

Pigment Extender Properties of Rice Husk Ash in Emulsion Paint

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ABSTRACT: This study investigated the applicability of rice husk ash (RHA) as a new pigment extender in emulsion paint by comparing the properties of RHA-filled emulsion paints with those of calcium carbonate used as the reference standard. RHA obtained by controlled incineration of rice husks in a muffle furnace at a temperature of 650°C and duration of 4hrs, was ground to obtain powdery RHA of smaller particle size. The RHA powder and calcium carbonate were sieved to obtain the same particle size range of approximately 32-63microns (μm) used in the formulation of emulsion paints. White emulsion paints were produced with varied compositional levels of RHA and calcium carbonate which ranged from 2 to 12% by weight and the physical and performance characteristics of the paints determined. Styrene-acrylic resin was used as binder in the paints to which paint additives were also added. RHA showed superiority to calcium carbonate in thickening effect at all extender levels but it was markedly greater at higher levels of 8-12%. RHA emulsion paints also surpassed those of calcium carbonate in viscosity, opacity, settling resistance, and drying properties but were similar to those of calcium carbonate in scrub resistance, weather resistance, storage stability, temperature stability and application properties.

KEYWORDS: emulsion paint, rice husk ash (RHA), pigment extender, calcium carbonate, silica, amorphous silica

I. INTRODUCTION

The search for new and cheaper raw materials with improved properties is an ongoing process in the paints industry. In recent times, 'renewability' has been included as a raw material requirement due to increased global sensitivity to environment protection. This has also stimulated research investigations into renewable (plant-based) resource as an alternative to non-renewable (fossil-based) resource for paints and other surface coatings [1][2][3]. Paint is a fluid material, which when spread over a surface in a thin layer, forms a solid, cohesive and adherent film [4]. Paints are primarily designed to protect and decorate surfaces and have three primary components; binder, diluent/solvent, pigment and a secondary component-the additives. Emulsion paints, are also called water-based paints because the diluent (dispersion medium) is water, which dries by evaporation on application of the paint on a substrate [5]. Pigments are coloured organic and inorganic insoluble substances used to give colour, opacity, consistency, build, durability and other properties to surface coatings [6][7]. Pigment extenders, often simply called 'extenders' or 'fillers' are inert, inorganic compounds that are added to paints in order to increase bulk, reduce cost and confer some special properties/ effects to the paint [7]. Extenders generally supplement the pigments by improving paint properties such as consistency, resistance to weathering, gloss, levelling, coverage etc [4][7] so can be referred to as 'auxiliary pigments'. Calcium carbonate is a widely used extender in surface coatings; others include kaolin (china clay), barytes, talc and silica flour [4].

Rice husk, an agro-waste generated in large amounts during rice-milling, has great potential as bio-resource for industrial applications due to its abundant availability, little or no cost and 'renewability'. The utilization of rice husks as industrial resource will minimize its environmental nuisance value. Rice husk ash (RHA) obtained by burning rice husks, has a high silica content compared to other lignocellulosic materials. The silica content of RHA ranges from 70 to 97% [8][9][10]. This high silica content enhances the value of RHA as a potential renewable alternative to natural silica (quartz) and synthetic silica for various industrial applications. Several researchers have explored the potentialities of RHA through various research investigations which include production of special cement and concrete mixes [11][12], as an adsorbent [13][14], production of heterogeneous catalysts [15][16], for zeolite synthesis [17][18][19] and more recently, as filler in wood adhesives [20]. Several studies [21][22][23] have shown that RHA

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 8, August 2015

obtained at a combustion temperature of 500-800⁰C is amorphous silica while RHA obtained above 800⁰C is predominantly crystalline silica, which has few practical uses. The transformation of silica from the amorphous to the crystalline form takes place at a temperature of about 800⁰C [24]. Crystalline and amorphous forms of silica have different properties hence the need to control the temperature and duration of combustion of rice husks so as to produce ash with the required properties for the desired end use. Amorphous silica has a disordered structural arrangement and is consequently porous, more reactive, has greater flexibility and larger surface area than crystalline silica which has an ordered silica network structure, thus has structural rigidity and is also far less reactive due to its electrical neutrality [24][25]. RHA used in this study is therefore predominantly amorphous silica.

In this work, the pigment extender properties of RHA in emulsion paint were studied. The objective of the study was to determine the suitability of RHA as a new extender in emulsion paint and to compare the physical, performance and application characteristics of RHA-filled emulsion paints with those of calcium carbonate, a widely used commercial extender.

II. MATERIALS AND METHODS

MATERIALS

Equipment for Production and Testing of Paint Samples

Mini stirrer (Diaf A/S Copenhagen NV. Denmark) ; I.C.I Digital Rotothinner Viscometer (Sheen 455N); pH meter (Mettler Toledo MP 220); Washability Tester: (Elcometer 1720) ; K – bar (Sheen) ; Digital Weighing scale (Sauter); Wt per litre (specific gravity) cup (Sheen)

Paint Raw Materials

The raw materials used to formulate the emulsion paints were grades designed for paint production and were all obtained from assured suppliers/ importers of paint raw-materials. They all met the required specifications when subjected to standard Quality Control tests.

METHODS

Washing and Drying of Rice Husks

Milled rice husks obtained from a rice mill located in Abakaliki, in Ebonyi State of Nigeria were washed several times to remove sand and stone contaminants. The washed rice husks were then spread on plastic trays and other extraneous materials like broken rice grains were removed by handpicking. The wet rice husks were dried at 100⁰C to a constant weight in an electric oven.

Production of Rice Husk Ash

A two-stage method, similar to that used by Sugita [26] was adopted for the production of RHA. The clean rice husks were put in a stainless steel pot, which was then placed on a gas stove and the rice husks incinerated until there was no further emission of fumes. The black, carbonized rice husk char obtained was put in medium-size crucibles which were placed in a muffle furnace. The muffle furnace was then switched on and left to attain the desired combustion temperature of 650⁰C, which was maintained until the required duration of 4 hours, was exhausted. The crucibles were withdrawn from the furnace and the milky-white, granular ash samples obtained, allowed to cool, then stored in a desiccator.

Preparation of Extenders for Paint Production

The granular RHA produced after incineration in the muffle furnace was ground with a ceramic mortar and pestle until the ash was reduced to fine particle size with a powdery texture (RHA powder). The RHA powder and calcium carbonate were passed through a standard sieve of aperture 63 microns (µm) to ensure a uniform particle size range of 32-63µm in the extenders as is required in the production of emulsion paints[27].

Formulae for Production of White Emulsion Paints [27]

The formulae for the white emulsion paints produced using RHA (RHAWEP) and calcium carbonate (CCWEP) extenders are presented in Table 1. The Table shows the different components of the white emulsion paints, their functions and weight percentages of RHA and CaCO₃ used in formulating the paints.

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 8, August 2015

Table 1: Formulae for Production of White Emulsion Paints using 2 to 12% by weight of RHA and CaCO₃

Component	Function	Components (in weight %) of RHA and Calcium carbonate White Emulsion Paints					
		2	4	6	8	10	12
Water	Solvent/diluent	43.15	41.15	39.15	37.15	35.15	33.15
Calgon PT	Wetting agent	0.15	0.15	0.15	0.15	0.15	0.15
Acticide Bx	Preservative	0.50	0.50	0.50	0.50	0.50	0.50
Berol 09	Emulsifier/surfactant	0.20	0.20	0.20	0.20	0.20	0.20
Antifoam	Defoamer	0.20	0.20	0.20	0.20	0.20	0.20
Coatex	Dispersant	0.20	0.20	0.20	0.20	0.20	0.20
*RHA/CaCO ₃	Extender, thickener	2.00	4.00	6.00	8.00	10.00	12.00
Titanium dioxide	White pigment	19.00	19.00	19.00	19.00	19.00	19.00
Styrene-acrylic resin	Binder	33.00	33.00	33.00	33.00	33.00	33.00
Natrosol	Cellulosic thickener	0.50	0.50	0.50	0.50	0.50	0.50
Ammonia	pH adjuster	0.10	0.10	0.10	0.10	0.10	0.10
Texanol	Coalescing agent	1.00	1.00	1.00	1.00	1.00	1.00
	Total	100%	100%	100%	100%	100%	100%

Procedure for Production of Emulsion Paints

The production method described is the same for all levels of extender but the quantities specified in the procedure are for the production of 300g of the emulsion paint which is obtained by multiplying the % by weight of each component by 3.

(i) The following components (weights in g) were loaded into a plastic vessel: Water (part), (15.15), Calgon PT (0.45) Acticide Bx (1.50), Berol 09 (0.60), Antifoam (0.30) and Coatex (0.60) (ii) They were stirred at low speed by means of a mini stirrer until Calgon PT dissolved. (approx 5 mins) (iii) The following ingredients were added into the same pot with low-speed stirring: RHA (24.00) Titanium dioxide (57.00) (iv) The mixture was then stirred at high speed (pigment dispersion stage) for about 20mins while intermittently scraping the sides of the vessel. When the dispersion was satisfactory, the stirring speed was reduced and the following components were added with slow stirring: Styrene - acrylic resin (99.00), Water (26.00) (vi) Slurry of the following was prepared in a separate container and added with stirring to the main pot: Water (70.35), natrosol(1.5). (vii) The following components were finally added and the mixture stirred until natrosol dissolved (approx 25mins): Antifoam (part) (0.30), Ammonia (0.30), Texanol (3.00). (iii) The paint was finally checked for satisfactory dispersion of the pigment with the aid of a small piece of a 100-mesh sieve and stored in sample cans.

PHYSICAL TESTS

Physical tests were carried out on white emulsion paints produced with RHA (RHAWEP) and CaCO₃ (CCWEP). The tests were carried out using standard testing methods [28] [29] with some modifications where necessary and include in-can assessment, pH, wt per litre and viscosity.

a. In-Can Assessment

The finished emulsion paint samples were put in plastic sample cans, gently and thoroughly stirred, then visually observed for colour, homogeneity/consistency and smoothness.

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 8, August 2015

b. Determination of pH of Emulsion Paints

The pH meter was switched on and the pH electrode was standardized with a buffer solution of pH 7.0. The glass electrode was rinsed with distilled water and dried with tissue paper. It was then dipped into the emulsion paint and the pH reading taken.

c. Determination of specific gravity (weight per litre)

The weight per litre cup was first weighed empty on a digital weighing scale. The value obtained was 'tared'. The paint sample was poured into the cup and any excess paint cleaned off from the hole in the lid. The cup with the paint was weighed to obtain the wt per litre value of the paint.

d. Determination of Viscosity of Emulsion Paints

The paint sample was poured into a sample can to a level of about 2.5cm from the top of the can. The can was then placed on the turntable of the rotothinner after it had been switched on. The disc was immersed into the emulsion paint inside the sample can. The disc was allowed to rotate inside the paint sample until the peak viscosity value was obtained. The viscosity reading was taken from the graduated scale around the turntable. The disc was raised and the sample can removed, following which the disc was thoroughly cleaned with a brush and water.

PERFORMANCE TESTS

The white emulsion paints were subjected to the following performance tests: opacity, drying properties, scrub resistance, settling resistance, storage stability and temperature stability.

e. Determination of Opacity of Emulsion Paints

The paint sample was scooped with a palette knife and spread evenly across the width of a Morest chart (black and white striped paper) about 4cm from the edge of the paper. The paint was then evenly applied down the length of the paper by means of the K-bar (stainless steel bar) and left to dry. A second coat was applied using the same application technique but with a space of 5cm left from the edge of the first coat. The opacity was assessed by the extent to which the black and white stripes were covered by the paint.

f. Drying Properties

The emulsion paint was applied on brush-out cards and the time it took for the film to become surface-dry and hard-dry respectively was measured.

g. Determination of Scrub resistance (Washability) [28][29]

This test is used to determine the number of wet brushing cycles that the emulsion paint film can withstand before any scratch is observed on the film. Two coats of RHA paint were applied to an asbestos panel of dimension 6"x 12" using a brush. The paint was allowed to dry under ambient conditions and allowed to age for seven days. The test panels were mounted on the washability (scrub resistance) testing machine fitted with nylon brushes. The machine was set at 5,000 cycles to determine if the paint would be washed off the asbestos panel after brushing the surface 5,000 times.

h. Determination of Settling Resistance

The emulsion paint samples were thoroughly stirred then poured into 500ml glass jars which were tightly covered. The samples were observed intermittently for any phase separation due to settling of the pigment and extender at the bottom of the container. The observation lasted for a period of six weeks at the end of which the height of the water phase at the surface of the paint samples was measured. The samples were then stirred to check the presence of hard settlement.

i. Determination of Storage Stability [28] [29]

The emulsion paint samples were put in plastic sample cans which were covered and the edge of the covers sealed with masking tape to ensure air tightness so as to prevent loss of moisture from the cans. Three samples of each emulsion paint variant were stored at $27\pm 2^{\circ}\text{C}$ (air-conditioned room) while others were stored at $0\pm 2^{\circ}\text{C}$ (in a refrigerator) for 12 months. The stored samples were periodically monitored for any adverse changes in the paint quality such as coagulation, changes in colour and odour, viscosity loss, pH drift, phase separation etc.

j. Determination of Temperature Stability

The paint samples were poured into glass jars to about three quarters full and kept in an electric oven at a temperature of $45\pm 2^{\circ}\text{C}$ and periodically monitored for any deterioration in quality for a period of 4 weeks.

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 8, August 2015

k. Determination of Weather resistance (Weatherability)

Two coats of the emulsion paint samples were applied on 20cm x 30cm asbestos cement sheets which, after drying were left exposed to weather conditions for 6 months at angles 90° and 45°. At the end of this period, the panels were washed and examined for defects such as cracking, colour fading, blistering, flaking and dirt retention.

APPLICATION PROPERTIES

l. Brushing Properties

The ease of application of the paint (after thinning) was determined using a brush which had been wet and excess water squeezed from it. The paint was applied on asbestos cement panels (300x100mm size) and the brushing/application properties observed.

m. Re-coating Properties

The paint was first thinned with water then applied by means of a brush on an asbestos cement panel (300x100mm size); three coats of paint were applied in all with 2hrs drying time between successive coats. The applied coats were observed for undesirable film properties like lifting of the underlying coats, sagging, flaking, cracking or colour separation.

III. RESULTS AND DISCUSSION

The results of various tests carried out on white emulsion paints produced with RHA (RHAWEP) and calcium carbonate (CCWEP) are discussed herein. Although the extender levels were varied from 2-12%, the higher levels (8-12%) had more pronounced effects on paint quality and form the basic framework for this discussion.

a. In-can Appearance

The formulated emulsion paints were all white, homogeneous viscous liquids with a smooth consistency. However, the CCWEPs samples had a more brilliant whiteness than RHAWEP which were off-white in colour. This slight colour disparity is a reflection of the colours of the extenders as RHA has an off-white colour while CaCO₃ is white or brilliant white in colour. This difference was observed only in white emulsion paints while for other colours there was no noticeable difference between the two extenders. This suggests that RHA can be used to produce all other colours of paint except brilliant white.

b. pH of Emulsion Paints

Table 2 shows the pH values obtained for the emulsion paints at 2-12% levels of RHA and CaCO₃. The pH values of CCWEPs were in the range of 9.00-9.50 and were slightly higher than those of RHA which ranged from 8.00-9.00 at all extender levels.

Table 2: pH Values of RHAWEPs and CCWEPs at 2-12% Levels of Extender

Type of Extender	pH Values of Emulsion Paints produced at 2-12wt% Pigment Extender Levels					
	2	4	6	8	10	12
RHA (RHAWEP)	8.99	9.08	8.98	8.93	8.88	8.88
Calcium Carbonate (CCWEP)	9.51	9.49	9.05	9.45	9.39	9.25

The pH values of RHAWEPs are within the pH specification of 7.00-9.00 for emulsion paints [29]. Also, the pH values showed no variation with increasing extender levels for both RHA and CaCO₃ which suggests that the extender level has little effect on pH. This is not unexpected because pH values of emulsion paints can be affected by many factors such as the pH of the resin and other components as well as amount of ammonia used. The pH of an emulsion paint can however, be adjusted to the desired level by the use of ammonia which is widely used for that purpose in emulsion paints.

c. Specific gravity (weight per litre) Values

The specific gravity, also known as weight per litre values of the formulated emulsion paints were found to be approximately in the range of 1.0-1.2 which is the range for premium quality emulsion paints [27]. However RHA

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 8, August 2015

paints had slightly higher values of 1.00-1.19 than those of CaCO₃ which ranged from 0.98-1.04. The S.G values did not show much variation with increase in extender levels for both RHA and CaCO₃. This may be attributed to several other factors which can affect the S.G of emulsion paint samples such as the specific gravity of the pigments, extenders and other ingredients, degree of dispersion of the pigment, amount of air bubbles trapped in the emulsion and amount of foam generated during the dispersion stage. These are interferences that affect the S.G of the paint which often varies but the degree of variation is usually within the range of ±0.2.

d. Thickening Effect of RHA and CaCO₃

The viscosity of emulsion paint is an important paint parameter as it affects the consistency, flow and application properties of the paint. Rice husk ash surpassed calcium carbonate in thickening effect at all levels in the formulation (2-12%). This was determined by viscosity measurements of RHAWEPs and CCWEPs which are presented in Table 3.

Table 3: Viscosity values of White Emulsion Paints Produced with RHA (RHAWEP) and CaCO₃ (CCWEP) at 2-12% Levels using a Digital Rotathinner Viscometer

Type of Emulsion Paint	Viscosity (poises) of RHAWEP and CCWEP at 0-12% Extender Levels						
	0	2	4	6	8	10	12
RHAWEP	-	2.8	3.5	3.8	4.3	4.9	6.4
CCWEP	-	2.6	3.2	3.5	3.6	4.0	5.0
Control (0% extender)	2.8	-	-	-	-	-	-

Fig. 1 shows plots of the viscosity values (in poises) of RHAWEPs and CCWEPs versus the level of extender. The thickening effect of both RHA and CaCO₃ was generally found to be markedly better at higher extender levels (8 - 12%) than at lower levels (2 - 6%).

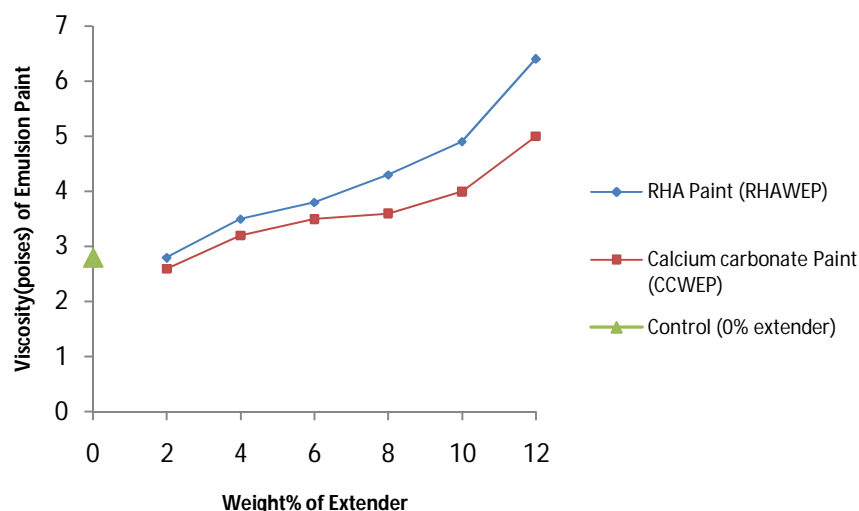


Fig.1. Plots of Viscosity values (in poises) of Emulsion Paints versus Weight% of Extender

The greater viscosity enhancement of the two fillers at higher levels implies that higher levels of extenders are required in the paint formulation for a marked increase in the viscosity of the paints. The superior thickening effect of RHA to CaCO₃ can be attributed to its lower S.G of 1.54 compared to 2.24 for CaCO₃ at the same particle size range of 32-63µm [30]. The low specific gravity of RHA implies that at the same weight of extender, RHA will have a greater number and volume of extender particles than CaCO₃ in the paint resulting in greater resistance to flow (viscosity). The lower S.G of RHA also results in a higher pigment volume concentration (PVC) of RHAWEPs and consequently its

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 8, August 2015

higher viscosity than CCWEPs. This is due to the fact that specific gravity of pigment/extender has an inverse relationship with PVC [5] such that RHA powder with lower S.G produces emulsion paint with higher PVC than CaCO_3 [30]. This increase in the volume of extender means that the extender particles spread out to a greater extent in the paint matrix, which invariably increases the viscosity of the paint. In addition, the high surface to volume ratio of RHA, the expanded conformation and disorderly nature of amorphous silica which is the major component of RHA confers greater surface area, flexibility and reactivity to RHA [24] [25] which translates into higher viscosity. The Control which had 0% extender had a value of 2.8 poises which is the same as that for 2% RHA but slightly higher than that of 2% CaCO_3 . This suggests that low levels of RHA and CaCO_3 have little effect on paint viscosity.

e. Opacity of Emulsion Paints

The opacity of the paint, which is also known as the hiding or obliterating power, is largely dependent on the nature and amount of pigment in the paint. Most extenders have little or no effect on the opacity of paint due to the narrow range of their refractive indices which are close to those of oils and resins [4]. A titanium dioxide level of 19.00% was used in both RHAWEP and CCWEP samples. Interestingly, RHAWEPs was observed to have a slightly better opacity than CCWEPs as it covered the most chart better in two coats. This can be attributed to the presence of small quantities of titanium dioxide found in RHA, in addition to other metallic oxides [9] which increased the total amount in the formulation resulting in the observed improved opacity. Titanium dioxide has excellent hiding power [4] so the presence of even small quantities in RHA made a slight but noticeable impact on its opacity.

f. Drying Properties

RHAWEP films displayed shorter surface drying times of 10 to 15 minutes than CCWEP which ranged between 20 and 23 mins. This can be attributed to the higher PVC values of the RHA paints which invariably reduced the volume of water (used as diluent in the formulation) since the volume of pigment has increased due to the lower specific gravity of RHA. The films of RHAWEP also dried hard faster (within 1hr) than CCWEP which took a longer time.

g. Scrub Resistance (Washability)

The RHAWEPs as well as CCWEP showed, excellent scrub resistance of over 5000 cycles at all extender levels. This implies that the dry paint films can be washed (scrubbed) over 5000 times without loss of the paint from the substrate. This shows that the paint is of premium (high) quality. The excellent scrub resistance is attributable to the type of resin (styrene-acrylic copolymer) and level of resin (33%) used in the formulation. Styrene-acrylic has very good binding properties which was evident in the strong adherence of the paint film to the substrate and resultant high resistance to scrubbing. This factor is largely dependent on the type of resin used. The two far exceeded the specification of a minimum of 501 cycles for premium quality emulsion paint [29].

h. Settling Resistance

Emulsion paints generally have an intrinsic tendency to settle at the bottom when left to stand for long periods due to the high level of pigment and extenders, which have higher density than water used as the solvent. This phenomenon often manifests in the form of phase separation in which the solvent (water) forms the top layer. However, on stirring, the paint is expected to be re-dispersed and thereby regain its homogeneity. RHAWEPs showed greater resistance to settling as they had smaller amounts of water on the surface (4.5, 4.0 and 3.5mm in height) than CCWEPs (9.0, 7.0, 5.0 mm in height) for 8, 10 and 12% extender levels respectively. This can be attributed to the lighter nature and lower density of RHA particles, which are more likely to remain suspended in the resin while the CaCO_3 particles which are denser are more likely to settle. On stirring, RHAWEPs were found to have less hard settling at the bottom of the container than CaCO_3 .

i. Storage Stability

RHA- and CaCO_3 -filled emulsion paints displayed good stability after 6 months of storage at $0\pm 2^\circ\text{C}$ and $27\pm 2^\circ\text{C}$. There was no evidence of biochemical deterioration in both RHA and CaCO_3 samples which often manifests as changes in colour, coagulation, viscosity loss, pH drift, development of offensive odour. This clearly shows the efficacy of the fungicides and bactericides (Acticide) used in the formulation. The low chemical reactivity of silica which is the major component of RHA is also a contributory factor to the stability of the RHA paint.

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 8, August 2015

j. Temperature Stability

RHAWEPs and CCWEPs showed good stability when stored at an elevated temperature of 45°C as they substantially retained their physical and chemical integrity. The stability of the samples was evident in the absence of offensive odour and deterioration in paint quality which can be attributed to the efficacy of the preservative used as well as the relative stability of silica, the predominant component of RHA.

k. Weather Resistance

RHAWEPs and CCWEPs displayed good resistance to external exposure as there were no noticeable defects on the films subjected to external exposure in comparison with the fresh films.

APPLICATION PROPERTIES

l. Brushing Properties

RHAWEPs displayed good application properties, similar to those of CCWEP, in terms of ease of application, flow and spread of the paint on the substrate when applied with both brush and roller. This is not unexpected since the two formulations are the same except for the type of extender, which evidently did not affect the brushing properties.

m. Re-coating Properties

RHAWEPs were similar to CCWEPs in having good re-coating properties. The second and third coats were applied on the first coat smoothly and with ease with the aid of a brush. There were no undesirable film properties such as peeling of underlying coat, sagging, flaking and cracking [29]

IV. CONCLUSION

This study has established that RHA is suitable for use as pigment extender in emulsion paints. The results also revealed that the RHA-filled emulsion paints displayed good performance and application characteristics in conformity with the requirements for premium quality paint. In addition, emulsion paints formulated with RHA surpassed those of calcium carbonate (the reference standard) in some attributes, namely, viscosity, opacity, settling resistance and drying properties but were similar to those of calcium carbonate in scrub resistance, weather resistance, storage stability, temperature stability and application properties. RHA therefore serves as a good, cheap, abundant, readily available, renewable extender for the production of emulsion paints.

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