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## **Original Article**

# Utilization of granite quarry dust extender in formulating anti-corrosive paints for the protection of steel

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#### ABSTRACT

Alkyd paints possessing anti-corrosive property for the protection of steel was prepared using granite quarry dust at extender contents, 0-80 wt.%. Granite quarry dust was analyzed for extender properties, and sieved to 75 µm particle size. Determinations showed that the extender consisted largely of silica (87.50%), with the other oxides present in smaller proportions. The prepared paints had good surface – and through-dry times; an indication that the incorporated granite quarry dust had no adverse effects on the paint properties. The paint dry films which were in the range, 0.10–0.20 mm exhibited good hardness, and impact strength thereby contributing to the paints anti-corrosive properties. Paints formulated using 60, and 80 wt.% granite quarry dust had maximum film hardness of 5H. The paint dry films generally performed satisfactorily on immersion in 3% NaCl, 3% Na<sub>2</sub>CO<sub>3</sub>, and distilled water; an indication of good performance of the paints in salty environment. These good performances of the formulated paints are attributed to the presence of inert oxides in granite quarry dust. The paint films, however, performed fairly in 3% NH<sub>3</sub>, and poorly in 3% H<sub>2</sub>SO<sub>4</sub>. The paint performance characteristics obtained with granite quarry dust are enough evidence to justify its use in the surface coatings industry, and in the process, contribute to an economic and efficient way of disposing the granite quarry dust waste.

Keywords: Extender, alkyd paints, particle size, granite quarry dust, steel, anti-corrosive paints

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## **INTRODUCTION**

Extenders are widely used in the paint industry where they modify certain paint properties such as gloss, flow characteristics, viscosity, and settling tendency.<sup>[1]</sup> Extenders also reduce the cost of paint production by substituting in parts the prime pigment requirement of paints. Though, insoluble in an oil medium, extenders are particularly transparent in the medium but differ from true pigments since they do not impart opacity to paints. Extenders are also needed to act as particle spacers to optimize hiding efficiency of titanium dioxide (TiO<sub>2</sub>) thereby, avoiding overcrowding.<sup>[2-4]</sup>

Most of the extenders used in coatings are processed minerals that are expensive, and not indigenously available.

These factors lead to increases in the cost of the resultant paint products.<sup>[5]</sup> The processing of most mineral extenders also lead to significant loss of materials.  $TiO_2$ , a white pigment is widely used in paint production because of its efficiency in scattering visible light. Consequently, the addition of  $TiO_2$  to paints imparts whiteness, brightness, and opacity to paints. It has been reported that about 4.6 million tons of  $TiO_2$  are consumed annually, a figure which no doubt, has increased substantially with increasing consumption of paints.<sup>[6]</sup> Although,  $TiO_2$  confers many benefits to paints, it is an excellent absorber of ultraviolet light which leads to photocatalytic degradation of organic binders in the formulated paints.<sup>[7]</sup> Like many other mineral pigments,  $TiO_2$  is expensive, and this leads to high cost of paint products.

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Paint formulators and scientists are investigating economically and technically viable extenders for use in paint production so as to bring down the cost of painting to acceptable limit. Fortunately, the recent advances in the technology of coatings have helped to increase the demand for paint extenders. The following materials have been investigated for possible use as extenders in paints with improved outcome: Dolomite ore,<sup>[8]</sup> fly ash,<sup>[9]</sup> clays,<sup>[10-13]</sup> silica fume,<sup>[14]</sup> and rice husk ash.<sup>[15]</sup>

The establishment of many granite quarrying sites in Nigeria has led to the accumulation of huge amount of granite quarry dust. The dust which results from granite stone processing is non - biodegradable, and have no end use applications. The dust have potentials to contaminate the air and water, and possess harmful effects on the growth of plants.<sup>[16]</sup> Therefore, proper utilization, and handling of granite quarry dust in an economic manner are needed largely to protect the environment from dust pollution and in the process, achieve a clean and green environment. There are efforts to develop innovative advanced materials with such industrial wastes. Thus, granite dust has been used as a filler for polycarbonate toughened epoxy resin,<sup>[17]</sup> vinyl ester resin,<sup>[18]</sup> polypropylene,<sup>[19]</sup> high density polyethylene,<sup>[20]</sup> concrete,<sup>[21,22]</sup> and cement.<sup>[23]</sup>

This study reports the utilization of granite quarry dust from Arab Contractors depot, Aba, Nigeria as an extender in formulating alkyd paints that exhibited good corrosion protection of mild steel. The granite quarry dust which was sieved to 75  $\mu$ m was used within extender contents, 0–80 wt.%. In an earlier report from our laboratory,<sup>[24]</sup> granite quarry dust was used to fabricate unsaturated polyester resin tiles that exhibited good hardness, impact, and tensile strength properties.

Granite quarry dust is a fine industrial waste that is not saleable, and is stockpiled in different quarrying sites in Nigeria. The use of such wastes for sustainable polymer products is generally cost-effective, and is gaining popularity.<sup>[16]</sup> Thus, the utilization of granite quarry dust in alkyd paint production is, therefore, expected to reduce not only the overall cost of painting but also, offer an economic way of disposing the waste thereby, ensuring a cleaner environment. The use of granite quarry dust in formulating alkyd (solvent-based) paints has not been reported in the literature to our knowledge.

## **MATERIALS AND METHODS**

#### Materials

A medium oil length soyabean alkyd resin was used as the binder in this study and was purchased from Jokens Industry Ltd, Onitsha, Nigeria. It has an acid value of 2.30 mgKOH/ gKOH. Granite quarry dust used as an extender was collected from Arab Contractors depot, Aba, Nigeria. It was sieved to 0.075  $\mu$ m particle size after purification. Lead and cobalt naphthenates used as driers were purchased from a chemical store at Onitsha, Nigeria. Lead naphthenate was used for through dry while cobalt naphthenate was used for surface dry of the applied paint films. The driers have metal contents of 36% lead and 12% cobalt, respectively.

#### Methods

#### Preparation of granite quarry dust extender

The granite quarry dust was first sundried, after which impurities were removed from it. It was further reduced to fine powder using a notarized grinder, calcined at 850°C for 6 h, and sieved to 0.075  $\mu$ m particle size. The granite powder was stored in a tight lid container for further use.

#### Characterization of granite quarry dust

The prepared granite quarry dust was characterized for: pH (ASTM D 1208 – 960), refractive index (ASTM D 1208 – 96), specific gravity (ASTM D 153 – 84), oil absorption (ASTM D 281 – 12), and chemical composition (ASTM D 5381 – 94). The resistance of the prepared granite quarry dust to chemicals was as described previously.<sup>[25]</sup>

#### Preparation of granite quarry dust alkyd paints

The paint samples were prepared using the following ingredients: Medium oil length soyabean alkyd resin, granite quarry dust,  $TiO_2$  (prime pigment), and xylene. A typical formulation used in the preparations is shown in Table 1. Cobalt naphthenate (3.30 g) and lead naphthenate (0.11 g) driers were used for surface, and through dry of the paint samples.

#### **Analysis of Paint Samples**

The following tests were carried out on the granite quarry dust extended paints: Surface – and through dry times using methods as described previously,<sup>[25]</sup> dry paint film thickness (ASTM D 1005 – 95), dry paint film hardness (ASTM D 3363 – 05); impact strength of dry paint films (ASTM D 2794 -- 93), and adhesion of paint films on mild steel panels. ASTM D 6677 – 07, 2021 was used to determine the resistance of dry paint films to 3% NaCl, 3% H<sub>2</sub>SO<sub>4</sub>, 3% Na<sub>2</sub>CO<sub>3</sub>, and distilled water.

Table 1: Formulations for	alkyd paint samples
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Ingredients(g)	Formulations					
	Ι	II	III	IV	V	VI
Alkyd resin	66	66	66	66	66	66
Titanium dioxide	29.70	23.80	17.80	14.80	11.90	5.90
Granite quarry dust	-	5.90	11.90	14.90	17.80	23.80
Xylene	11.90	11.90	11.90	11.90	11.90	11.90

## **RESULTS AND DISCUSSION**

#### **Characteristic of Granite Quarry Dust**

The properties of granite quarry dust used in this study are shown in Table 2.

The specific gravity of granite quarry dust was determined to be 2.45, a valve that is lower than that of  $\text{TiO}_{2}(4.20)$  used in this study. The low specific gravity of granite quarry dust is an indication that it can be incorporated into paints in high proportion without having adverse effects on paint properties. The specific gravity of some extenders is: Calcium carbonate 2.25,<sup>[26]</sup> China clay, (2.60) and talc 2.65–2.85.<sup>[9]</sup> In general, extender pigments that have low specific gravity invariably will have low tendency to settle in formulated paints. Conversely, extender pigments having low specific gravity will exhibit high oil absorption.<sup>[27]</sup>

Granite quarry dust has a low refractive index (1.51) when compared to TiO<sub>2</sub>(2.75). Commercial extenders such as talc, mica, and whiting have refractive indices of 1.40, 1.59, and 1.65, respectively. Although, extender pigments affect the scattering power of paints, they do not enhance the opacity of dry paint films or much color when added to clear coatings. It is important to note that the refractive indices of extenders are not markedly different from the values found in most dry paint films (1.45–1.70).<sup>[28]</sup>

Table 1 shows that granite quarry dust has an oil absorption value of 29.0 g/100 g, a value that is lower than that of TiO<sub>2</sub> which is 18.50 g/100 g. The oil absorption of some extender pigments are: Fly ash 19.0, China clay 30, talc 25–35, amorphous silica 29, and calcium carbonate 17.5.<sup>[29]</sup> The oil absorption of an extender is an indication of the amount of base resin it will require without compromising other coatings properties. This property depends on the physical structure of an extender (i.e., size and shape of the extender particles) and which in turn affects other coatings properties such as settling tendency, flow characteristics, and film durability.<sup>[6]</sup> In general, the less oil an extender absorbs, the less resin it will require in paint formulation.

#### **Chemical Composition of Granite Quarry Dust**

Silicon dioxide  $(SiO_2)$  (87.50%) was the major oxide present in granite quarry dust as determined by X-ray fluorescence technique. Thus, the granite is an acidic rock. Other oxides

Table 2:	Properties	of granite	quarry dust
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Parameter	Value
Specific gravity	2.45
pH	6.80
Refractive index	1.51
Oil absorption (g/100 g)	29.0

such as aluminum oxide  $(Al_2O_3)$  (2.67%), sodium oxide  $(Na_2O)$  (2.0%), potassium oxide  $(K_2O)$  (1.20%), calcium oxide (CaO) (1.06%), magnesium oxide (MgO) (1.83%), and titanium oxide (TiO<sub>2</sub>) (0.33%) are present in smaller proportions. SiO<sub>2</sub> is known to be chemically inert, non-toxic, and it is the most common mineral oxide that is resistant to weathering. The large presence of SiO<sub>2</sub> in granite quarry dust indicates that paint formulated with it will function as anti - corrosive paint due to its unreactive nature capable of slowing down the diffusion of reactive species into painted surfaces. Consequently, painted metallic surfaces will be protected from decay thereby, ensuring the durability of the painting.

#### **Chemical Resistance of Granite Quarry Dust**

Determinations showed that granite quarry dust was stable both in the cold and when heated to the following chemicals: toluene, chloroform, methanol, sulfuric acid, acetic acid, sodium hydroxide, and ammonia. However, it exhibited slight solubility, and color change when heated in hydrochloric acid. This later observation indicates that paints formulated with granite quarry dust should not be used in hydrochloric acid prone environment. The general stability of granite quarry dust in the chemicals studied is attributed to the large presence of SiO<sub>2</sub>. SiO<sub>2</sub> is insoluble in water and mineral acids, and has a high melting point (1713°C). Similarly, Al<sub>2</sub>O<sub>3</sub> which is next highest in quantity (2.67%) in granite quarry dust and has a melting point of 2054°C may also contribute to the thermal stability of granite quarry dust in the chemicals studied.

#### **Properties of Formulated Alkyd Paints** *Surface dry times*

The surface dry times of the prepared paint samples are illustrated in Figure 1. The surface dry time of the alkyd paint sample without granite quarry dust is 10 min. The granite quarry dust prepared paints exhibited surface dry times that were in the range, 10–20 min. The paint sample containing 10 wt.% granite quarry dust has surface dry time of 10 min, the same as paint sample without quarry dust. The Nigerian Industrial Standard (NIS)<sup>[30]</sup> stipulates that a gloss paint shall not exhibit surface dry time of more than 6 h from the time of application.

In general, the prepared paint samples had good surface dry times. Thus, the incorporation of granite quarry dust into the paint samples did not adversely affect their surface dry times.

#### Through dry times

The through dry times of the prepared paint samples illustrated in Figure 2 show that the through dry times increased with granite quarry dust content. The through dry time of the paint sample without granite quarry dust is 270 min. The paint samples exhibited good through dry times because according to NIS,<sup>[30]</sup> the through dry time of a gloss paint shall not exceed 24 h.

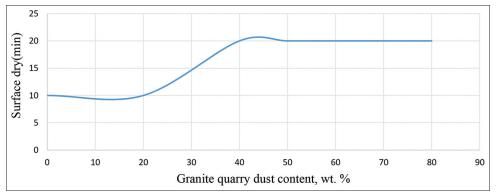


Figure 1: Effect of granite quarry dust content on surface dry times of formulated alkyd paints

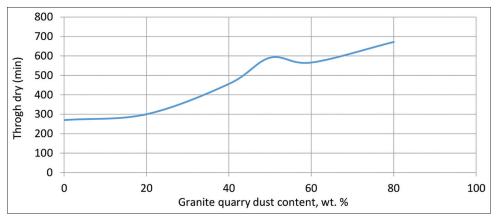


Figure 2: Effect of granite quarry dust content on through dry times of formulated alkyd paints

## Impact strength

The impact strength of the formulated paint samples was determined using Charpy impact tester, and all the formulations passed the test. This is an indication that the paints have the ability to resist crack or break due to mechanical loads and stress levels, and therefore, should be protective against corrosion or microbial action on application to metallic surfaces.

## Film hardness

The hardness of paint dry films of granite quarry dust formulated paint samples was observed to increase with granite quarry dust content, except for 80 wt.% granite quarry dust content where a decrease (4H) was observed. The paint sample without granite quarry dust had a film hardness of 6B, and this was the least film hardness obtained in this study. The maximum film hardness (5H) was recorded for paint samples containing 60, and 80 wt.% granite quarry dust. In general, the formulated paint samples exhibited good film hardness property which is an indication that the binder molecules, and pigment particles were in mutual attraction.

## Dry paint film thickness

The variation of dry paint film thickness with granite quarry dust content is illustrated in Figure 3. The figure shows a general decrease of paint dry film thickness with granite quarry dust content, except at 60, and 80 wt.% granite quarry dust content where an increase in film thickness was observed. The paint sample without granite quarry dust had film thickness of 0.45 mm. Paint samples containing 20, 40, and 50 wt.% granite quarry dust exhibited the least dry paint film thickness (0.10 mm). A film thickness of 0.20 mm was obtained for the paint samples containing 60, and 80 wt.% granite quarry dust. Thus, film thickness obtained for the formulated paint samples were in the range, 0.10–0.20 mm. It has been reported<sup>[9]</sup> that paint film thickness of more than 20  $\mu$ m performs well as barrier resistant to weathering. The above is an indication that film thickness obtained for granite quarry dust formulated paints will function as anti-corrosion paints. The dry paint film thickness affects its durability.

## Adhesion of paint films to substrate

Data on the adhesion of dry paint films to mild steel panels illustrated in Figure 4 show that there is no definite order of variation of dry paint films to substrate with granite quarry dust content. While the paint sample without granite quarry dust had an adhesion loss of 1.56%, the paint samples containing 40, and 80 wt.% granite quarry dust recorded an adhesion loss of 12.50%, the maximum loss obtained in this study. The least film adhesion loss (1.56%) was obtained with the paint

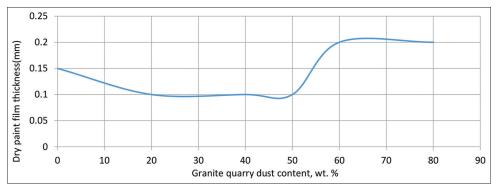


Figure 3: Effect of granite quarry dust content on dry paint film thickness of formulated alkyd paints

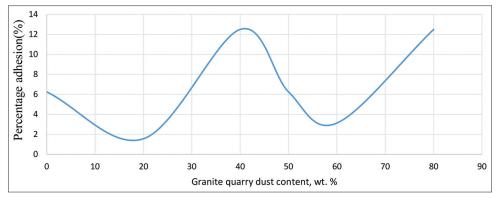


Figure 4: Effect of granite quarry dust content on adhesion property of formulated alkyd paints

Formulation	Resistance to				
	Distilled	3%	3%	3%	3%
	water	H <sub>2</sub> SO <sub>4</sub>	NaCO <sub>3</sub>	NaCl	NH <sub>3</sub>
AT - 100	1	4	0	1	3
AGD - 80/20	1	4	0	0	2
AGD - 60/40	0	3	0	1	1
AGD - 40/60	0	4	0	0	2
AGD - 60/40	1	3	1	0	2
AGD - 80/20	0	3	0	0	1

Table 3: Resistance of dry paint films to chemicals

Key: 0=No effect, 1=Very slightly effect (colour/wrinkling), 2=Slight effect (wrinkling), 3=Effect (blistering/peeling), 4=Definite effect (blistering/peeling)

sample containing 20 wt.% granite quarry dust. The present study reveals that the granite quarry dust formulated paints possess good adhesion property to mild steel surfaces and therefore, should function as anti-corrosive paints. The NIS<sup>[30]</sup> stipulates that a gloss paint shall not exhibit more than 50% removal of the dried paint films. In general, adhesion is an important property of paints that ensures that an applied paint remains adhered to the painted surface for long under severe environmental conditions. The durability and quality of a paint are dependent on the adhesion property of the paint.

#### Dry film resistance to chemicals

The visual changes that occurred on the formulated dry paint films after immersion in  $3\% H_2SO_4$ ,  $3\% NH_3$  solution, 3% NaCl,  $3\% Na_2CO_3$ , and distilled water are shown in Table 3.

This table shows that granite quarry dust formulated paints generally performed well in distilled water, 3% NaCl, and, 3% Na<sub>2</sub>CO<sub>2</sub>, and therefore, should function as anti-corrosive paints in salty environments. The good performances of granite quarry dust formulated paints to the chemical media studied are attributed to the presence of inert oxides (SiO<sub>2</sub>, Al<sub>2</sub>O<sub>2</sub>, etc.) capable of slowing down the diffusion of corrosive species into the film substrates, and in the process, function as a barrier between the metal substrate and applied paint films; hence, the anti-corrosion property. The performances of the formulated paints are influenced by the raw materials used, and the mutual attraction between them. The formulations showed that the amount of base resin (alkyd) and xylene used is constant except for the pigment (TiO<sub>2</sub>) and granite quarry dust, and this helps to quantify the effect of incorporating granite quarry dust on the anti-corrosion property of the paints.

Table 3 shows that the performances of the formulated paints were fairly good in 3% NH<sub>3</sub> but poor in 3% H<sub>2</sub>SO<sub>4</sub>. However, the addition of granite quarry dust into the formulations

helped to reduce the adverse effects of 3%  $H_2SO_4$ , and 3%  $NH_3$  on the painted panels at some formulations. Thus,  $H_2SO_4$  and  $NH_3$  prone environments are particularly not good environments for the application of granite quarry dust alkyd paints. Tiwari and Saxena<sup>[9]</sup> who studied fly ash extended solvent - borne coatings reported good performance of the coatings in 2%  $H_2SO_4$ , and that paint films of formulations based on whiting detached completely from the substrate in 2%  $H_2SO_4$ . The later observation was attributed to chemical reactivity of whiting.

## **CONCLUSION**

Alkyd paints based on granite quarry dust as an extender were formulated and found to possess anti-corrosive properties on mild steel.

- 1. The surface and through dry times of the prepared paints were within acceptable limits, an indication that the incorporated granite quarry dust did not adversely affect the drying of the paints.
- 2. The dry paint films exhibited good impact resistance, and hardness property; the latter generally increased with granite quarry dust content. The 60, and 80 wt.% granite quarry dust formulated paints had maximum film hardness of 5H.
- 3. Dry paint film thickness which were in the range, 0.10–0.20 mm were obtained for the paint samples, and is an indication that the paints will function as anti-corrosive paints.
- 4. The paint samples exhibited good adhesion on mild steel panels, and the sample containing 20 wt.% granite quarry dust had the least adhesion loss (1.56%).
- 5. The performance of the formulated paints in distilled water, 3% Na2CO<sub>3</sub>, and 3% NaCl were generally good; an indication of the anti-corrosive property of the paints. The good performance is attributed to the presence of inert oxides in the granite quarry dust. The formulations, however, performed poorly in 3% H<sub>2</sub>SO<sub>4</sub>, and fairly in 3% NH<sub>3</sub>.

The present study has highlighted the utility of granite quarry dust as an extender in formulating anti-corrosive paints for the protection of steel. Granite quarry dust which is locally available, easy to process, and generally stable to heat and chemicals will provide a cheap source of extender to the paint industry; and the utilization will be an economic and an efficient way of disposing the waste for a cleaner and greener environment.

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