# Full Length Research Paper

# Copolymerization of methylol urea with ethylol urea resin for emulsion paint formulation

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Accepted 23 May, 2011

In this work, copolymerization of urea formaldehyde (UF) and urea acetaldehyde (UA) was successfully carried out. Some physical properties such as gel time, viscosity, density, melting point, moisture uptake, refractive index and formaldehyde emission level were evaluated. The IR analysis of spectra from the pure amino resins and their copolymer (UF + UA) showed chemical interaction between urea formaldehyde and urea acetaldehyde resins. Resin from the copolymer possess better optical property, flexibility and softness, density, water resistance and better control of formaldehyde emission than the convectional urea formaldehyde resin. Some physical properties of the copolymer showed that properties such as viscosity, refractive index, moisture uptake, melting point and density are in agreement with the literature values of other type of binders use in paints. Therefore, the urea formaldehyde and urea acetaldehyde copolymer resin present itself as a potential binder for emulsion paint formulation.

**Key words:** Amino resin, copolymerization, binder, paint.

#### INTRODUCTION

Global coating and ink markets are in innovative mood. The innovation are driven by environment, health, safety, quality improvement and cost reduction. This is especially due to regulations against volatile organic compounds (VOC) from coating surfaces. The present situation is now challenging companies to find solutions in waterbased systems (Osemeahon et al., 2009). With the advent of the regulations against air pollution, and for safety consideration, there have been continued interests in searching for alternative raw materials and new formulations to reduce the overall volatile organic compounds in surface coatings (Gan and Tan, 2001). Recently, much research has focused on replacing paints solvent-based with water based (Osemeahon et al., 2009). The advantages of water borne paint include being non polluting, easy to handle, quick drying, economic and environmentally friendly. However, although most household paints are waterbased, this is not true of industrial paints. Because of the

**Abbreviations: UF**, Urea formaldehyde; **UA**, urea acetaldehyde; **VOC**, volatile organic compounds; **MU**, urea monomer; **EU**, ethylol urea.

special requirements of the industrial coatings, satisfactory water-based polymers with the required properties have not yet been developed (Gooch, 1997). Therefore, a significant challenge in this drive to reduce VOC is the need for the water-borne technology to deliver the enamel type properties characteristic of solvent-borne coatings (Osemeahon et al., 2009).

The acceptance of urea formaldehyde resin 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 (Barminas and Osemeahon, 2006; Osemeahon and Barminas, 2007a). These disadvantages limit its uses. However, UF resins offer a wide range of conditions that make synthesis of these resins with important properties such as gel time, tack and spreading ability of the uncured resin possible. Also, formaldehyde emissions and the durability of the cured resin can be controlled and specifically tailored for the final end use of the resins (Osemeahon and Barminas, 2006). Park et al. (2002) reported that the ultimate performance of a fully cured amino resin largely depends on its synthesizing parameters, including the ingredient mole ratio, catalyst, temperature, viscosity, reactivity and so on. These parameters are frequently adjusted

empirically to tailor the resin properties to specific production requirements such as the resin reactivity, formaldehyde emissions, water resistance, etc.

In our earlier experiments (Barmina and Osemeahon, 2007; Osemeahon and Barminas, 2007a, b), we reported that the synthesis of a new class of UF resin which has low formaldehyde emission, and moisture uptake while retaining the traditional disagreeable hardness and brittleness associated with UF. Also amino resins from different aldehydic groups namely formaldehyde, acetaldehyde, proparaldehyde and butaldehyde were synthesized. It was revealed that while the resin from formaldehyde aldehydic group was too brittle those of other aldehydic groups were on the other hand too soft and exist as semi solid in their respective cure state. Thus we are faced with amino resins of two extremes. Those two groups of amino resins from different aldehydic groups on their own do not meet the requirements of the coating industry as a binder for emulsion paint formulation (Osemeahon and Barminas, 2007a). To address the above problem, a copolymer composite of UF and UP was synthesized with the aim of obtaining a compromise product which may neither be too soft nor too hard (Osemeahon and Barminas, 2007b). Although, the UF + UP composite showed a great promise as a binder for emulsion paint formulation, its major drawback is that it takes a very long time (132 h) to cure. This limits its acceptance as a binder for the coating industry. However, the gel time of amino resin is a function of the aldehydic group involved (Osemeahon and Barminas, 2007a). The longer the alkyl length of the aldehydic group, the longer the rate of cure of the resin. It is therefore our felt thought that since alkyl group in acetaldehyde aldehydic group is shorter than that of the proparaldehydic aldehydic group, copolymerization of UF with UA will produce a copolymer composite with corresponding faster rate of cure than that of UF + UP composite. This experiment therefore aims at producing a copolymer composite of UF and UA as a way of developing a paint binder for emulsion paint formulation from amino resin.

#### **MATERIALS AND METHODS**

Urea, formaldehyde, acetaldehyde, sodium dihydrogen phosphate, sulphuric acid, sodium hydroxide pellets and sucrose were reagent grade products from the British Drug House (BDH). All materials were used as received.

#### Resin synthesis

Trimethylol urea monomer (MU) was prepared by the method described by Barminas and Osemeahon (2006). One mole (6.0 g) of urea was reacted with three moles (24.3 ml) of 37% (w/v) formaldehyde using 0.2 g of sodium dihydrogen phosphate as catalyst. The pH of the solution was adjusted to 6 by using 0.5 M H2SO4 and 1.0 M NaOH solutions. The solution was then heated in a thermostatically controlled water bath at 70 °C. The reaction was allowed to proceed for 2 h after which the sample was removed and

kept at room temperature ( $30 \, ^{\circ}$ C). Ethylol urea monomer (EU) was synthesized by reacting one mole (6.0 g) of urea with one mole (7.4 ml) of 97% (w/v) acetaldehyde at pH 8 (Osemeahon and Barmina, 2007a) to produce monoethylol urea. Other procedures followed same as described above.

#### Preparation of UF/UA composite films

Copolymer composite of UF and UA was obtained by preparing serial concentrations (0 to 80%) of UA in UF resin at 30 °C. The mixture was stirred and left for 24 h at room temperature (30 °C) before the commencement of the various tests. To obtain the films of UF + UA composites, the solutions were poured into different glass petri dish for casting. These were then allowed to cure and set for seven days at room temperature (30 °C) (Osemeahon and Barminas, 2007a, b). The physical properties of these films were then investigated.

#### **Determination of viscosity**

The viscosity of the polymer blend (UF/UA) was obtained from a previous method (Barminas and Osemeahon, 2006). In brief, a 100 ml Phywe made graduated glass macro-syringe was utilized for the measurement. The apparatus was standardized with 20% (W/V) sucrose solution whose viscosity is 2.0 m Pa.s at 30 °C. The viscosity of the resin was evaluated in relation to that of the standard sucrose solution at 30 °C.

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

The above properties were determined according to standard methods (AOAC, 2000). The density of the different resins was determined by taking the weight of a known volume of resin inside a density bottle using metler (Model, AT400) weighing balance. Five readings were made for each sample and average value calculated. The turbidity of the resin samples was determined by using Hanna microprocessor turbidity meter (Model, H193703) (Barminas and Osemeahon, 2006). The melting points of the different film samples were determined by using Galenkamp melting apparatus (Model, MFB600-010F). The refractive indices of the resin samples were determined with Abbe refractometer (Barminas and Osemeahon, 2006).

#### Determination of moisture uptake

The moisture uptakes of the different resin films were determined gravimetrically (Osemeahon and Barminas, 2007a). Known weight of the sample was introduced into a desiccator 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 each sample was then recorded as the moisture intake by resin. Triplicate determinations were made for each sample and the average value recorded.

#### Determination of formaldehyde emission

Formaldehyde emission test was performed by using the standard 2 h desiccator test as earlier reported (Osemeahon and Barminas, 2007a). The mold used was made from aluminium foil with a dimension of 69.6 x 126.5 mm and thickness of 12.0 mm. The emitted formaldehyde was absorbed in 25.0 ml water and analyzed

Figure 1. Copolymerization reaction of methylol urea and ethylol urea.

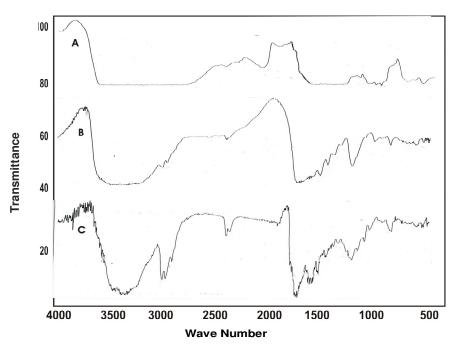


Figure 2. IR Spectra of (A) UF, (B) UF + UA and (C) UA.

by a refractometric technique using Abbe refractometer. Triplicate samples were used and average value taken.

#### Tensile test

Tensile properties (tensile strength and elongation at break) were measured as described by Osemeahon et al. 2007), using Instron Testing Machine (Model, 1026). Resin films of dimension 50 mm long, 10 mm wide and 0.15 mm thick were brought to rapture at a clamp rate of 20 mm/min and a full load of 20 kg. A number of five runs were done for each sample and the average elongation evaluated and expressed as the percentage increase in length. The copolymerization reaction could be summarized as shown in Figure 1.

#### **RESULTS AND DISCUSSION**

# IR spectra

The overlay spectra of UF, UA and UF + UA are shown in Figure 2. In the spectra of UF, the broad band stretching from 3600 cm<sup>-1</sup> through 3100 cm<sup>-1</sup> is due to O-H of methylol urea, 2913 cm<sup>-1</sup> is due to N-H, 1740 cm<sup>-1</sup> is due to C=O of urea, 1446 cm<sup>-1</sup> is due to -CH<sub>2</sub> of methylene bridge and 1086 cm<sup>-1</sup> due to C-O-C of ether linkage (Conner, 1996; Osemeahon and Barminas, 2007b). All the above basic structural peaks of UF also appeared

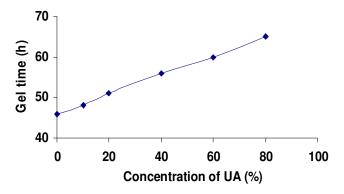


Figure 3. Effect of UA concentration on the gel time of UF.

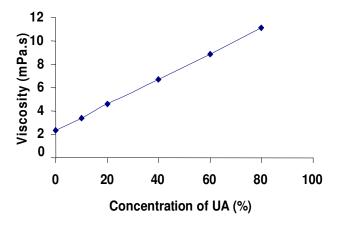
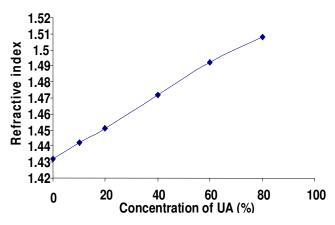


Figure 4. Effect of UA concentration on the viscosity of UF.



**Figure 5.** Effect of UA concentration on the refractive index of UF.

in the spectra of UA and UF + UA resins. This verifies the fact that UF, UA and UF + UA have the same basic structure and hence all belong to amino resin (Osemeahon and Barminas, 2007b). However, comparing the peak intensities of UF with those of UF + UA, the UF + UA peaks display higher intensities than

those of pure UF. On the other hand, comparing the peak intensities of UA with those of UF + UA, the UA spectral peaks display higher intensities than those of UF + UA. Thus, UF + UA spectra seem to occupy an intermediary position between the spectra of UF and UA. This may suggest chemical interaction between UF and UA to form UF+ UA.

#### Gel time and viscosity

Rheological properties such as the viscosity and the dynamic modulus can be directly correlated to the evolving physical and mechanical properties during resin cure (Hu et al., 2001). In the coating industry, an understanding of the viscosity of the paint binder is very important because it controls factors such as flow rate, leveling, sagging, thermal properties, adhesion and dry rate of paint film (Osemeahon et al., 2007). Thus an understanding of the viscosity is not only important but crucial.

Figures 3 and 4 show the effect of UA on the gel time and viscosity of UF, respectively. From Figure 3, the gel time of UF increased with increase in UA concentration. This result is due to decrease in reactivity which is a function of the increase in the size of the alkyl group attached to the carbonyl carbon resulting from the copolymerization of methylol urea with ethylol urea (Teware, 2000).

The greater the size of the alkyl group attached to the carbonyl carbon, the less the reactivity of the carbonyl group because the large groups around carbonyl group may hinder the attacking reagent due to steric hinderance (Teware, 2000). From Figure 4, it is observed that the viscosity of UF increases with increase in UA concentration. This development is attributable to increase in molecular weight as UF copolymerizes with the relatively large molecular weight UA (Osemeahon et al., 2007).

#### Refractive index

Paints are known to have varying degrees of opacity or transparency depending on the amount of light transmitted through or reflected from the surface of the paint surface. The gloss of a paint film is a function of refractive index of the surface and particle size (Barminas and Osemeahon, 2006). The effect of UA concentration on the refractive index of UF is shown in Figure 5. It is observed that the refractive index of UF increased with increase in UA concentration. This observation can be explained in terms of differences in molecular weight and molecular features of the copolymer (Tezza and Krochta, 2001). As the concentration of UA in the copolymer increases, the molecular weight of UF + UA also increases and hence the differences in the interaction with light.

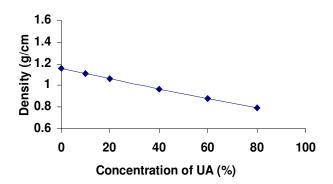
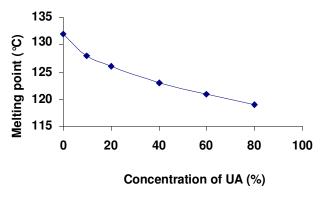


Figure 6. Effect of UA concentration on the density of UF.



**Figure 7.** Effect of UA concentration on the melting point of UF.

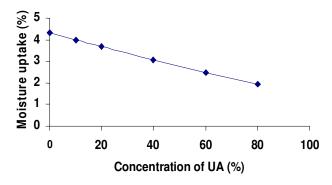


Figure 8. Effect of UA concentration on the moisture uptake of UF.

# Density

In the coating industry, the density of the paint binder has a profound influence on factors such as pigment dispersion, brushaility of paint, flow, leveling and sagging (Osemeahon and Barminas, 2006). Figure 6 shows the effect of UA concentration on the density of UF. The density of UF decreases with increase in UA concentration.

This result is attributable to the packing nature of resin molecules (Chain and Yi, 2001). Density depends on free volume and packing efficiency of molecular chains. The reduction in density with increase in molecular weight indicates inefficient molecular packing (Osemeahon and Barminas, 2007b).

# **Melting point**

The melting point of a polymer has a direct bearing to its thermal property, molecular weight, degree of cross linking and the level of rigidity of the polymer. One of the disadvantages of UF resins is that it is too hard and brittle to be used as a painter binder (Osemeahon et al., 2007). Figure 7 shows the effect of UA on the melting point of UF resin. The melting point decreases with increase in UA concentration. This trend can be explained in terms of the inherent nature of AU which is semi solid in its cured state (Osemeahon and Barminas, 2007b).

#### Moisture uptake

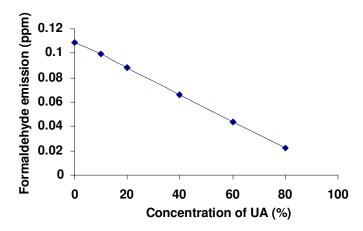
The interaction of structural network of polymer resins with water is both of fundamental and technical interest. One of the major drawbacks of UF resin is poor water resistance (Conner, 1996). Many resins and other polymer matrices absorb moisture by instantaneous surface absorption and diffusion (Barminas Osemeahon, 2006). In the paint making industry, the moisture uptake of the binder is very crucial because it is responsible for blistering and broominess of paint film (Osemeahon and Barminas, 2007a). Figure 8 shows the effect of UA concentration on the moisture uptake of UF The moisture of UF increases with UA concentration. The possible reason for this development may be due to increase in the molecular size holes in the polymer structure with increase in UA concentration (Hu et al., 2001). Water transport in polymer network is related to the availability of molecular size holes in the polymer structure and the polymer water affinity (Hu et al., 2001).

## Formaldehyde emission

The emission of the hazardous formaldehyde during cure of urea formaldehyde is one of the major setbacks of UF resin (Kim, 2001). In the development of paint binder from urea formaldehyde resin, serious effort must be made to reduce formaldehyde level to acceptable one (Osemeahon et al., 2007).

Figure 9 shows the effect of UA on the formaldehyde emission. It can be seen that the formaldehyde emission decreases sharply with increase in the UA concentration.

This result can be explained on the basis of gradual reduction in UF concentration with gradual increase in UA



**Figure 9.** Effect of UA Concentration on the formaldehyde emission of UF.

Table 1. Effect of UA on the tensile properties (elongation at break and tensile strength) of UF.

| Concentration of UA (%) | Tensile strength (kg/mm²) | Elongation at break (%) |  |  |
|-------------------------|---------------------------|-------------------------|--|--|
| 0                       | $0.229 \pm 0.021$         | $115.00 \pm 0.22$       |  |  |
| 10                      | $0.215 \pm 0.020$         | $123.24 \pm 0.03$       |  |  |
| 20                      | $0.202 \pm 0.020$         | $128.21 \pm 1.08$       |  |  |
| 40                      | $0.188 \pm 0.004$         | $137.50 \pm 0.01$       |  |  |
| 60                      | $0.159 \pm 0.010$         | $148.31 \pm 0.41$       |  |  |
| 80                      | $0.134 \pm 0.000$         | $152.00 \pm 0.03$       |  |  |

**Table 2.** Comparison of some physical properties of UF + UA copolymer with convectional urea formaldehyde resin.

| Dranautic                    | R                   | esin             | Reference                     |  |  |
|------------------------------|---------------------|------------------|-------------------------------|--|--|
| Property                     | UF + UA             | Convenctional UF |                               |  |  |
| Gel time (h) at 30 ℃         | $63.17 \pm 0.34$    | 36.0             | Barminas and Osemeahon, 2006. |  |  |
| Viscosity (mPa.s)            | $14.02 \pm 0.23$    | 1.2              | Barminas and Osemeahon, 2006. |  |  |
| Refractive index             | $1.5528 \pm 0.0100$ | 1.3465           | Barminas and Osemeahon, 2007. |  |  |
| Moisture uptake (%)          | $4.34 \pm 0.11$     | 18.0             | Ajayi et al., 2005.           |  |  |
| Melting point (°C)           | $137.00 \pm 0.82$   | 200.0            | Ajayi et al., 2005.           |  |  |
| Density (g/cm <sup>3</sup> ) | $0.82 \pm 0.00$     | 1.1764           | Ajayi et al., 2005.           |  |  |
| Formaldehyde (ppm)           | $0.020 \pm 0.000$   | 0.70             | Kim, 2001.                    |  |  |

concentration in the copolymer. The formaldehyde emission level is being reduced drastically by limiting the problem at source (Pizzi et al., 2002).

#### Tensile test

Elongation at break determines to what extent a material stretches before breaking and hence the ductility or flexibility of the material. The effect of UA on the tensile strength and elongation at break are shown in Table 1. It was observed that the tensile strength decreases while the elongation at break increases with increase in UA concentration. This result may be attributed to increase in alkyl length (Mohammed et al., 2001). It may also be as a result of differences in crystallinity or crystalline orientation of resin molecules (Xie et al., 2001; Chain and Yi, 2001). Table 2 compares some physical properties of UF + UA copolymer with conventional UF. It was observed that the moisture uptake dropped from 18 to

Table 3. Comparism of some physical properties of UF + UA resin with films from other paint binders.

| Type of resin   | Physical property |                      |                  |                                 |                      |                     |                         |                           |
|---|-------------------|----------------------|------------------|---------------------------------|----------------------|---------------------|-------------------------|---------------------------|
|   | Gel time (h)      | Viscosity<br>(mPa.s) | Refractive index | Density<br>(g/cm <sup>3</sup> ) | Melting<br>point (℃) | Moisture uptake (%) | Elongation at break (%) | Literature                |
| UF + UA   | 63.13.00 ± 1.00   | 14.50 ± 0.01         | 1.5528 ± 0.0082  | $0.82 \pm 0.02$                 | 137.00 ± 1.00        | 4.34 ± 0.10         | 157.08 ± 0.00           | This study                |
| Epoxy-based Divinyl Ester                                   | 0.8               | 38.0                 | ND               | 1.04                            | 197                  | ND                  | ND                      | Gawdzik Matynia, 2001     |
| Styrene Modified Epoxy                                      | 72                | ND                   | ND               | ND                              | 200                  | ND                  | ND                      | Yoon and Mc Grath 2001    |
| Maleic anhydrle grafted polypropylene bend with epoxy resin | ND                | ND                   | ND               | ND                              | 200                  | ND                  | 11.6                    | Shieh et al., 2001        |
| Epoxyfumerate Resins  | 0.96              | 45.0                 | ND               | 1.07                            | 110                  | ND                  | ND                      | Gawdzik et al., 2002      |
| Whey Protein Isolate biopolymer                             | ND                | ND                   | 1.4838           | ND                              | ND                   | ND                  | ND                      | Trezza and Krochta, 2001  |
| Styrene-Butadiene latex                                     | ND                | ND                   | ND               | ND                              | ND                   | ND                  | 220                     | Xie et al., 2001          |
| Aromatic amines-modified polyethylene                       | ND                | ND                   | ND               | 0.96                            | 133                  | ND                  | ND                      | Starostina et al., 2001   |
| Silicone-Modified styrene-butyl acrylate Copolymer latex    | ND                | ND                   | ND               | ND                              | ND                   | 8.6                 | ND                      | Wu et al., 2000           |
| Aqueus-based polyurethane                                   | ND                | ND                   | ND               | ND                              | ND                   | 25                  | 713                     | Huang et al., 2000        |
| Rubber Seed oil modified alkyd resins                       | 24                | 3.11                 | ND               | 0.95                            | ND                   | ND                  | ND                      | Aigbodion and Pilla, 2001 |
| Glycidyl methacrylate and piperazin                         | ND                | ND                   | ND               | ND                              | ND                   | ND                  | 1700                    | Hong et al., 2002         |
| Epoxy Resins  | ND                | ND                   | ND               | ND                              | 101                  | 0.04                | ND                      | Hu et al., 2001           |
| Aqueous Polyurethane  | ND                | ND                   | ND               | ND                              | ND                   | 17.2                | ND                      | Lee and Kim, 2001         |

ND: Not determined.

4.34%, melting point from 200 to 137°C, density from 1.1764 to 0.82, and formaldehyde emission from 0.70 to 0.021 ppm. These indicate positive developments in terms of water resistant, hardness, film adhesion and toxicity, respectively. The great increase in refractive index means

better gloss for the coating industry. Table 3 shows some physical properties of UF + UA resin and those from some literature paint binders. These properties include that of gel time agree with the literature values. Suggesting that, UF + UA may be acceptable as a binder for emulsion

paint formulation in the coating industry.

### Conclusion

Copolymerization of urea formaldehyde and urea

acetaldehyde amino resins was successfully carried out in this study. Some physical properties such as gel time. viscosity, density, melting point, moisture uptake, refractive index and formaldehyde emission level were evaluated. The IR analysis of spectra from the pure amino resin and their copolymer showed chemical interaction between urea formaldehyde and urea acetaldehyde resins. Resins from the copolymer have better optical property, density, water resistance, flexibility and softness and better control of formaldehyde emission than the convectional urea formaldehyde resins. Some physical properties of the copolymer showed that properties such as viscosity, refractive index, moisture uptake, melting point, gel time and density are in agreement with the literature value of other type of binders use in paint formulation. Therefore, urea formaldehyde and urea acetaldehyde copolymer composite present itself as a potential binder for emulsion paint formulation.

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