ISSN: 2230-9926

Available online at http://www.journalijdr.com

IJDR

International Journal of Development Research
Vol. 08, Issue, 10, pp.23159-23166, October, 2018

ORIGINAL RESEARCH ARTICLE OPEN ACCESS

EVALUATION OF COMPOSITES FROM DIMETHYLOL UREA AND HYDROXYLATED COCONUT OIL FOR POSSIBLE APPLICATION FOR WATER RESISTANT EMULSION PAINT

[1]Osemeahon, S.A., [1]Dass, P.M., *[1]Fadawa, F.G. and [2]Archibong, C.S.

[1]Department of Chemistry Modibbo Adama University of Technology Yola, Adamawa state
[2]Department of Chemical Science, Federal University Wukari, Taraba State

ARTICLE INFO

Article History:
Received 18th July, 2018
Received in revised form
20th August, 2018
Accepted 20th September, 2018
Published online 29th October, 2018

Key Words:

Copolymerization, Dimethylol urea, Hydroxylated coconut oil, Binder, Emulsion paint.

ABSTRACT

Copolymerization of dimethylol urea and hydroxylated coconut oil was carefully studied in this work. The FTIR spectroscopy of the pure DMU and DMU/HCNO showed chemical interaction took place. Some physico-chemical parameters such as melting point, gel time, moisture uptake, turbidity, viscosity, density, solubility, formaldehyde emission, and refractive index was 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/HCNO) resin will serveas a potential binder for emulsion paint formulation.

Citation: Osemeahon, S.A., Dass, P.M., Fadawa, F.G., Archibong, C.S. 2018. "Evaluation of composites from dimethylol urea and hydroxylated coconut oil for possible application for water resistant emulsion paint", *International Journal of Development Research*, 8, (10), 23159-23166

INTRODUCTION

The terms "paint" and "surface coating" are often used interchangeably. Surface coating is the more general description of any material that may be applied as a thin continuous layer to a surface. Paint was traditionally used to describe pigmented materials as distinct from clear films which are more properly called lacquers or varnishes. Basically, there are two major types of paint. Oil paint, which is oil based, and emulsion paint which is water based. Solvent paint 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. The evolutions of volatile organic compounds which have health and environmental hazards have been considered seriously. This has led to the restrictions imposed by authorities, driving the industries towards the production of paints with little or no volatile organic compounds (Lee, *et al*, 2011 and Yousefi, *et al*, 2011). Water based paints are gaining popularity with the public's efforts to avoid using paints that involve harmful solvents and dangerous chemicals in its application and cleanup. Urea formaldehyde (UF) resin adhesive is a polymeric condensation product of the chemical reaction of formaldehyde and urea, and considered as one of the most important wood adhesive. But its acceptance 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 limit its uses. Coconut oil predominantly contain medium chain triglycerides, with roughly 92% saturated fatty acids and 2% polyunsaturated fatty acids, unsaturation could further be increased by expoxidation and then hydroxylation to create more bonding sites for the oil. When this is achieved, blending of the hydroxylated coconut oil and the dimethylol urea will be a reality. When blending is achieved, the flexible coconut oil will reduce the rigidity associated with dimethylol urea.

****Corresponding author:*** **Fadawa, F.G.,**
Department of chemistry ModibboAdama University of Technology Yola, Adamawa state.

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 coconut oil.

Extraction of coconut oil: Method described by Hamid, *et al*, 2011 was used. Coconuts were collected and de-husked by removing the cap which was done by tapping lightly on a hard surface. The collected coconut meat was grated and milk extracted. This was done by adding water to the grated coconut and mixed well. The milk was squeezed out from the mix. This was done twice to extract more milk from the leftover. The collected coconut milk was boiled to evaporate water from the mix. The oil was seen bubbling on the surface of the heated milk. This was removed from the fire and the oily milk allowed to cool. Oil was collected from the cooled mixture by placing a piece of cloth over the heated mix and lightly pressing with the finger tips. The oil was absorbed by the cloth from the mix and this was squeezed into another container. However, this still contained lots of coconut flesh. The content was allowed to settle down for a few minutes, and pure coconut oil was seen floating up. The container was tilted slowly and the oil drained into another container.

Epoxidation of the coconut oils: Epoxidation was carried out using the method described by Goud *et al*., 2007. 200cm3 of the coconut 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 coconut oil. A hydrogen peroxide of molar ratio 1.5:1 to the coconut 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 about 1600rpm under isothermal condition at 50-60oC. The product was cooled and decanted in order to separate the organicsoluble compounds (epoxide coconut 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 triplicates.

Hydroxylation of the epoxidized coconut oil: Hydroxylation of the coconut oil was carried out using procedure described by Petrovic *et al*., 2003. 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 coconut 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 60oC 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 triplicates.

Resin synthesis: The one step process (OSP) as reported by Osemeahon and Barminas (2007) was adopted. 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 H_2SO_4 and 1.0M NaOH solutions. The solution was heated in a thermostatically controlled water bath at 70^{O}C. The reaction was allowed to proceed for 2hrs after which the resin was removed and kept at room temperature (30^{O}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^{O}C). For film preparations, resins from DMU and DMU/HCNO were poured into different petri dishes for casting using the solution casting method (Osemeahon and Archibong 2011). The resins were also allowed to cure and set for three days at 30^{O}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, 2011. 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/HCNO blends were determined by mixing 1ml of the resin with 5ml of distilled water at room temperature (27-30^{O}C).

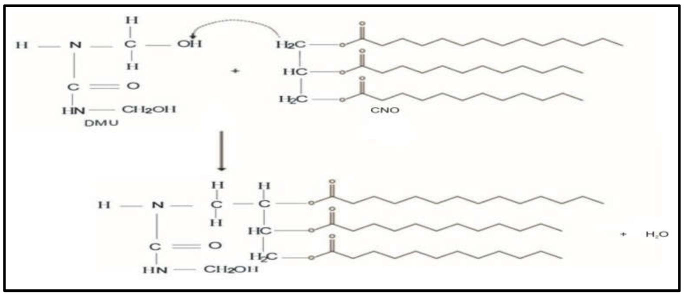
Determination of density, turbidity, melting point and refractive index: The density of the resins were determined by taking the weight of a known volume of resin inside a density bottle using Pioneer (Model PA64) weighing balance. Three readings were taken for each sample and average value calculated. 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 (AOAC), 2000).

Determination of viscosity and gel time: Viscosity and gel time were carried out according to method described by Osemeahon and Archibong, 2011. 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^{0}C. The viscosity of the resins was evaluated in relation to that of the standard sucrose solution at 30^{O}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 coconut oil.



FTIR spectral analysis: Figure 1a and 1b show the FTIR spectra of pure DMU, CNO, ECNO, HCNO, as well as that of a blend of DMU/HCNO. It is difficult to the differences by visual examination of the pure oil and its blends (Mariana-Atenna *et al*, 2012). The FTIR Spectra of pure DMU shows that DMU contains a hydroxyl group (OH), and an amine group (NH_2) between 3371cm^{-1} and 3300cm^{-1}. These peaks are also present in the FTIR spectra of pure coconut oil with a strong stretching absorption of O-H stretch at 3469cm^{-1} due to the hydrophilicity of CNO. This OH stretching is particularly due to the absorption of water which is intermolecularly bonded. A C – H stretching is also observed at wavelength 2951cm^{-1}. Bending absorption of methylene (CH_2) is observed at 1456cm^{-1}. The two peaks observed at 1747cm^{-1} and 1162cm^{-1} are due to stretching absorption of aldehyde (C=O) and esters (C – O) respectively (Mahmood, *et al*., 2009). In the ECNO, both the peaks of OH and NH diminished due to the epoxidation of the CNO. The concentration of O-H and N-H diminishes due to the weak bands. C=O bond was observed in ECNO and DMU as well. The C=O in CNO and ECNO is due to the aldehyde of fatty acid. But this is not observed in DMU which lies between 3500cm^{-1} to 3310cm^{-1}. The spectrum also shows a signal at 1746cm^{-1} which indicates the presence of a carbonyl compound and an ester bond in the region between 1640-1810cm^{-1} which is Sp hybridized (C = double bond), and indicates the presence of a fatty acid. At 1453.49cm^{-1} it shows a peak with medium signal attributed to C–H bonding (vibration of deformation) of an alkane (Sp3 hybridization) of a methyl group (CH_3), while S=O stretching of sulfonamide appears at 1363.70cm^{-1} and a CH_2 vibration of deformation of methylene was observed. There is no peak found at 3007cm^{-1} and 1655cm^{-1}. This is indicative of the presence of unsaturated fatty acids (Yaakob and Abdul, 2013). In Figure 1b which shows the DMU, HCNO and blend (DMU/CNO), the hydroxylation of the CNO does not affect the hydroxyl group in the CNO. This can be found between 3717cm^{-1} which is due to a free alcohol present in it, and a C–O indicative of an aldehyde of the coconut oil. The hydroxylation of the CNO has no effect on both the hydroxyl group and the aldehyde. Other peaks observed are at 2923cm^{-1} due to C–H stretching of an alkane, 2357cm^{-1} due to O=C=O of a strong stretching carbon dioxide. The peak at 1739cm^{-1} indicates the C=O strong stretching of an aldehyde, while that at 1454cm^{-1} is indicative of C-H of the medium bending of an alkane of a methyl group (CH_2) (Mahmood *et al*., 2009). In all, CNO and its blends C–H stretching of an alkane and alkenes were observed for both (Methylene and Methyl). The DMU/CNO blend shows the presence of O–H (hydroxyl), and a primary amine from the DMU (NH_2). Others are C–O stretching of an aldehyde which is a carbonyl compound with numerous C–H, C=C, C–C of methyl group and a rocking symmetry of CH_2. This could be attributed to the 1,2,3-trisubstituted compounds in the CNO. The presence of ether due to the epoxidation of compound with an overlap between 600-1200cm^{-1} (Coates, 2000). One of the powerful tools used in the identification and investigation of the presence of functional groups in a molecule is the FTIR spectroscopy, as each specific chemical bond has a unique energy absorption band, and can obtain structural and bond formation on a complex to study the strength and the fraction of hydrogen bonding and miscibility (Kaniappan and Latha, 2011; Saravanan *et al*., 2011).

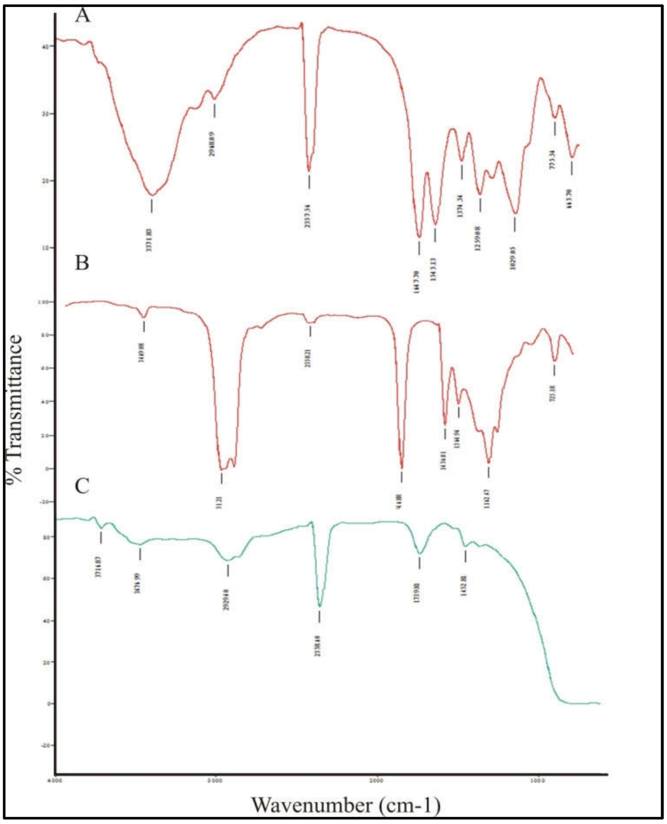


Fig. 1a. A – FTIR Spectra of DMU, B – FTIR Spectra of CNO, C – FTIR Spectra of ECNO

Effect of HCNO concentration on the viscosity of DMU/HCNO copolymer composite: Viscosity is an important rheological property of fluids. Rheology is a branch of physics that deals with deformation and flow of matter under stress. It has been used for many years as a semi-quantitative tool in polymer science and engineering (Oluranti, *et al.* 2011). Studying the rheological properties of fluids and gels is important.

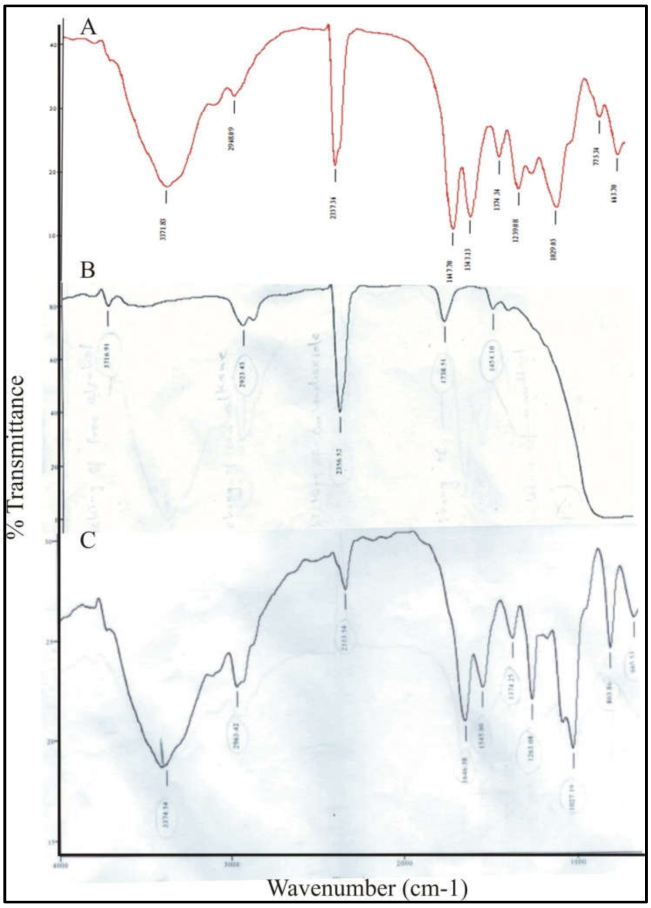


Fig. 1b. A – FTIR Spectra of DMU, B – FTIR Spectra of HCNO, C – FTIR Spectra of DMU/HCNO

This is because it is the key to design processing operations that depend on the way the product flows. For example, smoothness, creaminess, spread-ability, thickness, flow-ability, hardness and brittleness (Hussain and Nasr, 2010). Viscosity of a paint binder is very important in the coating industry. This is because it controls factors such as flow rates, sagging and leveling, thermal and mechanical properties, and dry rate of paint film and adhesion of the coating to substrate (Osemeahon and Barminas, 2007). Figure 2.shows the effect of HCNO concentration on the viscosity of DMU/HCNO composite. Initially, where the amount of HCNO is zero while that of DMU is 100, the viscosity is relatively low. However, with the inclusion of HCNO, there is an increase in the viscosity and reaches its peak at 50mls of HCNO. This difference in viscosity may be attributed to the variation of the different chain lengths, and the increase in molecular weight of the copolymer formed. Increase in molecular weight gives rise to increase in viscosity (Afzal *et al*., 2013). Further inclusion of HCNO after 50mls into the copolymer signals the overloading above the optimal level and therefore gave rise to polymer degradation resulting from the presence of COOH in the HCNO, which gives rise to fragmentation of long polymer chains to short oligomers, leading to a decrease in the viscosity as could be seen from the graph (Osemeahon and Dimas., 2014).

Effect of HCNO concentration on the density of DMU/HCNO copolymer composite: Density is a physical property of matter that expresses the ratio of mass to volume, and is an important physical parameter in polymer engineering processes. Therefore, density measurements are very useful for the identification and characterization of different substances. It is a factor that affects the production cost and profitability of the manufacturing process (Kazys and Rekuviene, 2011). In the coating industry, the density of the binder is important because it influences such factors as flow, leveling and sagging, pigment dispersion and brush-ability. Figure 3 shows the effect of HCNO concentration on the density of DMU/HCNO composite. It is observed that there's a gradual decrease in the density with the increase in HCNO concentration. This behavior may be attributed to the difference in molecular features and morphology which influences the packing nature of the composite molecules as the concentration of HCNO is increased. The density of DMU as can be seen is higher than that of HCNO. Therefore, it is expected that with increase in HCNO concentration, there will be decrease in the density (Osemeahon and Dimas, 2014).

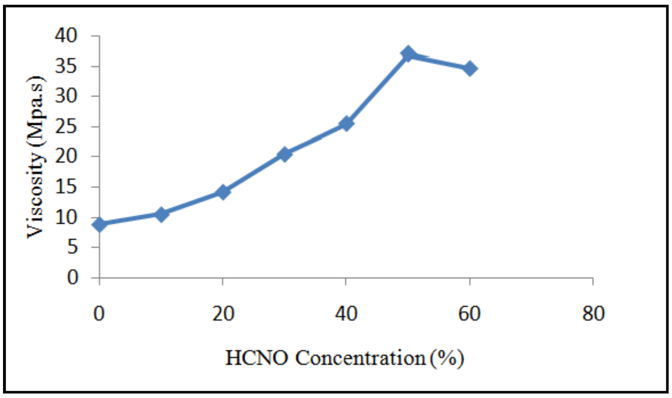


Fig. 2. Effect of HCNO concentration on the viscosity of DMU/HCNO copolymer composite

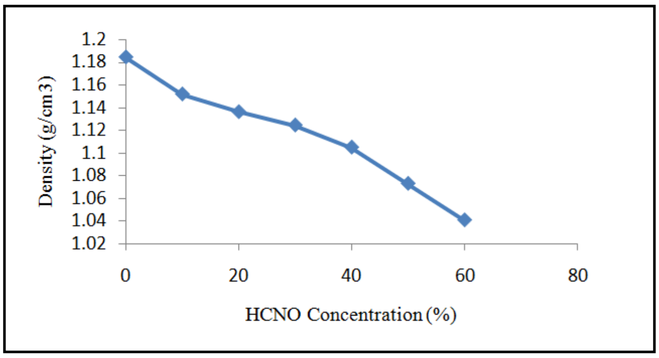


Fig. 3. Effect of HCNO concentration on the density of DMU/HCNO copolymer composite

Effect of HCNO concentration on the gel-time of DMU/HCNO copolymer composite: The function of a binder's gel-time among other factors determines the dry time of paint. Technically, gel-time enables a paint formulator to ascertain the optimum storage period of a binder before its utilization for paint formulation. It is also important in the determination of adhesion (Menkiti and Onukwuli, 2011; Desai *et al*, 2003).Figure 4.shows the effect of HCNO concentration on the gel-time of DMU/HCNO composite. From the graph, it can be observed that the gel-time increased with increase in inclusion of HCNO. This may be attributed to increase in molecular weight, reduction in intermolecular forces between the copolymer chains, which leads to increase in viscosity of the composite resin (Osemeahon and Dimas, 2014). Therefore, continuous increase in HCNO will lead to increase in cross-linking of the molecules.

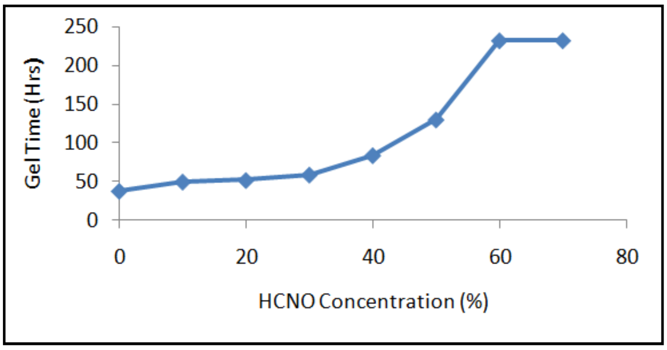


Fig. 4. Effect of HCNO concentration on the gel-time of DMU/HCNO copolymer composite

Effect of HCNO concentration on the turbidity of DMU/HCNO copolymer composite: Figure 5 shows the effect of HCNO concentration on the turbidity of DMU/HCNO composite. It can be observed that turbidity is relatively low at 0cm^3 HCNO. This is because pure DMU is clear and transparent. On addition of HCNO, turbidity increases. This may be due to the growth of large inter-polymer aggregate, as well as thermodynamics of mixing (mixing behavior of the copolymer composite) among other factors. The higher the HCNO concentration, the higher the light scattering and the higher the turbidity (Osemeahon and Dimas, 2014).Turbidity measurement of a binder is done in order to characterize the optical properties of the binder as it relates to the gloss property. The refractive index gives indication of the turbidity. The principle of turbidity was derived from light scattering. When there is homogeneity and there are few particles, there will be less scattering; hence higher scattering is observed when we have a non-homogenous system with a lot of particles.

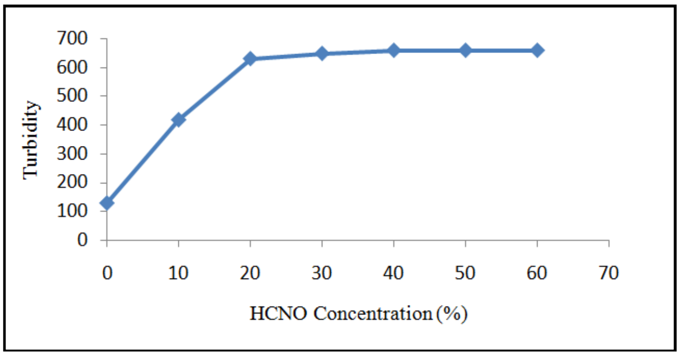


Fig. 5. Effect of HCNO concentration on the turbidity of DMU/HCNO copolymer composite

Effect of HCNO concentration on the refractive index of DMU/HCNO copolymer composite: Gloss is a very important property of coatings for the purpose of decoration and aesthetic value (Kaygin and Akgun, 2009; Yumiko *et al*., 2010). Gloss is one of the advantages of oil paint, and it shows how much paint reflects and shines. For emulsion paint to challenge oil paint, the gloss property of emulsion paint must challenge that of the oil paint. The effect of HCNO concentration on the refractive index of DMU/HCNO copolymer composite is shown in Figure 6. From the graph, it can be observed that refractive index increases with increase in HCNO volume. Pure DMU has a low refractive index. As HCNO is added to DMU, the optical property of DMU is changed. There is an alteration in the crystallinity of the system with HCNO loading due to increase in the number of various chain lengths that make up the copolymer. This may perhaps explain the above behavior (Jain, 2008). This result implicates that an emulsion paint with high gloss can be produced from DMU/HCNO copolymer paint binder.

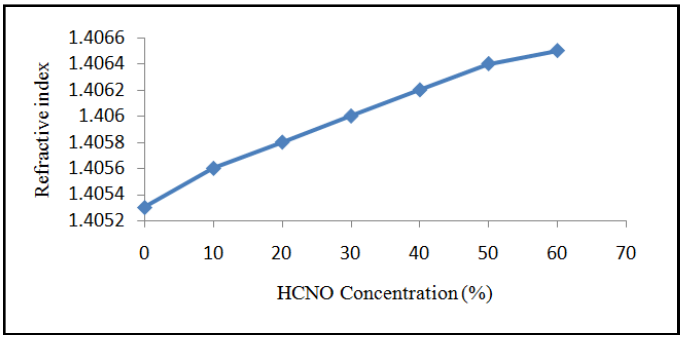


Fig. 6. Effect of HCNO concentration on the refractive index of DMU/HCNO copolymer composite

Effect of HCNO concentration on the formaldehyde emission of DMU/HCNO copolymer composite: The emission of hazardous formaldehyde during the curing process of urea formaldehyde resins has continued to pose a serious setback. Therefore, it is very important to determine the formaldehyde emission from synthesized urea-formaldehyde resin prior to its application (Derkyi, *et al*., 2008). The effect of HCNO concentration on the formaldehyde emission on DMU/HCNO composite is shown in Figure 7. As the concentration of HCNO increases, formaldehyde emission decreases. This may be attributed to the gradual decrease in DMU content, with HCNO loading in the blend. It may also be attributed to the reduction in stress during the curing process which reduces emission resulting from improved flexibility. HCNO serves as a hindrance, disrupting the continuity of the DMU matrix, making it difficult for the reactive groups in the DMU to interact with each other (Lee *et al*., 2011; Abdullahi and Park, 2010).

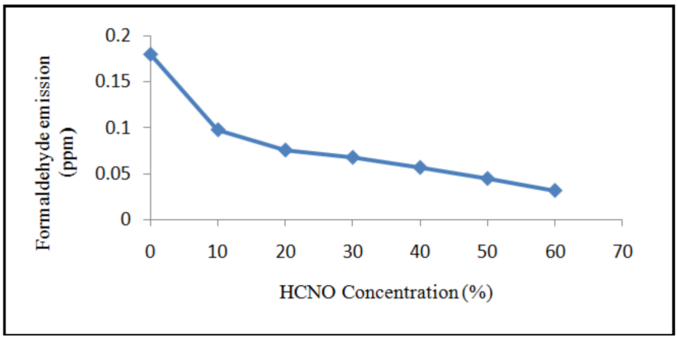


Fig. 7. Effect of HCNO concentration on the formaldehyde emission of DMU/HCNO copolymer composite

Effect of HCNO concentration on the melting point of DMU/HCNO copolymer composite: In the coating industry, the melting point of a binder is related to its thermal resistance as well as its brittleness. The thermal property, degree of cross linking, level of rigidity and molecular weight of the polymer is related to its melting point (Afsoon *et al*., 2011). Figure 8 shows the effect of HCNO concentration on the melting point of DMU/HCNO composite. The result shows a gradual decrease in the melting point from 0-40cm^3 volume of the HCNO in the blend. This may be attributed to the increase in flexibility due to increase in volume of HCNO which gave rise to increased molecular mobility. However, there’s a sharp increase in melting point observed at 50cm^3 HCNO inclusion in the blend. This behavior may be as a result of increase in molecular weight and cross-linking density, which makes the composite polymer harder. It can be observed from the graph that optimal hardness was obtained at 50cm^3 loading of HCNO. Also, at 60cm^3 HCNO inclusion in the blend, polymer degradation might have occurred as the rigid structure of the copolymer became flexible therefore making the molecules to gain mobility resulting in the decrease in melting point (Osemeahon and Dimas, 2014).

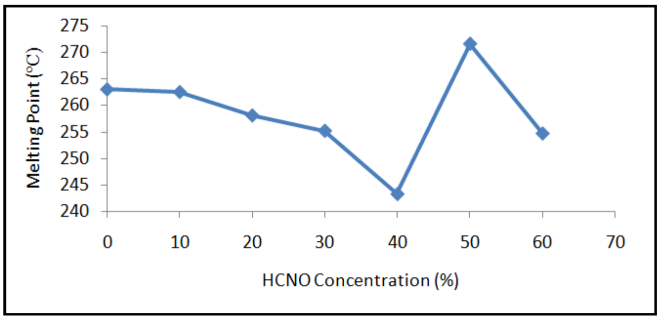


Fig. 8. Effect of HCNO concentration on the melting point of DMU/HCNO copolymer composite

Effect of HCNO concentration on the moisture uptake of DMU/HCNO copolymer composite: The binder is one of the major constituent of paint, as it plays a major role in the moisture transport properties of paint. A detailed knowledge of moisture transport is essential to understand the resistance of a material against attacks from its environment. One of the major setbacks of urea-formaldehyde resin is its high moisture uptake which has to be improved in order to satisfy the coating industry. Water induces chemical degradation of the network and also generates stress because of swelling and hence blistering of the coating film. It deteriorates thermo-mechanical properties as well as adhesion (Gonzalez *et al*., 2012; Toloel *et al*., 2013). Figure 9 shows the effect of HCNO concentration on moisture uptake of DMU/HCNO composite. It can be observed that the moisture uptake decreased with increase in the volume of HCNO. This may be attributed to the hydrophobic nature of HCNO. Therefore, increase in the volume of HCNO gives rise to increase in hydrophobicity of the polymer composite (Naghash *et al*., 2007). Hydrophobic components present in the binder give the best combination of improving water resistance of waterborne coatings (Emile, 2003; Bharath and Swamy, 2009).

Effect of HCNO concentration on the elongation at break of DMU/HCNO copolymer composite: Elongation tells how much a material will stretch before it breaks. It determines the adhesion between phases because of its sensitivity for load transfer between the phases (Hwang, *et al*., 2012: Cakir, *et al*., 2012). The gradual increase in the elongation at break with increase in the concentration of HCNO as shown in figure 10 may be attributed to the ability of HCNO to enhance flexible spacing, thereby increasing the free volume in the blend. Also, it may be due to the increase in the soft segments in the blend as HCNO is added (Akinterinwa, *et al*., 2016).

Water solubility: Table 1 shows the effect of HCNO concentration on the solubility of DMU/HCNO copolymer composite. It is observed that from the beginning up to 40cm^3 volume of the HCNO, the copolymer is in a hydrophilic state with DMU dominating. This allows the copolymer to stay in aqueous solution. But above 40cm^3 inclusion of HCNO, the copolymer is noticed to have adopted the hydrophobic nature due to the hydrophobicity of HCNO. Thereby the insolubility observed in this state (Osemeahon and Dimas, 2014). The development of amino resins for emulsion paint formulation requires an understanding of the solubility of the resin in water (Osemeahon *et al*., 2010). It is important from the technical point of view as well as from the processing point of view. This is more so because the solubility of DMU decreases with increase in concentration of HCNO concentration. It therefore means for a paint formulator using this binder, it is only 30cm^3 of oil that can beusedfor paint formulation, as this is the quantity that can go into solution.

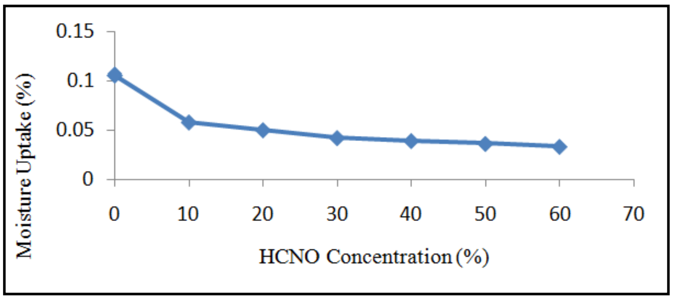


Fig. 9. Effect of HCNO concentration on the moisture uptake of DMU/HCNO copolymer composite

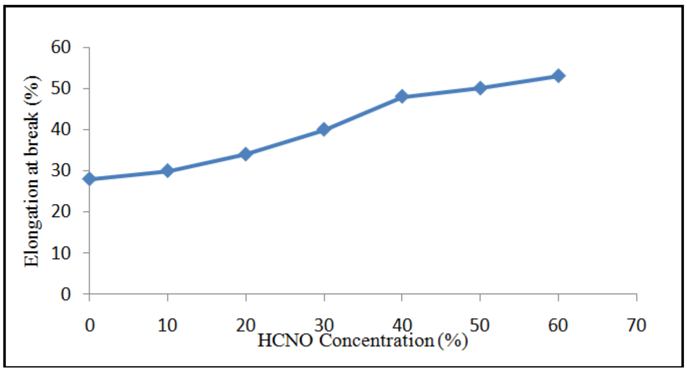


Fig. 10. Effect of HCNO concentration on the elongation at break of DMU/HCNO copolymer composite

Table 1. Effect of HCNO concentration on the solubility of DMU/HCNO copolymer composite

DMU/HCNO concentration	Solubility in water
DMU	Highly soluble
90/10	Soluble
80/20	Soluble
70/30	Slightly soluble
60/40	Insoluble
50/50	Insoluble
40/60	Insoluble

Conclusion

The copolymerization of dimethylol urea (DMU) and hydroxylated coconut oil has further reduced the moisture uptake, hardness/brittleness, and formaldehyde emission that are common attributes associated with dimethylol urea binders. This is due to the hydrophobicity, flexibility of coconut oil, and molar reduction of dimethylol urea by subsequent inclusion of coconut oil. Therefore dimethylol urea/ hydroxylated coconut oil resins can be recommended for use as binder for emulsion paint formulation.

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