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# Formulation and Production of Emulsion Paint Using Enyigba Clay as Extenders and Filler

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**ABSTRACT**

*The aim of this project work is to formulate and produce emulsion paint from local source, which can compete comfortably with the global raw materials for paint production. The major raw material was clay from Enyigba in Abakaliki, Ebonyi State, Nigeria. Other ingredients used were: Water, titanium (iv) oxide, calcium carbonate, kaoline, combizel, P.V.A or acrylic, ammonia, deformer, genippor and formalin. The clay in Enyigba was carefully dug out and physically cleaned. It was then grinded and soaked in water for three days for softening. This was followed by calcinations for four hours at 750°C. The calcined clay was sieved to 0.02 um particle size and further cleaned up with tetra oxo sulphate (iv) acid, $H_2SO_4$. The prepared paint sample was characterized using standard methods as follows: Specific gravity (ASTMD 153-84): Refractive index (ASTMD 281-12): Oil absorption (AS TMD 281-12): PH (ASTMD 1208-90): And chemical composition determined via Energy Dispersive X-Ray Fluorescence Spectrometer (EDX-RF). The emulsion paint sample produced was subjected to quality control tests based on the Standard Organization of Nigeria, (SON) and was certified correct of the following properties: Colour, hiding power, viscosity, density, cracking, drying time and PH test. It is obvious that Enyigba has a raw material (clay) capable of producing world class emulsion paint with specific gravity, PH and oil absorption of 2.46, 6.67 and 34.30 g/g respectively.*

**Keywords:** Paint; Enyigba clay; Extender; Drying time; Calcination; Emulsion

## INTRODUCTION

Paint can be classifies based on the type of solvent used. Water based paints (emulsion) and oil based paint (gloss). Another classification could also be based on the substrate part of application (exterior paints, interior paint, automotive paints and marine paints). Gloss paint (oil based paint) are paints that may be classified according to whether the drying mechanism is predominantly solvent evaporation, oxidation or some chemical reaction. Gloss paints which dry by solvent evaporation, can be applied on a fairly hard resin as the vehicle. It is applied to interior wood and metal-skirting boards, radiators, wood paneling etc. Gloss paint is available in water based or oil-based versions. Three kinds of Gloss paint are traditional gloss pant, satinwood paint and modern eggshell paint. Emulsion paints (water based paints) is a water based paint with resin added to make it hardwearing. It is normally wall paint or exclusively for interior walls and ceilings. Emulsion paint is made from water, resin, pigments and a tiny amount of additives or preservatives [1]. There different types of emulsion paint, which include; Flat matt emulsion, soft sheen emulsion and silk emulsion paint. Latex paint denotes a suspension of polymer particles in water. Emulsion paints consist basically of a combination of pigment and latex; hence it is called latex paint. White Wash may be made by mixing unslaked line with water and allowing it to stand for a few days before use or starting with calcium hydroxide in the first place. The applied coating converts to calcium carbonate. Certain additions, like glue, have been used for their cheapness. Cement Paint is used to a limited extent to masonry. It is supplied as a powder containing Portland cement and white or alkali-resistant-colored pigments. It is mixed for application. High pigment volume, the use of fast-drying vehicles such as low oil content resin combinations or oil [2]. Free synthetic resins and low-binding solvents such as petroleum fractions with distillation ranges from 100°C to 150°C are three factors responsible for the fast-drying characterizes of the road-stripping paints. Resin solutions like politicized chlorinated rubber or styrene but butadiene may be used. Some aromatic hydrocarbon solvent has to be included in these vehicles to maintain solubility. Antifouling paints that are used in ship bottoms are formulated with copper and mercury compounds as positions for marine growth, using binders which are not too permanent so that gradual breakdown of the film and release of position can take place (as tributyltin compounds are used as the toxic materials). A typical formulation calls for zinc oxide ferric oxide, magnesium silicate, cuprous oxide, mercuric oxide, rosin, oil, coal tar and solvent. The most commonly used type of paint is the emulsion

paint which constitute the solvent which is usually water, the pigment which impacts color to mixture, titanium (ii) oxide which is most popular pigment used in production, the extender-pigment which acts to reduce cost of pigment and increase durability of the mixture and lastly the additives like preservatives, defoamer added to improve other properties of the paint [3].

However, in recent years, the crucial effect of the extender pigment on the rheological properties of paint has been noted and also reduces the cost of paint production which positively affects the general economics of paint production. Different research shows that different compounds have been used as extender-pigment in the production of emulsion paint and as such have different effects on the properties of the paint. Some of these compounds are as following: barium sulfate in its natural crystalline form of barite, magnesium silicate known as talc and asbestine, ordinary silicon (ii) oxide is also used but the most commonly used extender-pigment is calcium carbonate [4]. Pigments for the formulation of paint are of submicron particles which most possess certain features such as opacifying strength (also called hiding power), brightness, resistance to chemical attack, good thermal suitability and resistance to UV degradation. Some pigments from mineral sources gives opacity and brightness because of their ability to scatter light. The degree to which their do this depends on the structure they form and their intrinsic index of refraction [5]. They can be found in paste or oxide. When colours are found in paste, it means they are in condensed liquid form (like your pomade) but when we say they are in oxide, it means they are in dust form (like your powder). Pigments like titanium dioxide have high refractive index and very strong absorption in the UV region, while calcined kaolin has a very similar property with titanium dioxide and hence can substitute titanium dioxide as a pigment. Pigment as one of the most important components of paint usually have a particle size of about 0.5 to 5 µms in a standard paint but most times have a lower value in size as 0.01 µms for some organic pigment.

Inorganic pigments have mineral origin and are mostly metal oxides. These metal oxides are naturally found such as red and yellow oxides. However, they can be synthesized to get the main colour. However, on the other hand, organic pigments are not naturally occurring but are made from petroleum by-products. They are brightly colored as used for clean colors red, yellow, bright green and blue, purple and magenta. Extender pigment is an integral part of all coating formulations and significantly contributes in various coating properties such as flow characteristics, gloss, resistance, abrasion and setting tendency. Since they don't impact opacity to the coating and are partially transparent in the oil medium, there are different from true pigments. Most of the extender used is such as talc, whiting and barites used in the production of paint are expensive due to their mineral origin and requires long processing which result to significant loss of minerals. Extender pigment is usually natural occurring minerals such as clay, mica talc and limestone. They are mostly used to reduce the high cost of paint. They are usually gotten by mining.

Anti-corrosive pigments are important area to study as steel requires the greatest protection against corrosion and the need to develop pigments to inhibit the corrosion of steel being considered for many years. Some of the anti-corrosive pigment which has been into use are red lead, $Pb_3O_4$. When combined with linseed oil, red lead is set to be the standard anti-corrosive primer for iron and steel. There are special purpose pigments like copper which can be used in anti-fouling paints for ships. Other special pigments are the non-leafing aluminium flake pigments and the special effect, iridescent, pearl affair pigments used in automotive finishes. Nevertheless, titanium dioxide is regarded as the most important of all the various pigments. This result to increasing demand of for titanium dioxide and its price has consequently been on the rise. In 2011, the price of titanium dioxide rose to 35% and is expected to increase at an average annual rate of about 7% for the near future. The above reason has made the search for an alternative pigment an important one. One of such alternatives is calcined kaolin with its physical and chemical properties compare favorably with that of the titanium dioxide.

Solvent is used to mix all the chemicals together *i.e.* it is used to dissolve or disperse different components used in paint formulation (e.g. pigment and resin). The type of liquid (solvent) used depends upon the other components of the given paint. Oil-based paints for example can use kerosene or fuel as the primary liquid while emulsion paints on the other hand; tend to use water as their liquid. Examples are Ketones, aromatics, aliphatics, alcohols, glycol ethers, glycol ether esters, glycols, glycol esters, chlorinated products, terpenes, etc. It helps to hold all the pigments together. It also helps the product to adhere to the surface it is being painted too without much stress. A good example is PVA, which stands for Poly Vinyl Acrylic.

Clay is a common name for a number of fine grained earthly materials that are plastic and tenacious when moist and permanently hard when fired or baked. Generally, clays are hydrated silicates of aluminum made of the composition of $Al_2O_3$, $2SiO_2$, $2H_2O$ and which mostly contain impurities like potassium, sodium, calcium, magnesium or iron in small quantities. They are one of the major minerals that are abundant on the earth's crust which are characterized by sheet silicate structures of composite layers stacked along the c-axis. Local clays are known to be layered silicates and their mineral platelet structure has the thickness of about one manometer, however, its dimensions in lengths and width can be measured in hundreds of nanometer (200 mm-400 mm) range after purification. These clays are naturally abundant, renewable and very cost effective and easy to process. For instance, according to the Nigeria ministry of solid minerals development, Nigeria has very large and economic deposit of kaolinite clay minerals with a reserve of about 3 billion tones in many locations [6].

## MATERIALS AND METHODS

### Materials

The solvent, binder, pigments and additives used in this study were obtained from paint factory, Alex-Ekwueme Federal University Ndufu Alike in Ebonyi state Nigeria. The Enyigba clay used in this project study was hand dug from the clay deposit at Abakaliki local government area, Ebonyi state, Nigeria.

**Raw materials for emulsion paint formulation:** The raw materials used in paint formulation are grouped into five: Extender, pigment, solvents, binder and additives.

### Preparation of clay extender

The Enyigba clay sample, was dug out from the clay deposit site and impurities removed physically. Then the clay was grinded and soaked in water for 3 days to softening it. Calcination at 750°C for 4 hrs followed. The calcined clay was sieved to 0.02 µm particle size and further cleaned up with $H_2SO_4$ to further removal of impurities.

### Preparation of emulsion paint sample

A series of emulsion paints were prepared using the Enyigba clay and calcium carbonate combined with kaolin. The calcium carbonate and kaolin were used as a reference extender. The specific formulation used in the preparation is show in Table 1.

**Table 1:** Formulation of Enyigba Clay Emulsion Paint (ECEP).

| S/N | Materials | Mass of sample A (Kg) (Ecep) | Mass of sample B (Kg) | Mass of sample C (Kg) (C and Kep) |
|---|---|---|---|---|
| 1 | Clay | 0.8 | 0.4 | _ |
| 2 | Calcium | - | - | 0.5 |
| 3 | Kaolin | - | - | 0.3 |
| 4 | Water | 1 | 0.5 | 0.5 |
| 5 | Genippor | 0.1 | 0.05 | 0.1 |
| 6 | PVA(Acrylic) | 0.2 | 0.01 | 0.2 |
| 7 | Combizel | 0.01 | 0.005 | 0.01 |
| 8 | Titanium oxide | - | 0.05 | 0.05 |
| 9 | Ammonia | 0.01 | 0.005 | 0.01 |
| 10 | Formalin | 0.01 | 0.005 | 0.01 |
| 11 | Deformer | 0.01 | 0.005 | 0.01 |
| Total | | 2.14 | 1.12 | 1.69 |
| **Note:** ECEP means Enyigba clay emulsion paint and C and KEP means calcium carbonate and kaolin emulsion paint. | | | | |

**Procedure for production of emulsion paint**

The following procedures were dully followed to produce Enyigba Clay Emulsion Paint (ECEP) and the reference paint produced with calcium carbonate and kaolin (C and KEP). The materials used were properly measured and weighed out in kilogram, with the aid of weighing balance. Firstly, a measured 1 kg and 0.5 kg of water were poured into two different containers (the reactor tank). Then the measured clay (0.8 kg) was poured into the container having 1 kg of water, likewise the kaolin (0.3 kg) and calcium carbonate (0.5 kg) mixed together were also poured in a separate container having 0.5 kg of water and mixing started immediately. After about 5 mins of mixing with a stirrer and stilling on a continuous mixing process we introduced Genippor into the reactors. This Genippor now softens and makes the clay, calcium carbonate and kaolin to dissolve more in the water by reducing its surface tension.

However, the measured quantity of PVA (0.2 kg) was poured into each of the two reactors and mixing continued for another 4 mins. This helped to bind the whole materials together as the mixing process continued. Furthermore, we poured in the measured thickener (Combizel 0.01 kg) into a very negligible amount of water and mixed properly before pouring it into each of the reactor tanks. This was to dissolve properly the combizel which serves as the catalyst before going into the tanks, thus to increase homogeneity of the mixture. All these were done in continues stirring process.

Hence, we poured in the measured quantity of ammonia (0.01 kg) and formalin (0.01 kg) as the mixing continued. At this stage, we divided the ECEP sample into two and added 0.05 kg of titanium dioxide while the same quantity of titanium (0.05 kg) was pour to the C and KEP sample which served as pigment or colorants. The mixture was stirred thoroughly for another 10 minutes to obtain a homogenous emulsion. The samples paints produced were kept for analysis and testing.

## RESULTS AND DISCUSSION

**Composition analysis of Enyigba clay**

The results of chemical composition of Enyigba clay were determined using Energy Dispersive X-ray fluorescence spectrometer (EDX-RF) [6] as represented in Table 2 below.

**Table 2:** Composition of Enyigba clay.

| Chemical composition | Composition, weight % |
|---|---|
| $SiO_2$ | 62.81 |
| $Al2O_3$ | 27.03 |
| $TiO_2$ | 1.82 |
| $Fe_2O_3$ | 2.81 |
| MgO | 0.08 |
| CaO | 0.17 |
| $Na_2O$ | 0.6 |
| $K_2O$ | 0.34 |
| MnO | 0.08 |
| PbO | 1.28 |
| ZnO | 0.1 |
| $ZrO_2$ | 1.06 |
| $Bi_2O_3$ | 1.58 |

These results showed the appreciable presence of Silica ($SiO_2$) and Alumina ($Al_2O_3$) in the clay, followed by Titanium Dioxide ($TiO_2$) and Iron (III) Oxide ($Fe_2O_3$), with other constituents present in smaller quantities. However, it is evident that Eniyigba clay contains less than 65% of silica as recommended by World Health Organization, for clays to be used for paint production. Paint containing more than 65% of silica can cause fibrosis, silicosis and lung cancer. The high proportion of silica and aluminium oxide in the clay 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 emulsion paint. The oxide contents ($SiO_2$ and $Al_2O_3$) of this clay which is 62.81% and 27.03% respectively were consistent with other Nigerian clays that are used to produce paint. Some of these clays include; Aningene clay 43.05%, 23.92% and Nwangene clay 66.2%, 27.50%. Ibere clay 52.06%, 27.87% and Oboro clay 60.21%, 19.05%. Mayo-Belwa clay 59.8%, 7.08%. Omankwo Afikpo clay 87.13%, 6.70%; Okposi clay 53.04%, 19.70%. The comparison between the quality of the produced pint and the standards are contained in Table 3. It is clear that the the produced paint complies with the standard from SON [7].

**Table 3:** Comparison between Enyibga clay and standard raw material standard of SON 2007.

| S/N | Property | Sample A (ECEP) without $TIO_2$ | Sample B ECEP with $TIO_2$ | Sample C (C and KEP) | Standard from SON |
|---|---|---|---|---|---|
| 1 | PH | 8.1 | 7.9 | 8 | 7.0-8.5 |
| 2 | Viscosity using I.C.I rotor thinner viscometer | 6.2 (poises) | 6.4 | 6.4 (poises) | 6.0 (poises) (using I.C.I Rotothinner viscometer minimum. |
| 3 | Opacity | High opacity observed at first coating and was able to cover very well | High opacity observed at first coating and was able to cover very well | High opacity observed after second coating | Good opacity and shall be able to cover well at not more than two coats with a minimum spreading rate of 10 $m^2$/litre. |
| 4 | Brushability | Very easy to brush. Brisuh marks not broadly pronounced | Esay to apply. | Easy of application of brush. Brush marks observed | Easy application of the successive coats by brushing. Brush marks produced during brushing application shall not be more pronounced than those of the first coat. |
| 5 | Drying properties | 18 hrs | 20 hrs | 21 hrs | Maximum 24 hours |
| i. | Surface drying | 20 hrs | 20 hrs | 20 hrs | 20 hrs |
| ii. | Hard drying | 1 hr 33mins | 1 hr 45 mins | 1 hr 50 mins | Maximum 2 hours |
| 6 | Specific gravity (kg/l) | 1.13 | 1.01 | 1.12 | 1.33-1.39 |
| 7 | **Resistant to external exposure** | | | | |
| 7i | Intense sun shines | No cracking and color change was observed | No cracking and the same color retained | No cracking and color change was observed | No film defect such as cracking blistering, flaking color fading and dirt retention |
| 7ii | Water (rainfall) | High wash ability resistance observed | Resistance to wash ability | High wash ability resistance observed | |

**Chemical composition:** X-Ray Fluorescence (XRF) analysis of Enyigba clay shows an appreciable presence of silicon Oxide ($SiO_2$) (62.81 wt.%) in the clay, followed by aluminium oxide ($Al_2O_3$) (23.07 wt.%) and Titanium Oxide ($TiO_2$) (1.82 wt.%) in that order. The quantities of Potassium Oxide ($K_2O$) and iron(iii)oxide ($Fe_2O_3$) are 0.34 and 2.18 wt.% respectively, with other oxides present in minute quantities. However, it is evident that Eniyigba clay contains less than 65% of silica as recommended by World Health Organization, (WHO) for clays to be used for paint production. Paint containing more than 65% of silica can cause fibrosis, silicosis and lung Cancer (WHO). According to Raheem and Olowu, a large presence of $SiO_2$ and $Al_2O_3$ in an extender indicates the kaolinite nature of the extender pigment and which will form the basis for its utilization in paint making [8].
**PH test and result:** The pH for the three samples was 8.1 (sample A), 7.9 (sample B) and 8.0 (sample C) respectively.
**Viscosity:** Viscosity of the emulsion paint samples are presented in Table 4 and it shows that the lower the clay content the less the viscosity.

**Table 4:** The viscosity results of paint samples produced.

| S/N | Paint Sample | Clay content (wt%) | Viscosity in pascal-seconds (Pa.s) (1Pa.s=10P) |
|---|---|---|---|
| 1 | Sample A | 80 | 1.13 |
| 2 | Sample B | 40 | 0.93 |
| 3 | Sample C | 0 | 0.92 |

**Density:** This is determined using a weight per gallon cup. 50 ml of paint was filled in the cup and used in the same way as a specific gravity bottle. The paint density was calculated for the three paint samples and was gotten as in Tables 5 and 6.

**Table 5:** The densities of paint samples produced.

| S/N | Paint sample | Clay content (wt%) | Density (ρ) (kg/l) |
|---|---|---|---|
| 1 | Sample A | 80 | 1.15 |
| 2 | Sample B | 40 | 1.01 |
| 3 | Sample C | 0 | 1.12 |

**Table 6:** Surface drying time.

| S/N | Paint sample | Clay content(wt%) | surface dryness time | Hard drying time |
|---|---|---|---|---|
| 1 | Sample A | 80 | 18 hrs | 105 min |
| 2 | Sample B | 40 | 20 hrs | 110 min |
| 3 | Sample C | 0 | 20 hrs | 93 min |

**Chalking test on paint sample:** The chalking tests carried out on the formulated paint samples indicated that none of the paint samples showed any sign of chalking. A good paint should not chalk. The three paint samples showed good quality in terms of chalking. This is due to the choice of PVA as binder, which helps the paint to stick to the wall for a very long time.

**Colour:** The colour of the three paint samples varies due to different materials used in the production. The colour of sample A (ECEP) was Beige colour while the colour of sample C was find to be pure white due to the addition of $TIO_2$ as pigment. However, the Beige colour of the sample A was due to the clay content which served as pigment with such fine colour. But when we divided the sample A into two and added $TIO_2$ as pigment to obtain sample B, the colour was fine to be Brown colour. Generally, it was observed that Enyigba clay can not only serve as extender and filler but can also serve as pigment as shown in our work [9-12].

**Drying time:** The paint samples were applied with brush on a panel surface and allowed to dry under normal condition. The time it took for each sample to dry very well was monitored and recorded for each sample [13-15].

## CONCLUSION

Enyigba clay obtained from Abakaliki Local Government Area of Ebonyi State; Nigeria, has been used successfully to formulate emulsion paints with improved qualities. The results from chemical composition analysis showed the appreciable presence of Silica ($SiO_2$) and Alumina ($Al_2O_3$) in the clay, followed by Titanium Dioxide ($TiO_2$) and Iron (iii) Oxide ($Fe_2O_3$), with other constituents present in smaller quantities. and it is evident that enyigba clay contains less than 65% of silica as recommended by world health organization, for clay to be used for paint production. however, the quality control test carried out on the paints indicated good properties associated with the paints such as, good opacity, moderate drying time, moderate pH, high viscosity and high density compared to the reference sample. The overall analysis carried out showed that the Enyigba clay served as extender, filler and at same time a pigment since it gave us a very unique beige colour; therefore, replacing $TIO_2$ in a very high capacity. More so, the cost evaluation carried out showed that Enyigba clay is almost free which is an added advantage on the use of Enyigba clay rather than expensive imported raw materials and high cost of production. It's more available and nearer to the users and most importantly, it is will motivate and encourage the local processors of the raw materials in the country which will at the same time improve the level of industrial and technological development in Nigeria.

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The authors declare no competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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