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PHYSIOCHEMICAL ANALYSIS OF CLAY SAMPLES IN SOME PARTS OF SOUTHEASTERN NIGERIA

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ABSTRACT: *Nigeria is enriched with abundant mineral and natural resources; besides hydrocarbon resources, the exploration and mining of Nigeria mineral resources has not been given adequate attention it requires. In lieu of that physiochemical analysis of clay samples collected in some parts of Southeastern Nigeria were carried out in other to ascertain the physical and chemical constituents of the clay samples. The clay samples were collected from three states in southeastern Nigeria namely; Ozubulu clay from Anambra State, Ngwulangwu clay from Ebonyi State and Ngwo White clay from Enugu State. The physical properties of these clay samples were determined under the following: Moisture Content, Ash Content, Iodine number, pH Reading, Surface Area, Bulk Density/Porosity and Loss of Ignition. The chemical analysis was carried out so as determine the metallic oxide present in the clay using Atomic Absorption Spectrometer (AAS). The result shows that Ngwo white clay has the highest percentage of moisture content of 8.00%, followed by Ozubulu clay 7.50% and then Ngwulangwu clay 7.00%. Also, high bulk density observed from these clays shows that they can be used as bleaching clay for high oil retention ability. The pH readings of the clay samples showed that they were all acidic. The iodine numbers of the three samples were high which shows that they can be used for agricultural purposes. Also, the result showed that the ratio of Na_2O to CaO is greater than one for Ozubulu clay and Ngwo white clay samples which indicates the presence of swelling bentonites. The Iron oxide of Ngwulangwu clay is higher when compared to Ozubulu and Ngwo white clays. The Ferrous oxide impacted a red colour on the fired sample of Ngwulangwu clay. This high iron oxides of Ngwulangwu clay made the clay attractive and suitable for structural engineering works due to its mechanical strength and plasticity. Finally, the results obtained from the study of the three clay samples showed that they can be used for industrial and commercial purposes. It was also recommended that further studies be carried out to explore other potentials of these clays for other industrial utilization.*

KEYWORD: Physiochemical, Clay, Refractory, Raw Materials, Resources, Nigeria

INTRODUCTION

Nigeria is enriched with abundant mineral and natural resources; besides hydrocarbon resources, the exploration and mining of Nigeria mineral resources has not been given adequate attention it requires. Delving into geological survey of Nigeria soil, it was reported that clay is one of the major Nigeria minerals deposits that covers an estimated proven reserve of millions of tones, and these minerals are discovered all over the states in the country Agha (1998). Although, Nigeria is blessed with these large deposits of clay but they are not properly harnessed due to lack of the awareness in knowing the importance of clays and its mineral content which can be used in the industries, laboratories and in agriculture. Clay is the major raw materials required for production of refractory materials. Refractory minerals are non-metallic materials that have unusual high melting temperature and maintain their structural properties at very high temperatures Nnuka and Agbo (2000). A material that has a very high melting point in addition to its physical, chemical, mechanical and thermal properties is said to be naturally refractory, which makes it suitable for use in furnace, kilns and other high temperature processes (Obadinma, 2000). Refractories are used almost in every industry where high heat is needed, such as cement, chemical, metallurgical, ceramic kilns, petrochemicals, steam boilers, and hot stoves (Jock *et al.*, 2013). The raw materials for the production of various refractory products include kaolinite (Al_2O_3, $2SiO_2$ and $2H_2O$), chromite ($FeOCr_2O_3$), magnesite ($MgCO_3$) and various types of clays, (Abdullahi and Samaila, 2007). Refractory materials are very useful and play very crucial roles in the industrial development of any nation. The Nigerian metallurgical industries are struggling today because of many factors which include short supply of refractory materials (Aliyu, 1996). Furthermore, there is enormous need for refractories in Nigeria industries; for instance, Borode, *et al.*, (2000) iterated that the Ajaokuta Steel Complex will require about 36,000 tons of refractory bricks on completion for furnace lining while Adondua, (1998) estimated that at full operation capacity, both Ajaokuta Steel Complex and Delta Steel will require 43,503 and 2500 tons/year of fire clay refractories for their activities respectively. Despite having extensive clay mineral deposits in Nigeria, the refractory needs of these industries were well over 300,000 tonnes as at the year 2000 (Omowumi 2001). Nigeria continues to depend on external sources of refractory materials for many of its industries (Aliyu, 1996). Nigeria imported about 27 million metric tonnes of refractory materials in 1982 showing how much Nigeria spends per year in the importation of refractory materials (Obadinma, 2003). Omowumi (2001) in his investigation concluded that the properties of refractory clay samples from onibode; Ara Ekity, Ibamajo and Ijoko compare favorably with imported fire clay refractories. Within last few decades, there have been tremendous research works towards creating awareness and developing refractory products from local clay deposits such works includes; Balogun *et al.*, (1980), Obikwelu, (1987), Loto, and Akeju, (1994), Onyemaobi, *et al.*, (1995) Agha (1998), Nnuka and Agbo (2000), Omowumi (2001) and John (2003). At the end of this research work the government and other multinational company will see the need to create awareness and develop refractory products from local clay deposits since it will aid in the development of this country through improving our economy and gainful employment.

The Aim of the Research

The aim of this work is to carry out physiochemical analysis of clay samples in some parts of Southeastern Nigeria**.** This was done in other to evaluate the type of clay and its mineral content that can be used in ceramic industries, cosmetics, pharmaceutical, paper and cement industries and the type that can be used in production of bricks and bleaching clay.

Materials and Methods

The Clay samples were collected from three states in Southeastern Nigeria namely:

Sample A: Ozubulu Clay from Anambra State

Sample B: Ngwulangwu Clay from Ebonyi State

Sample C: Ngwo White Clay from Enugu State

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The impurities like pebbles, grass roots were removed and some quantities of each sample were collected using polyether bags and sent to the laboratory for analysis. The samples were characterized in other to ascertain its physical and chemical properties.

The Physical Properties

The physical properties of the Clay samples were determined under the following:

i. Moisture Content

ii. Ash Content

iii. Iodine Number

iv. pH Reading

v. Bulk Density/Porosity

vi. Loss of Ignition

Moisture Content

Oven drying method was used in the determination of the moisture content of the samples. 1.0g of the sample was weighed in the Petri-dish. The weighed sample was placed in an oven and dried at about 105°C, for two hours (2hrs). The sample was brought out, cooled in a desiccator and weighed. The sample was reheated for 20 minutes, cooled and weighed. This process of heating for every 20 minutes, cooling and weighing was repeated until a constant weight was obtained.

The percentage of the moisture was calculated using the formula

$$\text{Moisture (\%)} = \frac{\text{Loss in weight on drying}}{\text{Initial weight}} \times \frac{100}{1}$$

Ash Content

0.1g of the sample was heated for 20 minutes, cooled in a desiccator and weighed. The cooled sample was heated again for another 20 minutes, cooled and weighed. This process was repeated till a constant weight was obtained which showed that the sample was totally ash.

Mathematically, the ash content was calculated using the formular

$$\text{Ash (\%)} = \frac{\text{Weight of Ash}}{\text{Weight of sample}} \times \frac{100}{1}$$

Iodine Number

To determine the amount of Iodine absorbed, 0.5g of the sample was weighed and put into a conical flask. 10m1 of 5% HCI was introduced into the flask and was shaked for proper blending until the sample was wetted. Then 100ml of the stock solution was added and agitated at a speed using an electric shaker for a period of one hour (1hr). The mixture was filtered through a sintered glass crucible.

An aliquot portion (20mI) was titrated with 0.1m sodium thiosulphate using starch as an indicator. The concentration of Iodine absorbed by the sample at room temperature was calculated as the amount of Iodine absorbed in Milligrams.

$$I(mg) = \left(\frac{B-S}{B} \times \frac{VM}{W} \times 253.81\right)$$

Where B = Volume of thiosulphate solution required for Blank

S = Volume of thiosulphate solution required for sample titration.

W = Mass of sample

M = Concentration (Mol) of Iodine solution 253.81 which is the atomic mass of Iodine

V= 20m1 aliquot.

Note: Preparation of stock solution 4.1g of potassium Iodide (KI) was weighed which was followed by 2.7g of Iodine crystals. The mixture was made up to 1 litre with distilled water. (KI was added first before Iodine crystals for proper dissolution).

pH Determination

0.25g of the sample was weighed and transferred into a test tube. 25m1 of distilled water was measured and added into the test tube. The mixture was stirred in an electric shaker for one hour (1hr). Then, the sample was allowed to stabilize or settled before the pH was measured using the pH meter.

Determination of bulk Density/Porosity

5g of the sample was weighed and placed into a dry measuring cylinder. The measuring cylinder was tapped at the top for the sample to settle or sediment at the bottom.

The final value or volume was read at the measuring cylinder.

Mathematically:

BD = Weight in gramme/ml

Loss on Ignition

0.5g of the clay samples each were weighed in a clean dry platinium crucible. The weight of the crucible was equally noted before weighing the sample. It was heated in a furnace to about 600°C for 3 hours, cooled in a dessicator and weighed.

$$LOI = \frac{\text{Weight Loss}}{\text{Weight of sample}} \times \frac{100}{1}$$

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Chemical Test Properties

Digestion of Clay Samples

0.1g of each of the samples was weighed into a TeFlon crucible and moistened with Aqua regia, and Hydrofluoric acid (HF) (Aqua regia is a mixture of Hydrochloric acid and Trioxonitrate V acid in the ratio of 3:1 by volume). The ratio of Hydrofluoric acid (Hf) and Aqua regia is 2:1 and this is added into the weighed sample. The mixture was covered and then heated in a fume chamber at 100°C until a clear Milky solution is obtained. As the heating is done, additional aqua regia and HF is added to avoid the crucible from drying up. The clear solution is allowed to cool and then transferred into a 250m1 volumetric Flask. The solution is made up to 250m1 mark with distilled water. The digested samples were used in the determination of the oxides.

Determination of the Oxides Using Atomic Absorption Spectrometer (AAS).

After the digestion of the sample, the recommended standard methods were used for the elemental quantitative analysis. AAS, Measurements were made using spectra AA-10 spectrometer, manufactured by varian spectra. Ari-acetylene flame was used for the analysis of each metal oxide except for Silicon (SiO_2), Calcium (CaO) and Aluminum (Al_2O_3) oxides, which required nitrous oxide-acetylene flame instead, and a flame temperature of 2250°C. The machine automatically controlled the ratio of the fuel and oxidant gas, and pure water used as the blank. An atomic absorption spectrophotometer model 1233 with air-acetylene flame was used to analyze Iron (Fe_2O_3), potassium (K_2O), sodium (Na_2O_3) and Magnesium (MgO) Oxides. From the results obtained, the percentages were calculated.

RESULTS AND DISCUSSIONS

The physiochemical characterization of clay samples involved both the physical and chemical analysis of clay samples in order to identify the various components present in the samples. The physical analysis involved six parameters: Moisture content, Ash content, Bulk density, pH, iodine number and Loss-on-ignition. The chemical characterization was carried out using Atomic Absorption spectrometry (AAS). Table 1 presents the result of three the physical properties analysis of the three clay samples.

Table 1: The Physical Properties Analysis of the three Clay Samples.

Sample A: Ozubulu Clay

Sample B: Ngwulangwu clay

Sample C: Ngwo White Clay

S/N	Parameter	Unit	Sample A	Sample B	Sample C
1.	Moisture content	%	7.50	7.00	8.00
2.	Ash content	%	3.00	11.00	1.00
3.	Bulk Density	g/ml	1.25	1.57	1.67
4.	pH	-	6.60	4.13	5.63
5.	Iodine Number	Mg/L	30.45	48.20	89.50
6.	Loss of Ignition	%	4.00	8.00	4.00

Physical Characterization

From the result as shown in table one, Ngwo white clay has the highest percentage of moisture content of 8.00%, followed by Ozubulu clay 7.50% and then Ngwulangwu clay 7.00%, which accounts for the plasticity and strength of the clay, this is in accordance with Murray (2000), which states that the plasticity and strength of clay depend on the amount of water present in the value of 1.2 to 14.26%. This makes the three samples suitable for molding, and when combined with sodium montmorilonite it can be used in the determination of the refractory ability of bricks.

The high bulk density observed in the three samples which fell within the range of 1.48 - 2.15g/I shows that the clay samples are suitable for bricks productions which is in line with works of Omotoyinbo and Oluwole (2008) and can also characteristic of fireclays, Omotoyinbo (2008). Furthermore, high bulk density observed from these clays samples also shows that they can be used as a bleaching clay for high oil retention ability which is in agreement with the works of Kirk and othmer (1972).

Moreover, the pH readings of the clay samples showed that they were all acidic with the values Ozubulu (6.60), Ngwulangwu (4.13) and Ngwo white (5.63). From our chemistry, an acidic component or substance is used in neutralizing any alkaline substance therefore the three samples can be used for alkaline or base soil treatment.

The Iodine number is used in the determination of the amount of iodine that is absorbed by the sample. The iodine numbers of the three samples were high as shown in table 1 which showed that the clays can be used for agricultural purposes as supported by the works of Nweke and Ugwu (2007)

Loss-on-Ignition is the combustion of volatile matter present in the clay. They are often required to be low. The loss-on-Ignition of the three samples is low which is lower than 18.00% specified for upper limit for refractory clays Nweke and Ugwu (2007)

Finally, the ash content of the Ngwo white is the smallest with a value of 1.00% followed by Ozubulu clay 3.00% and Ngwulangwu clay 11.00%. This showed that the non-combustible volatile matter content of Ngwo wrhite clay and Ozubulu clay is lower compared to that of Ngwulangwu clay. Table 2 shows the result of the oxide analysis of the three-clay sample using AAS

Chemical Characteristics

Table 2: The Result of the Oxide Analysis of the three Clay Samples.

S/N	Parameter	Sample A	Sample B	Sample C
	%			
1.	K_2O	0.16	0.605	0.121
2.	CaO	0.41	0.8	0.61
3.	SiO_2	40.00	58.9	53.2
4.	Fe_2O_3	0.56	1.721	0.444
5.	Na_2O	2.42	0.338	1.4175
6.	Al_2O_3	18.94	5.176	8.906
7.	MgO	0.25	0.20	0.75

From table two, the oxide analysis of the clay showed that the alumina (Al_2O_3) content for Ozubulu clay is 18.94%, this qualified the clay as a high melting clay but not as a refractory clay, this is because the oxide value of the alumina lies within the recommended range for melting clay as suggested by Nnuka and Agbo (2000). And most part of alumina is combined to form the structure of aluminosilicates as kaolinite. However, Ngwo white clay and Ngwulangwu clay did not meet the specified alumina value.

Also, the amount of SiO_2 content for Ngwulangwu clay and Ngwo white falls within the range for refractory clay (46.00% - 62.00%), (Abdullahi and Samaila 2007). This can be used for lining of heat treatment furnaces, melting furnaces for low melting point metals, liquid metal ladles and portions of blast furnaces Yami *et al.*, (2007). while that of Ozulubulu falls below the range for refractory clay. The large amount of SiO_2 present in Ngwo white clay and Ngwulangwu clay is associated with crystalline phase quartz when combined to Alumina in the alumino silicate structure as suggested by Borlini *et al.*, (1999). They further stated that when this is present in a clay, it decreases the intensity of the clay, although it may increase the refractoriness of the clay that has its standard to be 46-62%.

The high SiO_2 and Al_2O_3 content with low CaO content present in all the clays suggested that the clays are kaolinite in nature, this is in accordance with Ajemba and Onukwuli (2013). The clays equally showed considerable values of alkaline oxides, K_2O and Na_2O which contributes to the formation of the liquid phase. The earthy alkaline oxides MgO and CaO can also act as fluxes during the firing stage.

Also, the result showed that the ratio of Na_2O to CaO is greater than one for Ozubulu clay and Ngwo white clay samples which indicates the presence of a swelling bentonites and is in accordance with sun *et al*., (2006). The Iron Oxide of Ngwulangwu clay is higher compared to Ozubulu and Ngwo white clays. The Ferrous Oxide impacted a red colour on the fired sample of Ngwulangwu clay due to the conversion from Ferrous to Ferric compound. This colour variation is considered useful for the manufacture of flower pot and earthen ware as suggested by Rhode (1973). This high iron oxides of Ngwulangwu clay made the clay attractive and suitable for structural engineering works due to its mechanical strength and plasticity as suggest by Nnuka and Agbo (2000). Finally, when the results obtained from these clay samples are compared to that of Mayo-belwa clay, it suggested that these clays can be used as a binder in the absence of standard binder like phosphoric acid and is in accordance with work of Nwajagu and Aneke (2001).

CONCLUSION

It was shown in this work that on the basis of the physiochemical characteristics of clay samples, suitable components were discovered which can be used for local and industrial purposes. This was done using standard techniques. The oxides analysis obtained from high silica and alumina in the ratio that is more than one helped the researchers to obtain the mineral necessary for the synthesis of zeolite used as a catalyst in oil industry. From Ngwlangwu clay, its high Fe_2O_3 content gives the clay suitable property for brick and ceramic production. The high silica content also suggests presence of free quartz and kaolinite in Ozubulu clay and that when activated, can be used for the bleaching of palm oil and serve as substitute for imported commercial fuller’s earth. Furthermore, through characterization, useful insight was gained into the appropriate uses for these clays. It was recommended that further studies be carried out to explore other potentials of these clays for other industrial utilization.

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