

Production Of Emulsion Paint Using Synthesized Hydroxylated Sunflower Seed Oil/Poly Vinyl Acetate Copolymer As A Binder

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Abstract: Hydroxylated Sunflower seed oil was synthesized and copolymerized by blending with commercial polyvinyl acetate. The determined physical properties of the copolymer composite (i.e. SSO/PVA) compared to the pure PVA revealed improvements in its physical properties such as poor gloss, low water resistance and flexibility. Comparison of these results with the literature values shows that physico-chemical properties of the SSO/PVA copolymer binder were in conformity. The SSO/PVA was then used as a binder in the production of emulsion paint. The results of the physico-chemical analysis of the emulsion paint showed that the pH, the Viscosity, Flexibility, Adhesion, Opacity, Hardness test, Tackiness, Resistance to blistering, and the drying time were also within range of the standard. It showed good resistance to acidic, alkaline and salt medium compared to the pure PVA paint. This study may therefore, introduce a novel binder to the coating industry.

Keywords: Polyvinyl acetate, Sunflower seed oil, copolymer binder, copolymerization, epoxidation, hydroxylation.

I. INTRODUCTION

Painting and coating are unique human activities that have helped the human race to contribute significantly in earning a better livelihood and building a better and more beautified world for ourselves [1]. Furthermore, paints simply means organic coatings applied to surfaces with the sole aim of imparting both protective and aesthetic functions. Paints and other pigments come in a variety of gloss levels which corresponds to the level of a pigment dispersed in a substance usually called a vehicle or binder, usually polymeric in nature, which adheres to the substrate. The vehicle (i.e the binder) is one of the most important ingredients in paint formulation because it is a film-forming material or oil [2].

Oil-based paint is superior to emulsion paint in many respects such as water resistant, flexibility, gloss and durability. However, despite the superiority of oil paint over

emulsion paint many countries are now threatening to ban its usage through appropriate legislation due to its negative effect to the environment [3]. Many functional chemical ingredients in paints are not water soluble and require alcohol or other organic solvents to form solutions. These solvents may be costly, hazardous to handle, or toxic. Emulsions are useful because they allow ways to deliver active materials in water which is inexpensive and innocuous. A related advantage of emulsions is they allow dilution of these active ingredients to an optimal concentration [4]. Emulsions are commonly used in many major chemical industries. After an emulsion paint is applied, the water evaporates and the polymer particles pack closely and fuse together to form a continuous film. The use of water rather than an organic liquid means that emulsion paints produce fewer VOC (volatile organic compounds) when they are used. Emulsion is the most popular paint for walls and a ceiling due to the fact that it is water based and has less smell,

dries comparatively quickly and is easy to apply. This fact has necessitated the need to search for an appropriated paint binder which can challenge the good properties of oil paint on one hand and use water as a solvent on the other hand.

Poly (vinyl acetate) (PVA), is one of such binders commonly used for emulsion paint formulation. Contrariwise, paints derived from PVA are characterized by poor water, chemical and water resistances, flexibility, gloss and durability etc. Therefore, if these properties are to be improved, the PVA must be modified with a copolymer whose property can make the needed correction in PVA. Hence, the purposed of choosing sunflower seed oil as that copolymer.

II. MATERIALS AND METHOD

PVA, Sunflower seed oil, NaOH, HCl, sodium dihydrogen phosphate, sulphuric acid, acetic acid, formic acid, Hydrogen peroxide, NH₃, Kaolin, Butanol. Petri dishes, Beakers, Conical flasks, Measuring cylinders, three neck flask, hot plate, thermocouple, condenser, stirrer e.t.c. All materials were used as received. PVA and other chemicals were bought around Jimeta market, Adamawa, Nigeria.

A. RESIN SYNTHESIS

a. EPOXIDATION OF SUNFLOWER SEED OIL

The experimental method for the epoxidation step adapted from Goud *et al.*, [5]

b. HYDROXYLATION OF SUNFLOWER SEED OIL

The experimental method for the hydroxylation step adapted from Petrovic *et al.*, [10]

B. COPOLYMERIZATION

This was carried out by blending serial concentrations (0-100%) of SSO in PVA.

C. DETERMINATION OF MOISTURE UPTAKE

The resin films moisture uptake was determined gravimetrically as described by Osemeahon and Archibong [11]. Average value of triplicate determinations of each sample was recorded.

D. DETERMINATION OF VISCOSITY AND GEL TIME

Viscosity measurements were carried out using 100 ml graduated glass macro-syringe at room temperature [11]. 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.

E. 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 (30°C).

F. 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, HI93703). 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 [12].

G. PAINT FORMULATION

The method described by Karakas *et al.*, [13] 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 minutes using a mechanical stirrer.

Stage	Materials	Quantity in g
First	Water	185
	Anti-foam	0.2
	Drier	0.2
	Calgon	1.16
	Genepour	1.16
	Bermocoll	2.5
	Troystan	1.14
	Dispersant	0.2
	Butanol	5
	Ammonia	0.54
	TiO ₂	50
Millbase	Al ₂ (SiO) ₃	11.2
	Na ₂ CO ₃	0.58
	Kaolin	2.52
Letdown	CaCO ₃	123.0
	Binder	100
	Water	15
	Dispersant	0.2
	Nicofoam	0.2
	Anti-skinning agent	0.2
	Total	500 g

Table 1: Recipe for the formulation of emulsion paints from PVA and SSO/PVA binders

H. TEST PROCEDURES FOR PAINT SAMPLES

The analysis of the paint samples was done as described by standard organization of Nigeria methods [14].

a. VISCOSITY

Viscosity of paint sample was determined by using an I.C.I. Rotoviscometer. 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.

b. 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 Mohst Chart. The paint sample was applied on the Mohst 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.

c. 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 aluminium with the aid of paint applicator. The film was allowed to air dry under room temperature (27-30°C) for 7 days. 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.

d. 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 intersections 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.

e. 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.

f. 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 a 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.

III. RESULTS AND DISCUSSION

Table 2 presents a comparison between PVA, SSO/PVA resins and the acceptable level in the coating industry. A decrease was observed in density, viscosity, moisture uptake and melting point between the PVA and SSO/PVA; this signals positive developments in terms of film adhesion, flexibility, water resistance and hardness, respectively. The refractive index, gel time and turbidity increased as seen from Table 2, this indicates better gloss, consistency and high ability to withstand weather or service condition. The density and refractive index of both PVA and SSO/PVA are within the acceptable range in the coating industry.

Parameters	PVA	SSO/PVA	Acceptable Level in the coating industry (Osemeahon <i>et al.</i> , 2013).
Density (g/cm ³)	1.59	1.099	1.07 (min.)
Refractive index	1.41	1.44	1.4000 (min.)
Moisture uptake (%)	0.4129	0.1381	3.10 (max.)
Viscosity (MM ² /S/S)	383.42	159.19	3.11- 38.00
Melting point (°C)	185	171	200(max.)
Turbidity (NTU)	1308	3238	- -
Gel time (Min)	88	219	- -
Solubility	Soluble	Soluble	- -

Table 2: Comparison of some physical properties of PVA and SSO/PVA resins

A. SOME PHYSICAL PROPERTIES OF PAINTS FORMULATED FROM PVA AND SSO/PVA BINDERS

For paints to fulfill the basic utility aims it must exhibit some properties and these, at some required levels. The binder

is one of the components responsible for the exhibition and stability of these properties [1]. Some physical properties of PVA and SSO/PVA paints are shown in Table 5. As seen from the table, some properties such as pH, viscosity, flexibility and opacity of both PVA and SSO/PVA fall within the acceptable ranges for paints [16]. All the paints formulated from SSO/PVA and PVA shows good adhesion, tackiness and hardness properties. This behaviour can be due to high molecular weight and hence high crosslink density of both the pure PVA and SSO/PVA binders [7].

Both the paints from the PVA and SSO/PVA binders passed resistance to blistering test. These results indicate that SSO segment has been cross linked into the SSO/PVA copolymer thereby impacting hydrophobic character. The higher degrees of cross-linking give rise to less free volume and solvent molecules hardly pass through the cross linked network [4]. This implies that the paint can perform well when exposed to environmental conditions such as rain and sun. The pure PVA paint failed water resistance test as a result of the inherent property of the resin in addition to low molecular weight of the PVA binder used in the paint [8]. The drying time of PVA and SSO/PVA paints falls within the specified range [16]. The low drying time displayed by PVA paint is attributed to the low molecular weight and hence low crosslink density of the PVA binder while the SSO/PVA has high crosslinked density due to increased in molecular weight which enhanced drying [8].

Parameter	PVA	SSO/PVA paint	SON Standard
pH	7.32	8.12	7-8.5
Viscosity (poise)	14.7	11.2	6-15
Flexibility	Pass	Pass	Pass
Opacity	Pass	Pass	Pass
Adhesion	Pass	Pass	Pass
Hardness test	Pass	Pass	Pass
Tackiness	Pass	Pass	Pass
Resistance to blistering	Pass	Pass	Pass
Drying time(min)			
Touch	51	55.6	20
Hard	73	87	120

Table 3: Some physical properties of paint formulated from SSO/PVA Binders

B. CHEMICAL RESISTANCE

The ability of a coating to resist chemical deterioration or staining is an essential element in its evaluation [9]. This test provides a thorough method of determining the ability of a coating to resist discoloration from chemical reagents and other materials common in today's environment. Table 6 shows the resistance of the SSO/PVA coating system on exposure to a number of chemicals. From the results, both PVA and SSO/PVA were unaffected by the salt medium. PVA and SSO/PVA films were also unaffected by both the acid and alkali solutions. SSO/PVA has better chemical resistance due to high cross-linked density of network which decreases their exposure to environment [9].

Samples	Media		
	0.1M NaCl	0.1 M HCl	0.1 M NaOH
PVA	A	A	A
SSO/PVA	A	A	A

Table 6: Chemical resistance of PVA and SSO/PVA paint films

IV. CONCLUSION

SSO/PVA copolymer was synthesized by blending polyvinyl acetate with hydroxylated sunflower seed oil. The effect of concentration of the blends, composition of the blends on the miscibility of SSO/PVA blends were studied, based on, the viscosity, gel time, density, melting point, turbidity, moisture uptake, and refractive index measurements. It is concluded that SSO/PVA blend is found to be completely miscible from 0 to 40% while it is immiscible from 50 to 100% composition range. Thus, this resin may be a good paint binder in the coating industry. The emulsion paint formulated using SSO/PVA binder possessed good consistency, smoothness and uniformity. The paint showed increased gloss, adhesion and flexibility. The pH, viscosity, opacity and drying time of the SSO/PVA paint falls within the SON standard. It showed good resistance to acidic, alkaline and salt medium compared to the pure PVA paint.

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