene varieties were within the acceptable limits. The plaster of paris[P.O.P] produced was of good quality and they met the required standard both for industrial and medical applications. There is absolute need for greater focus to be given to the raw materials development because the

current level of development activity does not offer convincing signs for useful progress. All the government and private initiative on raw materials development are not yielding positive vibration due to lack of synergy between the Federal, State and other agencies that are required to motivate stakeholders and champion research and development in the sector. This is evident from the various government policies and institutional frame work that have failed to make progress from policy summersault to inadequate funding. The development of the nation's mineral raw materials, metallic and non-metallic, offers the greatest potentials for effective exploitation, utilization and industrialization of the country as they are used in a wide range of petroleum, cement, ceramics, glass, refractories, construction among others capable of providing employment for the majority of the populace.

4. CONCLUSION

The picture of ceramic raw materials development in Nigeria is absolutely black because there is no sustainable policy and investment profile that point to a positive direction. Any nation that is developed or developing positively must accept the fact that there are two major industries that drives the manufacturing sector and they are simply:

- the primary raw materials industries
- the secondary manufacturing industries.

Without a properly developed raw materials industries, the manufacturing sector can only wobble and often fail due to poor product delivery thus exposing the nation of material import dependent. Though government cannot do everything, its role is significant at this low level of our development and must provide the enabling environment to attract investment and industry such as stable power supply, roads/rail infrastructure, fund accessibility and human capital development.

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