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Effect of Catalyst Concentration on the Rheological Properties of Melamine Formaldehyde Resinated Cotton Fabric

[1]A.I. Ezeribe, [1]C.J. Chukwu, [2]N.A. Obasi, [2]G. Otuchristian and [3]U.A. Ibiam
[1]Department of Science Laboratory Technology, Federal Polytechnic Bauchi, Nigeria
[2]Department of Science Laboratory Technology, Federal Polytechnic Unwana, Afikpo, Nigeria
[3]Department of Biochemistry, Ebonyi State University, Abakaliki, Ebonyi State, Nigeria

Corresponding Author: N.A. Obasi, Department of Science Laboratory Technology, Federal Polytechnic Unwana, Afikpo, Nigeria Tel: +2347033364146

ABSTRACT

Synthesis of mono through hexa substituted derivatives of Methylol Melamine Resins (MFR) were carried out at various catalyst (KH_2PO_4) concentrations of 0.5, 0.75, 1.0, 1.25 and 1.50 g. Rheological/mechanical properties of resin-cotton network were investigated to determine the effect of catalyst concentration and degree of substitution of resins on the flow property such as viscosity. Other parameters determined include percentage yield, breaking load and crease recovery properties of resinated cotton fabric. The result revealed that the yield increased with increase in catalyst concentration and attained the highest yield of 53.7% at 0.75 g and 51.3% at 1.0 g, respectively. The damaging effects of high acid concentrations were observed particularly at 1.25 and 1.50 g KH_2PO_4. Yield increased as the degree of substitution increased with hexa substitution having 48.8 and 53.7% at 0.5 and 0.75 g, respectively. Shear viscosity increased as catalyst concentration increased from 0.5 to 1.0 g with hexa and penta substitution giving the highest shear viscosity of 45 poises and 35 poises, respectively. The dry crease recovery angle increased with increased catalyst concentration up to 0.75 g and attained maximum value of 125° and 115° in the warp and weft directions, respectively at 150°C. The effect of catalyst on breaking load of sample at 150°C showed an increase from 0.5-1.0 g of catalyst concentration. The highest tear strength of 10.8 and 10.2 kgf at 0.75 g catalyst concentration was observed in the warp and weft directions, respectively. Acid damage at higher concentration was also implicated for the reduced tear strength.

Key words: Synthesis, catalyst, rheological property, resin, cotton fabric, shear viscosity, tear strength

INTRODUCTION

Resination or fiber-resin complex is one of the many types of fiber finishing. Some researchers (Choi, 1992; Bertoniere *et al*., 1974; Hollies and Getchell, 1967) proposed durable press finishing by the polymerization cross-linking (PC) with a wet fixation process. The polymerization treatment used with a wet fixation process could improve breaking strength and abrasion resistance at high level of durable press properties. This allows time for resin penetration and deposition inside the fibers (Siriwan and Marjorie, 2002). Fabrics made from cellulose or their blends have the tendency to wrinkle after washing and during drying (Andrews, 1995).

Resination is a chemical process used to improve such fabric wrinkle or durable press performance (Siriwan and Marjorie, 2002).

The alignment or orientation of the cellulose molecules in a regular pattern forms the crystalline region of the cotton fiber, while random arrangements of cellulose molecules form the amorphous region. The relative amounts of crystalline and amorphous cellulose influence the properties of cellulosic fiber (Smith and Block, 1982). The crystallinity of cotton fiber is between 85 and 95%, giving the cotton fiber moderate strength and abrasion resistance.

This paper investigated the effect of catalyst concentration and degree of substitution of resins on the flow, mechanical and crease recovery properties of resinated cotton fabric in order to improve its durable press performance.

MATERIALS AND METHODS

Polymerization reaction of melamine formaldehyde resin: Polymerization reaction was carried out in two stages according to Trotman (1975) method. Soluble resin was made by the reaction of 1 mole of melamine with 1, 2, 3, 4, 5 and 6 moles of formaldehyde to produce mono, di, tri, tetra, penta and hexamethylol melamine, respectively. Exactly 126.12 g of melamine was dissolved in 30 cm^3 of formaldehyde to produce mono, di, tri, through hexamethylol melamine, respectively in 0.5, 0.75, 1.0, 1.25 and 1.50 g of potassium dihydrogen phosphate (KH_2PO_4). Few drops of sodium carbonate solution were added to maintain the pH at about 7-8. The reaction was carried out at 40°C for 8-12 h. The resulting syrup was filtered and washed with ethanol to remove any unreacted formaldehyde. The monomethylol melamine resin was dried in the oven for 2 h at 40°C. The above procedure was repeated for the preparation of di, tri, tetra, penta and hexa-substituted methylol melamine.

Viscosity test: This was carried out according to method outlined in ASTM D 5053-95 (ASTM, 1995). Ten percent (10%) of the resin samples were prepared in five different beakers and stirred using a bar magnet on an electric hot plate at 40°C for 30 min. The cone of the viscometer was inserted into the beaker (plate) which sheared through the mixture, the viscosity was recorded. The test was repeated for all resin samples of mono-hexa methylol melamine in 0.5, 0.75, 1.0, 1.25 and 1.5 g of KH_2PO_4 as catalyst. The viscosity values obtained were plotted against their respective concentration of catalyst.

Resin application (pad-dry-cure method): The mercerized samples were impregnated with 30 mL each of 10%, 0.5, 0.75, 1.0, 1.25 and 1.5 g KH_2PO_4 catalyst. This catalyzed mono through hexa-substituted methylol melamine for 30 min was followed by occasional padding on a smooth surface to achieve even treatment and also to remove excess resins. The samples were dried in an oven at 100°C for 15 min and cured at 150°C for 45 min (Siriwan and Marjorie, 2002).

Crease recovery measurement: The crease recovery property of treated fabric was carried out in accordance with ASTM (1994). The resinated fabric specimen measuring 5.0×1.0 cm was creased by application of load of 2 kg in a Shirly Crease Recovery Tester for 5 min. The load was removed and the specimen was allowed to recover for 5 min. The Shirly Crease Recovery Tester was calibrated by adjusting the knob to face the 0° mark. The specimen was then transferred to the Crease Recovery Tester to measure the crease recovery angle in the warp and weft directions.

Breaking load measurement: The sample measuring 1.0×5.0 cm was mounted on the Instron 1026 Tensile Tester. The gauge length of Instron was adjusted to accommodate the sample. The instrument was operated at a speed of 010 mm min^{-1} and the load extension values of the instrument were recorded for various 10% concentrations of mono through hexa substituted methylol melamine resins (ASTM, 1995).

RESULTS AND DISCUSSION

Table 1 shows the yield percent of methylol melamine from mono through hexa substitution at various concentrations of catalyst with the highest yield of 53.7% at 0.75 g and 51.3% at 1.0 g of catalyst. The result of the effect of catalyst concentration on shear viscosity of methylol melamine resin is shown in Figure 1. These results revealed an increase in shear viscosity between 0.5 to 0.75 g catalyst concentration with hexamethylol and pentamethylol melamine resins having the highest shear viscosity of 45 and 35 poises, respectively. High catalyst concentration to monomer ratio affects negatively the properties of resin due to acid damage of the polymer chains (Choi, 1992; Shin *et al.*, 1989). The Fig. 1 also show increase in the degree of substitution. This is likely due to higher molecular mass of hexamethylol melamine resin compared to that of methylol urea resins. This result is consistent with that of Ajayi (2002).

Figure 2 and 3 depict the effect of catalyst concentration on resinated cotton fabric at 150°C. The crease recovery angle increased with increased catalyst concentration even up to 0.75 and 1.0 g in the hexa and penta substituted methylol melamine resin, respectively. This is due to the lowering of activation energy of reactant molecules thereby allowing more molecules of the resin to participate in the reaction. Creasing is due to weak Van der Waals forces which characterized the amorphous regions of the cotton fabric.

Table 1: Percentage yield of methylol melamine derivatives at constant pH of 8.0

	Catalyst conc. (g)				
Degree of substitution	0.50	0.75	1.00	1.25	1.50
1	44.0	46.6	35.4	25.6	16.0
2	25.2	27.8	25.2	25.1	21.8
3	38.6	42.0	35.3	15.3	14.7
4	38.6	42.0	35.3	15.3	14.7
5	42.5	41.0	40.0	38.3	42.9
6	48.8	53.7	48.4	43.8	47.7

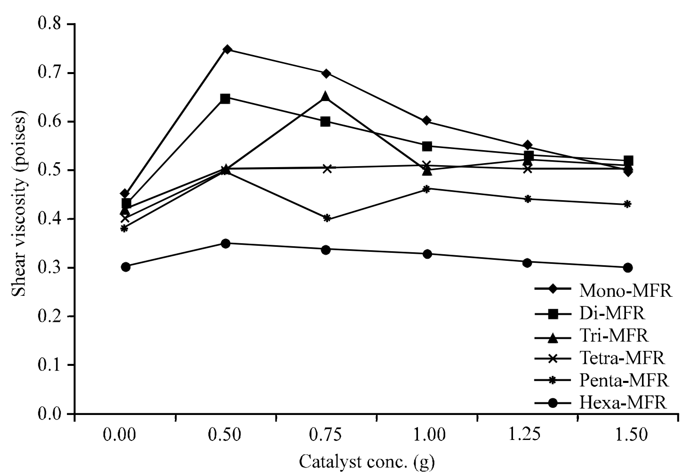

Fig. 1: Effect of catalyst concentrations on Shear viscosity of 10% methylol melamine resins (poises)

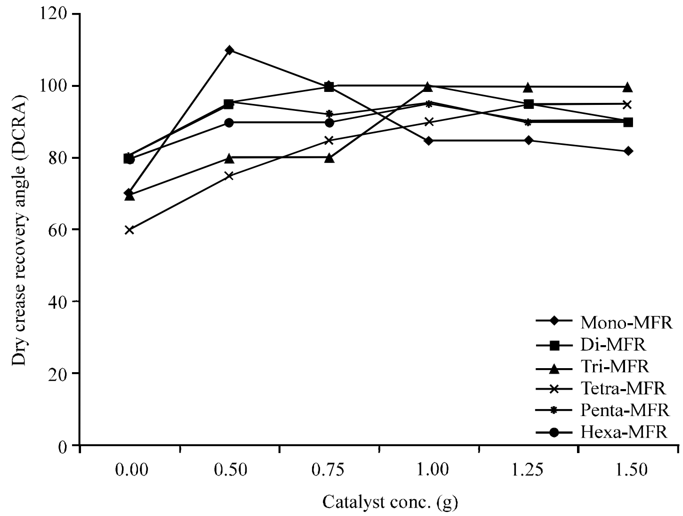

Fig. 2: Effect of concentration of catalyst and degree of substitution on dry crease recovery angle (DCRA) of 10% methylol melamine resinated cotton fabric cured at 15°C (warp)

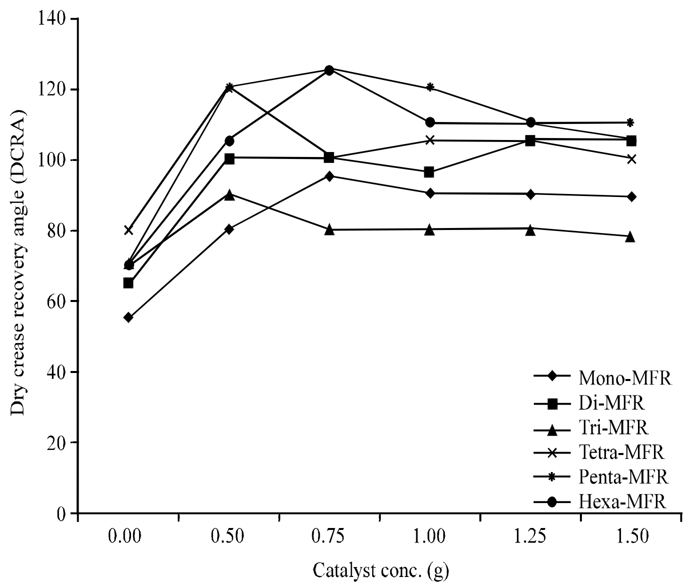

Fig. 3: Effect of concentration of catalyst and degree of substitution on dry crease recovery angle (DCRA) of 10% methylol melamine resinated cotton fabric cured at 150°C (weft)

Also, catalyst concentration of 0.75-1.0 g facilitated an increase in Dry Crease Recovery Angle (DCRA) and attained a maximum value of 125° and 115° in both warp and weft directions, respectively. Increase in DCRA is also not infinite since there are finite numbers of glucose residue per unit of cellulose molecule (Moji, 2000). This reason may account for the gradual decrease in DCRA as the concentration of the catalyst increased. This result is consistent with the result of Ajayi *et al.* (2001).

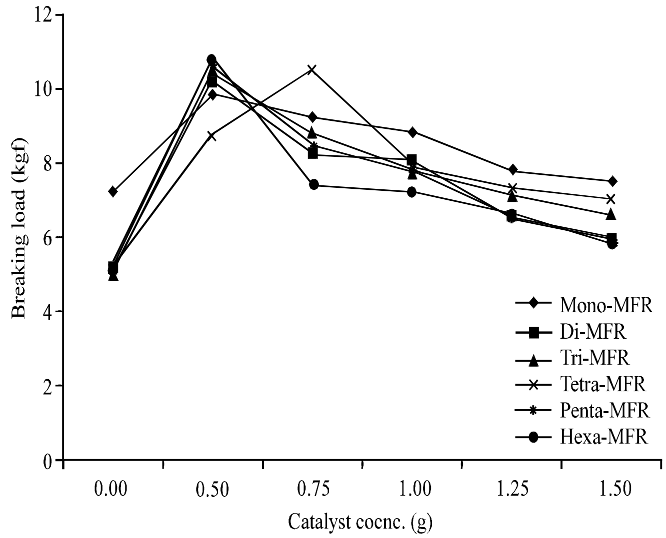

Fig. 4: Effect of concentration of catalyst and degree of substitution on breaking load (kgf) of 1% methylol melamine resinated cotton fabric cured at 150°C (warp)

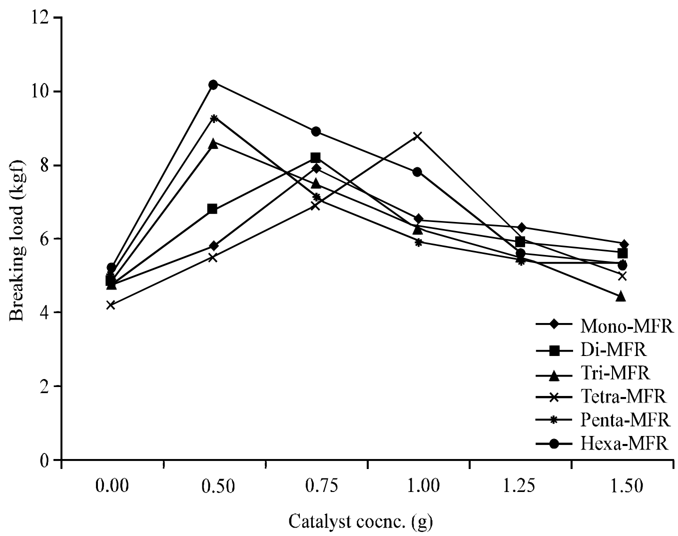

Fig. 5: Effect of concentration of catalyst and degree of substitution on breaking load (kgf) of 10% methylol melamine resinated cotton fabric cured at 150°C (weft)

The effect of catalyst concentration and the degree of substitution on the breaking load of resinated cotton fabric in warp and weft directions are as shown in Fig. 4 and 5. The graphs revealed that breaking load increased from 0.5-1.0 g of catalyst concentration. Hexa substituted methylol melamine have the highest breaking load of 10.8 and 10.2 kgf at 0.75 g catalyst concentration in warp and weft directions respectively. Amorphous regions of cellulose are filled up with resins and strengthened by chemical cross-link between cellulose and resin molecules (Norma *et al.*, 1979). Cross-linking provides anchoring points for the chains in the network (Ajayi *et al.*, 2001). The higher breaking load at curing temperature of 150°C can be ascribed to longer chain length of melamine compared to urea formaldehyde resins. This improves flexibility in the cotton fibers with improved mechanical properties (Andrews, 1995).

CONCLUSION

The effect of varying acid catalyst concentration was significantly visible throughout the test experiment. The yield increased as much as 54% at lower concentration compared to 41% at higher concentration level of catalyst. Acid damage of resin-cotton network was also visible in all the mechanical properties of resin tested. For better performance of the resin in the investigated area of application, resin may be prepared at 0.5-1.0 g of the catalyst. This fabric can be used for shirting and other light clothing with low stress requirements.

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