

## Analysis and Development of Igbokoda Clay as a Binder for Synthetic Moulding Sand

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### ABSTRACT

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Clay obtained from Igbokoda in the southwestern part of Nigeria has been analysed and developed as a binder for synthetic moulding sand. The analysis was compared with the analytical results obtained for the pre-treated bentonite clay imported from the U.S.A. and used by the Nigerian Foundries Ltd. (NFL). X-ray analysis indicated the presence of kaolin, illite and montmorillonite (parent mineral of bentonite), with kaolin having the highest percentage in the Igbokoda clay and montmorillonite was further confirmed by the flame test which shows the existence of exchangeable cations not present in kaolin. The effects of mixing varying amounts of clay, tempering water and sand on moulding properties were investigated. The green and dry strength and shatter index of the mixtures were determined. The green and the dry strength (both in shear and compression) of the Igbokoda test clay, mixed with the subangular lagoon silica sand and determined at optimum water content, give good values for synthetic moulding sand. The shatter index test shows a decrease in collapsibility as the water content decreases at constant clay addition. Igbokoda clay is more collapsible but less tough than bentonite as shown by the shatter index.

### INTRODUCTION

In general practice, moulding sand and dry sand are most commonly bonded with clay. In the natural moulding sands the clay occurs in association with the sand grains, whilst the synthetic sands are bonded with selected clays from separate deposits. Testing techniques for moulding-sand have been developed and the constitution and properties of foundry bonding clays have been comprehensively examined (Grim and Cuthbert, 1945; Taylor and Diran, 1952; Heine et al., 1958; Lawrence, 1961; Wenninger, 1963, 1968; Sanders and Doelman, 1967, 1969). However, the present work concerns the analysis and development of an available local clay deposit.

In this work, the composition of the clay was determined by X-ray diffrac-

tion and chemical analyses. Further to determine its suitability for use as a binder in synthetic moulding sand, mechanical and physical tests were carried out on green and dry sand test samples without any other additives. Efforts are still being made in the development of available local raw materials for industrial use, world-wide and particularly in the developing countries. This, no doubt, will contribute to the research interest created in this paper. Though clay is available in large quantities in different riverine areas of Nigeria, no particular industrial use has been made of it. There is also no known research on the analysis and development of the available clay to determine its suitability for industrial use.

This investigation is therefore a part of an on-going effort to develop an efficient foundry sand binder from available local clay deposits.

The clay used in this study was collected from Igbokoda in the riverine area of Ondo State in the southwestern part of Nigeria. The vast reserves of silica sand ( $\text{SiO}_2 > 98\%$ ) at Igbokoda make the area self-sufficient in moulding sand. A foundry industry using 100% local sand/clay is thus considered viable in the area if the available clay could be developed and found suitable as an efficient foundry sand binder. Further research work in the development of this clay for foundry use is anticipated.

## EXPERIMENTAL PROCEDURES

### *Preparation of test samples*

The as-received clay contains a large amount of coarse particles, mainly quartz. Because of their widely varying size ratios, the clay and the quartz were separated using gravity sedimentation techniques, followed by wet-sieving to remove light, coarse particles that might float in the suspension.

The as-received wet clay was dissolved in water and thoroughly dispersed by  $1.5 \text{ g/dm}^3$  Calgon (sodium hexametaphosphate). After stirring for about 30 min, the content of the jar was allowed to settle for about 5 min. The quartz was observed to be sedimenting and the clay particles to be in suspension. The quartz sediments and the clay in suspension were separated by decantation. The sediment was discarded while the suspension was retained for subsequent separation.

The suspension, which was 100% clay and silt, was sedimented using centrifugal sedimentation methods. The separation was carried out in a laboratory centrifuge. This consists of four  $55\text{-cm}^3$  tubes radially arranged and traces a 7-cm radius when rotating. The tubes were filled with the slurry obtained from the gravity separation suspension and allowed to rotate at 15,000 rev./min for 30 min. The centrifugal force was calculated to be 19 kN. The clear water on top was then decanted and the thickened slurry removed from the bowls for subsequent dewatering operations.

This slurry was dried in an oven at 50°C for 16 h. It was virtually impossible to increase the temperature so as to reduce the drying time because of the characteristic changes that might occur at higher temperatures. The dried mass was crushed in a laboratory mill and subsequently sieved (90  $\mu\text{m}$  sieve) to further ensure the purity of the clay.

#### *Loss on ignition test*

Five grams of the dry material was heated for 2 h at 1000°C in a glass crucible, using the laboratory muffle furnace. After ensuring constant weight after ignition, the residual clay was measured and the ignition loss calculated (Table I).

#### *X-ray analysis*

X-ray diffraction patterns of Igbokoda clay and NFL bentonite were obtained using a Phillip's diffractometer and chart recorder. Iron  $K\alpha$  radiations with a Mn filter were used. The scanning conditions were .28 kV and 12 mA, 2°  $2\theta$ /min scanning speed, 1 cm/min recording speed and a time constant of 4.0. The range was  $4 \cdot 10^2$  cps. The results obtained were analysed (Tables II, III).

#### *Classical analysis*

Comparative tests were carried out on the bentonite and Igbokoda clay (Table IV) with the aim of determining the intensity of the emitted radiation based on  $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{Ca}^{2+}$ . In the flame photometer, a suitably diluted solution of the clay samples was sprayed into a flame producing the characteristic radiation. The intensity of radiation was measured with a photomultiplier.

TABLE I

Physical properties of Igbokoda clay

Property	Value
% Clay bond	
(1) as received	78%
(2) quartz-free	100%
Specific gravity	2.22
Colour	grey
Loss on ignition	
% at 100°C	15.7

TABLE II

Diffraction angles and corresponding *d*-values of Igbokoda clay

Peak	$\theta^\circ$	$d(\text{nm}) \frac{0.96865}{\sin \theta}$	Related clay minerals	<i>d</i> -values of known minerals (nm)
1	3	1.847	Montmorillonite	2.15
2	7.5	0.732	Montmorillonite	0.754
3	12.3	0.453	Montmorillonite	0.453
4	13	0.432	Illite	0.431
5	15	0.374	Kaolinite	0.374
6	15.5	0.362	Illite	0.363
7	16.7	0.337	Kaolinite	0.338
8	21.8	0.260	Montmorillonite	0.259, 0.260

TABLE III

Diffraction angles and corresponding *d*-values of NFL bentonite

Peak	$\theta^\circ$	$d(\text{nm}) \frac{0.96865}{\sin \theta}$	Related clay minerals	<i>d</i> -value of known minerals (nm)
1	3.75	1.481	Montmorillonite	1.5
2	12.4	0.453	Montmorillonite	0.453
3	13	0.431	Illite	0.431
4	15	0.374	Kaolin	0.375
5	15.5	0.363	Kaolin	0.362
6	16.2	0.348	Kaolin	0.348
7	16.7	0.337	Kaolin	0.338

TABLE IV

Chemical composition of NFL bentonite and Igbokoda clay

		A (bentonite)	B (Igbokoda clay)
K <sup>+</sup>	(ppm)	100	300
	(%)	0.1	0.03
Na <sup>+</sup>	(ppm)	13,000	1710
	(%)	1.3	0.171
Ca <sup>2+</sup>	(ppm)	1360	1120
	(%)	0.136	0.112
Fe	(ppm)	6700	8080
	(%)	0.67	0.808

### *Testing of moulding sand*

Details of the tests, testing procedures and equipment used have been described in the AFS Foundry Sand Handbook (Anonymous, 1963). The silica sand (80, 85, 89, 91, 93, 95 and 98% by weight), clay binder (20, 15, 11, 9, 7, 5, 3% by weight), and tempering water were thoroughly mixed manually for 10 min and stored in covered plastic jars to prevent air-drying. At each addition of an amount of sand mixture adequate to form standard 50 mm diameter and 50 mm high, test pieces after three rams was measured out. After ramming three times, the test pieces were then used in standard green compression and shatter index test (Anonymous, 1963). The loading rate in the compression tests was 13.6 kg/min. The tests were repeated on three similar samples and the average determined. Also, three standard test pieces were made for the dry compression and shear tests. They were baked for 2 h at 160°C and cooled in air to ambient temperature for about 30 min before testing on a universal strength test machine. Averages of the test results were recorded.

## RESULTS AND DISCUSSION

### *X-ray analysis*

The X-ray diffraction pattern obtained ( $2\theta$  is 2°–40°) confirmed the presence of high-quality montmorillonite with small amounts of kaolin and illite (Tables II and III). Igbokoda clay (Table II) was shown to contain kaolin as the predominant mineral with appreciable quantities of montmorillonite and illite.

The imported bentonite (Table III) possessed the typical pattern of a three-layer mineral, while the Igbokoda clay diffractogram presented the typical pattern of a two-layer mineral. The degree of crystallinity and  $d$ -spacing varied in both cases due to random orientation of the grains and suppression of some peaks by impurities, especially in the quartz-free untreated Igbokoda clay.

### *Chemical analysis*

The chemical analysis (Table IV) shows the presence of Na<sup>+</sup>, K<sup>+</sup>, Ca<sup>2+</sup>, and Fe<sup>2+</sup> in both clays along with the alumina and silicates peculiar to all clays. Sodium is the most potent exchangeable cation in Igbokoda clay even though proportionately less than in the bentonite. It was not observed to swell when water was added (as does bentonite). K<sup>+</sup> and Ca<sup>2+</sup> are also present in lesser quantities. Its iron (Fe) content is greater than that of bentonite, presumably due to the fact that the Igbokoda clay was not pretreated to eliminate iron compounds and other likely impurities that do not contribute to the bonding

ability of the clay. But the bentonite, being a standard bonding clay, had been chemically treated to remove these impurities.

The high sodium content in the bentonite confirmed its swelling characteristic. Thus it can be classified as a swelling or sodium montmorillonite. Swelling is broadly due to two main causes; adsorption of water in the surface layers of the lattice, which is hydration concerned with the exchangeable cation; and a second stage in which the unit layers are forced apart as a result of repulsion which is similar to the forces due to osmotic pressure and known as osmotic swelling (Grim, 1953; Stephens and Waterworth, 1968).

The calcium ion  $\text{Ca}^{2+}$ , present in clays as an exchangeable cation, does not possess the swelling quality of  $\text{Na}^+$ . Therefore, the montmorillonite present in Igbokoda clay is probably  $\text{Ca}^{2+}$ -based, while the  $\text{Na}^+$  locked within the illite is also present. The analysis classifies the Igbokoda clay as sodium (calcium) aluminium iron hydroxide silicate. However, it could be said that kaolin, the predominant mineral in Igbokoda clay, is  $\text{Al}(\text{Si}_2\text{O}_5)(\text{OH})_4$ , which shows the degree of substitution and cation exchange capacity to be low. However, illitic clays usually contain more than 3–7%  $\text{K}^+$  (Worall, 1975). Analysis of Igbokoda clay indicates a very low illite content (0.03%  $\text{K}^+$ ). This then shows that it is not illitic clay.

### *Green strength*

The green strength of a clay test material is the compressive stress in  $\text{kN/m}^2$  necessary to cause rupture of a standard cylindrical specimen using a compression-testing machine. A green sand contains clay and water, as well as the principal sand constituent,  $\text{SiO}_2$ .

Figs. 1, 2 and 3 show the effect of the various clay and tempering water contents on the green and shear strength of both the Igbokoda clay and bentonite-bonded sands. Both increased steadily as the clay content increased, but decreased after reaching an optimum as the tempering water increased. For each clay type there was an optimum water content. Too much water causes excessive plasticity, and too little fails to develop adequate strength. These effects could be attributed to the polar characteristics of clay, as a result of which they are able to adsorb strong polar molecules, especially water molecules. The adsorption results in the formation of thin water layers around the clay particles and is thus responsible for the plasticity of clay (Taylor et al., 1966). The first layer adjusts to the electric field of the clay and a second layer may be attracted to the first and so on. The attraction becomes weaker from the second layer on and is progressively weaker for successive layers. Sodium montmorillonite reaches its maximum strength when the film is about three molecules thick, an excess of which makes the system lose its strength (Taylor et al., 1966). Calcium montmorillonite attains a maximum strength when the film is about four molecules thick. This confirms the higher water sensitivity

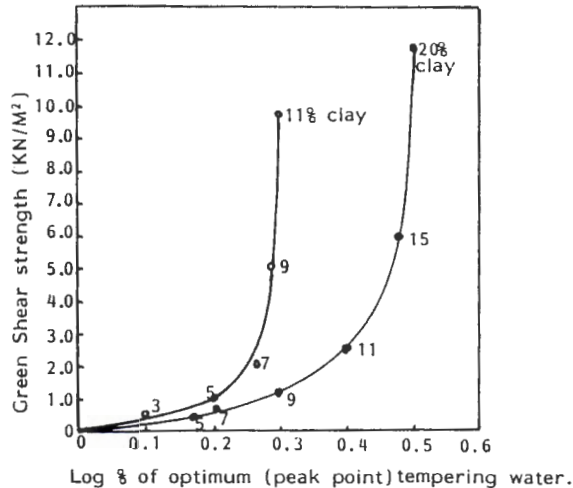


Fig. 3. Effect of optimum tempering water on the green shear strength of Lagos lagoon sand bonded with Igbokoda and NFL imported bentonite clays. ● = Igbokoda clay; ○ = bentonite clay.

of bentonite-bonded sand compared with the Igbokoda clay-bonded sands. However, the higher strength of the former for a particular clay content will be due to its better cation exchange capacity and hence higher bonding strength.

As the clay content increased, the differences in the green compressive and shear strengths of both clay bonds increased. This is probably due to the fact that with more clay the exchange capacity increased more rapidly in the bentonite than in Igbokoda clay, thereby enhancing the better bonding property in the former than in the latter. It could also be explained that the increasing discrepancy at high clay contents is due to the reduction in the dependence of the strength of the sand/clay mixture upon the sand strength.

It was also observed that the Igbokoda clay lost its moisture faster than bentonite when placed in the open. This seems to suggest that the bentonite clay has a comparatively higher adsorption and retentive ability than the Igbokoda clay.

#### *Dry strength*

The dry compression strength of the materials was obtained by drying the specimens in an oven at 100–110°C for 2 h as previously indicated. The universal testing machine was used to determine the strength following the same procedure as for the green strength, but with higher loads used.

Fig. 4 shows the effect of clay additions on the dry compressive and shear strengths of both samples. Both increased as the tempering water was increased. This dry-strength behaviour, in particular, is the reverse of that for the green strength which diminished with increasing water content after

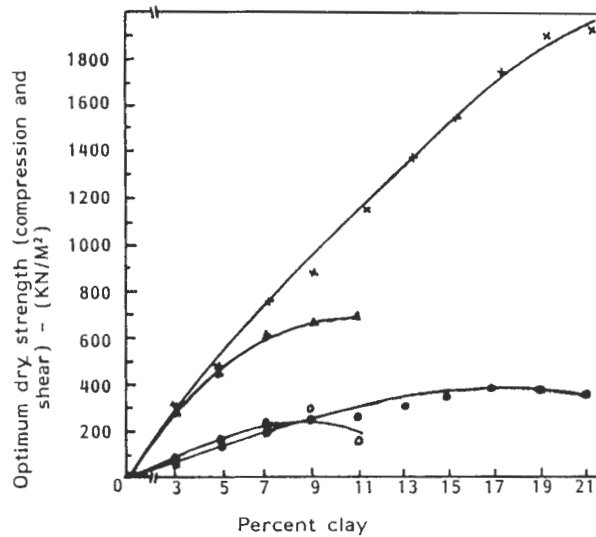


Fig. 4. The effect of percentage clay addition on the optimum dry compression and shear strength of Lagos lagoon sand bonded with Igbokoda and bentonite clays.  $\times$  = compression strength curve (Igbokoda clay);  $\bullet$  = shear strength curve (Igbokoda clay);  $\Delta$  = compression strength curve (bentonite clay);  $\circ$  = shear strength curve (bentonite clay).

reaching an optimum. That this behaviour of the dry strength was largely due to improved distribution of the binder and the higher bulk densities attainable (Beely, 1982), seems reasonable. Although the dry strength of bentonite-clay bonded sand was generally higher than that of Igbokoda clay bonded sand, differences at higher clay content did not increase as in the green strength. This was the case since water content does not contribute primarily to dry strength.

A possible source of bond strength is the surface tension of the water surrounding the clay and sand-clay particles (Caine and Toepke, 1967, 1968; Wenninger and Lang, 1969), and filling the capacity interstices, particularly those of the clay particles. It could thus be inferred that the bond strength is caused by the surface layers of water acting on a stretched membrane, forcing the particles together. As the water layer becomes thinner by drying, the forces holding the particles together increase (Taylor et al., 1966). This also explains the higher dry strength of the moulding sands than their green strength.

A decrease in the dry shear strength of bentonite-bonded sand was obtained at 11% clay addition (Fig. 4). Repeated tests gave the same result. This trend was considered to be due to clay saturation for a particular water content in which the transverse fracture on the stretched interparticle membrane easily overcame the polar bonds.



### Shatter index

Fig. 5 shows the effect of clay additions on the collapsibility of the green sands, while Fig. 6 shows the effect of clay additions on toughness, all as a measure of the shatter index.

The Igbokoda clay showed a continuous decrease in collapsibility (Figs. 5 and 6), both with the clay content and tempering water percentage. Similarly, the toughness increased with increasing clay and tempering water addition, changing from a concave shape to a convex curve. The Igbokoda clay did not reach saturation within the limit of the experimental period.

With the bentonites with low clay contents (3, 5, 7%), the collapsibility decreased with moisture content while toughness increased. At higher clay percentages, however, the collapsibility passed through a maximum.

The deformation or plasticity of moulding sand is determined from the green compression test (Beely, 1982). The property can be related to the potential resistance of moulds to hydrostatic pressure and to casting contraction.

The product of deformation and green strength has been used as an index of toughness (Beely, 1982). That is: moulding sand toughness number =  $d \times s \times 100$ , where  $d$  = deformation ( $\mu\text{m}$ ), and  $s$  = green compression strength ( $\text{kN}/\text{m}^2$ ). Hence, considering this direct relationship of green strength to toughness, the curves obtained both for the bentonite and Igbokoda clay bonded sands were not unexpected. Collapsibility measures the fragility of the moulding sand. Since collapsibility is the inverse of toughness, its tendency to increase with decreasing clay content could be justified.

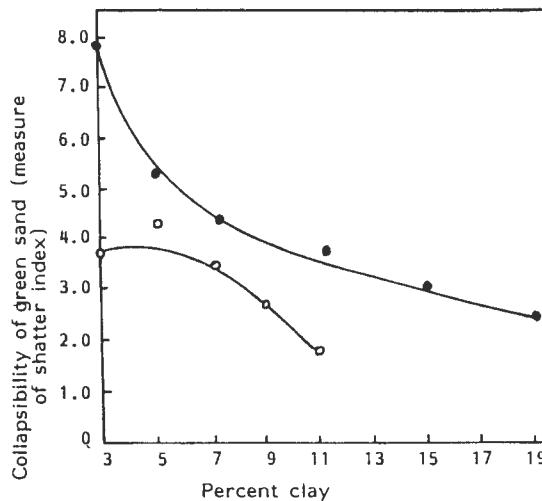


Fig. 5. The effect of clay addition on the collapsibility (shatter index measure) of Lagos lagoon sand bonded with Igbokoda and the NFL bentonite clays. ● = Igbokoda clay; ○ = bentonite clay.

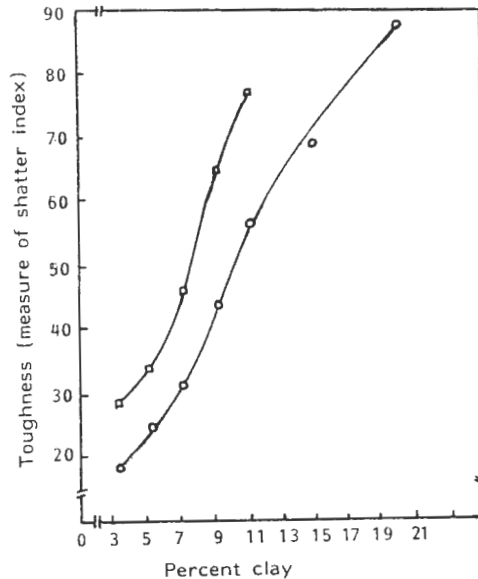


Fig. 6. The effect of clay addition on the toughness (measure of shatter index) of Lagos lagoon sand bonded with Igbokoda and the NFL bentonite clays. □ = bentonite clay; ○ = Igbokoda clay.

In the dry-strength analysis of bentonitic sand, the dry-shear strength passed through a maximum, as also the toughness. This phenomenon could be due to an optimum condition being reached in the sand-clay-water saturation system. The excess moisture increased the plasticity which made it easily deformable and hence a decrease in toughness with an attendant increase in collapsibility. Thus, plotting the optimum shatter index against clay content showed an increase in collapsibility of up to 5% and a gradual decrease as the clay content was increased. There was a steeper increase in toughness for bentonite-bonded sand because of its greater sensitivity to water additions.

#### CONCLUSIONS

(1) Igbokoda clay consists of shale minerals such as illite, kaolinite, montmorillonite, with kaolinite being the base clay and montmorillonite, most responsible for good moulding properties, present in a smaller quantity. Chemical analysis showed a fairly high iron content.

(2) The imported bentonite contains more sodium (swelling) montmorillonite than calcium (non-swelling) montmorillonite as is evident from the chemical and X-ray analysis and the high dry strength not attainable by calcium montmorillonite.

(3) Optimum green strength for the bentonite addition to sub-angular lagoon sand occurred at 9% clay addition and 2% tempering water. Igbokoda

clay exhibits an optimum green strength at 15% clay addition, and 3% tempering water.

(4) The shatter index properties of both clays are comparatively suitable as binders for synthetic moulding sand.

(5) The practical implication of these findings would seem to be that with a higher clay content, the Igbokoda clay could be substituted for the bentonite. With pretreatment of the clay, the optimum green strength could be attained with a lower percentage of clay addition; and a blend of both the bentonite and the Igbokoda clay would also give good moulding properties.

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## Determination of cations and anions in the ashes of some medicinally used tropical woods\*

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**Summary.** The ashes of some medicinally used tropical woods were characterised. These were *Azelia africana*, *Alstonia congensis*, *Antiaris africana*, *Terminalia ivorensis*, *Azadirachta indica*, *Ricinodendron heudelotti*, *Chlorophora excelsa* and *Daniellia ogea*. The ashes were obtained by treating wood in a muffle furnace, after which the ashes dissolved in distilled water. The very alkaline solutions obtained were analysed for  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$  and  $\text{PO}_4^{3-}$ . Due to wide medicinal application of these wood species experiments were made to determine the cations and anions which may be active ingredients in the compounds (drugs) used in the treatment of different ailments.

### Introduction

About one-third of the world's land surface is covered with forests with the equatorial rain forest accounting for about 18%. Among the large number of direct uses, the use of wood for fuel has probably been the most significant. Of the uses of industrial wood, lumber is the most important especially in construction of housing; furniture industry is also a major market for both softwoods and hardwoods. Besides these uses, most of these woods are of medicinal importance in the tropics where the bark, root and leaves are used in making drugs to cure several types of ailments. It is also possible to synthesise drugs from these compounds or intermediate compounds that may be extracted from the wood. (Irvine 1961; Tedder et al. 1975).

The preparation of macrocyclic compounds that may be of medicinal importance was found to be unsuccessful except in the presence of metal ions (Conlon et al. 1973). Yields of modified porphyrins are low in the absence of metal ions, while the use of a metal ion as a template is only applicable provided they can form metal complexes (Broadhurst et al. 1972). Transformation of these large macrocycles may involve the use of central metal ions as found for magnesium in chlorophyll and iron in haemoglobin. The introduction or presence of metals into or in macrocycles can cause striking variations in reactivity, and this may seriously affect the potency of the drug from such macrocycles.

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