Formulation of Emulsion Paint from a Copolymer Composite of Dimethylol Urea/Polystyrene

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Abstract
Dimethylol urea (DMU) with reduced formaldehyde emission was synthesized in this work and copolymerized by blending with polystyrene (PS) obtained by constituting waste expanded polystyrene into liquid form. The determined physical properties of the copolymer composite (i.e. DMU/PS) compared to the pure DMU revealed improvements in its disadvantageous properties such as hardness and moisture uptake. Paint samples were formulated using the pure DMU, DMU/PS and commercially obtained acrylic PVA binder resin, and analyzed. While the DMU/PS and APVA paints passed, the pure DMU failed such critical coating tests as flexibility, adhesion and blistering. Again, the DMU/PS and PVA paints show good chemical resistance while the DMU paint blisters in the alkaline medium. However, all paint samples passed tackiness, opacity and stability, and exhibit acceptable pH, viscosity, and drying time.
Keywords: Dimethylol urea, Polystyrenes, Copolymer composite, Emulsion paint

1. Introduction

Painting is unique human activities that have helped the human race to contribute significantly in earning a better livelihood, building a better and more beautified world for ourselves. Coatings on material items in our environment has presented either or combined functions including; protective, decorative/aesthetic, signal and atop all, durability [1]. As much as paint production and application is playing these vital roles, it has also come with quite some limitations. And scientists never gave up on minimizing, or even eliminating these limitations in the quest to build a safe and healthy world of paints and coatings. The binder is the paint's component responsible for the formation of the adherent and cohesive film. And based on the medium in which it's soluble or dispersible (i.e. organic solvent or water), paint can be classified as oil and water/emulsion based [2].

Even though of good performance and durable qualities such as good chemical, water, weather and heat resistance, adhesion, full gloss, flexibility, etc., the utility of oil paints has been gravely challenged by the evolution of volatile organic compounds (VOC) being the solvents typically used in their formulation. The evolution of VOC being both health and environmental issue has been seriously considered; hence the restrictions imposed on it by authorized regulatory bodies, driving the industry towards the production of paints with little or no VOC [3, 4].

Among the three basic methods that have been used in production and application of coatings with the omission of VOC, replacement of VOC with water in emulsion paints is the cheapest and most sustainable procedure compared to powder and UV-drying coating [5]. Water paints even though fundamentally inferior in their properties compared to the oil paint, is environmental friendly [6].

Urea formaldehyde (UF) is a cheap water soluble resin, which cures to form a clear, glossy and hard thermosetting film. It is suitable as a binder for water paint, but intrinsic properties such as formaldehyde emission, brittleness and moisture uptake limits its application [7, 8]. Durability problems associated with UF films is due to its brittleness and poor water resistance, while formaldehyde emission above some threshold concentration is hazardous [7, 9].

Being formaldehyde emission and moisture uptake reduction measure as reported [10]. DMU with lower formaldehyde to urea ratio was synthesized in this work, and copolymerized with PS resin obtained by constituting expanded polystyrene which is a waste product into liquid form. Formaldehyde emission and moisture uptake is expected to be further reduced in the DMU/PS copolymer composite due to the reduced amount of DMU, interaction between the components and the hydrophobic nature of the PS. PS is also expected to impact flexibility, hence reducing brittleness.

The aim of this research therefore is to produce a binder, emulsion paint from DMU/PS copolymer composite binder. This is hoped to potentially contribute in the reduction of VOCs in coating materials, and will also be economical while playing a sustainability role in waste recycling to useful and environmental friendly products.

2. Materials and Method

Formaldehyde, urea, sodium dihydrogen phosphate, sodium hydroxide, sulphuric acid, butanol, sucrose and ammonia are analytical grades (i.e. products from the British Drug House, BDH). Calgon, kaolin, anti-skining agent, nicofoam, genepour, bermocoll, anti-foam, drier, troystan, dispersant, sodium carbonate, titanium dioxide,

calcium carbonate, and aluminium silicate was obtained from Yola market, Nigeria. Gasoline was obtained from filling station in Yola. All materials were used as received. Polystyrene waste was collected from refuse dumps around Yola, Nigeria.

2.1 Resin synthesis

DMU was prepared by adopting the one step process (OSP) as reported by Osemeahon and Barminas [11] with some modifications. one mole (6.0 g) of urea to react with two moles (16.22 ml) of 37-41% (w/v) formaldehyde using 0.2 g of sodium dihydrogen phosphate as catalyst. The pH of the solution was adjusted to 6 by using 0.5M H_2SO_4 and 1.0M NaOH solutions. The solution was heated in a thermostatically controlled water bath at 70°C. The reaction was allowed to proceed for 2 h after which the resin was removed and kept at room temperature (30°C).

2.2 Formation of liquid polystyrene (PS)

Solid PS waste was cleaned with water and dried. Liquid PS was obtained by dissolving 5 g in 25 ml of gasoline (20%) as described by Osemeahon et al. [12].

2.3 Copolymerization

This was carried out by blending serial concentrations (0-70%) of PS in DMU.

2.4 Determination of formaldehyde emission

Formaldehyde emission test was carried out using the standard 2 h desiccator test as described by Osemeahon and Archibong [13]. Average value of triplicate determinations of each sample was recorded.

2.5 Determination of moisture uptake

The resin films moisture uptake was determined gravimetrically as described by Osemeahon and Archibong [13]. Average value of triplicate determinations of each sample was recorded.

2.6 Determination of viscosity and gel time

Viscosity measurements were carried out using 100 ml graduated glass macro-syringe at room temperature [13[. Average value of triplicate determinations of each sample was recorded. The gel point of the resin was determined by measuring the viscosity of the resin with time until a constant viscosity profile was obtained.

2.7 Elongation at break

The elongation at break was measured using Inston Tensile Testing Machine (Model 1026) based on ASTM D638. Five runs were carried out for each sample and the average elongation evaluated and expressed as the percentage increase in length.

2.8 Solubility in water

Solubility of the resins in water was determined by mixing 1ml of the resin with 5ml of distilled water at room temperature (27-30°C).

2.9 Density, turbidity, melting point and refractive index determination

Density was measured by taking the weight of a known volume of resin inside a density bottle using metler (Model, AT400) weighing balance. Average value of five readings was taken for each sample. Turbidity of the resin samples was measured using Hanna microprocessor turbidity meter (Model, H193703). Melting points of the film samples was determined by using Galenkamp melting point apparatus (Model, MFB600-010F). The refractive indices of the resin samples were determined with Abbe refractometer. The above properties were determined according to standard method [14].

2.10 Paint formulation

The method described by Karakas *et al.*, [15] was adopted for the paint formulation. The method spits the production process into three main stages as illustrated in table 1, while the mixture in each stage was stirred for 15 mins using a mechanical stirrer.

Table 1: Emulsion paint formulation recipe with DMU and DMU/PS as binders

Stage	Material	Quantity (grams)
First	Water	185.00
	Anti-foam	0.20
	Drier	0.20
	Calgon	1.16
	Genepour	1.16
	Bermocoll	2.50
	Troystan	1.14
	Dispersant	0.20
	Butanol	5.00
	Ammonia	0.54
Second (Millbase)	TiO_2	50.00
	Al_2SiO_3	11.20
	Na_2CO_3	0.58
	Kaolin	2.52
	$CaCO_3$	123.00
Third (Letdown)	Binder	100.00
	Water	15.00
	Dispersant	0.20
	Nicofoam	0.20
	Anti-skining agent	0.20
Total		500

2.11 Test procedures for paint samples

The analysis of the paint samples was done as described by standard organization of Nigeria methods [16].

2.11.1 Viscosity

Viscosity of paint sample was determined by using an I.C.I. Rotothinner viscometer. The 500 ml tin sample container of the instrument was filled with paint sample under test to 0.3 mm of the top rim of the depth gauge. The sample was stirred with a thermometer and the temperature maintained at 27°C. The container with the paint sample was placed in the processing ring on the turntable and the lever pulled down to switch on the motor automatically. The disc was allowed to run until a steady state is reached (5 minutes). At the end of the required time, the viscosity was recorded in poises. Triplicate determinations were made for each sample and mean value recorded.

2.11.2 pH and Opacity

The pH of paint sample was determined by using phywe pH meter model 18 195.04. Opacity was determined by using the standard Mohest Chart. The paint sample was applied on the Mohest Chart and allowed to dry for 24 h. The opacity was evaluated by comparing the dried sample film with the hiding power chart. Three determinations were made for each sample and mean value assessment recorded

2.11.3 Drying time and flexibility

Dry time was evaluated by applying the paint sample on a glass panel with the aid of bar applicator, and allowed to dry. Dry to touch was taken when the paint film is no longer sticking to the finger and dry to hard taken when the film resisted finger print. Triplicate evaluations were made for each sample and mean value assessment recorded. For flexibility test, paint sample was applied on a freshly degreased and chromate alumnium with the aid of paint applicator. The film was allowed to air dry under room temperature (27-30°C) for 7days. The panel with the film was bent through 180° with a smooth action (taking 1-2 seconds). The panel was removed and examined for cracking or loss of adhesion. Any crack or loss of adhesion indicates inflexibility or brittleness. Triplicate determinations were made at 27-30°C for each sample for quality assessment.

2.11.4 Adhesion property

Adhesion property of paint was carried out by applying a coat of paint film with film applicator on a degreased metal panel and allowed to dry for 48 hours. Two sets of lines, one crossing perpendicularly over the other were drawn with a crosshatch tester on the paint film. An adhesive tape was pressed firmly with the thumb covering all the interactions of the perpendicular line. The adhesive tape was held at its loose ends and forcibly removed from the panel. Removal of more than 50% of the square lines of the paint film indicates a poor adhesion. Triplicate determinations were made at 27-30°C for each sample for quality assessment.

2.11.5 Tackiness

This was carried out qualitatively on the dried film by hand feeling to find out if the paint film is sticky or not. Stickiness of a dried paint film is an indication that the film is tacky. Triplicate samples were used for each

determination and the average quality assessment recorded.

2.11.6 Resistance to blistering

Resistance to blistering was determined thus; undiluted paint sample was applied to a glass panel with an applicator to give a wet film thickness of about 120 μm, which was allowed to dry for 24 hours. At the end of this period 4 ml of distilled water in the form of circular drop was placed on the film. The presence of blistering, wrinkling, swelling or cracking within a period of 30 minutes indicates poor water resistance. Quality assessment recorded was the mean of triplicate determinations of each sample.

2.11.7 Stability test

This was carried out by fully sealing a paint sample in a container and allowing it to stay at room temperature (27˚C) for 4 months. At the end of this incubation period, the sample was re-examined for any change in viscosity or coagulation of the emulsion paint. Absence of coagulation or any change in viscosity is regarded as a pass.

2.11.8 Chemical resistance

The chemical resistance of the paint films was carried out thus; three flexible aluminium panels (150mm x0.3) was used as the test panels. A coat of paint with paint applicator was applied on the panel. One litter glass beaker was filled with 0.1 M NaOH solution to a depth of 150mm and the test pieces immersed for 48 hours to the depth of approximately 120mm. The test piece was removed, washed with running water and stood to dry for 2 hours. The above procedure was repeated using 0.1M HCl and 0.1M NaCl respectively. Poor chemical resistance was indicated by the presence of any surface defects such as cracking, blistering, peeling or changes in color.

3. Results and Discussion

Table 2 presents the physicochemical properties of the DMU and DMU/PS, compares the values on one hand, and with the accepted level in the coating industry on the other hand. The density of both the DMU and DMU/PS are acceptable in the coating industry. However DMU/PS exhibits lower density and this may be due to change in morphology of the composite resin with the incorporation of the bulky phenyl pendant group on PS chains leading to increase in specific and free volume in the composite resin [17]. The refractive indices of both resins are industrially acceptable, while the higher value hence gloss, obtained for DMU/PS can be attributed to the orientation and a state of aggregation established by PS, thereby creating light scattering boundaries and discontinuities in the molecular structure of the copolymer composite [18, 19]. The melting point of DMU/PS compared to DMU is significantly and favorably reduced. This indicates reduction in hardness hence the impaction of flexibility by copolymerization [20]. Moisture uptake and formaldehyde emission obtained for DMU in this work is lower compared to trimethylol urea reportedly used in coating application [11, 21, 22]. And these are further reduced below the maximum acceptable limit in DMU/PS.

Moisture uptake reduction in DMU/PS can be attributed to the manifestation of the hydrophobic property as possessed in PS and/or a possible interaction consuming the OH groups in the DMU and hence lowering affinity for moisture. The reduction in formaldehyde emission can be attributed to the reduced amount of DMU in DMU/PS and a possible interaction hindering the reaction leading to formaldehyde emission in DMU [11]. The viscosity of DMU/PS is increased beyond the acceptable limit, and this can be attributed to increase in molecular weight and/or a rise in internal friction due to the incorporation the bulky phenyl pendant group on PS chains [17, 23]. The significant and favorably rise in elongation at break in DMU/PS also signifies the impaction of flexibility to the DMU on copolymerization. This can be attributed to the flexible spacing in the DMU structure leading to change in morphology and crosslinking density [23, 24]. Reduced gel-time in DMU/PS can be attributed to increased crosslinking density and/or molecular weight [8, 25]. Reduced solubility can be attributed to a change in the interaction force in DMU/PS compared to DMU [26]. While the increased light scattering (i.e. turbidity) and opacity in DMU/PS can be attributed to haze impacted by the colored PS resin (brownish), change in morphology and increased molecular weight compared to the DMU [10, 27].

Table 2: Physicochemical properties of DMU, DMU/PS and the comparison with accepted levels in the coating industry

Property	DMU	DMU/PS	Accepted level in the coating industry [9, 21]
Density (g/cm^3)	1.1840	1.0583	1.07 (minimum)
Refractive index	1.4210	1.4245	1.4000 (minimum)
Melting point (C°)	261.4	205.70	200 (maximum)
Moisture uptake (%)	3.0	0.61	3.10 (maximum)
Formaldehyde emission (ppm)	0.0860	0.047	0.08 (maximum)
Viscosity (mPa.s)	10.30	149.45	3.11-38.00
Elongation at break (%)	26	123	125 (minimum)
Solubility	Soluble	Sparingly soluble	-
Turbidity (NTU)	112	597	-
Gel time (hrs)	216	67	-
Appearance	Clear	Creamy white	-

3.1 Physicochemical properties of paint samples formulated using DMU and DMU/PS as binder

Table 3 presents the results of some physicochemical parameters tested for the paint samples formulated using DMU, DMU/PS and commercially obtained acrylic PVA latex as binders. PVA which is an accepted binder in the coating industry is used in this production to check a possible effect of the formulation procedure on the paint properties. The Table also inter-compares the results for the different binders, and with the Standard Organization of Nigeria's (SON) specifications.

All paint samples passed the stickiness/tackiness test, indicating a good adhesion potential for all the paint samples [28]. The three paint samples are alkaline with the pH favorably falling within the SON specification range. The pH of the DMU/PS paint is however higher than that of the DMU, and this can be due to the lower amount of DMU in the copolymer composite resin. Depending on the nature of the inhabitant microbes in a particular environment, paint pH can be used to inhibit microbial activities in the film [29]. Paint pH has also been reported to be used in corrosion control [30].

DMU/PS and acrylic PVA paint samples passed the adhesion test while the DMU paint failed. This adhesion failure can be attributed to the lower molecular weight and crosslinking density of the DMU resin. The quality and durability of a coating is directly related to the nature of adhesion [28].

DMU/PS and acrylic PVA paint samples passed the blistering test while DMU paint failed. This result is consistent with that obtained for the adhesion, as blistering signifies adhesion failure [31]. DMU/PS paint exhibits the highest viscosity compared to DMU and PVA, but favorable falls within SON standard range. The higher viscosity of DMU/PS paint compared to DMU is attributable to effects earlier reported for the binders in this study. However, compared to acrylic PVA; it may be attributed to higher molecular weight and crosslinking density.

All paint samples pass the opacity test, indicating their ability to stabilize pigment dispersion. DMU failed the flexibility test, while DMU/PS and acrylic PVA paint samples passed. This further confirms the impaction of flexibility on copolymerization.

All the paint samples pass the stability test, which indicates good storage characteristics for the paint samples. Touch-dry and hard-dry are respective stages in coating dry-film formation. The touch-dry time is the period of particle coalescence and cohesion as the solvent evaporates, while hard-dry is the period of optimum adhesion and cohesion of the film to a stage if desired, further coat can be satisfactorily applied [32]. All the tested paint samples exhibits relatively longer touch dry time compare to SON standard. However, acrylic PVA paint exhibits the longest touch dry time, followed by DMU and DMU/PS respectively. The relatively short touch-dry time for DMU/PS paint can be attributed to the effects earlier discussed for the binder resin in this study. All the paint samples exhibits hard-dry time lower than the SON standards' maximum limit. In a different fashion, DMU paint exhibits the longest hard-dry time followed by PVA and DMU/PS respectively. The shorter hard-dry time for DMU/PS paint compared to DMU can be said to be consistent with the touch-dry time.

Table 3: Results and comparison of some physical properties of paints formulated from DMU and DMU/PS

Property	DMU	DMU/PS	PVA	SON Standard
Stickiness	Pass	Pass	Pass	Pass
pH	8.22	8.42	8.82	7 – 8.5
Adhesion	Fail	Pass	Pass	Pass
Blistering	Fail	Pass	Pass	Pass
Viscosity (Poise)	8.6	16.1	15.1	6 – 15
Opacity	Pass	Pass	Pass	Pass
Flexibility	Fail	Pass	Pass	Pass
Stability	Pass	Pass	Pass	Pass
Drying time (min)				
Dry to touch	55	42	57	20
Dry to hard	88	69	77	120

Table 4 presents the effects of the three typical mediums (i.e. NaCl; ionic, NaOH; alkaline and HCl; acidic) on the surfaces coated with the paint samples. All paint samples show no effect, hence a good resistance to this mediums except the DMU paint which blisters in the alkaline medium. This may be attributed to a relatively lower molecular weight and crosslinking density in DMU binder resin compared to DMU/PS and acrylic PVA. The condensation reaction in UF to form an infusible film is retarded in an alkaline medium [33]. Based on the amount, this effect will be more significant in the pure DMU compared to the DMU/PS binder resins. And this may be responsible for the blistering.

Table 4: Results and comparison of chemical resistance of DMU and DMU/PS

Sample	Media		
	0.1M HCl	0.1 M NaOH	0.1 M NaCl
DMU	No effect	Blistering	No effect
DMU/PS	No effect	No effect	No effect
PVC	No effect	No effect	No effect

4. Conclusion

Formaldehyde emission and moisture uptake has been minimized in the DMU synthesized in this work. Its copolymerization by blending with PS has further reduced this effects as well as its hardness. While other tested parameters has shown favorable changes in copolymer composite (DMU/PS). The formulation, analysis and comparison of paint samples of DMU/PS, DMU and commercially obtained acrylic PVA further confirmed superiority of DMU/PS resin compared to the pure DMU resin in coating application. Compared to the SON standards, the DMU/PS paint will potentially fit into the coating industry, while playing both the economic and environmental friendly roles, which are necessary to sustain qualitative living.

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