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Rice husk ash as flatting extender in cellulose matt paint

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Abstract: The high silica content of rice husk ash (RHA) promotes its attractiveness as an abundant, cheap and renewable resource for various industrial applications such as surface coatings formulation. This study investigated the applicability of rice husk ash (RHA) as a flatting extender in cellulose matt paint and compared its flatting effect with those of two commercial extenders; fumed silica (the standard flatting agent) and silica flour. RHA was obtained by controlled incineration of rice husks at a temperature of 650^0C and duration of 4hrs. The granular ash was then ground and sieved to obtain RHA flour with a particle size of ~32microns used in the formulation of the cellulose paints. Cellulose paint produced with fumed silica of 20µm-particle size was the reference standard while a sample produced with 0% extender served as the Control. The gloss values of the dry films of the paints were measured with a gloss meter at 20^0 angle of reflectance and their matt values calculated from their gloss values. The results showed that all three extenders substantially reduced the gloss levels of the cellulose paint samples with a resultant corresponding increase in their matt values. Although the flatting agents displayed varied degrees of flatting effect, their matt values were all over 100% higher than that of the Control. Fumed silica was highest in flatting performance, followed closely by RHA while silica flour ranked third. This trend was evident in the matt values (%) of 98.2±0.00, 94.7±0.06 and 86.9±0.11 obtained for cellulose matt paints produced with fumed silica, RHA and silica flour respectively. The Control had the lowest matt value of 42.9% showing its glossy nature due to the absence of a flatting agent.

Keywords: Cellulose Matt Paint, Rice Husk Ash, Flatting Extender, Fumed Silica, Gloss

1. Introduction

The paint industry is constantly in search of new, cheaper, more effective and more recently, renewable raw-materials through research and development efforts. The increased global sensitivity to environment protection as a result of recurrent climatic disasters linked to global warming has triggered a transition from the use of non-renewable (fossil-based) to renewable (plant-based) resources [1]. This trend has stimulated a new-found interest in fillers from agro-waste having potential applicability as extender in paints [2]. Rice husk, an agro-waste product, generated during rice-milling has a great potential as bio-resource for industrial applications due to its abundant availability, little or no cost and 'renewability'. Presently, there is very little domestic and industrial demand for rice husks showing the need to further explore its industrial potential through research. Rice husk ash, obtained by burning rice husks, has a high silica content compared to other lignocellulosic materials; values of silica content quoted in literature range from 70 to 97% [3-5].This high silica content makes RHA valuable as a renewable alternative to natural silica (quartz) and synthetic silica for various industrial applications. In a bid to establish the industrial applications of RHA, several researchers have used it in various research investigations, which include production of special cement and concrete mixes, [6-8] as an adsorbent, [9,10] production of heterogeneous catalysts [11,12] for zeolite synthesis [13-15] and more recently as extender in textured paint [16] and red oxide primer [17]. Extenders (also known as pigment-extenders or fillers) are incorporated into paints in order to increase bulk, reduce cost, supplement the pigment properties, and confer special properties/ effects to the paint [18, 19] such as flatting effect as is the case in this study.

Cellulose matt finish is an industrial paint used for coating the surfaces of items being manufactured [20] such as refrigerators, deep freezers, file cabinets (metal surfaces) and furniture (wood surface) for protective and decorative purposes. Cellulose paint, formulated without a flatting agent, has a glossy appearance (sheen) on a substrate. A shiny appearance may not be desirable in the said manufactured items, thus the need to substantially reduce the level of sheen by the addition of a flatting agent to the formulation to produce a low-gloss or completely flat appearance. Cellulose matt paint is so called because on application, it produces a low- gloss /matt effect on the substrate and also contains cellulose nitrate solution (often referred to as 'nitrocellulose' in paint industry). Paint films can exhibit any degree of gloss, from high gloss (98 to 100% reflectance) to dead matt (no reflectance) but between these two extremes are varying degrees of gloss and matt giving rise to other classifications such as semi-gloss, egg-shell gloss and egg-shell flat [18] based on the degree of light reflectance. Standard gloss measurements of dry paint films are taken with the aid of a gloss meter at any of these three angles of reflectance (relative to the normal to the surface): 20^o, 60^o or 80^o [18] depending on the level of gloss in the film of the surface coating. 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]. 80^o is for special effects [18]. RHA can be morphologically amorphous or crystalline depending on the combustion temperature. Several studies [22-25] have shown that burning rice husks at 500 to 800^0C yields amorphous silica and that a temperature greater than 800^0C, gives crystalline silica, thus RHA used in this study is predominantly amorphous silica. Silica flour is crystalline silica obtained by grinding pure silica sand to a fine powder [26]. Fumed silica (also known as pyrogenic silica) is produced in a flame and consists of microscopic droplets of amorphous silica fused into branched, chainlike, three-dimensional secondary particles [27].

This work investigated the applicability of RHA as a gloss-reducing (flatting) extender in cellulose matt paint and compared its flatting performance with that of fumed silica and silica flour. The objectives were to determine if RHA can be used as a new flatting agent in cellulose matt paint, reveal the extent of its flatting performance and ultimately establish if it can be used as a substitute for the expensive fumed silica which is used in the paint industry as a flatting agent. The comparison of RHA (amorphous silica) with silica flour (crystalline silica) was designed to reveal the effects of morphological differences between the two on their flatting performance.

2. Materials and Methods

2.1. Materials

Equipment**:** Muffle furnace (Labline), Laboratory electric oven, standard stainless steel sieve of aperture 32microns (BS410), flow cup (BSB4), ASTM Gloss meter (Sheen Tri-Gloss Master), Doctor blade (Sheen, 150 μm), Hagemann Gauge 5-60μm (G125 ICI PAT. PEND), Siphon feed Spray gun, (U-mate CH-202)

The commercial extenders used in this study (fumed silica of 20 μm-particle size and silica flour) as well as other raw materials used to formulate the cellulose paints 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 extraneous 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.

2.2.2. Production of Rice Husk Ash

A two-stage method, similar to that used by Sugita [28] 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 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, then stored in a dessicator.

2.2.3 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 flour). The RHA flour and silica flour were passed through a standard sieve of aperture 32 microns (μm) (0.032mm) to ensure fairly uniform particle size of ~32μm in the extenders as is required in the production of cellulose paints. The commercial fumed silica used as reference standard had a particle size of 20μm.

2.2.4 Production of Cellulose Matt Paints [21, 29]

The formulation adopted in the production of the cellulose paints using the three different extenders is given in Table 1. The only difference in the formulations is in the type of extender used. Cellulose matt paint produced with 0% extender was used as Control. The formulation for the Control is also included in Table 1.

Table 1. *Formula for Cellulose Matt Paints Showing the Components, their Functions and Weight Percentages [21, 29]*

		Cellulose Matt Paint		**Control (Cellulose Paint)**	
Components	Function	Wt%	Wt (g)	Wt%	Wt(g)
Short-oil alkyd resin	Binder	26.33	78.99	26.33	78.99
Xylene	Solvent	8.65	25.95	8.65	25.95
Methyl isobutyl ketone	Solvent	9.21	27.63	11.41	34.23
Flatting agent	Gloss control	3.20	9.60	0.00	0.00
Isopropyl alcohol	Solvent	2.99	8.97	3.99	11.97
Dioctyl phthalate	Plasticizer	0.57	1.71	0.57	1.71
Silicone fluid	Flow aid	0.34	1.02	0.34	1.02
Nitrocellulose solution	Resin/binder	26.59	79.77	26.59	79.77
White mill base	Pigment	21.00	63.00	21.00	63.00
Yellow oxide mill base	Pigment	1.12	3.36	1.12	3.36
Black mill base	Pigment	Trace	Trace	Trace	Trace
		100.00%	300.00g	100.00%	300.00g

2.2.5. Production Procedure [30]

The procedure described was used to produce 300g of each of the three cellulose matt paints using RHA flour, fumed silica and silica flour extenders at 3.2% by weight. The following components (in g) were loaded into a 1-litre plastic vessel and stirred thoroughly using a mini stirrer: short oil alkyd (78.99) xylene (part) (16.98), methyl isobutyl ketone (part) (12.00). The flatting extender (9.60) was added gradually under high speed to ensure proper dispersion after which the following components were added with slow stirring: isopropyl alcohol (8.97), xylene (balance)(8.97), dioctyl phthalate (1.71) silicone fluid (1.02) and nitrocellulose solution (79.77). When the mixture was homogeneous, viscosity was adjusted with methyl isobutyl ketone (balance) (15.63).The cellulose finish was then tinted to a deep yellow shade (magnolia) with white (63.00), yellow oxide (3.36) and black (trace) mill bases.

The cellulose paints formulated with the different extenders were coded as presented in Table 2.

Table 2. *Names and Codes of Different Cellulose Matt Paints*

Name of Cellulose Matt Paint	Code
Fumed silica cellulose matt paint	FSCMP
Rice husk ash cellulose matt paint	RHACMP
Silica Flour cellulose matt paint	SFCMP
Control (for cellulose matt paint)	CONCMP

2.3. Testing of Cellulose Matt Paint Samples

The following tests were carried out on the formulated cellulose matt paints using standard test methods as described below [29, 30].

2.3.1. In-Can Appearance [30]

The cellulose paint sample in the can was stirred with a spatula and examined for the colour, consistency, presence of lumps, coarse and foreign materials.

2.3.2. Fineness of Grind (Dispersion Level) [29]

The paint was spread across the length of the Hagemann gauge by means of a doctor blade and the dispersion level observed from the scale (in microns) on the guage.

2.3.3. Determination of Weight per Litre (Specific Gravity) of Cellulose Matt Paints

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

2.3.4. Determination of Viscosity of Cellulose Matt Paints [30]

The viscosities of the formulated cellulose paint samples were determined with a rotothinner. Each sample was poured into a 200ml-sample can to a level of about 2.5cm from the top of the cup. The rotothinner was switched on and the can placed on the turntable of the rotothinner. The disc was immersed into the emulsion paint inside the sample can which was then allowed to rotate 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.

2.3.5. Determination of Efflux Time of Thinned Cellulose Matt Paints [30]

The cellulose paint was first thinned down with methyl isobutyl ketone and its efflux time determined with a flow cup (BSB4). The flow cup (BSB4) was placed on the viscosity stand in a draught-free position such that the top of the cup was level. The forefinger was used to cover the orifice at the bottom of the flow cup. The thinned cellulose paint (at a temp of 27 ± 2^0C) was thoroughly stirred and poured into the flow cup to the brim such that the excess flowed into the groove around the top of the cup. A stop clock was used to record the time of flow. The finger was removed with simultaneous pressing of the knob of the stop clock for the commencement of timing. As soon as the flow of the paint had ceased, the timing was stopped, by pressing the knob on the stop clock. The time it took for the paint to flow out of the cup was recorded as efflux time.

2.3.6. Determination of Gloss Values of Cellulose Matt Paints [29]

The cellulose paint which had been thinned down to the required application efflux time of 23secs was sprayed evenly along the length of the glass panel. The applied paint film was allowed to dry hard under ambient conditions for 16hrs. For the gloss measurement, the gloss meter was switched on and allowed to warm up for a few minutes and then set to the required angle of reflectance for the gloss measurement (20^0).The gloss meter 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 glass panels to obtain four readings and the mean value calculated.

2.3.7. Determination of Dry Film Appearance

The cellulose paint was sprayed on metal panels by means of a spray gun and when dry, the film was observed for smoothness, gloss/ matt and presence or absence of bits.

3. Results and Discussion

The summary of the physical properties of cellulose paints formulated with RHA, fumed silica and silica flour are presented in Table 3.

Table 3. *Physical Properties and Gloss /Matt Values of Cellulose Matt Paints (CMPs) Produced with Different Flatting Extenders*

Physical Parameters of Cellulose Matt Paints	**Control (CONCMP)**	**Fumed silica (FSCMP)**	**RHA Flour (RHACMP)**	**Silica Flour (SFCMP)**
In-can appearance (before thinning)	Deep yellow, viscous liquid	Deep yellow, viscous liquid	Deep yellow, viscous liquid	Deep yellow, viscous liquid
Specific Gravity	1.55	1.59	1.59	1.56
Gloss values(%): 20^0				
Mean Gloss value (%)	57.1 ± 0.09	1.8 ± 0.00	5.3 ± 0.06	13.1 ± 0.11
Mean Matt value (%)	42.9±0.09	98.2±0.00	94.7±0.06	86.9±0.11
Increase in matt value relative to Control (%)		128	121	103
Effect of Matt values on dry film appearance	Semi-glossy; moderate sheen	Flat, dull; no sheen	Almost flat; very low sheen	Low sheen

3.1. In -Can Appearance

The cellulose matt paints produced from the three extenders all had a deep yellow colour due to same type and level of pigments used in all the formulations. They were free from bits, seeds and extraneous particles. All three therefore met the specification for this category of industrial paint. [29]

3.2. Wt per Litre (Specific Gravity) Values

The wt per litre values of the formulated cellulose paints before thinning were 1.55, 1.59, 1.56 and 1.59 for CONCMP, FSCMP, SFCMP and RHACMP respectively. The slightly lower value of the Control is probably due to the absence of extender resulting in lower solids content while the other finishes contained 3.2% extenders.

3.3. Viscosity and Efflux Time

The cellulose paint samples prepared from the three extenders all had a high viscosity of >15poises i.e. higher than the maximum value of 15poises on the Rotothinner scale. This can be attributed to the high pigment level of the coating relative to the solvent which is required for a good hiding/ obliterating power (opacity) of the product.

The paint samples were however thinned down to the required application efflux time of 23secs, which was determined with a BSB4 flow cup, before spraying.

3.4. Pigment Dispersion Level

The dispersion level indicates if the pigment(s) and extenders have been satisfactorily dispersed in the resin so as to give a good film appearance on application of the paint. A dispersion level of <5microns was obtained for all the paint samples, which is the required level for the cellulose finish [29]. This indicates the efficiency of the pigment/extender dispersion stage of the production process. The same levels of the extenders used and their closeness in particle size (20-32 μm) were contributory in maintaining the same dispersion levels in all the samples.

3.5. Gloss and Matt Values of Paint

The gloss value of paint quantifies the extent of its light-reflecting ability while the matt value gives the degree of its light-scattering capacity. Paint with high light-reflecting ability will have a high gloss value but a low matt value and consequently a shiny (high-sheen) appearance when applied while one with a low light-reflecting capacity (or high light-scattering ability) will invariably have a low gloss value, a high matt value and a flat (low-sheen/ no-sheen) appearance when applied on a substrate. 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% [18]. Semi-gloss finishes have varying degrees of gloss values with the corresponding matt values. The matt nature 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 completely glossy effect. This light-scattering, which is observed as matt effect, is due to the micro-roughness of the paint film brought about by extender particles dispersed in the paint [18]. The concentration and particle size of the extender will therefore affect the degree of light scattering of the paint and consequently, its gloss/matt appearance.

3.6. Matt Values of the Cellulose Paints

The dry films of the cellulose paints showed varying degrees of matt values obtained from the gloss measurements. The Control, (with 0% extender) expectedly, had the highest gloss value of 57.1%, corresponding to a matt value of 42.9%, thereby showing it belongs to the semi-gloss coatings category and had a moderate sheen level. The matt level of the Control film despite the absence of any extender can be attributed to titanium dioxide, a white pigment used in the formulation, which has very good opacity (hiding power) and some degree of flatting property. Fumed silica, the standard flatting extender is a very fine, thin, white powder with a very low bulk density and high surface area [27]. The fumed silica based paint (FSCMP) had the lowest gloss level of 1.8% corresponding to the highest matt value of 98.2 %, followed closely by that of RHA (RHACMP), which had a low gloss value of 5.3%, and a high matt value of 94.7%. Silica flour-based paint (SFCMP) ranked third from its gloss and matt values of 13.1% and 86.9 % respectively.

It is evident from these values that the addition of the different flatting agents to cellulose paint considerably reduced its gloss level or conversely increased its matt value correspondingly. The matt values translate into a percentage increase in matt level relative to the Control of 128%, 121% and 103% for fumed silica, RHA and silica flour respectively. This shows a remarkable reduction in the gloss level of the Control by the said extenders. The differences in the degree of flatting effect can be attributed to differences in their particle sizes and specific gravity [2] giving rise to different degrees of light scattering. The excellent flatting effect of fumed silica (98%) is due to its smaller particle size of 20µm compared to 32µm approximately for RHA and silica flour. The smaller particle size implies larger surface area, higher concentration and closer packing of the fumed silica particles than those of RHA and silica flour. These factors translate into fumed silica finish (FSCMF) having a greater volume of extender particles than SFCMP and RHACMP of the same weight and consequently greater light-scattering (matt) effect. The good flatting effect of RHA (95%) even at a higher particle size of 32µm suggests that at 20µm particle size, RHACMP would probably be at par with FSCMP in matt effect.

The superior flatting effect of RHA (amorphous silica) to silica flour (crystalline silica) despite the same particle size of ~ 32 µm and the same levels of pigment and extender in the two formulations, can be attributed to the lower specific gravity of RHA (1.54) than that of silica flour (2.18) at same particle size range of 32-63 µm [2]. The lower S.G gives higher volume of extender particles which results in greater light scattering of the RHA finish (RHACMP) and consequently, better flatting effect. Moreover, the disorderly nature of amorphous silica in RHA used in the formulation confers greater flexibility, reactivity and larger surface area to RHA particles compared to the ordered microstructure of the crystalline silica flour which imposes some rigidity and less reactivity to its particles [31]. This factor enhances the light-scattering ability of RHACMP with its resultant higher matt values than those of SFCMP.

3.7. Visual Effects of Matt Values

The gloss and matt values of the dry cellulose films reflected in the dry film appearance. Visual observation of the films clearly showed that The Control had the highest gloss/sheen attributable to the absence of flatting agent in the formulation. Fumed silica (FSCMP) finish had a completely flat, dull appearance without sheen. RHA had a near flat appearance but a slight sheen was observable on the film while silica flour finish had a noticeably low sheen. The degree of sheen desired in the paint film therefore dictates the choice of extender.

4. Conclusion

Rice husk ash evidently has very good flatting property as it produced almost flat (low-sheen) cellulose films (~ 95% matt) at a particle size (~32µm) larger than that of fumed silica (20 µm). This suggests that on further reduction of its particle size to 20 µm, RHA would probably be on a par with fumed silica which gave a completely flat paint film (98% matt). RHA surpassed silica flour in flatting effect at same particle size of ~32µm. This study has therefore proven that rice husk ash can be used as a good cheap, abundant and renewable substitute to the expensive fumed silica as a flatting agent for cellulose paint.

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