



## EFFECTS OF SOME PROCESS VARIABLES ON GEL TIME OF KERATIN MODIFIED UREA-FORMALDEHYDE RESIN

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### Abstract

The effect of some processes variables on the gel time of keratin modified urea formaldehyde (KMUF) resin was studied. The gel time of the keratin modified urea formaldehyde resin increased with increase in amount of catalyst to some extent, where the further increase in the amount of catalyst showed no substantial effect in the gel time. The result also indicated that the effect of the catalyst content on the gel time is more efficient in the resins of lower solid contents than those of higher solid contents. While the pH value of the resin system decreased as the gel time increases. It was observed that gel time of keratin modified urea formaldehyde resin decreased with increase in solid content. This study has revealed the effect of these variables on KMUF resin, therefore this results suggest a guide for it application in composite fabrication.

**Keywords:** Catalyst, gel-time, pH, resin, solid content, adhesive

### INTRODUCTION

Urea-formaldehyde adhesives are thermosetting, when heated; they become thermoplastic and set to an infusible and insoluble mass. These are condensation products of urea and formaldehyde (Sheikh *et al.*, 2008). Presently approximately one million metric tons of urea-formaldehyde resin are produced annually. More than 70% of this urea-formaldehyde resin is used by the forest product industry for a variety of purposes (White, 1995). The resin is used in the production of an adhesive for bonding particle board (61% of the urea-formaldehyde used by industry), medium-density fiber board (27%), hardwood plywood (5%), and a laminating adhesive for bonding (7%) for example, overlays to panels and interior flush doors. Urea-formaldehyde resins are the most prominent examples of the class of thermosetting resins usually referred to as amino resins (Williams, 1991; Updegraff, 1990). Urea-formaldehyde resin comprises of about 80% of the amino resins produced worldwide. The pH value, solid content and catalysts of urea formaldehyde resin plays a very important role in providing a combined pH environment at the interphase between wood and urea formaldehyde resins (Xing *et al.*, 2007). To obtain the optimum bond strength, the press time and temperature must be adjusted for the pH environment. If this correction is not precise, the glue line will be uncured or over cured, and this will result in poor bond strength. Some researchers have shown that wood extractives (Albritton and Short, 1979; Slay *et al.*, 1980), wood pH values and buffer capabilities (Guo *et al.*, 1998; Xing *et al.*, 2004), strongly affect the gel time of urea formaldehyde resins. Xing *et al.* (2005) reported that an optimal range of catalyst contents exists for the curing of urea formaldehyde resins.

Evidence exists for the idea that a higher catalyst content accelerates not only the rate of cure urea formaldehyde resins, but also their rate of hydrolysis after resin curing (Myers, 1985). The optimal catalyst content should generate an acceptable cure rate with the addition of less catalyst. Ammonium chloride ( $\text{NH}_4\text{Cl}$ ) is a common and effective harder used for accelerating urea formaldehyde resin curing. Little is available concerning the effect of the solid content of urea formaldehyde resin on the gel time. Therefore, the aim of this study is to investigate the effects of pH value, solid content and catalyst on the gel time of keratin modified urea formaldehyde resin.

### MATERIALS AND METHODS

The keratin modified urea formaldehyde resin used in this study was KMUF-09, as prepared by Dim (2009). The solid content of the resin was 70% as determined by a solid pan technique (ASTM, 1993). The pH value of the resin was 8.0. The catalyst was a 10%  $\text{NH}_4\text{Cl}$  solution and was of analytical grade. Six samples with different pH values were obtained by mixing of small drops of  $\text{NH}_4\text{Cl}$  into the keratin modified urea formaldehyde resin. Six keratin modified urea formaldehyde samples with different solid contents of 45, 50, 55, 60, 65 and 70% were prepared by dilution with distilled water. And 20 ml of catalyst was added to the six samples use to test for the effect of solid content on gel time of the resin. Eight samples with different catalyst contents ranging from 0- 40 (ml) were prepared by the addition of an  $\text{NH}_4\text{Cl}$  solution. The gel time of all the samples was measured using a Techne Gel Timer (Techne Ltd Duxford, Cambridge England) through the addition of 5 grams of the prepared samples to test tube and heated in a 100 °C glycerin solution. The pH values were measured with

a HACH 19200 pH meter (Hach Company Box 389, Loveland, and Colo.USA). Firstly, the pH meter was calibrated with standardized buffer solutions at pHs of 4 and 7. After which 200g of resin sample was measured into a 300 ml beaker, and the initial pH values of the resin and the solution, was adjusted by the gradual addition of ten percent  $\text{NH}_4\text{Cl}$ . Each of the samples was stirred for 5 min at  $20^\circ\text{C}$  and their pH values recorded. All measurement was duplicated for each sample.

### RESULTS AND DISCUSSION

From Table 1, it can be seen that the gel time of urea formaldehyde resin with respect to amount of catalyst decreased rapidly from 5700 to 210s as the amount of catalyst increased to 25 ml. This shows a little variation from finding reported by Xing *et al.* (2005). And this may possibly be as a result of environmental conditions and it can also be attributed to the presence keratin in the resin used. It can also be observed that further increase in the catalyst content gave no

substantial changes in the gel time of the resin. This is because the optimal catalyst amount for the reaction has been attained (Pinto and Pobleto, 1992). This is also in accordance with Xing *et al.* (2005) which stated that an optimal range of amount catalyst exists for the curing of urea formaldehyde resins. However the amounts of catalyst directly affect urea formaldehyde resin curing and the performances of final products (Pobleto and Pinto, 1993). As shown in Table 2, it can be observed that the gel time of the resin was strongly affected by its solid content. It can be seen that the gel time of the resin decreased with increasing resin solid content. This is based on the fact that the concentration of the reactants decreased with decreasing solid content. Therefore, the cure rate decreased, and this resulted in a longer gel time. Table 2 also indicates that the effect of the catalyst content on the gel time is more efficient in the resins of lower solid contents than those of higher solid contents (Xing *et al.*, 2007).

Table 1: Effect of Catalyst on Gel Time

Gel time (secs)	Amount of catalyst (ml)
5700	0
1420	10
830	15
420	20
210	25
210	30
160	35
160	40

Table 2: Effect of Solid Content on Gel Time

Gel time (secs)	Solid content
2050	45
1400	50
650	55
360	60
250	65
240	70

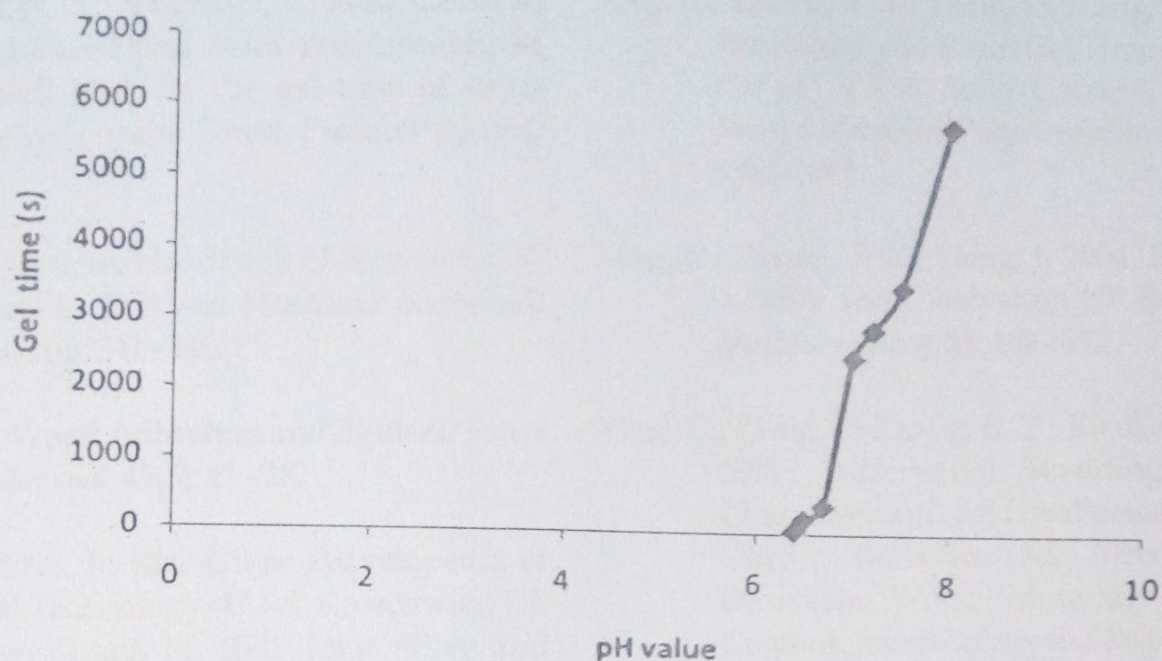


Figure 1: Effect of pH values on Gel time

Figure 1 shows the effect of pH value on the gel time of the resins. Since the pH of the original resin sample is 8.0 and the resin is an acid-catalyzed curing resin. Therefore to determine the critical pH at which the resin-curing rate begins to increase drastically. The gel times were measured by decreasing resins pH values. The samples were adjusted with  $\text{NH}_4\text{Cl}$  acid, the gel time decreased from around 5700 to 2500s at pH of 7.2. As shown in Figure 1, the pH value of the resin had to be reduced to around pH of 6.8 to achieve a gel time of 300s. The relationship of pH value caused by  $\text{NH}_4\text{Cl}$  and the gel time generated a linear regression model as shown in Figure 1. This proves that  $\text{NH}_4\text{Cl}$  has a strong catalyzing effect on reactants in the resins systems (Xing *et al.*, 2007).

### CONCLUSION

This study shows that the main effect of  $\text{NH}_4\text{Cl}$  on KMUF resin curing is catalyzing the reactants in the system. The gel time of the KMUF resin increased with increase in amount of catalyst to some extent, where the further increase in the amount of catalyst showed no substantial effect in the gel time. The gel time of the KMUF resin decreased with increasing resin solid content. The gel time of KMUF resins decreased as the pH value of the system was decreasing. Therefore, the gel time of KMUF resins decreases with increase in the amount of catalyst and solid contents and decrease in pH value of the system. Thus this study was important since the effects of pH value, solid content, and catalyst on the gel time of KMUF resins is essential to the establishment of effective processing parameters for applying these polymers in wood-based-composite manufacturing.

### REFERENCES

- Albritton, R. O.; Short, P. H. 1979. The effect of extractives from pressure-refined hardwood fiber on the gel time of urea-formaldehyde (UF) resin. *Forest Products Journal*, 29: 40-41.
- ASTM standard D4426-93; American Society of Testing and Materials: Philadelphia, PA, 1993.
- Dim, P. E. 2009. Production and Application of Keratin-Modified Urea-Formaldehyde Petrochemical. M.Eng. Thesis, Federal University of Technology, Minna, Nigeria.
- Guo, A. L.; Zhang, H. S.; Feng, L. Q.; Gao, X. X.; Zhang, G. L. 1998. pH Value and Buffering Capacity of 6 Shrub Species and Relevant Effect on Curing Time of UF Resin. *China Wood Industry*, 12: 18.
- Myers, G. E. 1985. In: wood Adhesives in 1985: Status and Needs; Christiansen, A. W. *et al.*, Eds.; Forest Prod. Res. Soc.: Madison, WI.
- Pinto, S. A.; Poblete, W. H. 1992. In Formaldehyde Release from Wood Products *Cienc Int Forestal*, 6: 259.
- Poblete, W. H.; Pinto, S. A. 1993 *Forest Products from Latin America. Bosque*, 14: 55-58.
- Sheikh, A. M.; Khokhar, L.; Anwar, M. S.; Khan, M. H.; Akhtar, H. 2008. Studies on Synthesis and Properties of Urea Formaldehyde Adhesives. *J. Chem. Soc. Pak.*, 30: 20-22.

Smy, J. R.; Short, P. H.; Wright, D. C. 1980. Catalytic effects of extractives from pressure-refined [hardwood] fiber on the gel time of urea-formaldehyde resin. *Forest Products Journal*, 30:22.

Uydegraff, I. H. 1990. In: *Handbook of Adhesives*, 3<sup>rd</sup> ed; Skeist, L, Ed; Van Nostrand Reinhold: New York, pp. 341 -346.

White, J. T. 1995. Wood Adhesives and Binders. *Forest Products Journal*, 45(3): 21 - 28.

Williams, L. L. 1991. In *Kirk-Othmer Encyclopedia of Chemical Technology*, 4<sup>th</sup> Ed; Kroschwitz, J. I. and Howe-Grant, M. (Ed); John Wiley and Sons: New York, NY; 1991, 2: 604 - 637.

Xing, C.; Zhang, S. Y.; Deng, J.; Wang, S. 2007. Urea-Formaldehyde-Resin Gel Time as Affected By the pH Value, Solid Content, and Catalyst. *Journal of Applied Polymer Science*, 103: 1566-1569.

Xing, C.; Zhang, S. Y.; Deng, J. 2004. Effect of Wood Acidity and Catalyst on UF Resin Gel Time. *Holzforschung*, 58: 408 - 412.

Xing, C.; Deng, J.; Zhang, S. Y.; Riedl, B.; Cloutier, A. 2005. Differential Scanning Calorimetry Characterization of UreaFormaldehyde Resin Curing Behavior As Affected By Less Desirable Wood Material And Catalyst Content. *Journal of Applied Polymer Science*, 98: 2027-2032.