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FORMULATION OF MATT WOOD VARNISH USING RICE HUSK ASH AS A FLATTING AGENT

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ABSTRACT

This study investigated the applicability of rice husk ash (RHA) as a flatting (gloss-reducing) agent in wood varnish and compared its flatting effect with those of two commercial extenders; fumed silica (the standard flatting agent) and silica flour. RHA was obtained by first heating rice husks in a gas stove to obtain rice husk char, which was then subjected to controlled incineration in a muffle furnace at a temperature of 650°C and duration of 4 hrs. The creamy-white granular ash was ground and sieved to obtain RHA powder with a particle size of ~32 microns (µm), which was used in the formulation of matt wood varnish. Silica flour was also sieved to the same particle size as RHA. Wood varnish produced with fumed silica of 20 µm-particle size was used as the reference standard while that produced with 0% flatting agent served as the Control. The gloss values of the wood varnish dry films were measured with a gloss meter at 60° angle of reflectance and their matt values calculated from their gloss values. The results showed that although the three flatting agents displayed varied degrees of flatting effect, they all substantially reduced the gloss levels of the wood varnish samples with a resultant corresponding increase in their matt values. 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 95.8 ± 0.08, 88.9 ± 0.14 and 68.55 ± 0.34 obtained for wood varnish produced with fumed silica, RHA and silica flour, respectively. The control had the highest gloss and lowest matt value of 34.4% ± 0.42. These values correspond to increases in matt values (%) higher than that of the control by 178.49, 153.43 and 99.27 for fumed silica, RHA and silica flour wood varnishes, respectively.

Key words: Matt wood varnish, Rice husk ash, Flatting agent, Fumed silica, Silica flour.

INTRODUCTION

The surface coatings industry is constantly in search of new, cheaper, more effective and recently, renewable raw-materials through research and development efforts. The increased global sensitivity to environment protection as a result of recurrent climatic

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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 also come into play in the surface coatings industry as it 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 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, which ranges from 70-97%[3-6]. 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 explore the applicability of RHA in the surface coatings industry, its extender properties have been studied in some coatings, namely, textured paint[7] red oxide primer[8] cellulose matt paint[9] and emulsion paint[10]. However, literature survey revealed no documented study on the use of RHA as flatting agent in wood varnish.

Wood varnish is an architectural coating applied on wood surface for protective and decorative purposes. Drying of the varnish could be by evaporation of the volatile solvents only or could involve subsequent oxidation, polymerization and crosslinking of the film-forming resin[11]. Wood varnish formulations are usually a blend of resins and solvents without a pigment and consequently impart on application, a glossy (shiny), hard, transparent film through which the grain of the wood can be seen. Wood varnish can also be designed to produce a semi-gloss or completely flat (matt) finish by substantially reducing its gloss level[12]. This is achieved by the addition of a flatting agent. Flatting agents used to reduce the level of lustre in wood varnish 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[13,14] such as flatting effect, as is the case in this study.

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[13] 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[13] 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[15].

RHA can be morphologically amorphous or crystalline depending on the combustion temperature. Several studies[16-18] have shown that burning rice husks at 500 to 800°C yields amorphous silica while a temperature greater than 800°C, gives crystalline silica, thus RHA used in this study is predominantly amorphous silica. Silica flour is 100% crystalline silica obtained by grinding pure silica sand to a fine powder[19]. 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[20].

This work investigated the applicability of RHA as a gloss-reducing (flatting) extender in wood varnish 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 wood varnish, reveal the extent of its flatting performance vis-à-vis fumed silica and ultimately establish if it can be used as a substitute for the expensive fumed silica, which is used in the paint industry as the standard flatting agent. The comparison of RHA (amorphous silica) with silica flour (100% crystalline silica) was designed to reveal the effects of morphological differences between the two on their flatting performance.

EXPERIMENTAL

Materials and methods

Equipment: Muffle furnace (Labline), Laboratory electric oven, standard stainless steel sieve (BS410), flow cup (BSB4), ASTM Gloss meter (Sheen Tri-Gloss Master), Doctor blade (Sheen, 150 µm), 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 wood varnish were grades designed for paint production and were obtained from assured suppliers/importers of paint raw-materials.

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 100^{0}C to a constant weight in an electric oven.

Production of rice husk ash

A two-stage method, similar to that used by Sugita[21] 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^{o}C, which was maintained until the required duration of 4 hrs, was exhausted. The crucibles were withdrawn from the furnace and the creamy-white, granular ash samples obtained, allowed to cool, then stored in a dessicator.

Preparation of extenders for wood varnish 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 silica flour were passed through a standard sieve to obtain a uniform particle size of ~32 microns (μm), which is the smallest particle size obtainable by sieving. The commercial fumed silica used as reference standard in the study had a known particle size of 20 μm.

Basic principles in the formulation of wood varnish

A number of basic principles were considered in the formulation of the wood varnish. Cellulose nitrate, often called 'nitrocellulose' in the paint industry is the major film-former or primary binder in the wood varnish formulation. However, nitrocellulose forms hard, brittle and non-flexible films and therefore must be modified with a plasticizer to provide a flexible film and a secondary binder to give increased solids (build) and gloss to the film[11,15]. The plasticizer used in the formulation is dioctyl phthalate while the secondary binder is non-drying short oil alkyd resin. Alkyd resin varnishes have replaced oleoresinous (drying oil-based) ones due to greater durability, less yellowing, ease of application and beauty[11]. The active (true) solvent used in the varnish is methyl isobutyl ketone while the diluents (thinners) are hydrocarbon solvents; toluene and xylene. The choice of correct solvent blend is vital to obtain the best film properties. This is usually a combination of active solvents and diluents to give a balance of adequate solvency in the wet paint and an evaporation range which will give the required drying characteristics[15]. Cost-effectiveness of the solvent blend is also an important formulation consideration.

Production of matt wood varnish[22]

Based on the foregoing formulation principles and after some trial formulations, the formula adopted in the production of the matt wood varnishes using the three different extenders is presented in Table 1. The formula for the Control, which contains 0% flatting agent is also included in Table 1.

Table 1: Formula for wood varnish production showing the components, their functions and weight percentages[22]

Components	Function	Matt wood finish		Control (wood varnish)	
		Wt%	Wt (g)	Wt%	Wt(g)
Methyl isobutyl ketone (MIBK)	Solvent	12.00	36.00	12.00	36.00
Toluene	Diluent	35.00	105.00	36.5	109.50
Nitrocellulose solution	Main binder	12.00	36.00	12.00	36.00
Short oil alkyd resin	Binder	12.00	36.00	12.00	36.00
Dioctyl phthalate	Plasticizer	2.00	6.00	6.00	18.00
Flatting agent	Gloss reduction	3.00	9.00	0.00	0.00
Xylene	Diluent	23.50	70.50	25.00	75.00
Silicone fluid	Flow aid	0.50	1.50	0.50	1.50
		100.00%	300.00 g	100.00%	300.00 g

Production procedure[22]

The procedure described was used to produce 300 g of each of the three samples of matt wood varnish using RHA powder, fumed silica and silica flour extenders at 3.0% by weight. The following components (in g) were loaded into a 1-litre plastic vessel and slowly stirred using a mini stirrer: MIBK (part) (18.00), toluene (100.00), nitrocellulose solution (36.00), short oil alkyd (36.00), dioctyl phthalate (6.00). The flatting agent (6.00) was added gradually under high speed to ensure proper dispersion after, which the following components were added with slow stirring: xylene (balance) (70.50), silicone fluid (1.50), MIBK (18.00) toluene (5.00).

The matt wood varnish samples formulated with the different flatting agents have been coded as shown in Table 2.

Table 2: Codes of Formulated Wood Varnishes

Name of matt wood varnish	Code
Fumed silica matt wood varnish	FSMWV
Rice husk ash matt wood varnish	RHAMWV
Silica Flour matt wood varnish	SFMWV
Control (for matt wood varnish)	CONMWV

Thinning of the wood varnish

A 100 g-sample of the wood varnish was weighed into a sample can and methyl isobutyl ketone was gradually added to it with stirring. The efflux time of the wood varnish was checked after each addition using a flow cup (BSB4), until a value of 23-27 secs was obtained, which is the required range for spray application of the wood varnish[21]. Physical tests were then carried out on the thinned wood varnish samples.

Tests on wood varnish samples

The following tests were carried out on the formulated wood varnish samples using standard test methods[22,23].

In-can appearance

The wood varnish samples were put in sample cans, stirred with a spatula and examined for colour, consistency and homogeneity.

Determination of dry film properties

The wood varnish was strained through a 200-mesh nylon, then a small quantity poured onto a glass panel and left to dry. The dry film was then observed for smoothness, presence/absence of foreign materials such as bits and seeds and surface defects.

Determination of weight per litre (specific gravity) of wood varnish

The weight per litre cup was first weighed on a digital weighing scale (W1). The matt wood 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 wt per litre value of the paint was obtained by deducting the wt. of the cup (W2-W1).

Determination of efflux time of wood varnish

The efflux time of the thinned wood varnish was determined with a flow cup (BSB4). The flow cup 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 wood varnish (at a temp of 27 ± 2^{o}C) 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 the efflux time.

Determination of gloss values of wood varnish[22,23]

The thinned-down wood varnish was scooped with a pallet knife and applied generously across the width of the glass panel about 2 cm from the edge, which had been taped off with a masking tape. With the aid of a spray gun, the wood varnish was sprayed evenly along the length of the glass panel. The applied film was allowed to dry hard under ambient conditions for 16 hrs. 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 (60^o) for the gloss measurement. 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.

Determination of wood varnish appearance on wood panels[22]

Four pieces of 6"x 4" plywood panels were first smoothened with sand paper then coated with sanding sealer to fill the pores on the surface of the wood. The sealer was allowed to dry for 20 mins, after which the surface of the wood was further smoothened with a 'smooth' sand paper. The wood varnish was then applied on the wood substrate by spray application with a spray gun. When dry, the film was observed for smoothness and level of gloss/matt.

Determination of settling resistance[22]

The wood varnish samples were thoroughly stirred then poured into 500 cm^3 plastic jars, which were tightly covered. The samples were left to stand for a period of 24 hrs at the end of which they were stirred to check the presence or absence of hard sediment and their ease of re-dispersion on stirring.

RESULTS AND DISCUSSION

Particle size of flatting agent

The particle size is an important physical parameter of a flatting agent as it affects the degree of its light-scattering ability, which is responsible for its flatting effect. The light-scattering performance increases with decrease in particle size of the extender due to increased surface area. Fumed silica with a particle size of 20 µm is the standard flatting agent used in matt wood finish[22] as it was found to give optimum flatting effect due to its small particle size. A particle size of 32 µm (instead of 20 µm) was used for both RHA and silica flour because it is the smallest particle size obtainable by sieving. The difference in particle size is expected to manifest in the light-scattering ability of the particles and hence their flatting effect and matt values.

In-can appearance

The control (CONMWV) had a glossy and transparent appearance due to the absence of flatting agent, which gave a homogeneous resin-in-solvent system. The wood varnish samples containing fumed silica, RHA and silica flour all had a cloudy appearance due to the presence of extender, which is in the form of suspended particles in the varnish, thereby forming a two-phase heterogeneous system, which prevents complete transmission of light but causes scattering of light rays instead.

Dry film properties

The four samples were all found to be free from bits, seeds and extraneous particles and therefore met the specification for this category of surface coating[23].

Thinning of wood varnish

The wood varnish after production was found to have a higher viscosity than the specified efflux time of 22-27 secs, required for the spray application of the wood varnish[21]. The stated range provides the right emission rate of the varnish from the spray gun to produce an even spread of the film on drying. The efflux time of a liquid is indicative of its viscosity (resistance to flow) as it is the time it takes for a given volume of liquid to pass through an orifice of known diameter; the higher the viscosity, the longer the efflux time expressed in seconds or minutes. Thus, the matt wood finish was thinned down to an efflux time of 23 secs (at 25^{o}C) before application. A higher or lower viscosity would impair the consistency of the film and produce a functionally and aesthetically unsatisfactory finish.

Weight per litre (specific gravity) values

The weights per litre values of the thinned wood varnish samples were found to be 0.89, 0.91, 0.91 and 0.91 for CONMWV, FSMWV, SFMWV and RHAMWV, respectively as presented in Table 3. The Control expectedly had a slightly lower value due to lower solids content arising from the absence of flatting agent. The other three had the same specific gravity due to similar solids content. The difference in value between the control and the other samples is not marked because the weight of flatting agent (3.0%) is relatively small.

Drying time

The tack-free (touch-dry) and hard-dry times of the wood varnish samples after applying on brush-out cards are presented in Table 3. The wood varnish is a blend of resin and solvent and dries solely by solvent evaporation. The cellulose nitrate used as the primary binder is non-convertible, thus, film formation does not involve any chemical reaction. The Control had longer drying times probably because of its relatively higher volume of solvent ((73.5%)), which will cause the evaporation process to take a slightly longer time than in the case of the matt wood varnish samples, which have lower amounts of solvents (70.5%). The difference in the drying times is however, not wide because the difference in the volume of solvents used is also not large.

Settling resistance

The settling resistance properties of the matt wood varnishes are presented in Table 3. The settling resistance (anti-settling property) is an important quality parameter of matt wood varnish as it affects its application properties. Matt wood varnish is a suspension (dispersion) of extender particles in a continuous phase made up of a high level of solvent and resin. Thus, being heterogeneous it is intrinsically susceptible to settling but is expected to be re-dispersed on stirring. FSMWV showed greater resistance to settling as there was little and soft sediment at the bottom of the container compared to RHAMWV and SFMWV, which had higher and slightly hard settlement when the samples were left to stand. In addition the FSMWV dispersed more easily than RHAMWV and SFMWV on stirring. This can be attributed to the smaller particle size and lower specific gravity of fumed silica, which makes the particles to have a greater tendency to remain suspended in the resin-solvent system unlike the larger-sized and denser silica flour and RHA particles, which are more likely to settle at the bottom of the container. SFMWV showed slightly greater sedimentation than RHAMWV due to its denser particles but they were both similar in the texture of sediment and re-dispersion properties. The control, which is a homogeneous resin-solvent system, had no perceptible sediment, which is attributable to the absence of any suspended particles.

Table 3: Physical and settling resistance properties of wood varnish samples produced with different flatting extenders

Physical parameters of wood varnish	Control (CONMWV)	Fumed silica (FSMWV)	RHA Flour (RHAMWV)	Silica Flour (SFMWV)
In-can appearance (after thinning)	Clear liquid	Slightly cloudy	Cloudy	Cloudy
Specific gravity	0.89	0.91	0.91	0.91
Touch-dry time (mins)	20	8	9	10
Hard-dry time (mins)	32	20	20	22
Degree of sedimentation	-	Low	Slightly high	Fairly high
Texture of sediment	-	Soft	Slightly hard	Slightly hard
Resistance to settling	-	Good	Fair	Fair
Re-dispersion on stirring	-	V. Good	Fairly good	Fairly Good

Gloss and matt values of paint films

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[15]. 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 on a substrate 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. 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[13]. 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 pigment and /or extender (flatting agent) particles dispersed in the paint[13]. The concentration and particle size of the flatting agent will therefore affect the degree of light scattering of the paint and consequently, its gloss/matt appearance.

Gloss and matt values of formulated wood varnish

The gloss and matt values of the matt wood varnishes and the Control are presented in Table 3 and Fig. 1. The dry films of the wood varnish samples showed varying degrees of matt values obtained from the gloss measurements. The Control, (with 0% extender) expectedly, had the highest gloss value of 65.6%, corresponding to a matt value of 34.4%, thereby showing it belongs to the semi-gloss coatings category and had a moderately high sheen level. Fumed silica, the standard flatting extender is a very fine, thin, white powder with a very low bulk density and high surface area[19]. The fumed-silica filled varnish (FSMWV) had the lowest gloss level of 4.2% corresponding to highest matt value of 95.8%, followed closely by that of RHA (RHAMWV), which had a low gloss value of 11.1%, and high matt values of 88.9%. Silica flour-filled varnish (SFMWV) ranked third from its gloss and matt value of 31.45% and 68.55%, respectively. It is evident that the addition of the different flatting agents to the wood varnish considerably reduced their gloss levels or conversely increased their matt values correspondingly. The matt values translate into a percentage increase in matt level higher than that of the Control by 178.49%, 158.43% and 99.27% for fumed silica, RHA and silica flour, respectively. This shows a remarkable reduction in the gloss level of the Control by the said flatting agents. The differences in the degree of flatting effect can be attributed to differences in their particle sizes and specific gravity giving rise to different degrees of light scattering.

Table 4: Gloss and matt values of control and wood varnish samples produced with fumed silica, rha and silica flour

	Control (CONMWV)	**Fumed silica (FSMWV)**	**RHA Flour (RHAMWV)**	**Silica Flour (SFMWV)**
Gloss values (%): 60^0				
Mean gloss value (%)	65.6 ± 0.42	4.20 ± 0.08	11.1 ± 0.14	31.45 ± 0.34
Mean matt value (%)	34.40 ± 0.42	95.80 ± 0.08	88.90 ± 0.14	68.55 ± 0.34
Increase (%) in matt value over control		178.49	158.43	99.27
Dry film appearance on wood	Glossy; Moderately high sheen	Flat, dull; no sheen	Almost flat; very low sheen	Slightly glossy; Low sheen

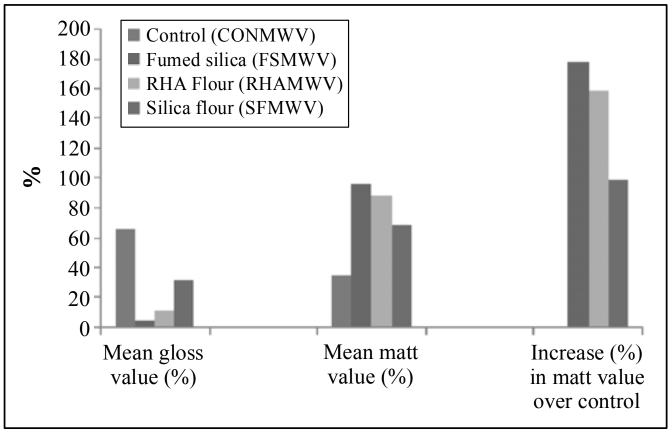

Fig. 1: Mean gloss and matt values of the control and matt wood varnishes produced with fumed silica, RHA, silica flour, as flatting agents

The excellent flatting effect of fumed silica (~96%) is due to its smaller particle size of 20 µm compared to 32 µm 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 FSMWV having a greater volume of extender particles than SFMWV and RHAMWV of the same weight and consequently greater light-scattering (matt) effect. The good flatting effect of RHA (~89%) even at a higher particle size of 32 µm suggests that at 20 µm particle size, RHAMWV would probably be on a par with FSMWV in matt effect since both RHA and fumed silica are amorphous in nature.

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 in the two formulations can be attributed to the lower specific gravity of RHA (1.54) than that of silica flour (2.17)[24]. The lower S.G gives higher volume of extender particles, which results in greater light scattering of the RHA finish (RHAMWV) and consequently, better flatting effect. In addition, 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 rigidity, reduced surface area and less reactivity to its particles[25]. These factors enhance the light-scattering ability of RHAMWV resulting in its higher matt values than those of SFMWV.

Effects of matt values on wood surface appearance

The wood varnish samples were sprayed on wood panels so as to observe the actual effects of the gloss and matt values of the dry varnish films on wood surface. Visual observation of the films clearly showed that the Control was glossy with a moderately high sheen attributable to the absence of flatting agent in the formulation. Fumed silica (FSMWV) finish had a completely flat, dull appearance on wood surface; 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 choice of flatting agent is therefore dependent on the degree of sheen desired on the wood surface. If a flat appearance is desired, fumed silica would be the preferred flatting agent. RHA is suitable for a low-gloss finish while silica flour is preferable for a semi-gloss finish.

CONCLUSION

Rice husk ash evidently has very good flatting property as it produced almost completely matt (low-sheen) wood varnish (~ 89% matt) even at a larger particle size of 32 μm. This suggests that on further reduction of its particle size to 20 μm, RHA would probably be on a par with fumed silica, the standard, which gave an almost completely flat finish (~96%). RHA surpassed silica flour in flatting effect at same particle size of 32 μm due to the higher specific gravity of silica flour particles as well as the morphological differences between amorphous and crystalline silica present in RHA and SF, respectively. 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 wood varnish designed to produce a matt finish.

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