

*Full Length Research Paper*

# Study of some physical properties of urea formaldehyde and urea proparaldehyde copolymer composite for emulsion paint formulation

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**Copolymerization of urea formaldehyde (UF) and urea proparaldehyde (UP) was successfully carried out. Physical properties which include gel time, viscosity, density, melting point, moisture uptake, refractive index and formaldehyde emission level were evaluated. The infrared analysis of the pure amino resins and their copolymer (UF + UP) showed chemical interaction between UF and UP resins. Resin from the copolymer showed better results on optical property, flexibility, softness, density, water resistance and lower formaldehyde emission than the conventional UF resin. Physical properties of the copolymer such as viscosity, refractive index, moisture uptake, melting point and density are in agreement with values obtained in literature for other types of binders used in paint formulation. However, the gel time is relatively too high and hence the need for inclusion of a curing agent to increase the rate of cure of the copolymer resin. UF and UP copolymer resin present itself as a potential binder for emulsion paint formulation.**

**Key words:** Paint binder, amino resins, urea formaldehyde, urea proparaldehyde, copolymerization.

## INTRODUCTION

Thermosetting polymers such as urea formaldehyde (UF) and melamine formaldehyde are the most widely used amino resins (Conner, 1996; Pizzi et al., 2001; Updegraff, 1990). However, the acceptance of amino resins as a universal material in many engineering areas such as the coating industry is impeded by some of its inherent qualities such as brittleness, poor water resistance and formaldehyde emission (Lowel, 1990; Conner, 1996). Conner (1996) reported that the procedure for the synthesis of UF resins offer a wide range of conditions which make the synthesis of the uncured resin having important properties such as gel time, tack and spreadability possible. Also, formaldehyde emission and the durability of the cured resin can be controlled and specifically tailored for the final end use of the resin.

In our earlier experiments (Barminas and Osemeahon, 2007), the synthesis of urea formaldehyde resin through a new synthetic route and some physical properties of

urea proparaldehyde resin (UP) were reported. It was stated therein that amino resin from aldehydic group of formaldehyde is too hard and brittle while that from proparaldehyde on the other hand is too soft and exists as semi solid in its cured state. These two amino resins from the two different aldehydic groups on their own, do not meet the requirements of the coating industry. However, polymer systems consisting of components with different properties are usually known to possess some advantages over the separate polymers (Osemeahon and Barminas, 2007). Therefore, it is our felt thought that if UF and UP monomers are copolymerized, a compromise product may be obtained as a copolymer of UF and UP resins. This copolymer is expected to share properties of both monomers, and if this happens, an amino resin which is neither too soft nor too hard could be obtained and this may satisfy the needs of the coating industry. This experiment therefore aims at producing a copolymer composite of UF and UPS as a way of developing a paint binder for emulsion paint formulation from amino resins which may meet the requirements of the coating industry.

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## MATERIALS AND METHODS

### Materials

The chemicals used include urea, formaldehyde, proparaldehyde, sodium hydroxide pellets, sulphuric acid, sodium dihydrogen phosphate and sucrose. These were obtained from British Drug House.

### Resin syntheses

Trimethylol urea was prepared by the method described by Barminas and Osemeahon (2007) 6.0 g of urea was reacted with 24.3 ml of 37% (w/v) formaldehyde using 0.2 g of sodium dihydrogen phosphate as catalyst (Chen et al., 2001). The pH of the solution was adjusted to 6 by using 0.5 M H<sub>2</sub>SO<sub>4</sub> and 1.0 M NaOH solutions. The solution was then heated in a thermostated water bath at 70°C. The reaction was allowed to proceed for 2 h after which the sample was removed and kept at 30°C.

Propylol urea was synthesized by reacting 6.0 g of urea with 7.4 ml of 97% (w/v) proparaldehyde at pH of 8 to produce monopropylol urea (Barminas and Osemeahon, 2007). Other procedures followed same as described for the synthesis of methylol urea. Copolymerization of UF with UP was carried out by preparing serial concentrations (0 - 80%) of UP in UF at 30°C. The solutions were allowed to stand for 24 h before the commencement of the various tests.

### Film preparation

Films of the different resins obtained at various PU concentrations were cast on Petri dishes by using solution casting method (Mirmohseni and Hassanzadeh, 2000). The resins were then allowed to cure and set for seven days at 30°C. Some physical properties of these films were investigated.

### Determination of viscosity and gel time

The viscosity of the resin was evaluated by the method described by Barminas and Osemeahon (2007). Five different readings were taken for each sample and the average values were calculated. The gel point of the different resins obtained at the different UP concentrations was determined by monitoring the viscosity of resins with time until a constant viscosity profile was obtained (Barminas and Osemeahon, 2007).

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

The above properties were determined according to the procedures outlined by AOAC (2000). The densities of the different resins were determined by taking the weight of a known volume of resin in a density bottle using a Mettler AT400 weighing balance. Five readings were made for each sample and the average values were calculated. The turbidity of resin samples were determined by using Hanna microprocessor turbidity meter model H193703. The melting point of the different film samples were determined by using a Galenkamp melting point apparatus model MFB600-010F. The refractive index of resin samples were determined using Abbe refractometer.

### Determination of moisture uptake

The moisture uptake of the different resin films was determined gravimetrically according to the methods of Osemeahon and Barminas

(2007). Known weights of each sample were introduced into a desiccator containing a saturated solution of sodium chloride. The wet weight of each sample was then monitored until maximum weight was obtained. The difference between the wet weight and dry weight of each sample was then recorded as the moisture uptake by resin. Triplicate determinations were made for each sample and the average value was taken.

### Determination of formaldehyde emission

Formaldehyde emission test was performed by using the standard 2 h desiccator test as we earlier reported (Osemeahon and Barminas, 2007). The evaluation of the absorbed formaldehyde by the 25.0 ml of water was obtained from a standard calibration curve derived from refractometric technique using Abbe refractometer.

In brief, resin prepared from one step-process (OSP) was aged for 2 days. At the end of this period, the resin was poured into a mold made from aluminium foil with a dimension of 69.6 mm x 126.5 mm and thickness of 1.2 mm. The mold and its content were then placed inside a desiccator along with 25 ml of water, which absorbed the formaldehyde emitted. The set up was allowed to stand for 2 h at 25°C after which the 25 ml water was removed and analyzed for formaldehyde content. Triplicate samples were used for each determination and average values were taken.

### Tensile test

Tensile properties (tensile strength and elongation at break) were measured using Instr testing machine model 1026 as earlier reported by Osemeahon and Barminas (2007). Resin films of dimension 50 mm long, 10 mm wide and 0.15 mm thick were brought to rupture at a clamp rate of 20 mm/min and a full load of 20 kg. Five runs were taken for each sample and the average elongation taken and expressed as the percentage increase in length.

### Infrared (IR) spectroscopy

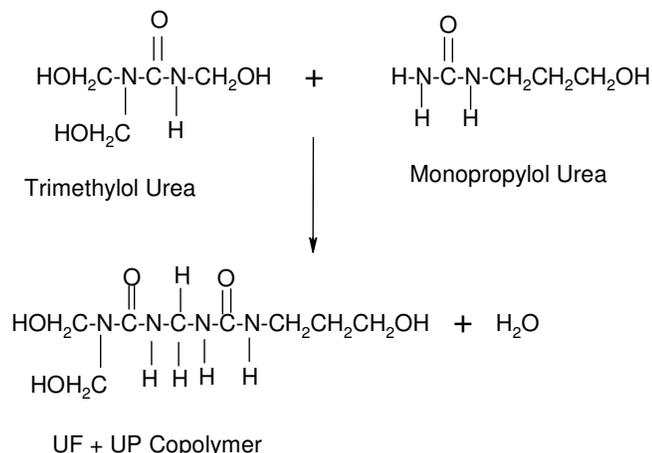
The IR spectrum of the dried resin films were run by direct scanning of the film using Buck Scientific 500 M Spectrophotometer between 600 - 4000 cm<sup>-1</sup>.

## RESULTS AND DISCUSSION

### IR analysis

The overlay spectra of UF, UP and UF + UP are shown in Figure 2. In the spectra of UF, the broad band stretching between 3600-3100 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; Gan and Tan, 2001; Ahmad et al., 2001; Park et al., 2001). All the above basic structural peaks of UF also appeared in the spectra of UP and UF + UP resins. This confirms that UF, UP and UF + UP have the same basic structural features and hence all belong to amino resins.

However, the UF + UP peaks display higher intensities than those of pure UF. On the other hand, it is observed that UP spectral peaks display higher intensities than those of UF + UP. Thus, UF + UP spectral peak intensi-



**Figure 1.** Copolymerization reaction of methylol urea and propylol urea.

ties seems to occupy an intermediate position between those of UF and UP. This suggests chemical interaction between UF and UP to form UF + UP (Figure 1).

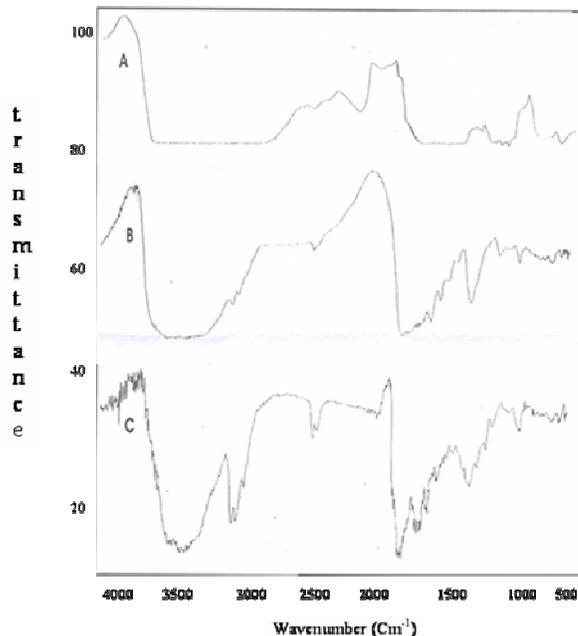
### Gel time and viscosity

The processing of thermosetting resins requires understanding of the rheology and the polymerization reaction kinetics during cure. The gel time is one of the most important kinetic characteristics of curing, because it describes the attainment of certain critical conversion responsible for the transition from liquid to solid state of the curing process (Vilas et al., 2000). The gel point is characterized by the appearance in the reactive system of a macromolecule with an infinitely large molecular weight. The kinetic scheme of the cured paint binder is of considerable importance both for the manufacturing processes, pot stability and rate of cure of the paint film (Achi, 2003).

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). Viscosity of a paint binder affects flow properties such as leveling and sagging (Lowel, 1990). Film forming property, adhesion property and drying rate of paint film are also functions of viscosity.

Figures 3 and 4 show the effect of UP on the gel time and viscosity of UF respectively. From Figure 3, it can be observed that the gel time of UF increases with increase in UP concentration. This is due to decrease in reactivity which results from the increase in the size of the alkyl group attached to the carbonyl carbon of the copolymer (Teware, 2000). The greater the size of the alkyl group attached to the carbonyl carbon, the less is the reactivity of the carbonyl group due to steric hindrance (Teware, 2000).

From Figure 4, it is observed that the viscosity of UF



**Figure 2.** IR spectra of (A) UF, (B) UF + UP and (C) UP.

increases with increase in UP concentration. This development is attributable to increase in molecular weight as UF copolymerizes with the relatively large molecular weight UP (Markovic et al., 2001).

### Refractive index

Gloss is an important quality factor of many coating products. The gloss of paint coatings with or without pigments is a function of refractive index of the surface, the angle of incidence of the beam of light, the nature of light and the nature of the material (Trezza and Krochta, 2001). The effect of UP concentration on the refractive index of UF is shown in Figure 5. It is observed that the refractive index of UF increases with increase in UP concentration. This observation can be explained in terms of differences in molecular weight and molecular features (Trezza and Krochta, 2001; Johnson and Wilkes, 2001).

### Density

In the coating industry, the density of a paint binder has a profound influence on factors such as pigment dispersion, brushability of paint, leveling and sagging (Lowel, 1990). Figure 6 shows the effect of UP concentration on the density of UF. It can be observed that the density of UF decreases with increase in UP concentration. This result is attributable to the packing nature of resin molecules (Sekaran et al., 2001; Chain and Yi, 2001). Density depends on free volume and packing efficiency of mole-

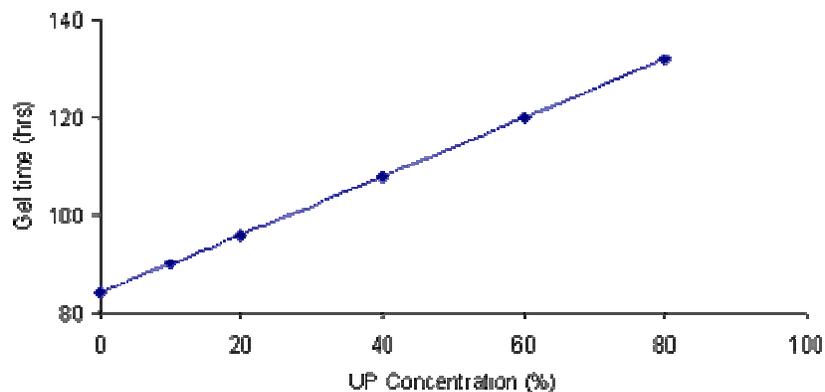


Figure 3. Effect of UP concentration on the gel time of UF

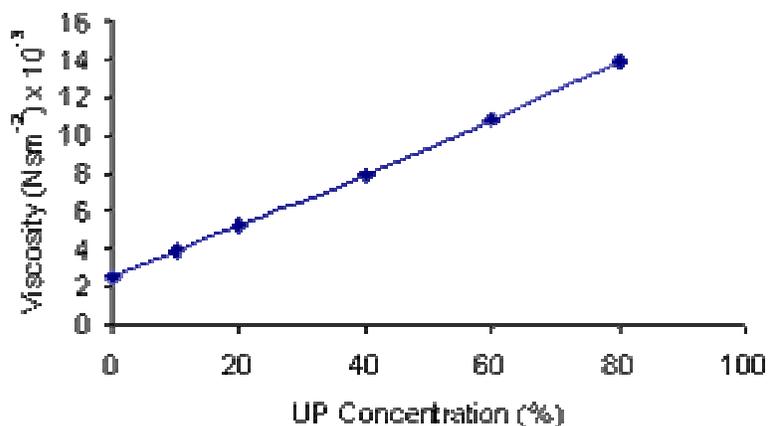


Figure 4. Effect of UP concentration on the viscosity of UF.

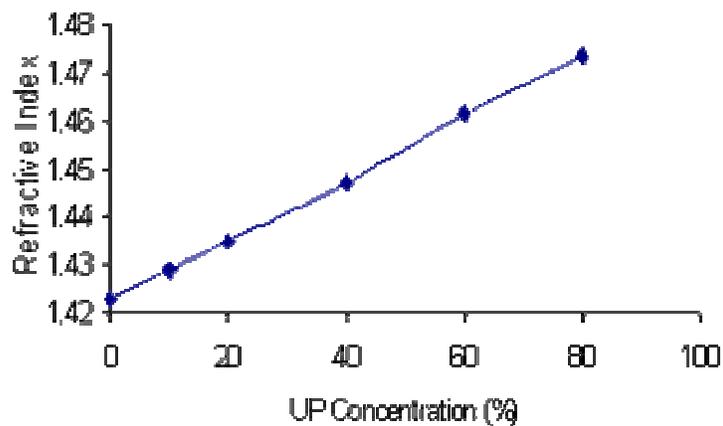


Figure 5. Effect of UP concentration on the refractive index of UF.

cular chains. The reduction in density with increase in molecular weight indicates inefficient molecular packing (Osemeahon and Barminas, 2007).

**Melting point**

The melting point of a polymer has a direct bearing to its

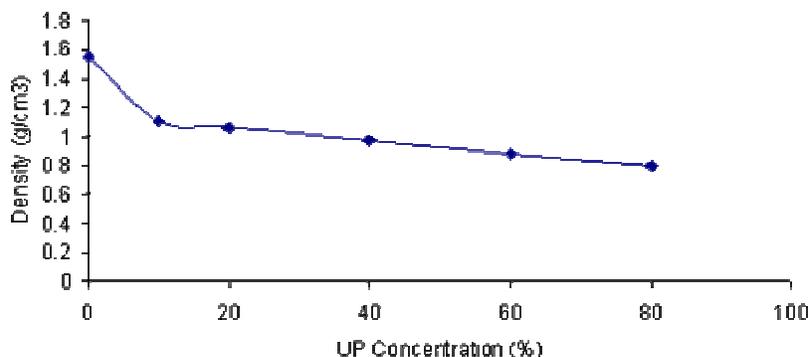


Figure 6. Effect of UP concentration on the density of UF.

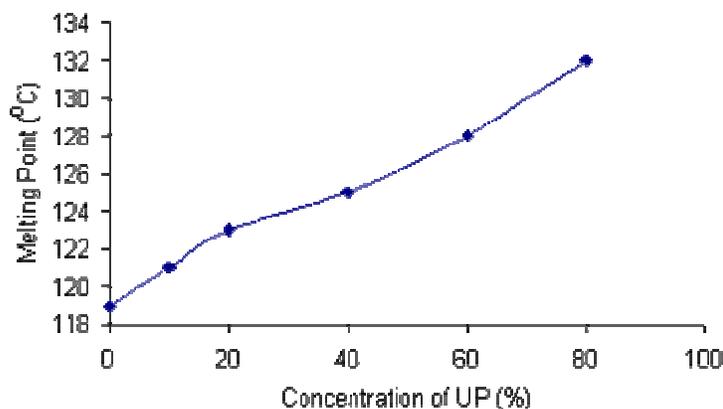


Figure 7. Effect of UP concentration on the melting point of UF.

thermal property (Bindu et al., 2001). It is related to its molecular weight, degree of cross-linking and level of rigidity of the polymer (Park et al., 2001). In the coating industry, the melting point of a binder is related to its thermal resistance as well as to its brittleness. Figure 7 shows the effect of UP concentration on the melting point of UF resin. It can be observed that the melting point of UF resin increases with increase in UP concentration. This trend can be explained in terms of increase in molecular weight (Markovic et al., 2001; Park et al., 2001). This is further supported by the viscosity result obtained in this study.

### Moisture uptake

The interaction of structural network of polymer resins with water is both of fundamental and technical interest (Barminas and Osemeahon, 2007). Water uptake affects vital properties of the polymer material such as the physical, mechanical, thermal and structural properties (Hu et al., 2001; Nogueira et al., 2001). One of the major drawbacks of UF resin is poor water resistance (Conner,

1996). 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.

Figure 8 shows the effect of UP concentration on the moisture uptake of UF resin. It is observed that the moisture uptake of UF increases with increase in UP concentration. The possible reason for this development may be due to increase in the molecular size holes in the polymer structure with increase in UP concentration (Nogueira et al., 2001; 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 (Nogueira et al., 2001).

### Formaldehyde emission

One of the major drawbacks of urea formaldehyde resin is the emission of the hazardous formaldehyde during cure (Kim, 2001; El-Naggar et al., 2001; Pizzi et al., 2001). Therefore in the development of paint binder from urea formaldehyde resin, serious effort must be made to reduce formaldehyde emission to acceptable level. Fig-

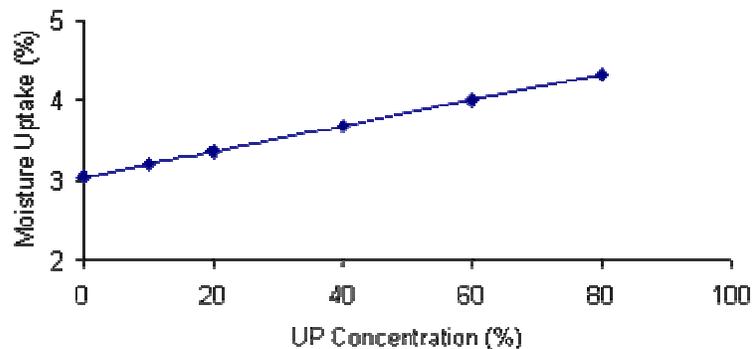


Figure 8. Effect of UP concentration on the moisture uptake of UF

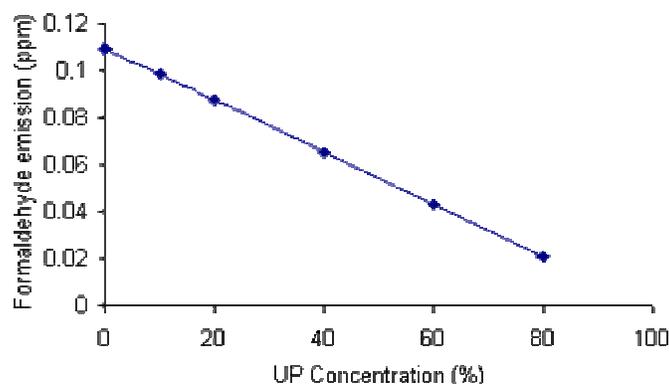


Figure 9. Effect of UP concentration on the formaldehyde emission of UF

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

Conc. of UP (%)	Tensile strength (kg/mm <sup>2</sup> )	Elongation at break (%)
0	0.214	110.03
10	0.200	115.34
20	0.187	122.11
40	0.161	132.20
60	0.134	143.01
80	0.107	162.31

ure 9 shows the effect of UP concentration on the formaldehyde emission of UF. It can be seen that the formaldehyde emission decreases sharply with increase in UP concentration. This result can be explained on the basis of gradual reduction in UF concentration with gradual increase in UP concentration in the copolymer. The formaldehyde emission level is being reduced by drastically limiting the problem at source (Pizzi et al., 2001).

### Tensile test

Elongation at break determines to what extent a material stretches before breaking and hence the ductility or flexi-

bility of the material. One of the shortcomings of UF resin is that it is too hard and brittle and hence poor resistance to crack propagation (Lowel, 1990). In the coating Industry, a paint binder must be able to withstand stresses emanating from variation in environmental factors. Therefore, in developing paint binder from amino resins, tensile property such as elongation at break must be taken into consideration.

The effect of UP on the tensile strength and elongation at break are shown in Table 1. It is observed that the tensile strength decreases while the elongation at break increases with increase in UP concentration. This may be attributed to increase in alkyl length (Teware, 2000). It

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

Property	UF + UP	UF (References)
Refractive index	1.4733	1.3465 (Barminas and Osemeahon, 2007)
Moisture uptake (%)	4.32	18.0 (Ajayi et al., 2005)
Melting point (°C)	132	200 (Ajayi et al., 2005)
Density (g/cm <sup>3</sup> )	0.79	1.1764 (Ajayi et al., 2005)
Formaldehyde emission (ppm)	0.021	0.70 (Kim, 2001)

**Table 3.** Comparison of some physical properties of UF + UP resin with films of other paint binder.

Types of resin	Physical properties							Reference
	Gel time (h)	Viscosity (Nsm <sup>-2</sup> )	Refractive index	Density (g/cm <sup>3</sup> )	Melting point (°C)	Moisture uptake (%)	Elongation at break (%)	
UF + UP copolymer	132.00	13.90	1.4733	0.79	132.00	4.32	162.2	Present study
Epoxy-based divinyl ester	0.80	38.00	ND	1.04	197.00	ND	ND	Gawdzik and Matynia (2001)
Styrene modified epoxy	72.00	ND	ND	ND	200	ND	ND	Yoon and McGrath (2001)
Maleic anhydride grafted polypropylene bend with epoxy resin	ND	ND	ND	ND	200	ND	11.6	Shieh et al. (2001)
Epoxyfumarate resins	0.96	45.00	ND	1.07	110.00	ND	ND	Gawdzik et al. (2000)
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	220.0	Xie et al. (2001)
Aromatic amine-modified polyethylene	ND	ND	ND	0.96	133.00	ND	ND	Starostina et al. (2001)
Silicone-modified styrene-butyl acrylate copolymer latex	ND	ND	ND	ND	ND	8.60	ND	Wu et al (2000)
Aqueous-based polyurethane	ND	ND	ND	ND	ND	25.00	713.0	Huang et al. (2000)
Rubber seed oil modified alkyd resins	24.00	3.11	ND	0.95	ND	ND	ND	Aigbodion and Pilla (2001)
Glycidyl methacrylate and piperazin	ND	ND	ND	ND	ND	ND	1700.0	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	17.20	ND	Lee and Kim (2001)

ND: Not Determined.

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 + UP copolymer with conventional UF resin. The copolymer showed a decrease in the moisture uptake, melting point, density and formaldehyde emission. These indicate positive developments in terms of water resistance, 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 + UP resin and those from some other paint binders. It could be observed that the physical properties of the copolymer are in agreement with those of other paint binders. The low cure rate of UF + UP may be overcome by using a curing agent or by increasing the viscosity of UF before copolymerization.

## Conclusion

Copolymerization of urea formaldehyde and urea proparaldehyde 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 resins and their copolymer showed chemical interaction between urea formaldehyde and urea proparaldehyde resins. Resin from the copolymer possesses better optical property, density, water resistance, flexibility, softness and lower formaldehyde emission than the conventional 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 types of binders used in paints. However, the gel time is relatively too long and hence there may be a need for the inclusion of an appropriate curing agent to increase rate of cure of the copolymer resin. In conclusion therefore, the urea formaldehyde and urea proparaldehyde copolymer composite present itself as a potential binder for paint formulation.

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