Performance Evaluation of Local Clay-Extender Pigment on Alkyd Paint Formulations

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Abstract: The performances of Nsu clay extender pigment in alkyd paint formulations have been studied. Xylene was used as the solvent while TiO₂ formulated alkyd paint sample served as a reference in this study. The three particle sizes of the clay used in this study are 0.075, 0.15, and 0.30 mm. The physic-mechanical properties of the paint samples were evaluated according to ASTM. The study showed that paints formulated with Nsu clay had higher viscosities which increased with increases in clay contents at any clay particle size investigated while 100 % TiO₂ formulated alkyd paint had the least paint viscosity. The specific gravities of the formulated alkyd paint samples decreased with increases in clay contents and clay particle sizes. The 100 % TiO₂ formulated alkyd paint had the highest specific gravity. The drying properties of the paint samples were generally good. The alkyd paint formulated with 0.075 mm clay particle size had low drying times than those formulated with 0.15 and 0.30 mm clay particle sizes at any clay content. The dry film thickness of the formulated paint samples were in the range of 0.29 - 0.39. The dry paint film hardness of the formulated alkyd paints increased with increases in clay contents at the three clay particle sizes studied. With the exception of TiO_2 formulated paint sample which exhibited no settling tendency, the formulated paint samples exhibited moderate settling tendencies for 0.075 mm clay particle size when compared with the other clay particle sizes. There was no mildew formation observed on all the paint samples after exposure to rain and sunlight for eight months. There was no evidence of rust in all the formulated paint samples. Generally, all the paint samples exhibited good resistance to distilled water, 3 % H₂SO₄, and Na₂CO₃. All the formulated paint samples were affected on 3 % NH₃ immersion which resulted to wrinkles, with the larger particle sized clay adversely affected. The performance characteristics obtained from the formulated paint samples with the indigenous Nsu clay has highlighted the improved paint properties obtainable with the local clay which should justify their utilization in the surface coatings industries for a variety of different applications

Key words: Nsu clay, calcination temperature, extender pigments, particle sizes, titanium dioxide, alkyd paint, drying properties.

I. Introduction

Environmental concern has become one of the most important topics in the coatings industry which has led researchers to continually search and develop coatings system that are non-toxic and has less volatile organic compound (VOC) recognized to cause serious problems in air pollution. In the coatings industry, clays have been used for many years as renewable natural extender pigments for various coating applications which includes lacquers, varnish and paints.

Extender pigments are an integral part of almost all coating formulations, contributing significantly towards modifying various coating properties such as flow characteristics, abrasion resistance, settling tendency, specific gravity hardness and thickness of dry paint films. The selection and proper blending of suitable extenders helps to optimize several engineering properties and the aesthetics of coatings. Titanium dioxide (TiO₂), a prime pigment, is used extensively in the paint industry. TiO₂ pigment used in paints is of mineral origin and requires long

processings such as grinding, levigation, chemical treatment, etc from ore to final stage which results to significant loss of materials [1]. These processed pigments are expensive and not indigeneously available thereby leading to high cost of paint products. The incoporation of TiO₂ pigment in paints has a deliterious effect on the properties of paint films, and therefore on the applied paint durability. However, TiO₂ is an excellent absorber of ultraviolet light, chemically active and contributes not only to the high cost of coating products but also, increases the rate of photo-catalytic degradation of surface coatings [2,3]. Thus, there have been efforts to find economically fine sized extenders of indigenous origin such as clay that would replace TiO₂ in parts in coating formulations and bring down the cost of coating products to acceptable limits and overcome the disadvantage of paint film degradation caused by TiO₂. The replacement of TiO₂, one of the most expensive ingredients of paint with relatively cheap extenders such as calcined clays without any adverse effect on the service properties of paint is a very

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important research topic to paint researchers [4]. The use of local clay extenders has the potentials of widening coating products for a variety of different applications.

The surface of many object corrode in the air and are damaged by the effect of weather and wear if not protected and taken care of. For example, iron rusts and wood deteriorates in the atmosphere [5]. As many materials become available, the need to modify their surface continues to grow. Many surfaces widely believed to be satisfactory without coating are actually attacked by weather, chemicals, atmospheric pollution or other factors and must be protected before certain uses. The wide variety of surfaces which must be protected and decorated has given rise to an infinite number of coating agents such as paints and vanishes not only to protect such surfaces but also to decorate them [6].

Ewulonu et al [7] studied the performance of local clay-Titanium dioxide core-shell extender pigments in alkyd paints. Results showed that the paint samples formulated using the core-shell pigments generally had low specific gravity (1.30 - 1.380). The dry paint films thickness were in the range of 0.24 - 0.39which indicate that the paint can serve as anticorrosive paint. The adhesion properties were generally good. The use of local clays, Okposi clay from Ohazara Local Government Area, and Amankwo Afikpo clay, both in Ebonyi State, Nigeria in alkyd paint formulations was reported by Igwe and Ezeamaku [8]. The formulated paint samples exhibited good drying properties and compared with commercial whiting formulated paint. The viscosity of the formulated paint samples increased with increases in clay content up to 18.9 wt % of clay content and thereafter decreased with any further increase in clay content. Fly ash has been used as an extender pigment in solvent borne-coatings [9]. However, there were certain disadvantages that limit the use of fly ash in water based coatings [10]. The major problems associated with such coatings are their greater tendency towards increasing corrosion of substrate material owing to the presence of water. The coatings showed improved corrosion and abrasion resistance, and better resistance to chemicals and organic solvents. Saxena and Dhimole [10] characterized and utilized copper tailings as an extender in paint production. Results obtained showed that the developed extender environmentally clean, and cost effective. Ahmed [3] studied the differences in the performances of natural kaolin, thermally treated kaolin (calcined kaolin), and chemically treated kaolin in alkyd based paint in the protection of steel. The result from the study showed that calcined kaolin performed better in the protection of steel than kaolin, while chemically treated kaolin varied in its performance according to the

concentration of the modifier used. The result suggested that kaolin could be used as a reinforcing agent in making other polymer composites such as rubber, and plastic composites.

The present study is aimed at optimizing the key processes of an integrated concept for cost effective extender pigment that will form the basis for architectural paints. The concept integrates clay activation and the conversion of naturally occurring clay mineral into valuable extender pigments for paint production.

II. Materials and Methods.

2.1 Materials

The local clay used in this study was sourced from Nsu, in Ehime Mbano Local Government Area within the South-Eastern region of Nigeria. Analytical grade of titanium dioxide from JHD was used. The extender pigment was prepared using local clay of 0.075, 0.150 and 0.30 mm particle sizes which was found to be the major sizes present in the clay.

2.2 Preparation of Extender Pigments [12]

The Nsu clay was hand dug from the deposit area and sundried. Impurities were removed from the clay before crushing. The crushed clay was calcined at $850~^{\circ}\text{C}$ for three hours. The calcined clay samples were sieved to 0.075, 0.15, and 0.30 mm clay particle sizes which were the major particles contained in the kaolin clay and stored in containers with tight lid for subsequent use.

2.3. Preparation of the Extender Pigmented Alkyd Paints

Alkyd paint samples were formulated based on the prepared extender pigments and ${\rm TiO_2}$. Paints prepared using ${\rm TiO_2}$ served as reference paint. Xylene was used as the solvent while long oil modified alkyd resin based on soya bean oil purchased from Eddyson Nigeria Ltd., Onitsha, Nigeria was used in this study as the binder. The different alkyd paint formulations studied are shown in Table 1. Lead and Cobalt naphthanate with metal contents of 36 % Pb and 12 % Co were used as driers. Cobalt naphthanate was used for surface dry, while lead naphthanate drier was used for through dry.

2.4. Preparation of Paint-Drier Mixtures

A given quantity of the prepared paint sample was weighed, and calculated amounts of lead napthanate and cobalt naphthanate driers were added. The paint-drier mixture was mixed thoroughly using a glass rod. The amount of driers added represents 0.60 wt%

lead (Pb), and 0.06wt% cobalt (Co) which are the amount of these metals normally used in the surface coatings industry [13]. The amount of driers that gave

the above percentage of metals in the paint-drier mixtures was calculated as follows:

Required amount of drier =
$$\frac{\text{weight of resin in paint X \% of metal required}}{\text{\% of metal in drier}}$$
 (1)

2.5. Property Determination on the Prepared Alkyd Paint Samples

The American standard testing methods (ASTM) was used to evaluate the prepared alkyd paint samples for viscosity (ASTM D 1200-10), specific gravity (ASTM D 1475-13), hardness of dry paint films (ASTM D 3363-05), thickness of dry paint films (ASTM D 005-95), settling and skinning tendencies (ASTM D 869-85). The settling tendencies of the paint samples were rated in accordance to the degree of settling on a scale that ranged from 10 to 0 (Table 2). Intermediate conditions were given appropriate odd numbers. The Nigerian Industrial Standard (NIS) procedure was used to study the mildew formation resistance (NIS 278:1990), and media resistance (NIS 268:1989) of the prepared alkyd paint samples. Mild steel panels were used as the coating surfaces for the casting of paint-drier mixtures. The panels were prepared according to ASTM D 609-00 procedure, and were taped to 0.40 mm thickness for the casting of the paint-drier mixtures. The paint-drier wet film thickness was assumed the same as the thickness of the taped layer used. The paint-drier mixtures were cast immediately after their preparations on the taped-off portions of the mild steel panels. The wet paint-drier films were leveled off using a short glass rod. The coated plates were placed on a level surface and were allowed to dry at room temperature. Subsequently, the drying properties were studied.

Table 1.Alkyd Paint Formulation with TiO₂ and Extender Pigments (0.075, 0.15, 0.30 mm)

Constituent, wt. %	T100-NC0	T80- NC20	T60 – NC40	T50 – NC50	T40 – NC60	T20 – NC80
TiO ₂	48.2	38.6	28.9	24.1	19.3	38.6
Calcined clay	0	9.6	19.3	24.1	28.9	9.6
Alkyd resin	147	147	147	147	147	147
Solvent	30	30	30	30	30	30
Total binder	147	147	147	147	147	147
Total pigment	48.2	48.2	48.2	48.2	48.2	48.2
P/B ratio	0.32	0.32	0.32	0.32	0.32	0.32
Drier	3.2	3.2	3.2	3.2	3.2	3.2

Table 2. Rating on Degree of Settling of Paint Samples

S/N	Rating	Description of Paint Condition					
1.	10	Perfect suspension, no changes from original condition of paint.					
2.	8	A definite feel of settling and a slight deposit brought on spatula.					
3.	6	Definite cake of settled pigments spatula drops through cake to bottom of the container under its own weight.					
4.	4	Spatula does not fall to bottom of the container under its weight, difficult to move spatula through cake sidewise and slight edge resistance.					
5.	2	Definite edgewise resistance to movement of spatula.					
6.	0	Cake that cannot be reincorporated with the liquid to form smooth paint by stirring manually					

III. Results and Discussions

3.1. Properties of the Local Clay and Prepared Extender Pigment

The X-ray fluorescence (XRF) determinations on the clay samples indicates the presence of silica (SiO_2) and aluminium oxide (Al_2O_3) in high proportions while the other constituents are present in very small proportions which indicates the kaolinite nature of the clay [12]. The oxides contents of the local clay were observed to increase with increased calcination temperature. The particle morphology of the calcined Nsu clay (extender

www.ijmret.org	Page 32
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pigment) obtained from scanning electron microscope (SEM) shown in Figure 1indicates the presence of subangular shaped structures with sharp edges of different sizes with a total of 67 particles [12]. It was observed that as the clay was calcined, new structures (regular structures) were formed which have the clay appeared as pure and fine particles in the base matrix. The local clays have the following physical properties: pH (5.1), oil absorption (59), refractive index (1.7), and specific gravity (2.54) [12]. The clay has shown great improvement in the specific gravity, and refractive index of extenders and can be considered to impart opacity to polymer coatings. High oil absorption observed with the clay indicates high resin demand without compromising other coating properties [14]. The oil absorption depends chiefly on the particle size, with lower particle sizes being associated with increased oil absorption [15], which also affects several other coating properties including flow characteristics, setting tendency, film durability [7]. The effects of heat on the solubility and colour stability of Nsu clay shows that the clay was not soluble in hydrochloric acid, methanol, toluene, ethanol, 2-propanol, and chloroform, except for the slight solubility of the clays observed in acetic acid when heated. Similarly, the colour of the clay was not affected in the media investigated either in the cold or when heated except for slight colour change in the presence of hydrochloric acid on heating [12]. Therefore, paints formulated with this clay may not be suitable for use in hydrochloric prone environment. The same recommendation applies for the clay in acetic acid (CH₃COOH) prone environments.

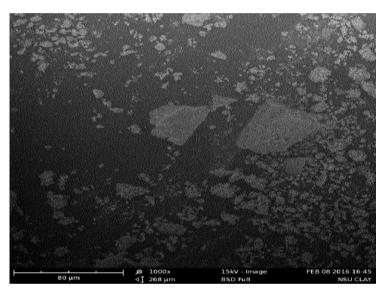
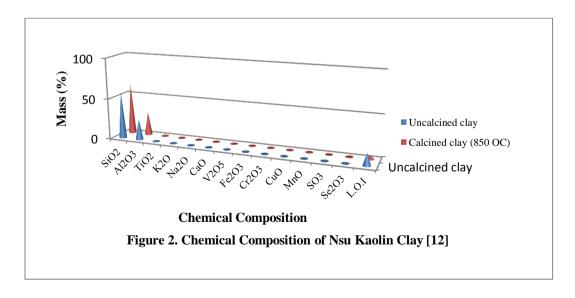


Figure 1. SEM micrograph of calcined Nsu kaolin clay [12]



3.2 Viscosity

The viscosity of the formulated alkyd paint samples are presented in Table 3 and illustrated graphically in Figure 3. The viscosities of the formulated paint samples were observed to increase generally with increases in clay content at any clay particle size considered. Similarly, at any clay content considered, the viscosities were observed to decrease with increases in clay particle sizes. Similar result was reported by Ewulonu et al [7] who studied the performance of local cay-titanium dioxide core-shell extender pigments in alkyd paint and found that the paint viscosities increased with increases in extender pigments. Also Igwe and Ezeamaku [7] who studied the use of local clays in alkyd paint formulations reported increases in paint viscosities with increased clay contents. For many systems such as decorative brush-applied paints and high build protective systems, a relatively high paint viscosity is required. In such systems, extender pigments are added to the paints during formulation to increase the paint viscosity in a way as not to adversely affect the flow and leveling characteristics of the formulated paints [16]. The viscosity of any coating system is the result of interactions between its various constituents and particle shape [17]. In the present study, the contents of TiO₂ and the extender pigment was varied without changing the proportions of other ingredients and this gives an idea of the effect of clay content on paint viscosity. Viscosity affects the application and flow properties of a coating and is generally adjusted according to the intended application [13]. According to the Nigerian Industrial Standard NIS 268: 1989, the minimum viscosity for gloss paint shall be 22 centipoise. Thus, the kaolin clay formulated paint samples in this study satisfied the viscosity requirement for an oil paint. The range of viscosities observed in this study makes them suitable to be applied by spray or brush [9].

Table 3. Coating Properties of Nsu Clay Formulated Alkyd Paints

Formulation Code	Viscosity (poise)	Specific Gravity	Drying Time (min)			Film Thickness	Pencil Hardness
Couc	(poise)	Gravity	Dust	Tack Free	Through	(mm)	Tai uness
			Free		Dry		
			0	.075 mm			
T100 – NC0	15.2	1.31	38	98	150	0.32	1 (6B)
T80 - NC20	28.4	1.28	36	105	160	0.32	8 (H)
T60 - NC40	36.9	1.25	39	108	163	0.35	9 (2H)
T50 – NC50	43.1	1.25	43	106	165	0.35	10 (3H)
T40 – NC60	49.8	1.23	50	109	169	0.38	9 (2H)
T20 - NC80	51.7	1.20	65	130	175	0.39	11 (4H)
			().15 mm			
T80 - NC20	23.4	1.25	50	107	164	0.30	3 (4B)
T60 - NC40	24.3	1.23	45	110	165	0.31	5 (2B)
T50 – NC50	38.5	1.21	39	116	167	0.34	5 (2B)
T40 – NC60	41.6	1.21	51	119	170	0.36	5 (2B)
T20 - NC80	46.4	1.20	67	132	174	0.34	10 (3H)
0.30 mm							
T80 - NC20	23.8	1.23	49	126	166	0.27	2 (5B)
T60 - NC40	22.9	1.22	48	128	169	0.32	3 (4B)
T50 – NC50	26.6	1.20	47	129	173	0.33	2 (5B)
T40 – NC60	37.3	1.19	59	131	177	0.35	2 (5B)
T20 - NC80	45.8	1.19	67	134	179	0.36	9 (2H)

^{*}NC – Nsu clay, T – Titanium dioxide

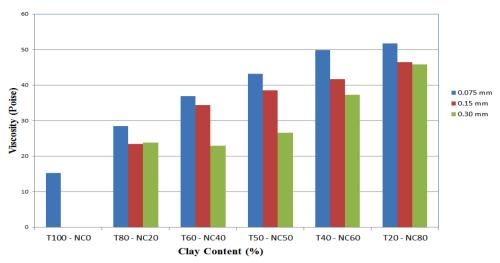


Figure 3. Effect of Nsu Clay Content on the Viscosity of Alkyd Paint Formulation

3.3 Specific gravity

The specific gravity of Nsu clay formulated alkyd paints presented in Table 3 is illustrated graphically in Figure 4. Generally, the specific gravity of the local clay formulated paint samples were observed to decrease with increases in clay contents and clay particle sizes. Thus, the alkyd paints formulated using clay of particle size 0.075 mm exhibited the highest specific gravity than alkyd paints formulated with clay of particle sizes 0.15 and 0.30 mm. The alkyd paints formulated using 80 % clays and 20 % TiO₂ exhibited the least specific gravity.

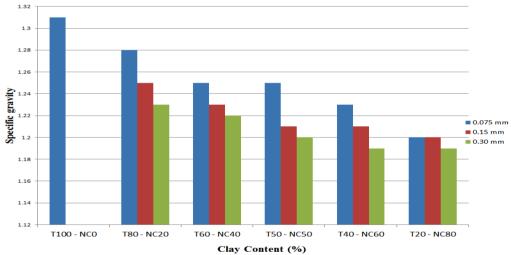


Figure 4. Effect of Nsu Clay Content on the Specific Gravity of Alkyd Paint Formulation

The 100~% TiO₂ formulated alkyd paint exhibited the highest specific gravity (1.31) when compared to those formulated with the local clay extender pigment at the three clay particle sizes studied. The observed result is attributed to the high specific gravity of TiO₂ (4.20) [16] as compared to the specific gravity of the clay used in this study. The alkyd paints formulated with 80 % clay exhibited the lowest specific gravity values. Specific gravity is used to determine the coverage of paint products on a substrate. The study further revealed that alkyd paints formulated with 40 and 50 % clay of 0.075 mm clay particle size, and 20 % clay of 0.15 mm clay particle size exhibited the same specific gravity (1.25). Similarly, alkyd paints formulated with 60 % clay of 0.075 mm clay particle size, 40 % clay of 0.15 mm clay particle size, and 20 % clay of 0.30 mm clay particle size exhibited the same specific gravity (1.23). Furthermore, alkyd paints formulated with 50 and 60 % clay of 0.15 mm clay particle size and 60 and 80 % clay of 0.30 mm clay particle size had the same specific gravity values of 1.21 and 1.19 respectively.

3.4 Drying studies on paint samples

The drying properties of the prepared paint samples shown in Table 3 are illustrated graphically in Figures 5-7. The drying properties studied are dust-free dry, tack-free dry, and through-dry.

3.4.1Dust - Free Dry Times

The dust free dry times of Nsu clay formulated alkyd paints were observed to increase with increases in clay content for clay particle size, 0.075 mm. There was no observed trend on the variation of dust free dry times of the formulated alkyd paints with increased clay contents for 0.15 and 0.30 mm clay particle sizes. The formulated paint samples generally had appreciable higher – dust free dry times at clay contents, 80 % than the other clay contents investigated in this study at any clay particle size considered. The 20 % clay content of 0.075 mm clay particle size had the least dust free dry time.

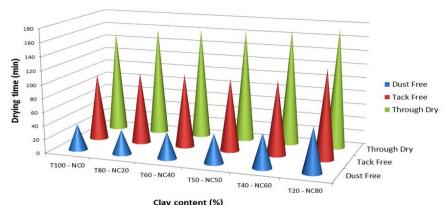


Figure 5. Effect of 0.075 mm particle sized clay content on the drying properties of Alkyd Paint Formulations

3.4.2 Tack – Free Dry Times

An applied paint film is considered tack-free when it did not stick to foreign objects on application of little pressure. The tack-free dry times of the formulated alkyd paints exhibited dry times that increased with increases in clay contents at any clay particle size considered and increased with increases in clay particle sizes. The TiO₂ formulated paint sample exhibited the least tack-free dry time of 98 minutes while the 80 % (particle size 0.30 mm) had the highest tack-free dry time of 134 minutes. The tack-free dry times of the formulated paint samples were generally good. According to Nigerian Industrial Standard (1989), the tack-free dry time of gloss paint shall not exceed 6 hours from the time of application.

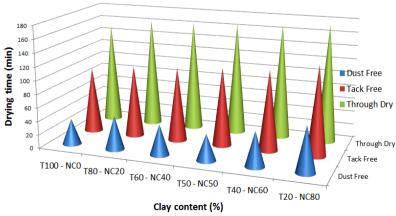


Figure 6. Effect of 0.15 mm particle sized clay content on the drying properties of Alkyd

Paint Formulations

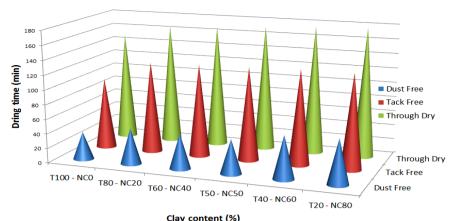


Figure 7. Effect of 0.30 mm particle sized clay content on the drying properties of Alkyd
Paint Formulations

3.4.3 Through – Dry Times

The through dry times of the formulated paint samples as illustrated graphically in Figures 5-7 shows that TiO_2 formulated paint sample exhibited the least through-dry time (150 minutes) while the 80 % clay formulated paint sample (clay particle size, 0.30 mm) had the highest through dry time of 179 min. Like was noted in the dust-free and tack-free dry times of the prepared paint samples, the through dry times of the local clay extender pigment formulated paint samples increased with increases in the amount of clay incorporated into the paints at the three clay particle sizes investigated. Generally, the through-dry times of the formulated paint samples increased with increases in clay particle sizes at any clay content considered.

3.5 Thickness of dry paint films

The thicknesses of the dry paint films presented in Table 3 are illustrated graphically in Figure 8. The thickness of the formulated alkyd paints were observed to increase with increases in the clay contents at the three clay particle sizes investigated except for decreases with clay content 80 % which occurred at clay particle sizes 0.15 and 0.30 mm. The figure further show that the thickness of the paint samples decreased with increases in clay particle sizes at any clay content considered. The thickness of the formulated alkyd paint samples were in the range of 0.27-0.39 mm. The film thickness of 20 % clay formulated alkyd paints at particle sizes, 0.15 and 0.30 mm were less than that of TiO_2 formulated alkyd paint (0.32 mm). The 80 % clay formulated alkyd paint (clay particle size 0.075 mm) exhibited the highest film thickness (0.39 mm) when other clay paints were considered. It is important to note that weight loss due to weathering is proportional to film thickness up to 20 μ m, above which the rate of weight loss becomes independent of the thickness. A film thickness of more than 20 μ m performs well as a barrier resistant to weathering [13]. Thus, the film thicknesses of the formulated paint samples in this study which are greater than 20 μ m is an indication that the local clay extender pigment formulated alkyd paints can function as anti-corrosive paints.

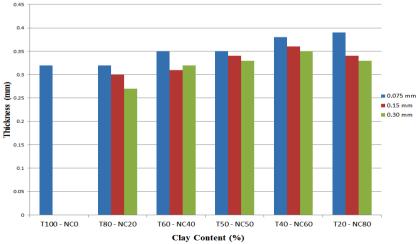


Figure 8. Effect of Nsu Clay Content on the Thickness of Alkyd Paint Formulations

3.6 Hardness of Dry Paint Films

The hardness of dry paint films presented in Table 3 is illustrated graphically in Figure 9. The hardness of the formulated alkyd paints (clay particle size, 0.075 mm) was observed to generally increase with increases in clay content except for the decrease observed with clay content 60 %. The hardness of the formulated paint samples (clay particle size, 0.15 mm) did not exhibit a general order with increases in clay contents. The 20 % clay formulated alkyd paint sample exhibited the least dry paint film hardness (6B) (1). The formulated paints containing 40, 50 and 60 % clay exhibited the same dry paint film hardness (2B) (5). Like was noted for 0.15 mm clay particle size, the alkyd paints formulated with 0.30 mm clay particle size did not exhibit any desirable order with increases in clay content. The 20, 50 and 60 % clay extender pigment formulated paints exhibited the same dry paint film hardness of 5B (2). The 40 % clay formulated paint sample had dry paint film hardness of 4B (3). The 80 % clay content exhibited the highest paint film hardness at the three clay particle sizes studied while the 100 % TiO₂ formulated paint had the least paint film hardness of 6B (1). The paints samples formulated with 0.075 mm clay particle size were generally hard, while the paints formulated with 0.15 and 0.30 mm clay particle sizes exhibited soft except the 80 % clay content which exhibited hard at the two clay particle sizes of 0.15 and 0.30 mm. The observed increase in paint film hardness with increased clay content is an indication of improved crack and scrub resistance of Nsu clay extender pigment in the formulated alkyd paints. Generally, at any clay content or clay particle size considered, the 0.075 mm clay particle size formulated alkyd paints exhibited the highest dry paint film hardness than the other clay particle sizes studied and the observed order of hardness is 0.075 mm > 0.15 mm > 0.30 mm formulated alkyd paint samples. It is important to note that dry paint film hardness is an important property that is used to describe dry paint films as this property gives indication on the effectiveness of the binder molecules, pigment particles, and additive materials in attracting one another. Generally, high degree of pigmentation gives strength and stiffness to paint films [18]. This is because the pigment particles can act as a load bearing part of the film thereby restricting movement within the film.

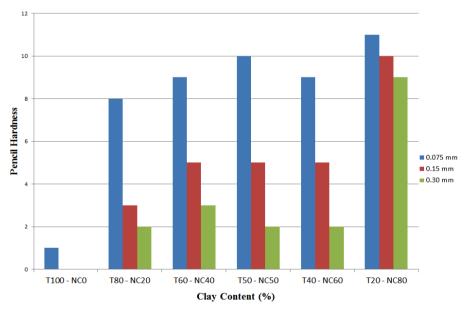


Figure 9. Effect of Nsu Clay Content on the Hardness of Alkyd Paint Formulations

3.7 Mildew Formation and Light Fastness of Dry Paint Films

All the formulated alkyd paint samples did not show any sign of mildew formation during the eight months of exposure to rain and sunlight. This is an indication that Nsu clay extender pigment performed well in the formulated paints. The light fastness of paint is its ability to resist deterioration under the action of sunlight. Deterioration becomes evident as a colour change in the applied paint. In the present study, the formulated alkyd paint samples shaded their colours on outdoor exposure to rain and sunlight. However there was no evidence of rust in any of the exposed alkyd paint film samples studied. This is an indication that the Nsu local clay formulated alkyd paints can withstand environmental challenges and therefore, can be applied in a corrosive environment.

3.8 Skinning and Settling Tendency

The formulated alkyd paint samples did not exhibit any sign of skin formation within the eight months of storage. However, various degrees of settlings were observed in the paint samples during the eight months of paint storage, Table 4. The table shows that 100 % TiO₂ formulated alkyd paint did not show any settling tendency at the duration of eight months. The Nsu clay formulated paint samples of particle sizes 0.075 and 0.15 mm exhibited moderate settling property. Conversely, all the paint samples formulated with the local clay extender pigment at clay particle size, 0.30 mm had fair settling tendency. Pigments of low specific gravity generally have low settling tendencies in paint. Conversely, pigments of high specific gravity are more prone to rapid settlement [16]. The wetting of pigments and extenders in paint media has an important bearing on the settling tendency of paints. Also the oil absorption of pigments generally affects the settling tendencies of paints. The high oil absorption exhibited by the local clay extender pigment and consequently clay particle sizes may have contributed to the various settling tendencies observed in the study.

3.9 Media Resistance Tests on Dry Paint Films

The visual changes that occurred on the formulated alkyd paint films after immersion in $3\%~H_2SO_4$, $3\%~Na_2CO_3$, $3\%~NH_3$, and distilled water are presented in Table 4.

Table 4. Me	dia Resistance	e of Nsu Ka	olin Clay I	Ory Paint Films

Formulation Code	3 % H ₂ SO ₄	3 %	3 % NH ₃	Distilled	Settling Test		
		Na ₂ CO ₃		Water			
	0.075 mm						
T100 – C0	0	0	1	0	10		
T80 - C20	0	0	2	0	8		
T60 - C40	0	0	2	0	8		
T50 - C50	0	0	2	0	6		
T40 - C60	0	0	2	0	6		
T20 - C80	0	0	2	0	4		
		0.15 mm					
T80 - C20	0	0	2	0	8		
T60 - C40	0	0	3	0	6		
T50 - C50	0	0	3	0	6		
T40 – C60	0	0	3	0	4		
T20 - C80	0	0	3	0	3		
0.30 mm							
T80 - C20	0	0	4	0	6		
T60 - C40	0	0	4	0	4		
T50 - C50	0	0	4	0	4		
T40 - C60	0	0	4	0	4		
T20 - C80	0	1	4	0	3		

From the table, it was clearly observed that all the formulated alkyd paint samples performed satisfactorily in 3% H₂SO₄, 3% Na₂CO₃ and distilled water as no film defect was observed in any of the immersed samples. The dry paint film samples were affected differently in 3% NH₃ depending on the clay particle size. The paint samples formulated with clay particle size 0.075 mm showed slight wrinkles on the surface on immersion in 3% NH₃; for the dry paint films formulated with clay particle size of 0.15 mm, the wrinkles were more pronounced when the dry paint films on mild steel panels were immersed in 3% NH₃. However, the formulated paint sample containing 20% of clay content shows slight wrinkles on the dry paint surface. The paint samples formulated with clay of particle size, 0.30 mm exhibited shrinkages on mild steel panels on immersion in 3% NH₃. The 100% TiO₂ formulated alkyd paint sample dry film was very slightly affected in 3% NH₃ as few wrinkles were observed on the film surface.

The present study has shown that clay particle size is an important factor that affects paint behaviour in NH₃ prone environment. The good performance of the clay formulated alkyd paints in 3 % H₂SO₄ and 3 % Na₂CO₃ is attributed to the presence of inert oxides in the clay [9]. It is important to note that water in one way or the other is the common enemy to most materials of construction. Improved water resistance has been a request of the consumer for exterior applications in order to protect the underlying substrate from water penetration. Less obvious to the consumer perhaps is the need for the paint film to be permeable to any water coming from within

the substrate to prevent cracking and blistering [19]. The absorption of water by paint films can lead to loss of components from the film, and hence, deteriorates the paint film. Generally, the performance of any coating system is the product of its individual raw materials and their mutual interactions [20]. The slight change in the chemical reactivity of paints formulated using varying amounts of Nsu clay at the three clay particle sizes of 0.075, 0.15, and 0.30 mm in NH_3 solution should restrict the application of the paints in areas where high durability against corrosive atmosphere is required.

3.10 SEM Micrographs of Formulated Alkyd Paint Samples

The SEM micrographs of the local clay extender pigment formulated alkyd paint samples of different clay particle sizes are shown in Figures 10 to 12. All the figures show uniform distribution of particles throughout the base matrix. These particles, which appeared angular shaped [12] were seen to increase with increases in clay particle sizes.

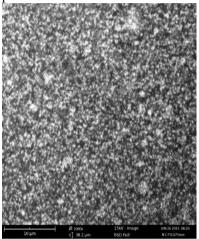


Figure 10. SEM Micrograph of 20 % Nsu Clay Formulated Paint at 0.075 mm Clay Particle Size



Figure 11. SEM Micrograph of 20 % Nsu formulated paint at 0.15 mm Clay particle size

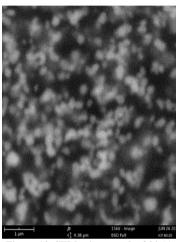


Figure 12. SEM micrograph of 20 % Nsu formulated paint at 0.30 mm clay particle size

The angular-shape observed in this clay exerts reinforcing effects of the clay. More so, the angular shaped particles were seen to overlap each other which made it difficult for water to penetrate the paint films [7] and this resulted to good chemical resistance, thick and hard paint films observed in this study.

IV. Conclusion

Alkyd paints have been prepared using Nsu local clay extender pigment. TiO2 formulated paint sample served as a reference alkyd paint sample. The viscosities of the formulated paint samples increased with increases in clay contents and decreased with increases in clay particle sizes. The 100 % TiO2 formulated paint sample had the least paint viscosity while paint samples containing 80 % of clay content had the highest paint viscosity at the three clay particle sizes studied. The specific gravities of the formulated paints decreased with increases in clay contents at the three clay particle sizes studied. The 100 % TiO₂ formulated paint sample had the highest specific gravity. The thickness of the dry paint film samples increased with increases in clay contents. The paint formulated with 0.075 mm clay particle size had the best dust-free, tack-free and through-dry times at any clay content. Similarly, the formulated paint samples with 0.075 and 0.15 mm clay particle sizes had moderate settling tendencies while paints formulated with 0.30 mm clay particle size exhibited fair settling tendencies. The 100 % TiO2 formulated paint sample did not exhibit any settling tendency.

There was no formation of mildew on any of the formulated paint samples studied after outdoor exposure to rain and sunlight. However, all the dry paint film samples shaded their colours on exposure to sunlight. The dry paint film samples generally were unaffected after immersion in distilled water, 3 % H₂SO₄, and 3 % Na₂CO₃. All the formulated alkyd paint dry films were slightly affected in 3 % NH₃, with 0.30 mm clay particle size formulated paint film samples being adversely affected in the medium. The present study has highlighted the utility of Nsu local clay extender pigment in alkyd paint formulation. The clay is indigenously available and easy to process with insignificant loss of material. It is expected that Nsu clay find utilization in the surface coatings industry as this will reduce the cost and dependence on imported extender pigments.

References

- [1] Morgans, W. M. (1990). *Outlines of paint technology*. London: Edward Arnold, 43-51.
- [2] Paint and Coating Industry Magazine (PCI, 2005). A comprehensive understanding of TiO_2 pigment durability.

- http://www.pcimag.com/articles
- [3] Ahmed, N. M. (2013). Comparative study on the role of kaolin, calcined kaolin and chemically treated kaolin in alkyd-based paint for protection of steel. *Pigment and Resin Technology*, 42(1), 3-14.
- [4] Ciullo, P. A. (1996). *Industrial minerals and their uses*. Westwood, New Jersey: Noyes Publication, 125-136.
- [5] Udeozo, I. P. Umedum, N. L., Okoye, N. H. and Kelle, I. H. (2013). Formulation of Glossy Emulsion Paint. International Journal of Science and Technology, 13(1), 822-828.
- [6] Lambourne, R. Paint and Surface Coatings Ellis Horwood Ltd., Chichester.
- [7] Ewulonu, C. M., Igwe, I. O. & Onyeagoro, G. N. (2016). Performance of local Claytitanium dioxide core-shell extender pigments in alkyd paints. *Advances in Nanoparticles*, 5, 90 102.
- [8] Igwe, I. O. &Ezeamaku, L. U. (2010). The use of local clays in alkyd paint formulation. *Malaysian Polymer Journal*, *5*(1), 81-94.
- [9] Tiwari, S. &Saxena, M. (1999). Use of fly ash in high performance industrial coating. *British Corrosion Journal*, *34*, 184-191.
- [10] Wicks, Z. W., Jones, F. N. I. & Pappas, S. P. (1992). Organic coatings: science and technology. New York: Wiley, 208-217.
- [11] Sexena, M. & Dhimole, L. K. (2005). Utilization and value addition of copper tailing as an extender for development of paints. *Journal of Hazardous Materials*, 129(1-3), 50-57.
- [12] Chukwujike, I. C. &Igwe, I. O. (2016). Extender properties of some Nigerian clays. *Journal of Minerals and Materials Characterization and Engineering*. 4(5), 1-13.
- [13] Tan T. D. (1995).Durability and TiO₂ pigments.*Paintindia Annual Journal*, 84-93.
- [14] Osabor, V. N., Okafor, P. C., Ibe, K. A. &Ayi, A. A. (2009). Characterization of clays in Odukpani South Eastern Nigeria. *African Journal of Pure and Applied Chemistry*. 3(5), 79-85.
- [15] Vanderbilt, R. T. (2013). An introduction to mineral fillers for paints and coatings.www.vanderbiltminerals.com.
- [16] Boxall, J. & von-Fraunhofer, J. A. (1986). *Concise Paint Technology*. New York: Chemical Publishing.
- [17] Enrique, M. S. (2005). *Impact of particle morphology on the rheology of coatings*. Atlanta, Georgia: Institution of Technology.
- [18] Nelson, G. L. (1995). *Adhesion, in paint and coating testing manual,* (14thed), In J. V. Koleske, Philadelphia P.A: ASTM, 44.
- [19] James, V. & Leger, P. (2011). Mordern

- formulations for architectural paints. Retrieved from www.coatingsgroup.com 05/04/14.
- [20] Tulloch, R. C. (1994). Coating titania particles. *Paintindia*,44(10), 35-40.