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Miscibility Studies of HNSO/PVA as Binder for the Emulsion Paint Production

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ABSTRACT

Hydroxylated neem seed oil (HNSO) was synthesized and blended with polyvinyl acetate (PVA) to develop HNSO/PVA copolymer binder for emulsion paint formulation. Some physical properties of the copolymer resin such as viscosity, gel time, density, melting point, refractive index, moisture uptake, elongation at break and water solubility were determined. The results from studies indicated that viscosity, refractive index, gel time and elongation at break increases with increase in HNSO concentration in PVA/HNSO copolymer composite. However, density, melting point, water solubility and moisture uptake decreases with HNSO concentration in the composite. This result is a pointer to the fact that the traditional problems associated with PVA can be address or reduce by HNSO/PVA blend. The results of the physico-chemical properties of HNSO/PVA copolymer binder were in conformity with the results in the literature values.

Keywords: Epoxidation, Hydroxylation, Blend, Paint, Binder.

I. INTRODUCTION

We can't imagine our life without synthetic polymeric materials these days. Application of polymer products found in every field; ranging from engineering, physical and life sciences. The outstanding properties of synthetic materials over natural materials comes from their unique versatility, reliability, workability and durability (Yelwa et al, 2017). Polymer synthesis is done through polycondensation and by catalytic, ionic or free-radical polymerization. Synthetic resins or binders are distinctive due to their Flexibility, glossiness, hardness, durability and resistance to water, alkali and other chemical (Yelwa et al, 2017).

Painting is one of the ancient human activities. It has been experimented with different methods of protecting the outer surface of their sea vessel. Paints appeared approximately 30,000 years ago when cave dwellers used crude paints to make images of their daily lives, showing pictures of animals and pictures of them hunting animals. However, archeologist have found pigments of and paint grinding equipment in Zambia thought to be between 350,000 and 400,000 years old (Croll, 2009). The Egyptians were also the first to further develop paints, as they were the first to develop a synthetic pigment known today as Egyptian Blue (Cacusic Olo) (Croll, 2009; ACA, 2014). The Greeks and Romans (600 BC – 400 AD), used varnishes base on drying oils with the understanding that paint could not only be used to decorate objects but also to preserve it (Croll, 2009; ACA, 2014): However, we have oil-based and water-based paints. Oil paint produces volatile organic solvent (VOC) on application. VOC depletes the ozone layer of the atmosphere resulting to global warming and hence the negative effect of climate change while water paint, although not as good when compared to the oil paint in terms of characteristic, it is environmentally friendly as it evaporates water to the environment on application (Yelwa et al, 2017). These facts made necessary, the 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. Nevertheless, paints derived from PVA are characterized by poor water resistant, flexibility, gloss and durability. If these short comings are to be corrected, the PVA must be modified with a copolymer whose property can make the needed correction in PVA (Yelwa et al, 2017). Neem seed oil is one of the vegetable oils and it is non-drying oil. However, it is hydrophobic, flexible and relative appreciable gloss compare to PVA. Hydroxylation of neem seed oil will introduce a hydroxyl(OH) group into the chemical structure of this oil. Therefore, by mixing both the PVA and neem seed oil together, a chemical interaction is expected to occur between the oil and the PVA polymers thereby forming a copolymer of NSO/PVA. The presence of the oil in PVA matrix will introduce hydrophobicity, gloss and flexibility into the NSO/PVA copolymer matrix (Yelwa et al, 2017). Thus, emulsion paint from this copolymer binder will share the good properties of the oil (water resistant, gloss, flexibility) and those of PVA (water solubility, drying, adhesion etc). This is expected because similar research has been conducted by Yelwa et al, 2017, with the sunflower seed oil and PVA.

II. METHODS AND MATERIAL

A. Materials/Reagents

PVA, Neem seed oil, NaOH, HCl, sodium dihydrogen phosphate, sulphuric acid, acetic acid, formic acid, Hydrogen peroxide, NH_3, 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. PVAC and other chemicals were bought around Jimeta market, Adamawa, Nigeria.

B. Sample Collection and Preparation

The Neem seeds were collected locally from the forest around Yola metropolis. The fruits were then washed, air dried at room temperature and cracked tore move the hard shell in order to obtain the seed. The seeds were washed with distilled water and air dried at room temperature, after which they were crushed/pounded using mortar and pestle to paste. The pounded materials were stored in a closed container pending use in the extraction processes.

C. Extraction of Neem Seed Oil

The crushed seeds were manually/traditionally treated with hot water and continuously stirred in other to get the oil.

D. Epoxidized and Hydroxylation of Neem Seed Oil

E. Epoxidation

200 cm^3 of the neem seed oil was placed in a 500cm^3 three neck flask equipped with a reflux condenser and a thermocouple (adopted from Goud et al., 2007). The flask was placed on a hot plate with temperature control. Acetic acid and formic acid at a molar ratio of 0.5:1 to the oil and sulphuric acid catalyst 3% weight of hydrogen peroxide as an oxygen carrier was added into the neem seed oil. A hydrogen peroxide molar ratio of 1.5:1 to the oil was added drop-wise into the mixture. This feeding strategy is required in order to avoid over heating the system since the epoxidation reaction is an exothermic reaction. The reaction were made to maintained uniformity by using a magnetic stirrer which runs at about 1600rpm under isothermal condition at 50-6°C. The product was then cooled and decanted in order to separate the organic-soluble compounds (epoxidized oil) from water-soluble compounds. The epoxidized oil has been washed with warm water (in small aliquots) in order to remove residual contaminants. The procedure was repeated 3 times.

F. Hydroxylation

The hydroxylation reaction (adopted from Petrovic et al., 2003) was performed in a 1000cm^3 three neck flask equipped with a reflux condenser and a thermocouple. The flask was placed on a hot plate with temperature control. 150 cm^3 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. 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.

G. Preparation of NSO/PVA blends and films

Blend of NSO/PVA was prepared by adding varying amount of NSO in PVA 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 (Yelwa et al, 2017).

H. Determination of viscosity and gel time

The standard method of viscosity determination was employed using the viscometer bath capillary inserted into the viscometer bath. 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 and Archibong (2011).

I. Determination of density, turbidity, melting point and refractive index

Determination of the density of the resin was done by taking the weight of a known volume of resin inside a measuring cylinder and weighed on a weighing balance. The density was then calculated using mass over volume relationship. Three readings were taken for each sample and average value was 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 Galen kamp 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).

J. Determination of moisture uptake

Determination of the moisture uptakes of the resin films was done gravimetrically. Known weight of the sample was introduced into desiccators containing a saturated solution of sodium chloride. The increase in weight (wet weight) of the sample was monitored until a constant weight was obtained. The difference between the wet weight and dry weight of the sample was also recorded as the moisture uptake by resin. Three determinations were made for the sample and the average value recorded (Yelwa et al, 2017).

K. 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 rapture at a clamp rate of 20 mm/min and a full load of 20 kg. A number of five runs were carried out as Osemeahon and Dimas did for each sample and the average elongation evaluated and expressed as the percentage increase in length.

L. 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) (Yelwa et al, 2017).

M. FTIR analysis

The infra-red (IR) analysis of PVA, HNSO, and HNSO/PVA was carried out using infra-red spectrophotometer (Buck scientific infrared spectrophotometer, Model 530) within 500 to 4000 cm^{-1}.

III. RESULTS AND DISCUSSION

A. FT-IR

FT-IR is the spectroscopy that deals with the infrared region of the electromagnetic spectrum. It covers a range of techniques mostly based on absorption spectroscopy, the infrared radiation of absorbed by organic molecules and converted into energy of molecular vibration, either stretching or bending. Different types of bonds and thus different functional groups, absorbs infrared radiation of different wavelength. An IR spectrum is a plot of wave number (X-axis) Vs percentage transmittance (Y-axis).

Figure 1, shows the FT-IR spectra of PVA, HNSO/PVA and HNSO. In the spectra of PVA, -C=O stretching vibration of the acetate at 1731.98 cm^{-1} is observed. Wave length of absorption at 1249.72 cm^{-1}, 1163.78 cm^{-1} and 1068.53 cm^{-1} correspond to the characteristic C-O-C stretching vibration of acetate. FT-IR spectrum of HNSO/PVA, shows disappearance of –OH stretching vibration at 3464.32 cm^{-1}. This shows that a condensation reaction between hydrogen in the PVA and –OH groups from the HNSO leading to a PVA/HNSO copolymer.

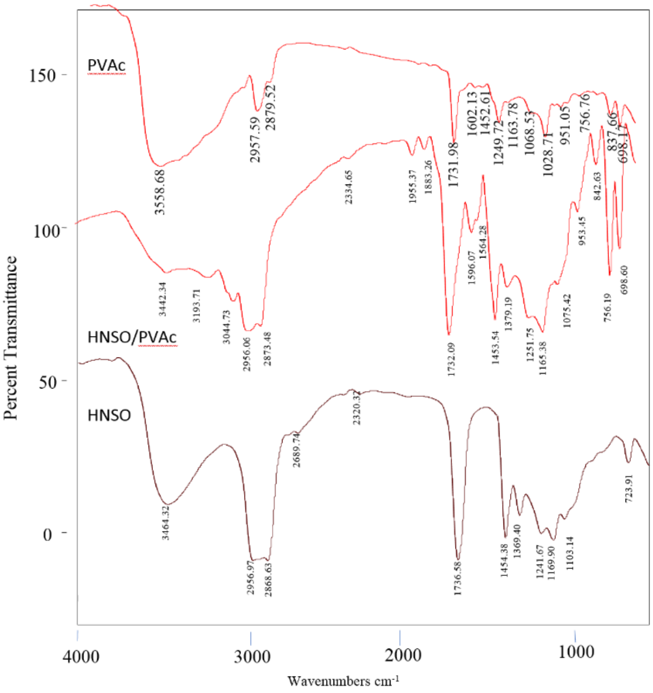

Figure 1: FT-IR spectra of PVAC, HNSO/PVA and HNSO

B. Effect of HNSO concentration on the Density of HNSO/PVA resin

Density is a physical property of matter that expresses a ratio of mass to volume and is an important physical parameter in polymer engineering processes. Density depends, on the atomic mass of an element or compound. Since different substances have different densities, density measurements are very useful for the identification and characterization of different substances and significant factor that affects the production cost and profitability of the manufacturing process. (Kazys and Rekuvience, 2011). The density of a paint binder in a coating industry has an important influence on factors such as pigment dispersion brush ability of paint, flow, leveling and sagging (Dimas, 2013).

The effect of HNSO concentration on the density of HNSO/PVA resin is shown in figure 2. Increase in concentration of HNSO lead to gradual decrease in the density of HNSO/PVA resin. Density of pure

PVAC is 1.034glcm^3, pure HNSO has a density of 0.856g/cm^3. The density of HNSO is lower compared to the density of pure PVA. As the concentration of the lower density HNSO increases in the pure PVA, the density of the blends is expected to fall between the density of PVA and HNSO (Yelwa *et al,* 2018). The gradual decrease in density of the blends can be as a result of differences in molecular features and morphology which influenced the packing nature of resin molecules as the concentration of HNSO increases (Dimas, 2013).

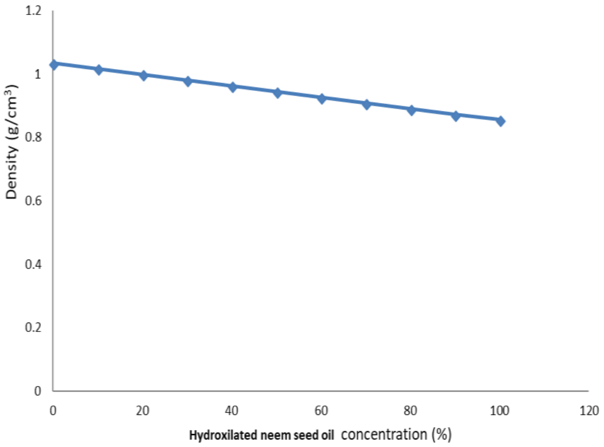

Figure 2 : Effect HNSO concentration on the density of hydroxylated NSO/PVA.

C. Effect of HNSO Concentration on Melting Point of HNSO/PVA Resin

Thermal properties, molecular weight, degree of cross-linking and the level of rigidity of the polymer is related to its melting point. In coating industry, the melting point of a binder is related to its thermal resistance as well as to the brittleness. (Afsoon *et al,* 2011). Figure 3 shows relationship between melting point of the HNSO/PVA resin and the concentration of HNSO. PVA is rigid, this is due to the strong forces of attraction holding or existing between the chains of the PVA. These forces of cohesion existing between the PVA chains is high enough to prevent its mobility at room temperature. At higher temperature, the PVA chains will gain enough energy, and that energy will make it possible to resist the forces preventing it from moving. At this point, of high temperature condition, the PVA chain becomes mobile. This chain mobility can affect the mechanical properties of the PVA to change from rigid to a soft rubbery material.

HNSO molecules are mobile that means the force3s of attraction between molecules are lower compared to that in PVA, HNSO therefore appears as a liquid at room temperature and pressure. Adding HNSO in PVA will interfere with the strong cohesive forces of attraction in the PVA chains. The interaction or interference will lead to a lowering of this strong cohesive force between the chains of PVA. Therefore, if this cohesive force in the PVA chain is reduced, the melting will also reduce. This explains why, the melting point of HNSO/PVA reduces as the concentration of HNSO in PVA increases.

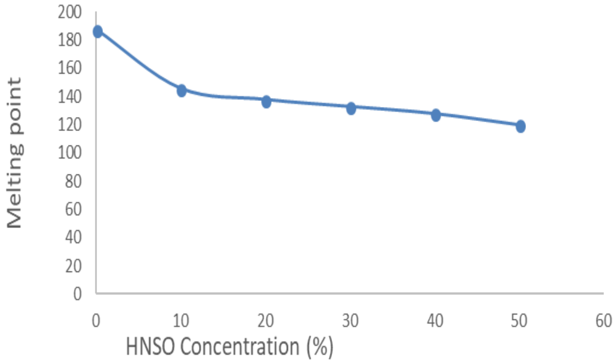

Figure 3 : Effect of HNSO concentration on the melting point of hydroxylated NSO/PVA

D. Effect of the HNSO Concentration on the Refractive Index of HNSO/PVA Resin

Refractive index and the age of incident lighting are factors that affect the gloss of a material. Gloss is an optical property which is based on the interaction of light with physical characteristic of surface. It is usually the ability of a surface to reflect light into a specular direction. Gloss is a necessary coating property when the purposes is for surface aesthetic or decoration (Yumiko *et al*; 2010). The refractive index of the binder is a crucial factor in the ultimate gloss property of the paint formulation. Figure 4 describe the effect of HNSO concentration on refractive index of HNSO/PVA resin. It is observed that the refractive index increases with HNSO concentration. Pure HNSO has a refractive index of 1.502, while pure PVA is 1.413. The refractive index of pure HNSO is higher compared to the refractive index of pure PVA. Refractive index of a liquid is an optical property and is affected by the type of atoms present, and the way or how these atoms are connected or arrange in a molecule. Refractive index of HNSO/PVA increases as the concentration of HNSO in PVA increases. This is because the optical property responsible for higher refractive index in HNSO is gradually and continually transferred or added to PVA, to form HNSO/PVA resin of higher gloss than pure PVA.

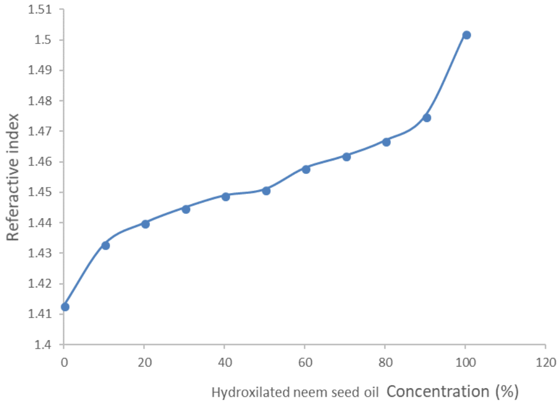

Figure 4 : Effect of HNSO concentration on the refractive index of hydroxylated NSO/PVA

E. Effect of HNSO Concentration on the Water Solubility of HNSO/PVA Resin

Emulsion paint is a paint in which the pigment is suspended in a resin that is dispersed in water as an emulsion. Water solubility can help to know the ability of the resin to form emulsion in each blend. In table 1, it is observed that increase in concentration of HNSO causes a decrease in its water solubility. Results showed that at concentration of 10, 20, and 30% HNSO the resin is soluble. This is because the resultant hydrophilic effect of the PVA in the HNSO/PVA resin in these blends is higher (Yelwa *et al,* 2017). At concentrations of 40 and 50% HNSO, resin is partially soluble, and this is because the hydrophobicity of HNSO and hydrophilicity of the PVA are roughly equal. Above 50% HNSO the hydrophobicity of HNSO dominates and the mixture become insoluble. This result therefore advised a copolymerization ration of 40% HNSO and 60% PVA.

Table 2 : Effect of HNSO concentration on the solubility of HNSO/PVAC Resin

HNSO concentration (%)	Water solubility level
0 (PVAC)	Soluble
10	Soluble
20	Soluble
30	Soluble
40	Partially soluble
50	Partially soluble
60	Insoluble
70	Insoluble
80	Insoluble
90	Insoluble
100 (HNSO)	Insoluble

F. Effect of HNSO Concentration on the Moisture Uptake of HNSO/PVAC Resin

The adhesion of coating to substrates is strongly influenced by absorption of water vapor. The mechanism of this influence proceeds as follows:

Absorption of water molecules on coating film
Inclusion of water in the interface between film and substrate
Blister formation
Corrosion/erosion of the substrate
Flaking or pelling of the film.

Although there is no fixed relationship between water absorption and water permeability generally the higher the water absorption, the more permeable the films will be to water vapor.

Normally the permeability measurement is applied on fresh applied films. It should be noted with progressive aging and weathering, films become more cross linked and in the case of water sensitive binders, the water-soluble additives are washed out by exposure to rain and dew. Moisture uptake is the amount of moisture absorbed when a coating film is exposed to moisture.

Figure 5, shows that the moisture uptake of the PVA resin at HNSO concentration of 0% is 1.26%. The moisture uptake of HNSO/PVA resin increased to 1.29% on addition of 10% HNSO concentration. This could be due to increase in the void-space of the resin. The moisture uptake shows continual decrease above the HNSO concentration of 10%. This could be due to a contraction in the void-space which reduces the diffusion of water vapor in the resin.

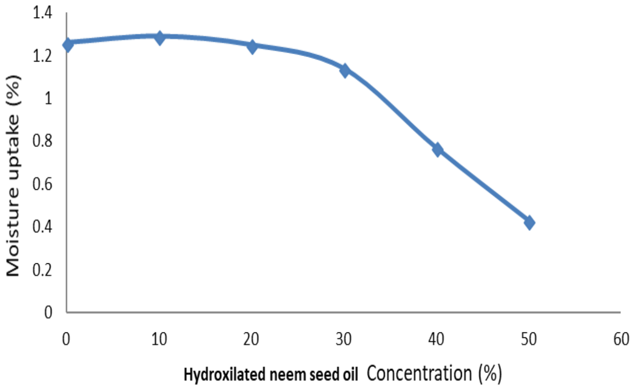

Figure 5 : Effect of HNSO concentration on the moisture uptake of hydroxylated NSO/PVA

G. Effect of HNSO Concentration on the Viscosity of HNSO/PVA Resin.

Viscosity is described as the internal resistance of a fluid to flow and may be considered as a measure of fluid friction. Viscosity is an important parameter in the paint industry processing stage. Measuring viscosity is an effective way of determining the state (properties of matter) or fluidity of a liquid. Viscosity measurement allows the most sensitive or convenient way of detecting changes in color, density, stability, solids, content and molecular weight. Figure 6 show the effect of HNSO on the viscosity of HNSO/PVA resin. The result showed that, there is a steady increase in viscosity on addition of 10, 20, and 30% of HNSO. This arises as a result of variation in respective chain length. The viscosity of the HNSO/PVA resin remain constant at HNSO concentration of 40 and 50%. As the concentration of HNSO is increased to 60%, a sharp rise in viscosity is observed. This is due to increase in molecular weight which give rise to increase in viscosity due to increase in chain cross-linking between HNSO molecules, and PVA chains. Above HNSO concentration of 60% a rapid fall in viscosity is observed. Viscosity shows continues decrease on addition of more HNSO above 60%. This fall in viscosity is due to addition of more HNSO above the optimal level leading to polymer dissociation. Polymer dissociation results to fragmentation of long chain polymer into shorter chains with corresponding decrease in viscosity, (Osemeahon and Barminas, 2007).

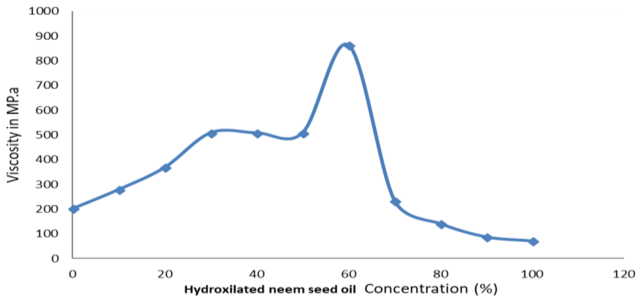

Figure 6 : Effect of HNSO concentration on the viscosity of hydroxylated NSO/PVA

H. Effect of HNSO on the Gel-Time of HNSO/PVA Resin

Gelation is the change in macro molecular structure of resin, which is usually expressed as an increased of viscosity and leads finally to the loss of flow-ability. (Suuperl *et al.,* 2006). Gel time is a time when a resin will begin to become more viscous until it reaches a state when it is no longer a liquid and has lost its ability to flow. (netcomposites.com). the resin will continue to harden after it has gelled, until at some time later it has obtained its full hardness and properties (netcomposites.com). The dry time of any paint is a function of its binder'sgel-time among other factors. On the technical front, gel-time enable paint formulator to ascertain the optimum storage period of a binder before it's utilization for paint formulation. (Desai *et al.*, 2003; Menkiti and Onukwuli, 2011). Gelation is characterized by the incipient formation of a material of an infinite molecular weight and indicates the conditions of the process ability of material. Prior to gelation system is soluble, but after gelation, both soluble and insoluble materials are present. As gelation is approached, viscosity increases dramatically and molecular weight goes to infinite. (Dimas, 2013). Figure 7 shows that the HNSO concentration increases, the corresponding gel time also increase. Gel time of HNSO is relatively longer compared to the gel time of PVA. Increasing the concentration of HNSO in the PVA will lead a gradual and steady increase in gel time of the system. The result showed that at HNSO concentration of 10,20,30,40 and 50%, the resin gelled and dried up within two weeks of casting. The gel time of HNSO/PVA resin at 50% HNSO concentration is 206 minutes. Therefore, it is advice the binder should be used within this period.

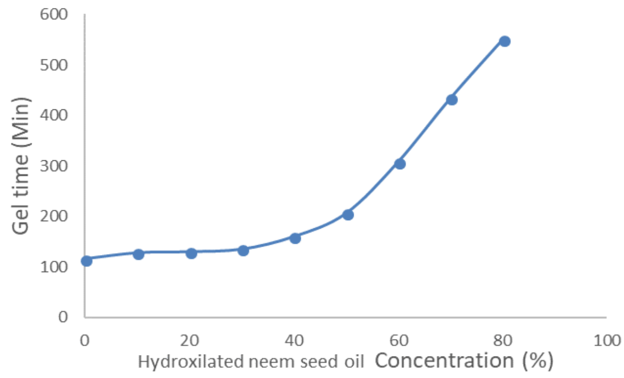

Figure 7 : Effect of HNSO concentration on the gel time of hydroxylated NSO/PVA

I. Effect of HNSO concentration on the elongation at break of HNSO/PVAC Resin

Elongation at break is the length which a material is stretched before it breaks. To perform properly in use, a coating must possess the proper amount of flexibility and toughness to withstand cracking when subjected to stress produced by shrinking or swelling, forming mechanical abuse and weathering. Flexibility is the ability to of a material to bend or flexed without cracking or undergoing other failure. Toughness is the strength or resilience of a material. It is the materials ability to withstand great strain imposed in a short time period without tearing, breaking or rupture (Mark, 1995). The effect of addition of the HNSO to PVA on elongation at break is given in figure 8. The blends results showed great increase in elongation and flexibility. However, the elongation at break showed a continual decrease on addition of more HNSO. The rapid or sharp increase on elongation is due to introduction of flexible HNSO molecules into the rigid PVAC chains. Addition of more HNSO, into the PVA chain increases the softness of the resin which results in poor elongation at break. One of the shortcomings of PVA is its brittleness and hence cracking propagation of its paint film. This experiment has therefore demonstrated an erosion of this negative property of PVA thereby promising a better paint film which will be able to withstand any seasonal variation.

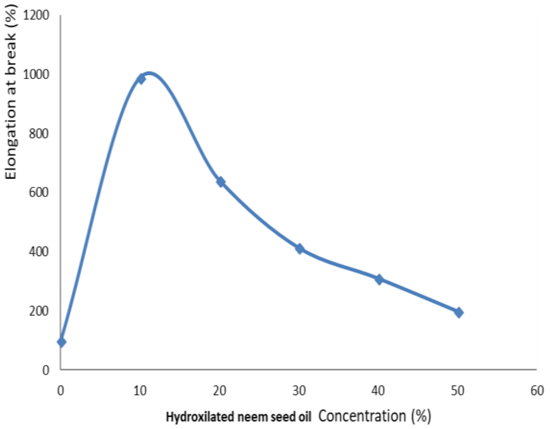

Figure 8: Effect of HNSO concentration on the elongation at break of hydroxylated NSO/PVA

J. Comparison between PVA and HNSO/PVA resins

The comparison between PVA and HNSO/PVA resins and the acceptable level in the coating industry is shown in Table 3. A decrease was observed in density, viscosity, and melting point between the PVA and HNSO/PVA; this shows improvement in terms of film adhesion, flexibility, water resistance and hardness, respectively. The refractive index, gel time and turbidity increased as seen from Table 3, this indicates better gloss, consistency and high ability to withstand weather or service condition. The density and refractive index of both PVA and HNSO/PVA are within the acceptable range in the coating industry.

Table 3 : Comparison of some physical properties of PVA and HNSO/PVA resins.

Parameters	PVA	HNSO/PVA	Acceptable Level in the coating industry (Osemeahon *et al.,* 2013).
Density (g/cm3)	1.59	1.3	1.07 (min.)
Refractive index	1.41	1.431	1.4000 (min.)
Moisture uptake (%)	0.4129	1.14	3.10 (max.)
Viscosity (MM2/S/S)	383.42	146.76	3.11- 38.00
Melting point (^{0}C)	185	176	200(max.)
Turbidity (NTU)	1308	3288	-
Gel time (Min)	88	235	-
Solubility	Soluble	Soluble	-

K. Comparison of some physical properties of HNSO/PVAC Resin with Resin from other paint

Table 4 compares some physical properties of HNSO/PVA copolymer binder with other paint binders. The viscosity, density, refractive index, melting point, moisture uptake of the HNSO/PVA copolymer, are all within the range of other binders. This results place HNSO/PVA copolymer resin as a competitive binder among other traditional resins for the coating industry.

Table 4 : Comparison of some physical properties of HNSO/PVA resin with resins from other studies

Parameters	Density (g/ cm3)	Moisture uptake (%)	Gel time (Min)	Refractive index	Viscosity (MM2/S/S)	Melting point (^{0}C)	Turbidity (NTU)	Elongation at break	Literature
NSO/PVA	1.31	1.14	235	1.431	146.76	176	3288	414	This study
TMU+PS	1.0990	1.01	120	1.4250	0.40	262	0.0233		Osemeahon and Dimas (2013)
UF+UP	079	4.23	132	1.473	13.90	132	ND	-	Osemeahon and Barminas (2007)
UF+PE	1.3362	0.0080	190	1.432	32.60	130	0.0142		Osemeahon and Archibong(2011)
SSO/PVA	1.38	0.1381	219	1.41	159.19	171	3238	-	Yelwa *et al* (2017)
DMU+PS	1.1953	0.26	1440	1.4295	35.6	181.42	611	-	Osemeahon et al (2015)
Commercial UF	ND	2	54.2	ND	451	ND	ND	-	Suurprele *et al* (2006)
Waste PS	0.84	ND	ND	1.464	500	168	ND		Osemeahon et al (2013)
PVA	0.929	ND	ND	1.40	0.40	ND	ND	ND	Habib (2011)

IV.CONCLUSION

The miscibility studies between hydroxylated neem seed Oil and polyvinyl acetate was successfully carried out. The flexibility, gloss, adhesion and elongation at break of the prepared resin were improved in the experiment. This work would add value to the neem seed oil and serve as raw material for coating application.

CONFLICT OF INTEREST

There is no conflict of interest declared.

V. REFERENCES

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