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The Impact of Fine Sand Calcined at 450°C and 750°C on Textcoat and Emulsion Paints, Respectively

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Abstract

This study investigated the suitability of Nawgu clay as an alternative pigment extender in emulsion paint production. Clay collected from Ifiteora Village, Nawgu, Anambra State, Nigeria, was washed, dried, pulverized, sieved, and separately calcined at 450°C and 750°C for 2–3 hours. The raw clay was characterized using X-ray fluorescence and X-ray diffraction analyses, while the formulated paints were evaluated through physicochemical and quality-control tests. XRF analysis showed that the clay contained predominantly silica (49.79%), alumina (14.04%), and iron (III) oxide (6.21%), while XRD confirmed the presence of quartz and kaolinite. Two emulsion paint samples were produced using the calcined clays as extenders and compared with a

commercial control paint. The 450°C and 750°C formulations recorded moisture contents of 61.735% and 70.862%, ash contents of 28.998% and 21.850%, viscosities of 26.256 and 8.844 centistokes, pH values of 9.55 and 10.27, and specific gravities of 1.080 and 1.226, respectively. The 450°C formulation demonstrated superior performance to the 750°C sample, particularly in viscosity and moisture retention, although the commercial control exhibited better overall properties. The findings indicate that Nawgu clay has potential as a low-cost, locally available extender, but cannot yet completely replace conventional industrial extenders without further improvement in formulation and processing.

Keywords: Nawgu Clay, Emulsion Paint, Calcination Temperature, Pigment Extender, Physicochemical Properties

Introduction

In a growing economy like Nigeria, the need to sort for sustainable raw material for its industries should be paramount. The coating industry plays an important role in the growth of the economy and as such, there is need for sustainability in the industry. Over the years, there has been ongoing research to find alternative raw materials to primitive ones used in paint production.

Paint can be seen as any liquid pigmented, liquefiable or solid mastic composite, that after application to a surface in a thin layer, converts to a solid film. It often serve as protective, colouring pigment, and sometimes, provide thin cover to material surfaces [1, 2]. Paints can be made in varieties of colours which may be bought or produced locally in quite a number of varieties, such as water colour or synthetic paints. Paints are generally stored, sold, and applied as a liquid, however, most types dry into a solid pigment. Generally, paints are either oil-based or water-based and each have unique characteristics which are greatly influenced by the nature of extenders used in its formulation [3].

Extenders are inert substances which do not impact opacity to paints but are incorporated into paints for a variety of reasons. They modify various coating properties such as viscosity, adhesion, hardness, gloss, abrasion resistance, flow characteristics, settling tendencies, etc. The selection and proper blending of suitable extenders help to optimize several mechanical properties and the aesthetics of coatings.

Extender pigments are added in order to reduce the cost of a paint formulation. They are also used to modify the flow (viscosity), sedimentation stability and film strength. Most extenders appear white and possess a refractive index similar to commonly used binders. Majority of them occur naturally and others can be produced synthetically [4, 5]. Clay, Aluminium silicate, magnesium silicate (talc), silica, calcium carbonate (synthetic and natural) and barium sulfate are some commonly used extender in paints and coatings.

Most of the extender pigments used in coatings such as titanium dioxide (TiO_2) are of mineral origin and require long processing, including grinding, levitation, chemical treatment, etc, from the ore to the final stage, resulting in significant loss of materials.

Similarly, these processed extenders are expensive, and are not available in abundance, and this has led to increases in the cost of resultant coatings products. Titanium dioxide (TiO_2), a prime pigment which is used extensively in the paint industry is chemically active and contributes not only to the high cost of paints, but also, increases the rate of degradation of paint surfaces. Efforts are being made to find economically and technically viable extenders for use in coatings, and thus, bring down the cost of coatings products to acceptable limits. The aim of this study is to use Nawgu clay calcined at 450°C and 750°C as Pigment-extender in commercial paint formulation.

Materials and Methods

Equipment

For this project research work, the following materials were used; benchtop Energy Dispersive X-ray Fluorescence elemental analyzer Cotex XRD(cctenex,England), Electric Oven(Frigidaire GCRE3060AF, Japan) Electric Furnance (Winchester WE30B4D, Belgium) The ASTM international standard was used to characterize the extender pigments for specific gravity (ASTM D153-84), Uniscope PHS- 3E pH meter (Surgifriend medicals, England), DV2T Brookfield Viscometer (Brookfield Engineering Laboratories, USA), Digital weighting balance(WW-Y, China) stirrer, plastic sieves, crucibles, bowls.

Reagents

Hydroxylethyl Cellulose (Nitrosol), Titaniumdioxide (TiO_2), Calcium Carbonate (CaCO_3), Water (H_2O), Ammonia(NH_3), Polyvinylacrylate ($\text{C}_2\text{H}_4\text{O}$), Hexasodium hexametaphosphate (Calgon) ($\text{Na}_2[\text{Na}_4(\text{PO}_3)_6]$) Biocide (weight ratio of the hypochlorite to the peroxide was about 10:1)and formalin (CH_2O).

Sample Collection and Preparation

The local Nawgu clay sample was washed, sundried and ground afterwards. The fine powdered clay was then sieved and divided into three portions. First portion was calcinated at 450°C , the second calcined at 750°C and the third portion was left uncalcined. The metallic and oxygen composition properties of the uncalcinated portion were determined via X-ray fluorescence and X-ray diffraction. The calcined samples were used in the formulation of two separate emulsion paint. Sample A had the calcined clay at 450°C incorporated into it while sample B had the calcined clay at 750°C incorporated into it.

Formulae for the production of Emulsion Paints

The formulation of the emulsion paints with Nawgu clay calcined at 450°C and 750°C labeled as sample A and B respectively are given in the table below denoted as Table 1. The table shows the list of materials used in the production process, their function and the weight percentage in the formulation.

Table 1: Formulation of Emulsion paints with Calcium carbonate, Nagwu clay calcined at temperatures of 450°C and 750°C

Components	Functions	Calcined at 450°C Sample A (%)	Calcined at 750°C Sample B (%)
Water	Solvent	40	40
Calgon	Surfactant	1.5	1.5
Poly vinylacrylate	Binder	7.5	7.5
Genapol	Emulsifier	2.0	2.0
Formalin	Fungal growth Retardant	2.0	2.0
Biocide	Preservative cellulosic	2.0	2.0
Natrosol	Thickener	3.0	3.0
Ammonia	pH adjuster	2.0	2.0
Clay (450°C)	Extender	40	-
Clay (750°C)	Extender	-	40
Total;		100%	100%

Procedure for Production of Emulsion Paints

Here, the materials to be used were properly weighed out in gram and the weight of the crucible and beakers were noted.

- Water was added to an empty clean bowl, calgonPT (0.05) was dissolved and stirred for 2-5Minutes.
- Polyvinylacrylate was added and stirring continued.
- Calcium carbonate (1.75g) was added, this was followed by addition of Titanium dioxide (0.375g) and the stir continued for another 10minutes.
- The natrosol (0.1) was dissolved in water with another container for few minutes before being added, this was followed by addition of genapol (0.075), biocide (0.1), anti-fungal agent (0.1) and ammonia (0.075) respectively to the mixture. The mixture is stirred thoroughly for another 10minutes to obtain a homogeneous emulsion. The same paints produced were kept for analysis and testing.

Quality Control Test

The quality control test were carried out on the emulsion paint serving as control, Nawgu clay calcinated at (450°C) and calcinated at (750°C). The tests and calculations were carried out using standard testing methods (NIS, 2015) with some modifications where necessary. The tests carried out includes specific gravity, Density, viscosity, pH Test, ash content and moisture content.

pH Test

The test solution and calibration solution was prepared to have the same temperature followed by the Washing of the pH electrode with distilled water, rewashing of the electrode was done using the digested solution of the three sample under analysis separately.

The electrodes (pH electrode and thermometer) were then dipped into the test solution while stirring the solution with a glass rod to ensure that the test solution and calibration solution were at the same temperature.

This process was repeated on the three sample, Clay solution calcined at 450°C , Clay solution calcined at 750°C and fine coat accuracy emulsion paint. Results obtained were (9.55, 10.27 and 8.96) respectively.

Moisture Content

The weight of empty beakers, were recorded as W_1 for each of the sample since the process was repeated separately.

The wet sample was then added to the beaker on the weighing balance and recorded as W_2 for each sample.

The sample was oven-dried in the beaker for 18hrs at 105°C After which it was cooled in then the desiccator, the weight taken and recorded as W_3 for each sample.

Calculation of the moisture content was done using the formula weight of sample after drying divided by weight of sample used multiplied by 100 in percentage.

The result obtained for calcined clay at 450 °C, 750 °C and fine coat emulsion paint are: (61.735%, 70.862%, and 90.638%) respectively.

Ash Content

The weight of empty crucibles was recorded and labeled as W_1 for each sample. samples were then added separately into the crucible on the weighing balance, for each sample and recorded as W_2 Then placed in the furnace set at 450, 750 and 600 degrees for two hours. Where the sample heated at 600 degrees was the fine coat emulsion paint and the 450°C and 750 °C were the local clay from Nawgu. Results are as follow:

Sample (450 °C) = 28.9995%; Sample (750 °C) = 21.9502%;
Fine coat Emulsion = 2.1678%

The samples were then removed from the furnace, cooled in the desiccators and the weights taken separately and recorded as W_3 .

$$\frac{W_2 - W_3}{W_2 - W_1} \times 100 = \text{moisture content (\%)}$$

Calculation of ash-content was done using the formula; weight of sample after ashing divided by weight of sample used multiplied by 100 percentage.

Viscosity

The method used for measuring viscosity here was based on using the capillary tube viscometer method. Here, the time it took for a volume of liquid to pass through the length of a 100mL volumetric tube was measured. The process was repeated three times concomitantly for each of the paint solution and their readings recorded correctly.

$$\frac{W_3 - W_1}{W_2 - W_1} \times 100 = \text{ash content (\%)}$$

The result so obtain are as follows; (26.2560, 8.8440 and 9.7530) for the paint solution of 450°C, 750°C and fine coat Control respectively. Readings were given in centistokes as their unit.

Specific gravity

This measurement was based upon the fact that a floating body is buoyed up more by a heavy liquid than by a light one. The method is more remarkable for speed than

accuracy, but still is sufficiently exact. A hydrometer was used, It consists of a float with a sinker at one end and a graduated tube and a rod at the other end. It is made of metal or glass. Generally two liquids are required, one for liquids ranging in sp. g. from 1.000 to 2.000, and another, which will indicate a sp. g. between 0.700 and 1.000. The range depends on the size of the instrument. For special work, in which variations within narrow limits are to be determined, more delicate instruments with a narrower range are made.

After these processes the following results were obtained for the paint solution of sample 450 °C, 750°C and Fine coat emulsion paint respectively are as follows; (1.0797, 1.2259 and 1.1870).

Density

The density of each of the paint sample was measured using a 100ml volumetric flask. Each of the paint sample was poured to fill a 100ml volumetric flask whose weight has be taken and recorded as W_1 for each of the paint solution. The filled volumetric flask was then reweighed and the new reading recorded as W_2 . Since the volume of the flask is already know, the weight of the paint for each of the solution of interest was obtained by subtracting the weight of the empty flask from the weight of the filled flask divided by 100 (volume of the 100ml volumetric flask).

This is given as: $\frac{1}{100} [W_2 - W_1]$.

Results

The results and properties of the emulsion paints, varied with various extender pigment used in the production; The Local paint incorporated with Nawgu clay calcined at 450°C, Calcined Nawgu clay at 750°C and the standard control Fine Coat Emulsion Paint. The Variation in their properties forms the basic framework of this discussion.

Compositional Analysis

X-ray fluorescence

The X-ray fluorescence (XRF) determinations of the uncalcined Nawgu local clay is presented in Table 2 in alphabetical order. These data are illustrated graphically in Fig 1.

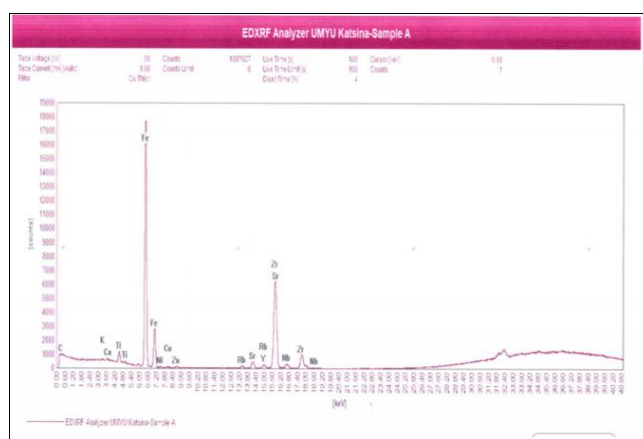


Fig 1: XRF spectra of Nawgu clay

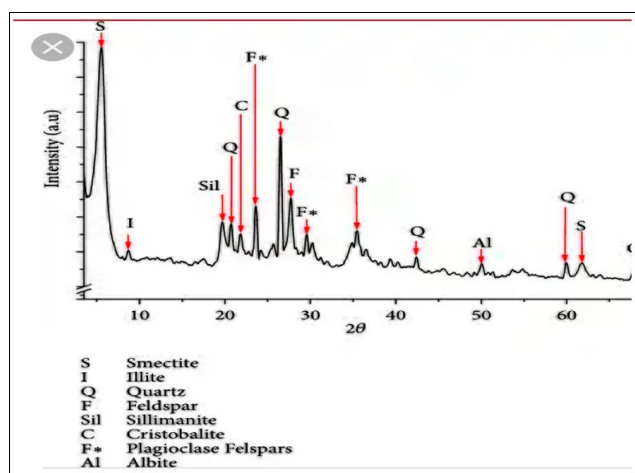
Table 2: Results of Uncalcined Nawgu Local Clay (UNLC)

Z	Chemical Composition	UNLC (Wt%)
1.	Al ₂ O ₃	14.04
2.	CaO	0.73
3.	Cr ₂ O ₃	0.012
4.	Fe ₂ O ₃	6.21
5.	K ₂ O	0.75
6.	MgO	3.77
7.	Na ₂ O	-
8.	P ₂ O ₅	0.25
9.	Rb ₂ O	-
10.	SiO ₂	49.79
11.	SO ₃	0.095
12.	TiO ₂	1.63
13.	V ₂ O ₅	0.03
14.	ZrO ₂	-

The result indicates the presence of silica (SiO₂), Iron (III) oxide (Fe₂O₃) and Aluminum oxide (Al₂O₃) in high proportions while the other constituents are present in smaller proportions as shown in Fig 1. The Uncalcined Nawgu clay was found to contain 49.79% of SiO₂, 14.04% of Al₂O₃ and 6.21% of Fe₂O₃; this explains its dark gray coloration with less than 30% for other constituents. The high proportions of Silica and aluminum is indicative of the kaolinite nature of the clay and the basis for the type of clay to be used for the production of clay paint (Rasheem *et al.*, 2013). The oxide contents (SiO₂ and Al₂O₃) of this clay were consistent with other Nigerian clays. Some of these clays includes; Obowo clay 67.08%, 21.60% and Ihite-Uboma Clay 56.4%, 29.3% (Ewulonu *et al.*, 2016), Ibere clay 52.06%, 27.87% and Oboro clay 60.21%, 19.05% (Mark.U, 2010), Mayo-Belwa clay 59.8%, 7.08% (Haruna *et al.*, 2014). The presence of the unreacted oxides in the clays is indicative of the fact that paints formulated with the clays will function as anti-corrosive paints since the unreactive oxides will slow down the division of corrosive species thereby delaying the phenomenon of corrosion in painted surfaces (Hare *et al.*, 2001).

X-Ray Diffraction (XRD)

X-ray diffraction pattern of raw clay sample is shown in Fig 2. The figure shows that clay is majorly composed of kaolinite and Quartz mineral.

**Fig 2**

The main reflection was at 26.758° which is a typical signal of quartz. Other absorbance peaks signifying the presence of

quartz include 20.998°, 26.758°, 35.053° and 36.250°. The high SiO₂ reported in XRF as discussed in 4.1 correlates with the quartz content as shown in Fig 2 which plays as impurity in the production of the paint. The presence of quartz will impact on the industrial value of Nawgu clay depending on the application.

Absorption peak signifying the presence of Kaolinite is located at 12.496°, 19.982°, 25.018°, 33.087°, and 36.656°. The chemical formula of kaolinite is Al₂O₃·2SiO₂·2H₂O (39% Al₂O₃, 46.5% of SiO₂ and 14.0% of H₂O) and its structure possess strong binding forces between the layers which resists expansion when wetted.

There was no piece of solid or hard material in the paint produced usually referred to as 'lump'. There was no thin layer on the surface as membrane usually referred to as 'Skin'. No residue settled at the base of the container and no bubbles of air at the surface of the paint normally referred to as 'foaming'. The local clay paint produced did not congeal, that is, it does not become thick and partly solid called 'coagulate'. The result conforms to NIS 273: 1990 (Raheem and Oluwa, 2013).

Table 3: Physicochemical properties of paint production

S. No	A-450°C	A-750°C	Control
Moisture content (%)	61.735	70.862	90.638
Ash Content (%)	28.998	21.850	2.168
Density (g/cm ³)	1.604	1.171	1.334
Viscosity(centiStoke)	26.256	8.844	9.753
pH	9.55	10.27	8.96
Specific Gravity	1.080	1.226	1.187

Data obtained on the viscosity of the prepared emulsion paints are illustrated on the Table 3 the table shows an increase in viscosity as the temperature of calcination decreases. Generally, the formulated emulsion paint exhibits higher paint viscosity than samples without clay. The viscosity of the formulated paint obtained conforms to the Nigerian NIS standard. Generally it is required that the minimum viscosity for emulsion paints shall be 600 centiStoke (Igwe *et al.*, 2017).

The density of the paints produced ranges from 1.2625 to 1.8103 g/cm³. All the paints satisfies this standard as provided by the NIS 273:1990 requirements of 2g/cm³ maximum. Similarly, all the paints produced met the paint surface and hard dry time 20 and 120 minutes as recommended by NIS standard 273:1990.

Specific gravity of paints is used to determine the coverage of paint products on substrates. The specific gravity of paint produced from Nawgu clay was determined to be 1.080 and 1.226 a value which is less than the commercial whitening (2.70) (Igwe *et al.*, 2017), and many other used conventional extenders. This gives the clay the advantage of being able to be used in high proportions without having adverse increase in the bulk density of the formulation (Igwe *et al.*, 2017).

Discussion

The X-ray fluorescence (XRF) analysis showed that uncalcined Nawgu clay contained mainly silica (SiO₂, 49.79%), alumina (Al₂O₃, 14.04%) and iron(III) oxide (Fe₂O₃, 6.21%). The predominance of silica and alumina is consistent with the aluminosilicate composition reported for Nigerian clays and supports investigating Nawgu clay as a potential paint extender [6, 7]. Iron oxide may contribute to the clay's dark-grey colour. However, composition alone

does not establish suitability; particle size, dispersion and final coating performance also matter.

X-ray diffraction identified kaolinite and quartz, with the main quartz reflection at 26.758° and kaolinite-associated peaks at 12.496°, 19.982°, 25.018°, 33.087° and 36.656°. Quartz is consistent with the high silica content measured by XRF. Its amount and particle characteristics should be considered during formulation. Kaolinite's layered structure and limited swelling may be useful in coatings, although these results do not quantify its contribution to paint performance^[6, 7].

The paints containing clay calcined at 450°C and 750°C differed from the commercial control. Their pH values were 9.55 and 10.27, respectively, compared with 8.96 for the control. The 450°C sample had higher viscosity (26.256 cSt) than the 750°C sample (8.844 cSt) and control (9.753 cSt). Densities were 1.604, 1.171 and 1.334 g/cm³, respectively, while specific gravities were 1.080, 1.226 and 1.187. These differences suggest that calcination temperature and extender composition may influence paint properties, as reported for other local-clay extenders^[6, 8].

The absence of lumps, skin, settled residue, foaming and coagulation suggests acceptable visual stability. However, the viscosity interpretation requires caution: The values are far below the document's cited minimum of 600 cSt, so the claim of compliance should be checked against the instrument, units, method and applicable standard. Density below the stated NIS maximum of 2 g/cm³ alone does not establish compliance. Further testing of hiding power, adhesion, drying time, scrub resistance, storage stability and weathering is needed before confirming Nawgu clay's commercial suitability.

Conclusion

Nawgu clay and a little measure of calcium carbonate was successfully incorporated into emulsion paint and compared with standard emulsion paint through the Quality control test. The local paint formulated from the distinct calcined clay at temperature 450°C and 750°C showed appreciable difference in viscosity. While the later has its viscosity lower than the former and the former lower than the control, the moisture contents from the two calcined paint products were lower than the control with the calcined clay at 450°C having the lowest moisture content. Also, the ash content from the local paints were much higher than that of the control. It can be generally concluded from these results that the local paint made with Nawgu Clay Calcined at 450°C is much better than that produced with the clay calcined at 750°C. However, the control was a better paint than the two, hence, Nawgu clay cannot sufficiently replace current industrial extenders.

It is important to note however, that the metallic compositions of Nawgu clay as shown from the electron scan analysis do not impact harmful effects on the water from where it was dug hence, the natives of Nawgu, should adopt her stream as a suitable source of water for drinking and other domestic activities.

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