

# Optimization of the Processing Viscosity of PVA/HNSO Composite, obtained by the Reactive Blending of Polyvinyl Acetate and Hydroxylated Neem Seed Oil

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**Abstract:** The effect of polyvinyl Acetate viscosity on a composite obtained from the reactive blending of PVA and Hydroxylated Neem Seed Oil was adequately looked at in this work. Some physico-chemical properties of the Polyvinyl Acetate obtained at different PVA viscosities (14.55-29.60 mpa.s) were evaluated. The Density, PVA emission, melting point, refractive index were seen to increase with increasing PVA viscosity. PVA viscosity below 23.68 mpa.s was seen to produce PVA/HNSO blend soluble in water and beyond this value the composite became insoluble. The processing of PVA/HNSO composite by formulators for the development of an emulsion paint could therefore be suggested below the value.

**Keywords:** Hydroxylated Neem Seed Oil, Polyvinyl Acetate, Viscosity, Blending.

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## Introduction

In the constituents for production of solventless paint, PVAC binder usually takes between 17 to 33% total cost of production depending on paint type and this raw material is not available locally, therefore imported (Huntsman, 2013). Hence, high cost of this raw material (binder) and consequently high cost of production due to the use of expensive binder. In addition, PVAC is hard and brittle, has poor water resistance and non-biodegradability which can contaminate the environment if not properly handled. Outside the production of paint PVAC emulsions are also used as adhesives for porous materials, particularly for wood, paper, and cloth, and as a consolidant for porous building stone, in particular sandstone (Surajudeen and Zebulu, 2015), hence, the need to improve on its qualities.

The amalgamation of two different organic functionalities into a single polymer offers a unique combination of distinctive properties of both constituents viz reduction in the emission of volatile organic compound, good processability, improved toughness, flexibility, chemical resistance and some other properties like weather ability, UV resistance and thermo oxidative stability, etc. The adoption of such amalgamation has become a sign qua non, especially with the environmental demands that have led to nationwide programme to limit volatile organic compounds (VOCs). Recent research developments using chemical design to achieve pollution prevention have resulted in totally new synthetic routes which eliminates standard feedstocks.

Vegetable oil represents one of the cheapest and most abundant biological feedstock available. Its use as blending tools offers numerous advantages such as low toxicity, gloss and inherent biodegradability. The double bonds in the vegetable oil are used as reactive sites in coatings and they can also be functionalized by epoxidation (Osemeahon and Archibong, 2011). Neem seed oil a typical vegetable oil, will impart valuable properties to the coating but in order to furnish these properties, the oil must be incorporated into the formulation, this can be achieved by first hydroxylating the already epoxidized neem seed oil to create OH bonding sites to enable proper blending.

The main advantages of using this neem seed oil is that the need for traditional solvent as white spirit can be effectively eliminated which will leads to the following benefits: Zero volatile organic content, Eliminate healthy risk associated with solvent in respiratory systems. Environmental friendly, solvent free and 100% biodegradable in nature, Brushes and other equipment can be easily cleaned with water, minimize fire risk (Ezeagba *et al.*, 2014).

This research work will seek to determine the viscosity at which the copolymer composite PVA/HNSO will be most suitable for the formulation of an emulsion paint.

### Materials and Method

The materials used were PVA, Neem seed oil, NaOH, HCl, sodium dihydrogen phosphate, sulphuric acid, acetic acid, formic acid, Hydrogen peroxide, Ammonium hydroxide, Butanol are analytical grades (i.e. products from the British Drug House, BDH).

### Preparation of Hydroxylation Neem seed oil

The hydroxylation reaction was performed in a 1000cm<sup>3</sup> three neck flask equipped with a reflux condenser and a thermocouple. The flask was placed on a hot plate with temperature control. 150 cm<sup>3</sup> of the epoxidized neem seed oil was hydroxylated using alcohol (methanol and isopropanol) with a molar ratio of 4:1 to the oil and water at a molar ratio of 2:1 was mixed with the epoxidized oil and sulphuric acid catalyst in the reactor. The reaction was performed at a fixed temperature of 60°C for 5 hours. Uniformity was maintained using a magnetic stirrer which runs at about 1600rpm (Petrovic *et al.*, 2002). The product was then cooled and decanted in order to separate the organic-soluble compounds from water-soluble ones. The product was washed with warm water (in small aliquots) in order to remove residual contaminants. The procedure was repeated 3 times.

### Preparation of NSO/PVA blends and films

Blend of HNSO/PVAC was prepared by adding varying amount of HNSO in PVAC resin. The mixture was stirred and left for 24 h at room temperature (30°C) and then poured into a glass Petri dish for casting. The resin was also allowed to cure and set for seven days at (30°C). The physical properties of the resin films were investigated using the method as explain by (Osemeahon *et al.*, 2010).

### Determination of viscosity and gel time

The standard method of viscosity determination was employed using the viscometer bath capillary inserted into the viscometer bath. A 15 ml of the resin was measured and poured into the U-tube viscometer with capillary and then corked. The U-tube was suspended into the viscometer bath containing water and the temperature of the bath was 33°C. The cork was removed and the time taken for the content to run up starting from the top mark to the middle mark was noted using a stop watch. From this result the viscosity of the sample was calculated. Three different readings were taken for each sample and the average value

calculated. Determination of the gel point of the resin was done by monitoring the viscosity of the resin with time until a constant viscosity profile was obtained (Osemeahon *et al.*, 2010).

### **Determination of density, turbidity, melting point and refractive index**

The mettle Toledo XS 64 was used in determining the density of the cured resin via Archimedes principle, three readings were taken for each sample and average values were calculated. The turbidity of the resin samples was determined by using Hanna microprocessor turbidity meter (Model, H193703). Determination of the melting points of the film samples was done 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 methods (AOAC, 2000).

### **Determination of moisture uptake**

The classic laboratory method of measuring high level moisture of solid or semi-solid material loss on drying (LOS) was adopted. In this technique a sample of material is weighed, heated in an oven for an appropriate period, cooled in dry atmosphere of a desiccator and then reweighed. The difference between the weighed and the reweighed sample were then recorded as the moisture intake by the resin. Triplicate determinations were made for each sample and mean value recorded.

### **Elongation at break**

The elongation at break was measured using Inston Tensile Testing Machine (Model 1026). Resin films of dimension 50 mm long, 10 mm wide and 0.15 mm thick was brought to rupture at a clamp rate of 20 mm/min and a full load of 20 kg. A number of five runs were carried out (Dimas *et al.*, 2013).

**Water solubility:** The solubility of blend in water was obtained by mixing 1 ml of the resin with 5 ml of distilled water at room temperature (30°C) (Dimas *et al.*, 2013).

### **Preparation of NSO/PVA blends and films**

Blend of HNSO/PVAC was prepared by adding varying amount of HNSO in PVAC resin. The mixture was stirred and left for 24 h at room temperature (30°C) and then poured into a glass Petri dish for casting. The resin was also allowed to cure and set for seven days at (30°C). The physical properties of the resin films were investigated using the method as explain by (Osemeahon *et al.*, 2010).

## **Results and Discussion**

### **Effect of PVA Viscosity on the PVA emission of PVA/HNSO**

Table 1. Shows the effect of PVA viscosity on the PVA emission of PVA/HNSO composite. It is observed that the Hazardous PVA emission increases with increase in PVA viscosity. This trend can be attributed to two reasons; 1. It may be due to the result in the rate of condensation reactions with increase in PVA viscosity thereby increasing the rate of emission of acetate in the process (Akinteriwa Ayodele, 2015). 2. It may be as a result of increase in stress during resin cure with increase in PVA viscosity. Low PVA viscosity gives rise to low molecular weight which favours molecular chain mobility that enhances flexibility of polymer network; flexibility reduces stress during curing, and reduction in stress reduces emission (Akinteriwa Ayodele, 2015). Even when an increase in formaldehyde emission was recorded with increase in PVA viscosity in this work. The maximum value (0.0435 ppm)

recorded is still within acceptable limit of 0.1ppm as stipulated by agency against environmental pollution (Igwe and Ezeamaku, 2010).

**Table 1. Effect of PVA Viscosity on the polyvinyl acetate emission of PVA/HNSO**

PVA Viscosity (mpa.s)	Polyvinyl acetate emission (ppm)
14.55	0.0520
19.60	0.0527
23.68	0.0535
26.42	0.0543
29.60	0.0552

### Effect of PVA viscosity on the solubility of PVA/HNSO in water

The development of amino resin for emission paint formulation require an understanding of the solubility of the resin in water (Akinteriwa Ayodele, 2015). It is important both from technical and processing ground. This is more so because the solubility of PVA decreases with increase in viscosity (Osemeahon *et al.*, 2010).

Table 2 shows the effect of PVA viscosity on the solubility of PVA/HNSO composite in water. Below a viscosity of 23.68mpa.s the PVA/HNSO composite is soluble in water and beyond this point the copolymer resin is insoluble in water. This is attributed to differences in molecular weight and cross link density Osemeahon and Archibong (2011). 23.68mpa.s seems to represent the gel point of the copolymer resin, thus processing of PVA/HNSO composite for emulsion paint formulation is recommended below this viscosity.

**Table 2. Effect of PVA viscosity on the water solubility of PVA/HNSO copolymer composite**

PVA viscosity (mpa.s)	Solubility
14.55	Soluble
19.60	Soluble
23.68	Partially soluble
26.42	Insoluble
29.60	insoluble

### Effect of PVA viscosity on the reactive index of PVA/HNSO

Gloss is an important factor of many coating products. The gloss of a coating with or without pigment is a function of refractive index of the surface. The angle of incidence of the beam of light and the nature of material (Sarujudeen *et al.*, 2015). Table 3. Shows the effect of viscosity on the refractive index of PVA/HNSO composite. It is noticed that the refractive index increases with increase in PVA viscosity. This is due to the increases in molecular weight and cross link density of the different viscosity (Surajudeen and Zebulu, 2015).

**Table 3. Effect of PVA viscosity on the refractive index of PVA/HNSO**

PVA Viscosity (mpa.s)	Refractive index
14.55	1.4350
18.50	1.4370
22.08	1.4420
24.44	1.4450
27.80	1.4532

### Effect of PVA viscosity on the density of PVA/HNSO

The density of a paint binder in a coating industry has a profound influence on factors such as pigment dispersion, brushibility of the paint, flow leveling and sagging. Table 4 shows the effect of PVA viscosity on the density of PVA/HNSO composite. The density increases with increase in PVA viscosity which is as a result of the increase in molecular weight. This observation suggests that when the copolymer resin is applied in paint the density behavior may be established readily (Onukwli, and Igbokwe, 2008).

**Table 4. Effect of PVA viscosity on the density of PVA/HNSO**

PVA Viscosity (mpa.s)	Density (g/cm <sup>3</sup> )
14.55	1.1830
19.60	1.1860
23.68	1.1869
26.42	1.1894
29.60	1.1898

### Effect of PVA viscosity on the melting point of PVA/HNSO

The melting point of a polymer is related to its molecular weight, degree of cross linking and the level of rigidity of the polymer (Osemeahon and Archibong, 2011). Table 5 shows the effect of PVA viscosity on the melting point of PVA/HNSO composite.

The melting point of PVA/HNSO composite is observed to increase with increase in the Viscosity of PVA, this is attributed to differences in the molecular weight and cross link density of the copolymer networks at different PVA viscosities. At the onset the differences between the melting point with increase in PVA viscosity is much but stabilizes as optimum growth is obtained.

**Table 5. Effect of PVA viscosity on the melting point of PVA/HNSO**

PVA viscosity (mpa.s)	Melting point (°C)
14.55	146
19.60	152
23.68	156
26.42	161
29.60	163

### Effect of PVA viscosity on the moisture uptake of PVA/HNSO

The interaction of structural-network of polymer resin with water is both of fundamental and technical interest. Water uptake affects vital properties of the polymer material such as physical, mechanical, thermal and structural properties.

One of the major drawbacks of PVA resin is their poor water resistance. In paint making industry, the moisture uptake of the paint binder is very crucial because it is responsible for blistering and broominess of the paint film.

From Table 6, the effect of PVA viscosity on the moisture uptake of PVA/HNSO copolymer composite is presented. The copolymer exhibits a gradual increase in moisture uptake with increase in PVA viscosity. This trend is attributed to increase in molecular weight and cross link density as the viscosity of PVA resin increases (Osemeahon and Archibong, 2011).

**Table 6. Effect of PVA viscosity on the moisture uptake of PVA/HNSO**

DMU Viscosity (mpa.s)	Moisture uptake (%)
14,55	0.0055
19.60	0.0160
23.68	0.0170
26.42	0.0180
29.60	0.0194

### Effect of PVA Viscosity on the elongation at break of PVA/HNSO

One of the short coming of PVA resin is that it is too hard and brittle and hence poor resistance to crack propagation (Ezeagba *et al.*, 2014). Table 6 shows the effect of PVA viscosity on the elongation at break of PVA/HNSO composite. It is observed that that the elongation at break reduces gradually with increase in PVA viscosity until after PVA viscosity of 23.68mpa.s that a rapid reduction of elongation at break is observed. This trend of result is attributed to the increase in molecular weight and hence cross link density of PVA/HNSO copolymer with increase in PVA viscosity.

### Conclusion

The effect of DMU viscosity on some physico-chemical parameters of PVA/HNSO were been investigated. The result obtained showed that PVA viscosity has a pronounced influence on the properties of PVA/HNSO copolymer composite. The processing of PVA/HNSO composite for emulsion paint formulation could be suggested below viscosity of 23.68mpa.s because at a viscosity beyond this value the copolymer composite becomes insoluble in water. Also at a viscosity beyond 23.68mpa.s the ductility of the composite is lost.

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