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Flatting Effect of Rice Husk Ash in Red Oxide Primer in Comparison with Calcium Carbonate

Clementina D. Igwebike-Ossi[1] and Nwabueze I. Elom[2]
[1,2] Department of Industrial Chemistry, Ebonyi State University, P.M.B. 053, Abakaliki, Nigeria

Abstract: *Rice husk ash (RHA) has a high silica content which promotes its attractiveness as potential bio resource for the surface coatings industry. This work investigated the applicability of rice husk ash (RHA) as a flatting (gloss-reducing) extender in red oxide primer and compared its flatting effect with that of calcium carbonate, the standard gloss control agent. RHA was obtained by controlled incineration of rice husks at a temperature of 650^{0}C and duration of 4 hrs. The granular ash obtained was ground to obtain powdery RHA of smaller particle size. The powdered RHA and calcium carbonate were sieved to obtain a fairly uniform particle size range of 32-63microns (µm) used in the formulation of the red oxide primers. The gloss levels of the dry films of the primers were measured with a gloss meter at 60^{o}angle of reflectance and the matt values of the primers calculated from their gloss values. The RHA-filled primer had the highest matt value (%) of 96.8±0.05 while that of calcium carbonate was 83.6±0.08 and the Control (without extender) had the lowest matt value of 9.07±0.05.Both primers had matt values which were over 800% higher than that of the Control. It is evident from these results that RHA showed superiority to calcium carbonate in flatting effect while the Control, gave a high-gloss film.*
Keywords: *flatting effect, gloss, matt, red oxide primer, rice husk ash, silica.*

I. Introduction

The surface coatings industry is constantly in search of cheaper, more effective and recently, renewable raw materials through research and development efforts. The gradual global transition from the use of non-renewable (fossil-based) to renewable (plant-based) resources has stimulated a new-found interest in extenders from agro-waste having potential applicability in paints and other surface coatings [1]. Rice husk, an agro-waste product, generated during rice-milling has great potential as bio-resource for industrial applications due to its abundant availability, little or no cost and 'renewability'. Rice husk ash, obtained by burning rice husks, has a high silica content compared to other lignocellulosic materials and therefore can be used as a source of silica for silica-based industrial applications. The silica content values of rice husk ash quoted in literature range from 70 to 97% [2-5]. It is this high silica content of RHA that makes it attractive to researchers as a renewable alternative to natural (quartz) and synthetic silica. Studies have shown that RHA is useful in various industrial applications which include production of special cement and concrete mixes [6,7] as adsorbent, [8,9] production of heterogeneous catalysts [10,11] zeolite synthesis [12,13] and as filler in adhesives [14,15]. More recently, the applicability of RHA as extender in the surface coatings industry has been investigated in some paints namely, textured [16] emulsion [17,18] , cellulose matt paint [19] and matt wood varnish[20].

A flatting agent is used to reduce the level of sheen in a glossy coating. The flatting agents used in paints and other surface coatings are usually extenders. Extenders (also known as 'pigment-extenders' or 'fillers') are chemically inert, inorganic compounds that are added to surface coatings in order to increase bulk, reduce cost, augment the pigment properties and confer some special properties to the paint [21, 22] such as flat (matt) appearance, as is the case in this study. Red oxide primer is an industrial paint, applied as undercoat on metal surfaces to prevent corrosion. It produces a matt appearance when applied on a substrate. Standard gloss measurements of dry paint films are taken with the aid of a gloss meter at any of these three angles of reflectance: 20^o, 60^o or 80^o [21] depending on the level of gloss in the paint. Thus, high gloss films are measured using a low angle (20^o) which gives minimum reflected light while low gloss films are measured at a high angle (60^o) of reflectance [21]. The 80^o angle is used for special effects [23].

In this work, the applicability of RHA as a flatting (gloss-reducing) extender in red oxide primer was investigated. Some of the quality parameters of the primer were assessed and compared with that produced with calcium carbonate, the standard flatting extender used in red oxide primer. The use of RHA as a flatting agent in a primer introduces novelty to this investigation.

II. Materials and Methods

2.1 Equipment for Production of Rice Husk Ash

Muffle furnace; Labline,
Laboratory Electric Oven
Standard stainless steel sieve of aperture 63 microns (BS410), Gas stove
Equipment for Production and Testing of Paint Samples
Gloss meter, (Sheen Tri-Gloss Master , 20-60-85)
Mini stirrer, Diaf A/S Copenhagen NV Denmark
Analog Rotothinner (viscometer): Sheen
Wt per litre (specific gravity) cup
Digital Weighing Scale: Sauter

Paint Raw Materials

The paint raw materials used in this study to produce the red oxide primers were grades designed for paint production and were obtained from assured suppliers/ importers of paint raw-materials.

2.2 Methods

2.2.1 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 materials like broken rice grains were removed by handpicking. The wet rice husks were dried at 100^0C to a constant weight in an electric oven.

Production of Rice Husk Ash

A two-stage method, similar to that used by Sugita [24] was adopted for the large-scale 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 furnace was then switched on and left to attain the desired combustion temperature of 650^0C, 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 and stored in a dessicator.

Preparation of RHA and Calcium Carbonate Extender

The granular RHA produced after incineration in the muffle furnace was ground with a mortar and pestle until the ash was reduced to fine particle size with a powdery texture. The powdery RHA and calcium carbonate were sieved with a standard sieve with a mesh size of 63microns(µm) to obtain a uniform particle size range of 32-63 µm in both extenders as required for the production of red oxide primers.

Production of Red Oxide Primers [25]

The formula used for the production of the red oxide primers using calcium carbonate and RHA as flatting agents is given in Table 1.0

The red oxide primers formulated with the two extenders and the Control were coded as follows:

Name of Red Oxide Primer	**Code**
Calcium carbonate red oxide primer (standard)	CCROP
RHA red oxide primer	RHAROP
Control Red Oxide Primer	CONROP

Table 1.0 Formula for Production of Red Oxide Primers Using Calcium Carbonate and Rice Husk ash as Flatting Extender [25]

Component	**Function**	**Control CONROP**	**Red Oxide Primer CCROP/RHAROP**	
		Weight %	**Weight%**	**Weight(g)**
Medium oil alkyd resin	Binder	59.2	44.20	221.00
Kerosene	Solvent	30.3	25.3	126.50
Soya lecithin blend	Dispersant	0.50	0.50	2.50
Easi gel	Anti-settling agent	2.50	2.5	12.50
Red (Ferric) oxide pigment	Colourant / anticorrosion	4.00	4.00	20.00
Extender ($CaCO_3$/RHA)	Flatting agent	0.00	20.00	100.00
Calcium drier	Surface dry	0.30	0.30	1.50
Cobalt drier	Through - dry	0.25	0.25	1.25

Zirconium drier	Through -dry	0.25	0.25	1.25
Silicone fluid	Flow aid, coalescing agent	0.20	0.20	1.00
Methyl Ethyl Ketoxime	Anti-skinning agent	2.50	2.50	12.50
		100%	100%	500.00g

Production Procedure

The quantities stipulated are for the production of 500g of the primer. The following components were loaded into a 1-litre plastic vessel and stirred slowly: medium oil alkyd (part) (71.00g), kerosene (part) (76.50g), soya lecithin blend (2.50g) and Easigel (12.50g). The red oxide pigment (20.00g) and flatting extender (100.00g) were then added to the vessel with slow stirring: The pigment and extender were dispersed at high speed with bits by means of a mini stirrer. When the dispersion was satisfactory (20 – 30microns), the mixture was run at low speed and the following materials were added: medium oil alkyd (150.00g), kerosene (50.00g) calcium drier (1.50 g) cobalt drier(1.25g), zirconium drier (1.25g), silicone fluid (1.00g) and methyl ethyl ketoxime (12.50g) to give a total weight of 500g of primer.

Thinning of Red Oxide Primers

The red oxide primers, after production, all had a high viscosity of >15 poises, so had to be thinned down to the required application viscosity of 3.0 poises/ RT/ 25^0C before their physical properties, such as wt per litre, drying time and gloss values were determined. This viscosity is the required viscosity for proper flow and spread of the primer during spraying with a spray gun. Exactly 100g of the primer was poured into a sample can. A weighed amount of kerosene was gradually added and the viscosity checked in the rotothinner until the required viscosity of 3.0 poises was attained.

Determination of Viscosity of Red Oxide Primers [25, 26]

The viscosity sample can was filled with the primer to within 2.5cm of top of can. The rotothinner was switched on. The sample can was then placed on the turntable of the rotothinner and the paddle immersed into the primer inside the sample can. The rotor with the sample was allowed to rotate until the peak value was obtained. The reading (in poises) was taken from the graduated scale around the turntable. The paddle was raised and the sample can removed and washed. The viscosity determination was done three times for each primer for reproducibility.

Determination of weight per litre (specific gravity) Values of the Primers

The weight per litre cup was first weighed on a digital weighing scale (W1). The primer was poured into the cup and the excess cleaned off from the hole in the lid. The weight of cup and primer was recorded (W2). The wt per litre value of the primer was obtained by deducting the wt of the cup (W2-W1).

Determination of Dry Film Appearance

Two coats of the primer were sprayed on 6” x 4” steel panels, allowing 10 mins interval between coats. The film on drying was observed for matt, presence/absence of haze, bits and seeds.

Determination of Drying Time of Red Oxide Primers [25, 26]

The red oxide primer was applied on 6” x 4” steel panels and the time it took for the applied coat to become tack-free (non-sticky to the touch) was recorded. The time it took for the coat to dry hard was also recorded.

Determination of Gloss and Matt Values of Red Oxide Primers [25,26]

The red oxide primer, which had been thinned down to the required application viscosity of 3.0 poises was scooped with a pallet knife and applied generously across the width of the glass panel about 2cm from the edge of the glass panel which had been taped off with masking tape. With the aid of a doctor blade, the paint was applied evenly along the length of the glass panel by holding the two ends of the blade and moving it down the length of the glass panel. The primer was allowed to dry hard under ambient conditions for 16hrs. The gloss meter was switched on, allowed to warm up for a few minutes and set to the required angle for the gloss measurement (60^0). It was then placed on the surface of the dry paint film on the glass panels and the reading taken. The gloss meter was placed at different positions on the gloss panels to obtain four readings and the mean value taken.

III. Results and Discussion

3.1 Comparison of Properties of Red Oxide Primers Produced using Calcium Carbonate (standard), and RHA as Flatting Agents

The determined physical properties of the red oxide primers are presented in Table 2.0. Red oxide primer is applied as undercoat on metal surfaces to help promote surface adhesion of the topcoat, usually gloss paint, to the substrate. The primer fills tiny crevices on the metal surface which helps to ensure smoothness of the topcoat (gloss) when applied. For good filling and sanding properties, primers are loaded with a relatively high level of extenders as is the case in this formulation where 20% of the flatting agents ($CaCO_3$/RHA) were used in formulating the red oxide primers. Extenders are dispersed as tiny particles in the resin and cause scattering of light which is responsible for the flatting effect. The red oxide primers produced with calcium carbonate and RHA extenders had a reddish-brown colour due to the ferric oxide pigment used in the product. The primers had the same specific gravity values of 1.04 which indicates that the individual specific gravity values of RHA and calcium carbonate had little or no effect on the S.G of the primer. The dispersion levels of the two primers were all found to be 25 µm which is the required level for satisfactory film properties in a primer when applied. The dry films of the primers were free from bits and seeds.

3.2 Drying Time of Primers

The tack-free (touch-dry) of 13 and 9 mins and hard-dry time of 23, and 18mins for calcium carbonate and RHA primers respectively show that RHA has an edge over calcium carbonate in drying properties despite the same levels of driers used in both primers. The Control had longer drying times of 27 and 35 mins probably because of its lower solids content and relatively higher volume of resin and solvent which causes the evaporation process to take a slightly longer time than in the case of the red oxide samples which have lower amounts of solvents.

3.3 Gloss and Matt Values of Calcium Carbonate and Rice Husk Ash Red Oxide Primers

The gloss and matt values of the primers are presented in Table 2 and Fig.1. The matt value of paint is calculated from its gloss value which is obtained from gloss meter readings. The gloss value of paint quantifies the extent of its light- reflecting ability while the matt value gives the degree of its light-scattering ability [23]. The matt appearance of paints is caused by the scattering of the light rays that are incident on the applied paint film by solid particles in the paint. This means the light that illuminates the substrate is diffracted, otherwise a perfect reflection would produce a gloss effect. This light-scattering, which is observed as flatting effect / appearance is due to the micro-roughness of the paint film brought about by extender particles dispersed in the paint. The concentration and particle size of the extender will invariably affect the degree of light-scattering of the paint and consequently its matt value. Paint with high light-reflecting ability will have a high gloss value and a shiny (glossy) appearance when applied, and a correspondingly low matt value while one with a high light-scattering property will invariably have a high matt value and a flat (non-glossy) appearance. Semi-matt and semi-gloss finishes have varying degrees of gloss and matt values. Since the two effects are opposites, the matt value (in %) of a paint can therefore be determined indirectly by subtracting the gloss value from 100 [21].

Table 2.0 Physical Properties, Gloss and Matt Values of Control and Red Oxide Primers Produced with Calcium Carbonate and RHA as Extenders

Physical parameters of Red Oxide Primers	**Control CONROP**	**Calcium carbonate (standard) CCROP**	**RHA Powder RHAROP**
Appearance	Reddish-brown liquid	Dark reddish-brown liquid	Dark reddish-brown liquid
Specific gravity (27±2°C)	1.03	1.04	1.04
Drying Time (mins) Tack-free Hard - dry	 27 35	 13 23	 9 18
Gloss values (%): **60^0** after 16hrs Mean Gloss value (%) **Matt Value**	 90.93±0.05 **9. 07**±0.05	 16.4±0.08 **83.6**±0.08	 3.2±0.05 **96.8**±0.05
Increase in Matt value relative to Control (%)		821.7	967.0

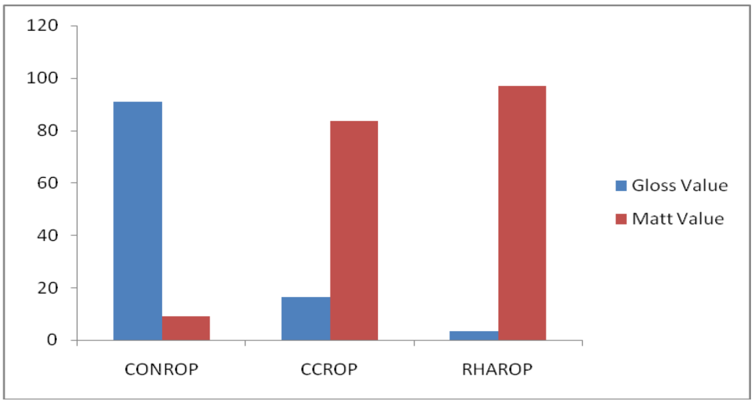

Fig.1 Gloss and matt values of the Control (CONROP), Calcium carbonate (CCROP) and RHA (RHAROP) Primers

The results reveal that RHAROP had the lowest gloss value of 3.2% which corresponds to the highest matt value of 96.8% at 60^0 angle while CCROP had a gloss value of 16.4% and consequently a matt value of 83.6 %. The Control, on the other hand, had the highest gloss value of 90.93% but lowest matt value of 9.07% due to the complete absence of extender in it which is responsible for light scattering that results in matt appearance.

Since the same particle size range was used in the formulation of the primers, the difference in flatting effect of RHA and $CaCO_3$ is not attributable to their particle size but to differences in their specific gravity. Thus, the superior flatting effect of RHA to that of calcium carbonate can be attributed to its lower specific gravity of 1.54 compared to the higher specific gravity of 2.24 for calcium carbonate at a particle size of ~63µm [1, 27]. The lower specific gravity of RHA extender produces primers containing a relatively greater volume of RHA particles than that of $CaCO_3$ which has smaller volume of extender particles. This is because the pigment/extender volume concentration increases with decrease in specific gravity [20]. Thus the larger volume of RHA extender particles in the primer translates into greater light scattering of the RHA film and consequently, higher matt value. Conversely, in the case of $CaCO_3$ however, the lower volume of extender particles translates into lower degree of light-scattering of its film and consequently lower matt value. In addition, the RHA sample used in the study is amorphous silica which has a disordered microstructural arrangement. This confers flexibility and large surface area [28] to RHA particles, which further increases the degree of light scattering and hence matt effect of the RHA film.

3.4 Visual Effects of Matt Values

The gloss and matt values of the primers reflected in their dry film appearance. Visual observation of the dry films clearly showed that the Control had a noticeably high sheen attributable to the absence of flatting agent in the formulation. RHAROP had a flat appearance while CCROP had a near flat appearance with a slight sheen.

IV. Conclusion

It is evident from the matt values of the red oxide primers that RHA displayed excellent flatting effect that surpassed that of calcium carbonate used as the reference standard. Its additional advantages of little or no cost, abundant availability and 'renewability' make it a preferable flatting extender in red oxide primer.

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