



Evaluation of composites from Dimethylol urea and Hydroxylated Sorrel oil for possible application for water Resistant Emulsion paint

S.A. Osemeahon, F.G. Fadawa*

Department of Chemistry, ModibboAdama University of Technology, Yola, Adamawa State, Nigeria

Abstract The copolymerization of dimethylol urea (DMU) and hydroxylated sorrel oil (HSO) was studied carefully in this work. FTIR spectroscopy of the pure DMU and DMU/HSO showed some chemical interaction. Some of the physico-chemical parameters such as melting point, gel time, moisture uptake, turbidity, viscosity, density, solubility, formaldehyde emission, and refractive index were evaluated. Results of the composite showed remarkable improvement in terms of formaldehyde emission, flexibility and softness, optical properties and water resistance compared to the hardness and brittleness, poor water resistance and formaldehyde emission associated with dimethylol urea. Therefore, this composite (DMU/HSO) resin will serve as a potential binder for emulsion paint formulation.

Keywords Copolymerization, dimethylol urea, hydroxylated sorrel oil, emulsion paint

Introduction

Paint is any liquid, liquefiable or mastic composition which after application to a substrate in a thin layer is converted to a solid film [1]. It is a mixture of binders and solvents that form a continuous film that can be protective, decorative or both. It is a liquid that dries to form a protective film on the surface. It is also the term used to describe a number of substances that consist of a pigment suspended in a liquid or paste vehicle such as oil or water. It is essentially a suspension of fine particles of pigments in a fluid [2].

Paint is made up of three major constituents. Each type of finish has its own special blend of constituents that enhances its performance. The constituents include; Binder, pigment, solvent and other additives for a specific purpose [3].

Basically, there are two major types of paint. Oil paint or solvent based paint, which is oil based, and emulsion paint which is water based. Paints and coatings can also be classified into the following major categories according to their end use; such as architectural coatings [4].

Solvent based paints 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 paint has been gravely challenged by the evolution of volatile organic compounds (VOCs) being the solvents typically used in their formulation [5-6]. This VOCs causes environmental problems one of which is global warming.

Volatile organic compounds (VOCs) are organic chemicals that have a high vapor pressure at ordinary room temperature. Their high vapor pressure results from a low boiling point, which causes large numbers of molecules to evaporate or sublime from the liquid or solid form of the compound and enter the surrounding air, a trait known as volatility [7].

Urea formaldehyde is a non-transparent thermosetting resin or plastic produced from urea and formaldehyde. These resins are used in adhesives, finishes, particle board, and molded objects [8]. The acceptance of urea formaldehyde as a universal material in many engineering areas such as in the coating industry is impeded by



some of its inherent qualities such as brittleness, poor water resistance and formaldehyde emission. These disadvantages have limited its uses.

The sorrel (*Hibiscus*) seed oil contains polyunsaturated fatty acids and has a great potential for industrial applications such as in paint and surface coatings, bio-lubricant and production of biodiesel [9]. The sorrel seed oil is rich in unsaturated fatty acids and is also a rich source of lipid soluble antioxidants [10]. Unsaturation of sorrel oil can be increased by epoxidation to create an epoxy group, and subsequently hydroxylation to create more bonding sites along the fatty acid chain of the oil. Copolymerization of the hydroxylated sorrel oil and dimethylol urea will reduce the disadvantages associated with dimethylol urea.

Materials and Methods

Urea, formaldehyde, sodium dihydrogen phosphate, sulphuric acid, sodium hydroxide, sucrose, distilled water, hydrogen peroxide, acetic acid, formic acid, iso-propanol, methanol (all of analytical grades), and sorrel oil obtained from sorrel seeds.

Extraction of Sorrel Oil

Extraction of sorrel seed oil was done using the cold press method, as described by Bamaiyi, *et al.*, [11]. Dry seeds of sorrel were collected and cleaned up. The seeds were pounded in a mortar. The paste was mixed with cold water and allowed to stand for some hours, after which the oil was decanted. The oil was heated in a pan in order to evaporate the excess water present. The oil collected was then kept at room temperature ready for use.

Epoxidation of Sorrel Oil

Epoxidation was carried out using the method described by Goud *et al.*, [12]. 200cm³ of the sorrel oil was introduced in a 500cm³ three necked flask equipped with a reflux condenser and a thermocouple. 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 sorrel oil. A hydrogen peroxide of molar ratio 1.5:1 to the sorrel oil was added drop wise into the mixture. This feeding strategy is required in order to avoid overheating the system since epoxidation is an exothermic reaction. The uniformity of the reaction was maintained by using a magnetic stirrer which runs at 1600rpm under isothermal condition at 50-60°C. The product was cooled and decanted in order to separate the organic-soluble compounds (epoxide sorrel oil) from water-soluble compounds. Warm water was used to wash the epoxidized oil (in small aliquots) in order to remove residual contaminants. This procedure was repeated in triplicates.

Hydroxylation of the Epoxidized Sorrel Oil

Hydroxylation of the sorrel oil was carried out using procedure described by Petrovic *et al.*, [13]. The reaction was performed in a 1000cm³ three necked flask equipped with a reflux condenser and a thermocouple. The flask was placed on a hot plate with temperature control. 150cm³ of the epoxidized sorrel 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 5hrs. Uniformity was maintained by using a magnetic stirrer which runs at 1600rpm. The product (polyol) was cooled and decanted in order to separate the organic-soluble compounds from water-soluble compounds. Warm water was used to wash the polyol (in small aliquots) in order to remove residual contaminants. This procedure was repeated in triplicates.

Resin Synthesis

The one step process (OSP) as reported by Osemeahon and Barminas [14]. One mole of urea (6.0g) was made to react with two moles of formaldehyde (16.22ml) 37-41% (w/v), using 0.2g of sodium dihydrogen phosphate as catalyst. The pH of the solution was adjusted to 6 by using 0.5M H₂SO₄ 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 2hrs after which the resin was removed and kept at room temperature (30°C).



Copolymerization

This was carried out by blending different concentrations (10-60%) of oil in DMU. The mixture was stirred with glass rod and left for 24hrs at room temperature (30°C). For film preparations, resins from DMU and DMU/HCNO were poured into different petri dishes for casting using the solution casting method [15]. The resins were also allowed to cure and set for three days at 30°C. the physical properties of the film were investigated.

Determination of Formaldehyde Emission

Formaldehyde emission was carried out using the standard 2hr desiccator test as described by Osemeahon and Archibong, 2011. The mold used was made from aluminium foil with a dimension of 69.9 x 126.5mm and thickness of 12.0mm. The emitted formaldehyde was absorbed in 25.0ml of water and was analyzed by a refractometric technique using Abbe refractometer. Triplicate determinations were made for the samples and the average value taken.

Determination of Moisture Uptake

The moisture uptake of the resin films were determined gravimetrically, according method described by Osemeahon and Archibong [15]. Known weights of the samples were 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 recorded as the moisture uptake by the resin. Triplicate determinations were made for each sample and the average value recorded.

Determination of Water Solubility

The solubility of DMU and DMU/HSO blends were determined by mixing 1ml of the resin with 5ml of distilled water at room temperature (27-30°C).

Determination of Density, Turbidity, Melting Point and Refractive Index

The densities of the resins were determined by taking the weight of a known volume of resin inside a density bottle using Pioneer (Model PA64) weighing balance. Three readings were taken for each sample and average value calculated. The turbidity of the samples were determined by using Supertek digital turbidity meter (Model 033G). The melting points of the film samples were determined by using Galenkamp melting point apparatus (Model MFB600-010F). The refractive indexes of the samples were determined with Abbe refractometer. The above properties were determined according to standard methods [16].

Determination of Viscosity and Gel Time

Viscosity and gel time were carried out according to method described by Osemeahon and Archibong [15]. A 100ml Phywe made of graduated glass macro-syringe was utilized for the measurement. The apparatus was standardized with 60% (W/V) sucrose solution whose viscosity is 5.9mpa.s at 30°C. The viscosity of the resins was evaluated in relation to that of the standard sucrose solution at 30°C. Three different readings were taken for each sample and the average value calculated. The gel time of the resin was determined by monitoring the viscosity of the resin with time until a constant viscosity profile was obtained.

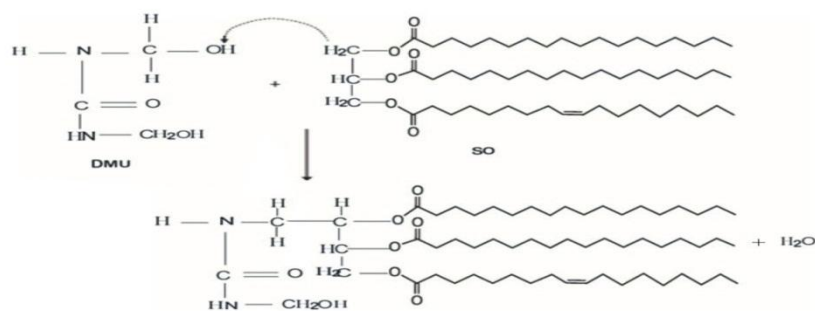
Determination of Elongation at Break

The elongation at break was determined using Inston Tensile Testing Machine (Model 1026). Resin films of dimension 50mm long, 10mm wide and 0.15mm thick was brought to rupture at a clamp rate of 20mm/min and a full load of 20kg. Three runs were carried out for each sample and the average elongation evaluated and expressed as the percentage increase in length.

Result and Discussion

Reaction between dimethylol urea and pure sorrel oil.





FTIR Spectral Analysis

Figure 1a and Figure 1b show the FTIR spectra of pure DMU, SO, ESO, HSO, and a blend of DMU/HSO. The DMU spectra show a peak between 3371 to 663cm^{-1} . The strong and broad O–H stretching of an intermolecularly bonded alcohol, an amine group with a carbonyl ($\text{C} = \text{O}$), while others are CH_2 rocking vibration. The $\text{C} - \text{O} - \text{H}$ in plane bending is typical of the DMU. SO shows a peak of $\text{C} = \text{O}$ and $\text{O} = \text{C} = \text{O}$ at 2928.58cm^{-1} . The $\text{C} - \text{H}$ stretching at 2868cm^{-1} is of rocking vibrations as well as unsymmetric bends. The methylene (CH_2) rocking vibrations were also observed in the 1367cm^{-1} [17]. The epoxidation of the SO does not affect the $\text{C} - \text{O} - \text{C}$ of the SO the $\text{C} - \text{H}$ stretching of an alkane at 2868cm^{-1} of the SO is also unaffected. The bending of the $\text{C} - \text{H}$ was confirmed by the presence of $\text{C} - \text{H}$ bending of an alkyl group [18]. The hydroxylation of SO (HSO) shows a stretching O–H at 2945cm^{-1} which indicates the inductive nature of the hydroxyl group while $\text{C} - \text{H}$ of alkyl and alkanes, $\text{C} = \text{C}$ still remains unreacted. A $\text{C} = \text{O}$ of a carbonyl compound in SO is reflected by the peak at 1747cm^{-1} . The DMU/SO blend (90:10) shows an O–H stretching of a free alcohol due to the inductive effect of the SO due to the attack on the unsaturated carbon center. A carbon dioxide stretching of $\text{O} = \text{C} = \text{O}$ at 2374cm^{-1} the $\text{C} = \text{C}$ stretching of a conjugated alkene/vinylidene was observed at 1649.40cm^{-1} . The peak at 1553.10cm^{-1} indicates the presence of a nitro compound due to the $\text{N} - \text{O}$ stretching vibration while others at 1377.12cm^{-1} to represent the O–H bending of a derivative of alkene. For 1027.13cm^{-1} and 1265.56cm^{-1} , the two wave numbers represent, a $\text{C} - \text{N}$ stretching of amine while the other is for the $\text{C} - \text{N}$ stretching of an aliphatic amine [19].

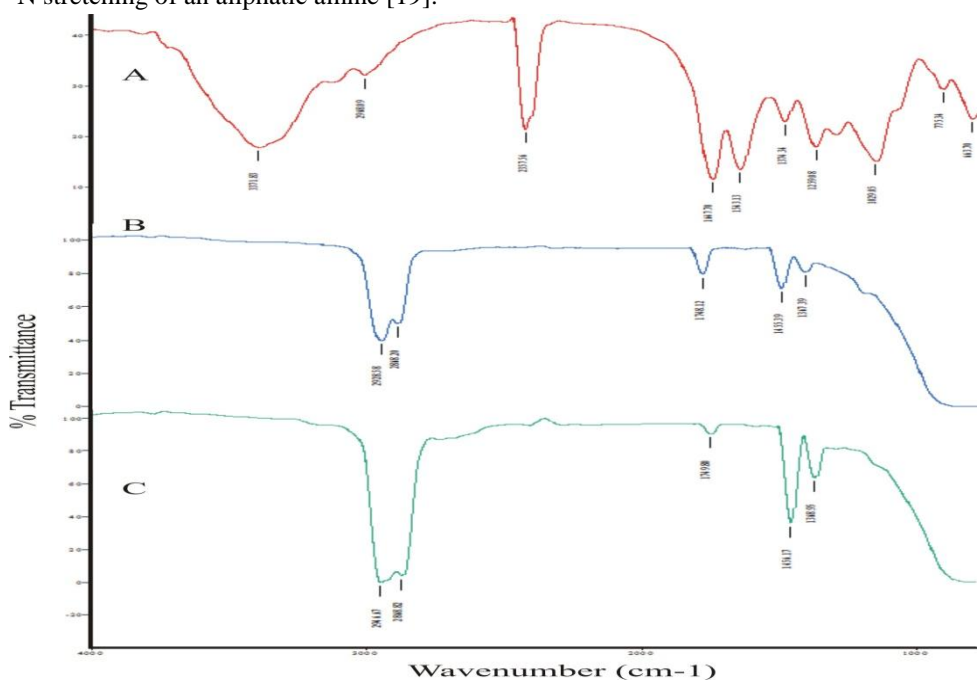


Fig: 1a

A – FTIR Spectra of DMU, B – FTIR Spectra of SO, C – FTIR Spectra of ESO.



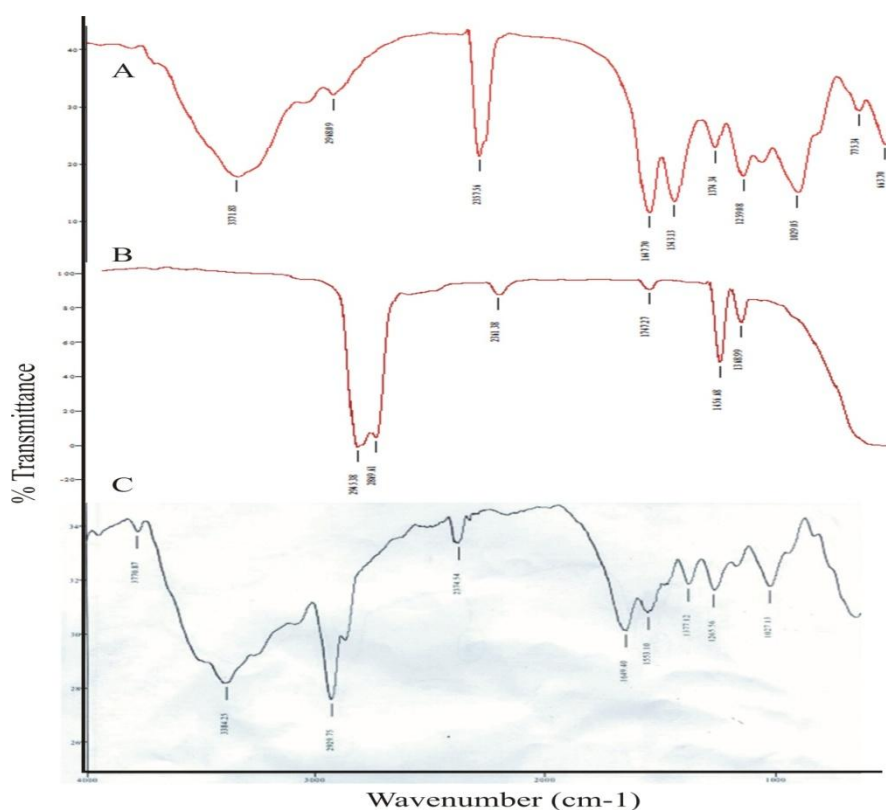


Fig: 1b

A – FTIR Spectra of DMU, B – FTIR Spectra of HSO, C – FTIR Spectra of DMU/HSO.

Effect of HSO Concentration on the Viscosity of DMU/HSO Copolymer Composite

Viscosity is a property of fluid which opposes the relative motion between the two surfaces of the fluid that are moving at different velocities. It is a measure of a fluid's resistance to gradual deformation by shear stress or tensile stress. In simple terms, viscosity means friction between the molecules of fluid. It corresponds to the informal concept of “thickness”. Viscosity of a paint binder affects flow properties such as leveling and sagging, film forming property, adhesion property and drying rate of paint [14].

Figure 2 is a graph that shows the effect of HSO concentration on the viscosity of DMU/HSO copolymer resin. From the graph, it is observed that viscosity increases with increase in inclusion of HSO. This is attributable to the increase in molecular weight as DMU copolymerizes with the relatively large molecular weight of HSO [20]. From the graph, it is observed that viscosity reaches its peak with 40cm³ inclusion of HSO after which it begins to fall. This is due to the dissociation of the molecules in the blend.

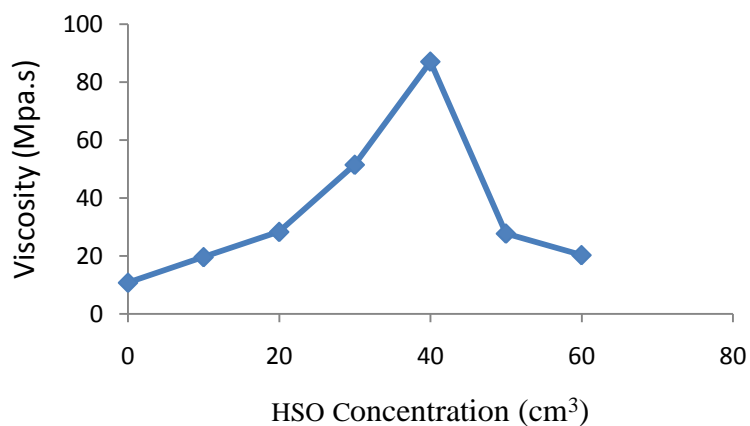


Figure 2. Effect of HSO concentration on the viscosity of DMU/HSO copolymer composite



Effect of HSO Concentration on the Density of DMU/HSO Copolymer Composite

Density is defined as the ratio of an object's mass to its volume. It increases with increasing mass or with decreasing volume. Objects with higher density are invariably heavier than low-density objects of similar appearance.

In the coating industry, the density of the paint binder has a great influence on such factors as flow, leveling and sagging, and brushability of paint [21].

The effect of HSO concentration on the density of DMU/HSO copolymer resin is shown in Figure 3. There's a gradual decrease observed with increase in concentration of HSO. This is because the physical properties of a polymer depend on the chain length and content of the soft and hard segments. The decrease observed is due to increase in the soft segments [22]. The densities of the blends are expected to decrease between the density of DMU resin and HSO, since pure SO has a lower density than pure DMU [23].

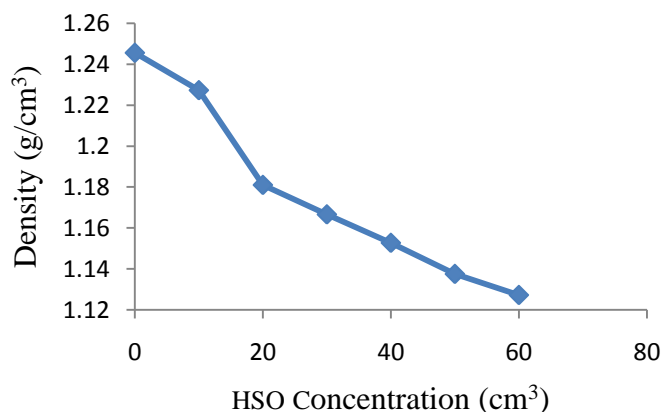


Figure 3: Effect of HSO concentration on the density of DMU/HSO copolymer composite

Effect of HSO Concentration on the Gel-time of DMU/HSO Copolymer Composite

The gel-time is one of the most important characteristics of curing. This is because it describes the attainment of certain critical conversion responsible for the transition from liquid to solid state of the curing process [24]. The gel-point is characterized by the appearance in the reactive system of a macromolecule with an infinity large molecular weight [25].

Figure 4 shows the effect of HSO concentration on the gel-time of DMU/HSO copolymer composite. It is observed that the gel-time increases with increase in HSO concentration. This is due to the decrease in reactivity which results from the increase in the size of the alkyl group attached to the carbonyl carbon of the copolymer. Also, the non-drying nature of SO can be a contributory factor.

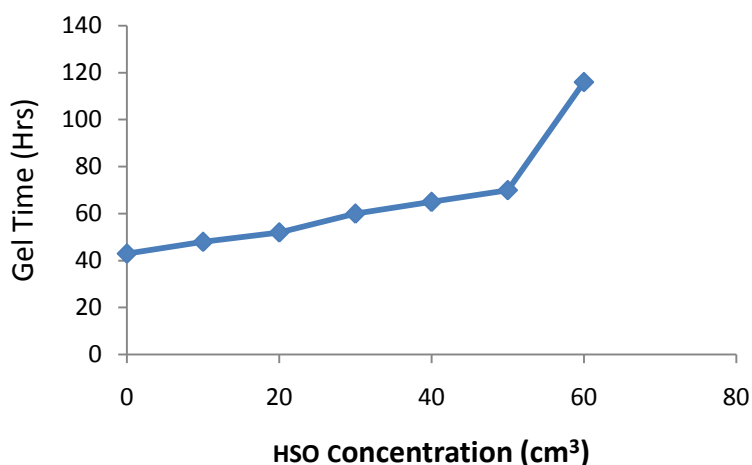


Figure 5: Effect of HSO concentration on the gel-time of DMU/HSO copolymer composite



Effect of HSO Concentration on the Turbidity of DMU/HSO Copolymer Composite

In the coating industry, the optical properties of the binder such as the turbidity are very important. This is because it is related to the gloss properties of the paint [26].

Figure 6 shows the effect of HSO concentration on turbidity of DMU/HSO copolymer composite. The turbidity of pure DMU is low. But with inclusion of HSO, the turbidity is observed to increase greatly. This may be explained in terms of the differences in molecular morphology and orientation. The behavior of the resins is also attributable to the differences in the level of crystallinity and crystalline orientation of the resin molecules [27].

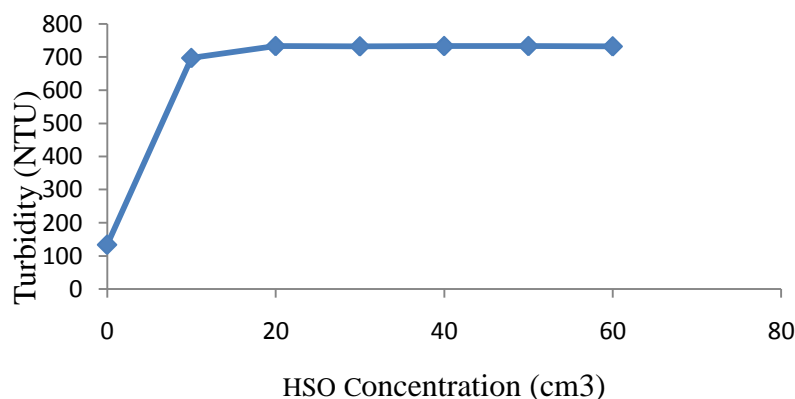


Figure 6: Effect of HSO concentration on the turbidity of DMU/HSO copolymer composite

Effect of HSO Concentration on the Refractive Index of DMU/HSO Copolymer Composite

Gloss is an important factor of many coating materials [28]. the gloss of a paint coating with or without pigments is a function of the refractive index of the surface, the angle of incidence of the beam of light and the nature of the material [26].

Figure 7 shows the effect of the concentration of HSO on the refractive index of DMU/HSO copolymer composite. It can be observed that increase in inclusion of HSO increases the refractive index of the copolymer composite. This is attributable to the increase in molecular weight of HSO concentration, and molecular features [26]. DMU has a lower refractive index than HSO. Therefore as HSO is added to DMU, the optical property of DMU with regards to refractive index increases.

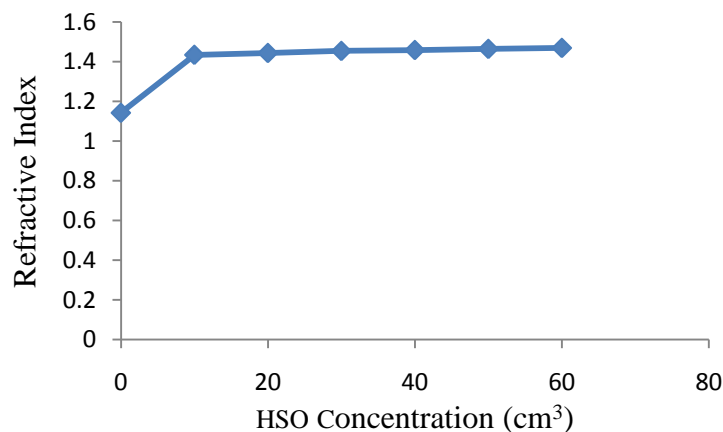


Figure 7: Effect of HSO concentration on the refractive index of DMU/HSO copolymer composite

Effect of HSO Concentration on the Formaldehyde Emission of DMU/HSO Copolymer Composite

The emission of formaldehyde during the curing process of a resin is known to be one of the major drawbacks of urea formaldehyde [29-30]. Serious efforts must be made to reduce formaldehyde to acceptable levels in the development of paint binders from urea formaldehyde resins [31].

Figure 8 show the effect of HSO concentration on the formaldehyde emission of DMU/HSO copolymer composite. It can be observed that formaldehyde emission decreases with increase in inclusion of HSO



concentration. This can be explained on the basis of gradual reduction in urea formaldehyde concentration with increase in HSO concentration in the copolymer [32]. This may also be as a result of reduction in the curing process which reduces formaldehyde emission resulting from improved flexibility. HSO also serves as an obstacle which disrupts the continuity in the DMU matrix, making it difficult for the reactive groups in the DMU to interact [5, 33].

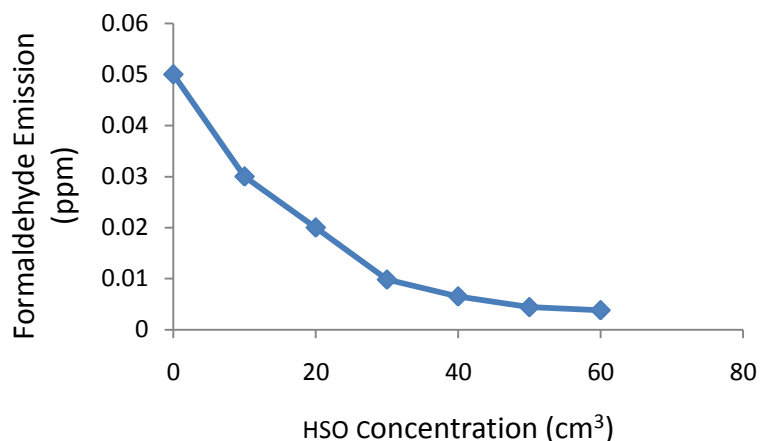


Figure 8: Effect of HSO concentration on the formaldehyde emission of DMU/HSO copolymer composite

Effect of HSO Concentration on the Melting Point of DMU/HSO Copolymer Composite

In the coating industry, the melting point of a binder is related to its thermal resistance as well as to the brittleness of the coating film [31]. The melting point of a polymer has a direct bearing to its thermal property. It is also related to its molecular weight, degree of crosslinking and the level of rigidity of the polymer [30, 34].

Figure 9. shows the effect of HSO concentration on the melting point of DMU/HSO copolymer composite. At the initial stage, a gradual decrease in melting point of the copolymer is observed, and eventually becomes very sharp at 40cm³ inclusion of HSO concentration. This can be explained in terms of increase in molecular mobility, differences in molecular feature, crystallinity and crystalline orientation [35-37]. However, at 50cm³ inclusion of HSO, a sharp increase in the melting point is observed. This behaviour can be attributed to increase in molecular weight, as well as polymer association.

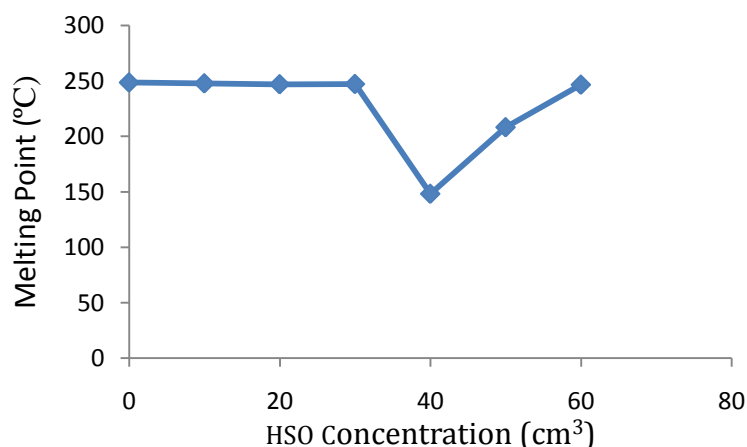


Figure 9: Effect of HSO concentration on the melting point of DMU/HSO copolymer composite

Effect of HSO concentration on the moisture uptake of DMU/HSO copolymer composite

In the coating industry, the moisture uptake of the paint binder is very important because it is responsible for blistering and broominess of paint [31]. The interaction of structural network of polymer resins with water is



both of fundamental and technical interest [30]. Water uptake affects vital properties of the polymer material such as the mechanical, physical, structural and thermal properties [38-39]. However, one of the major setbacks of urea formaldehyde resin is its poor water resistance [31].

Figure 10. shows the effect of HSO concentration on the moisture uptake of DMU/HSO copolymer composite. It is observed that the moisture uptake decreases with increase in the inclusion of HSO concentration. This can be explained in terms of the differences in chain topology which is related to the molecular size holes in the polymer structure which also depend on morphology and crosslinked density [38-39]. The transport of water in a polymer network is related to the availability of molecular-sized holes in the polymer structure and polymer-water affinity. The availability of holes depend on polymer structure, morphology and crosslink density. The polymer-water affinity is related to the presence of hydrogen bonding sites along the polymer chains, which create attractive forces between the polymer and the water molecules [39]. The hydrophobic nature of HSO is also responsible for this trend. This development will help to address one of the major drawbacks (poor water resistance) of DMU.

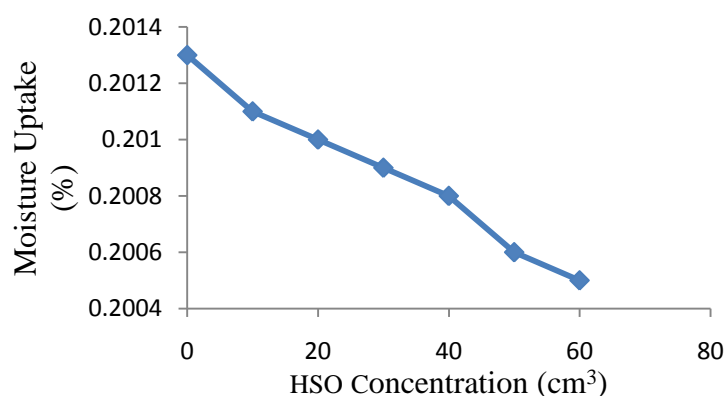


Figure 10: Effect of HSO concentration on the moisture uptake of DMU/HSO copolymer composite

Effect of HSO Concentration on the Elongation at Break of DMU/HSO Copolymer Composite

One of the major setbacks of urea-formaldehyde is its brittleness, which has hindered its use as a binder [40]. The effect of HSO concentration on the elongation at break of DMU/HSO copolymer composite is shown in Figure 11. The gradual increase in elongation at break of the copolymer composite with increase in the concentration of HSO shows an increase in the soft segments in the copolymer as more HSO is added. This may also be due to the HSO to enhance flexible spacing in the copolymer. A change in molecular weight, morphology and crosslinking density in the copolymer with change in HSO concentration may also be responsible for this behaviour [41-42].

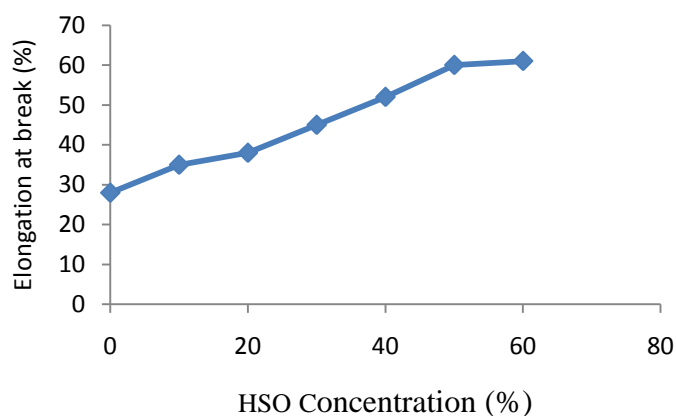


Figure 11: Effect of HSO concentration on elongation at break of DMU/HSO copolymer composite



Effect of HSO Concentration on Solubility of DMU/HSO Copolymer Composite

Water solubility is an important parameter in the consideration of any resin as a binder for emulsion paint formulation. The binders in oil paint are water insoluble, while that for emulsion paint are water soluble.

Table 1 shows the effect of HSO concentration on the solubility of DMU/HSO copolymer composite. It is observed that from the beginning up to 40cm³ loading of HSO, the copolymer is soluble. This is because of the fact that DMU which is highly hydrophilic dominates the solution. However, above 40cm³ inclusion of HSO, the copolymer became hydrophobic and therefore became insoluble in water.

Table 1: Effect of HSO concentration on the solubility of DMU/HSO copolymer composite

DMU/HSO Concentration	Solubility in Water
0	Highly soluble
90/10	Soluble
80/20	Soluble
70/30	Slightly soluble
60/40	Slightly soluble
50/50	Insoluble
40/60	Insoluble

Conclusion

Copolymerization of dimethylol urea (DMU) and hydroxylated sorrel oil (HSO) has been carried out. A reduction in the moisture uptake, brittleness, and formaldehyde emissions which are the major disadvantages attributed to dimethylol urea adhesives. This is due to the hydrophobicity of sorrel oil, flexibility of sorrel oil, and reduction in the molar ratio of dimethylol urea by the inclusion of sorrel oil. Therefore, dimethylol urea/hydroxylated sorrel oil resins can be recommended for use as binders for emulsion paint formulation.

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