

Effects of Temperature and Catalyst Mass on the Mechanical Properties of Urea Formaldehyde Resinated Cotton Fabrics

Ezeribe A.I.¹, K.A.Bello², Adamu H. M.³ boryo D. E. A.¹ Omar, Pan⁴

¹ Department of SLT., Federal Polytechnic, Bauchi.

² Department of Textile Technology A.B.U Zaria

³ Department of Chemistry, A.T.B.U. Bauchi.

Department of General Studies, Federal Polytechnic, Bauchi

Abstract: - The dipole forces which facilitate reaction are temperature dependent. Allowing fabrics to over cure degrades the cotton due to excessive heat and distort the attractive force between polar groups of resin – cotton network. Effects of temperature and catalyst mass on the breaking load (BL) and dry crease recovery angle (DRCA) of mono urea formaldehyde resin (MUFR) through Tetra urea formaldehyde resins (TUFR) resinated fabrics were carried out on chemically treated cotton fabrics, and was cured at 40°C to 170°C. The result obtained reveal the combined effects of temperature and catalyst mass on the monomethylol urea resinated fabric samples cured at 120°C(MUFR 120) gave a BL of 7.84kgf at 120°C and 7.40kgf at 100°C along the warp direction, between 0.75g to 1.0g of catalyst. Dimethylol urea resinated cotton fabrics samples cured at 150°C (DURF150) gave a BL of 10.01 kgf, this is followed by DSUFR 120 sample cured at 120°C which gave a BL of 8.21kgf. In fig. 27, trimethylol urea resinated samples cured at 80°C (TRUFR 80°C) gave the highest BL of 11.32kgf between 0.75g to 1.0g of catalyst. Further increase in curing temperature and mass of catalyst was counterproductive and reduced the tensile strength of samples by 8.0% ; 48% and 38% in DUFR, TRUFR and TUFR samples respectively in the warp axis. The trend replicated itself in weft axis, which gave the maximum BL of 10.01kgf at 150°C but failed around 9.26kgf at 170°C representing a loss in tensile strength of 8% in the weft axis. The DCRA increased with temperature and catalyst mass but reduced as catalyst mass exceed 1.0g. Resins prepared with 0.75g to 1.00g of catalyst and it resinated fabrics cured between 150°C to 155°C would improve the DCRA and BL of cotton fabrics.

Keywords: - Breaking Load (BL), Dry Crease Recovery Angle(DCRA) Stensile strength, Combined Effect of Catalyst Mass,

I. INTRODUCTION

Vale and Taylor(1964) reported that monomeric or dimeric methylol urea react at elevated temperature in the presence of alkaline catalyst to yield only short chain polymers. The polymers are solubilized by attached methylol group. The methylol group is protonated under acid conditions giving rise to a reactive intermediate which is resonance stabilized carbonium imonium (Othman, 1978). The present of catalyst in reactions, increase number of collisions between active centers and monomers. These lead to production of resins, with higher molecular masses and viscosity. Catalyst provides an additional path or mechanism for the reaction. This is further explained in terms of pole vaulting analogy (Robert et al; 1975). When catalyst is added, there is a new path, hence a different activation energy barrier, this new reaction path corresponds to a new reaction mechanism that permits the reaction to occur via different activated complex. Thus more particles can get over the new lower energy barrier and reaction rate is increased (Robert et al; 1975).. The nature of the product is greatly dependent on the reactants (Fred, 1984). Additional liberation of formaldehyde by thermal or hydrolytic cleavage are reduced by the type and amount of catalyst used (Peterson, 1997). Polymer sample exhibit glass-like behavior at low temperature and high frequency with young modulus of $10^9 - 10^{10}$ N/m² and will break or flow at strains greater than 5%. At high temperature, the same polymer may be rubber – like with modulus of 10^6 to 10^7 N/M² withstanding large extensions of about (100%) with permanent deformation (Jame *et al*; 1975). Still at higher temperature, permanent deformation occurs under load and behaves like a highly viscous liquid.

The reaction of urea by Holzer(1884) and the treatment of regenerated cellulose to improve the strength (Eschewer, 1979),gave birth to successful preparation and application of resins to textile fabrics. When resinated samples are cross-linked, the dimensional stability is improved, the creep propensity is lowered, resilience increases and resin structure becomes less prone to heat distortion due to increase in rigidity of molecular chain, strong intermolecular forces and subsequently increase in glass transition temperature (T_g). There is also great restriction in chain slippage due to the dimensional network structure (Billimeyer, 1984). Fabric finishing consists of mechanical, chemical and thermal processes, applied to fabrics to produce or impact specific performance level. Resination or fiber-resin complex is one of such fabric finish. Apparel made from cellulose,

regenerated cellulose/ blend of these with synthetic fibers have the tendency to wrinkle badly after washing and tumble drying or wearing.

Resination improves fabric wrinkle resistance durable press performance (Siviwan, 2002).

The relative amounts of crystalline and amorphous portion of cellulose influence the properties of cellulose fiber. The crystallinity of cotton is between 85 and 95 percent, giving cotton fiber moderate strength and abrasion resistance. Temperature and time of cure are important variables control as to obtain good cross-linking and low formaldehyde release in treated cotton fabric. Fabric treated with urea formaldehyde resins are easy to cure and should be cured as soon as the fabric is dried, to avoid reactants self cross-link. Curing temperature of 130°C to 150°C is strongly recommended for methylol urea resins (Cook *et al*; 1982b).

II. MATERIALS AND METHODS

Preparation of urea formaldehyde resins

Polymerization reaction of urea formaldehyde resins was carried out in two stages (Trotman, 1975), the low molecular mass, fusible and soluble resins was made by the reaction of 1 mole of urea with 1,2,3,4 moles of formaldehyde to produce mono-, di-, tri-, tetra-, methylol urea respectively. 1mole urea was dissolved in 30cm³ of formaldehyde to produce mono-, di-, tri and tetra methylol urea respectively in 0.5g, 0.75g, 1.0g, 1.25g and 1.50g of potassium dihydrogen phosphate (KH₂PO₄). 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 hours. The resulting syrup was filtered and washed with ethanol to remove any unreacted formaldehyde. The mono methylol urea resin was dried in the oven for 2 hours at 40°C.

Resin Application

The mercerized samples (5x1cm) were impregnated with 30mls each of 0.5g, 0.75g, 1.0g, 1.25g and 1.5g KH₂PO₄ catalyzed mono through tetra methylol urea for 30 min. followed by occasional padding on a smooth surface to achieve even treatment and also to remove excess resins. The curing reaction, leading to the cross-linking of substrate molecule with resin was carried out by exhaustion method at 40°C, 60°C, 80°C, 100°C, 120°C and 170°C in the oven for 45 minutes.

III. MEASUREMENT OF MECHANICAL PROPERTIES

Crease recovery measurement

The crease recovery property of treated fabric was carried out in accordance with British Standard specification (1974), the resinated fabric specimen measuring 1.0 cm by 5.0 cm was creased by application of a load of 2 kg in a Shirley Crease Recovery Tester for 5 min. The load was removed and the specimen was allowed to recover for 5 min. The Shirley Crease Recovery Tester was calibrated by adjusting the knob to face the 0° mark, and 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 cm x 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 10 mm/min and the load extension values of the instrument were recorded for various mass of mono through Tetra, catalysed methylol urea resins respectively. The breaking load values of the instrument were recorded at various concentration of mono through tetra catalyzed methylol urea resins, cured at 40°C through 170°C respectively.

Effect of temperature on resinated cotton fabric

Resinated samples were dried and cured in Gallen Kamp electric oven at 40°C, 60°C, 80°C, 100°C, 120°C, 150°C and 170°C for 30 min. These samples of dimension 5.0 cm x 1.0 cm were separately mounted on the Instron 1026 Tensile Tester at constant gauge length of 5 cm and speed of 010 mm/min. the breaking load values of the instrument were recorded at various mass of mono through tetra catalyzed methylol urea resins respectively.

IV. RESULTS

Figs 25, 26, 27, and 28 show the effects of temperature and catalyst mass on breaking load of mono methylol, tri methylol, tetra methylol resinated cotton fabrics (warp) respectively at pH of 8.0. Also, figures 29, 30, 31 and 32 reveal same on the weft direction. Figs 14 and 17 depict the effect of catalyst mass on the dry crease recovery angle (DCRA) of dimethylol urea resinated cotton fabrics in warp and weft axis.

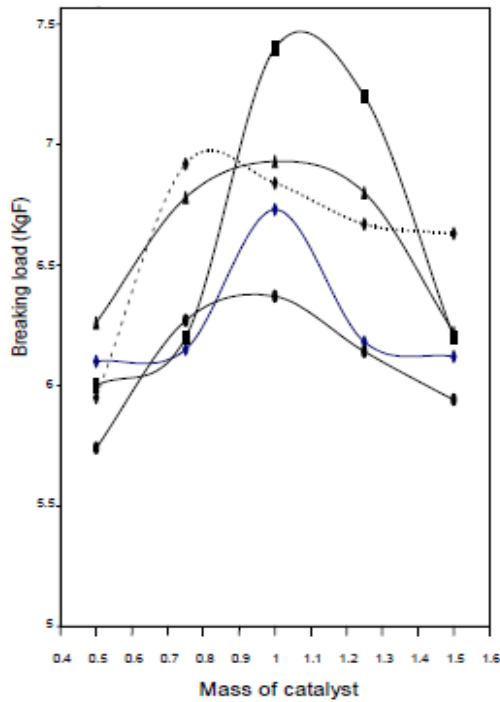


Fig. 25: Effect of temperature on breaking load of mono substituted resinated cotton fabric at pH of 8.0

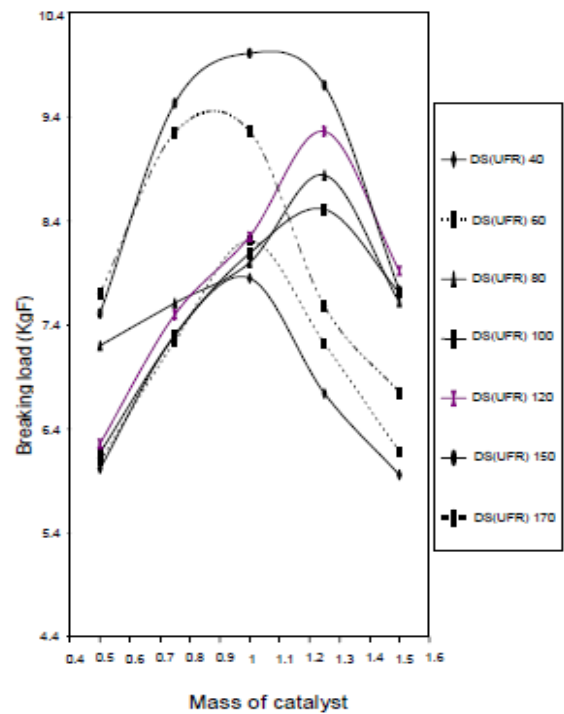


Fig. 26: Effect of temperature on breaking load of disubstituted resinated cotton fabric at pH of 8 (WARP)

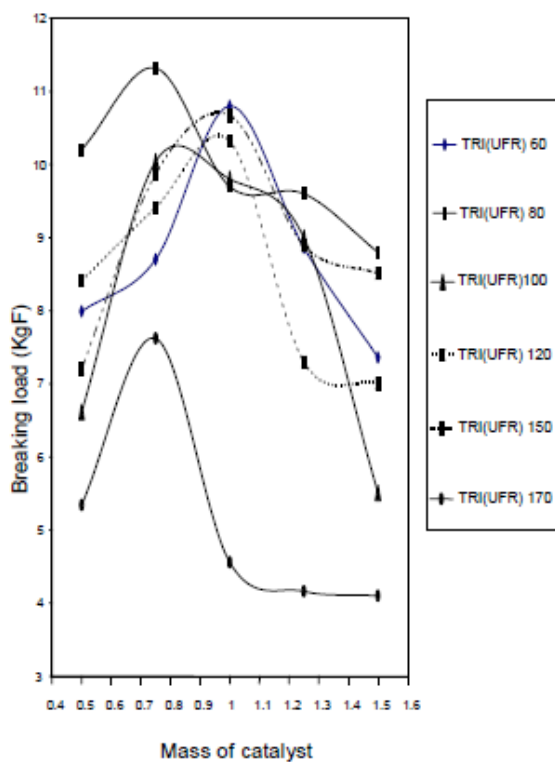


Fig. 27: Effect of temperature on breaking load of trisubstituted resinated cotton fabric at pH of 8 (WARP)

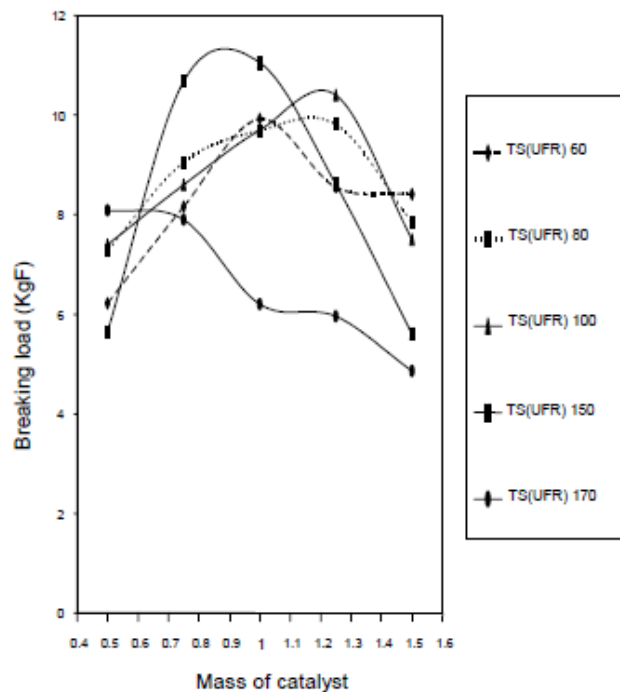


Fig. 28: Effect of temperature on breaking load of tetrasubstituted resinated cotton fabric at pH of 8 (WARP)

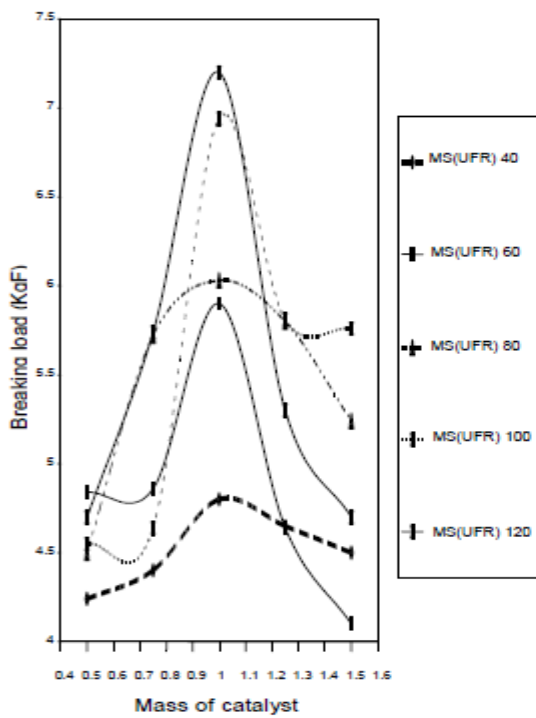


Fig. 29: Effect of temperature on breaking load of mono substituted resinated cotton fabric at pH Of 8 (WEFT)

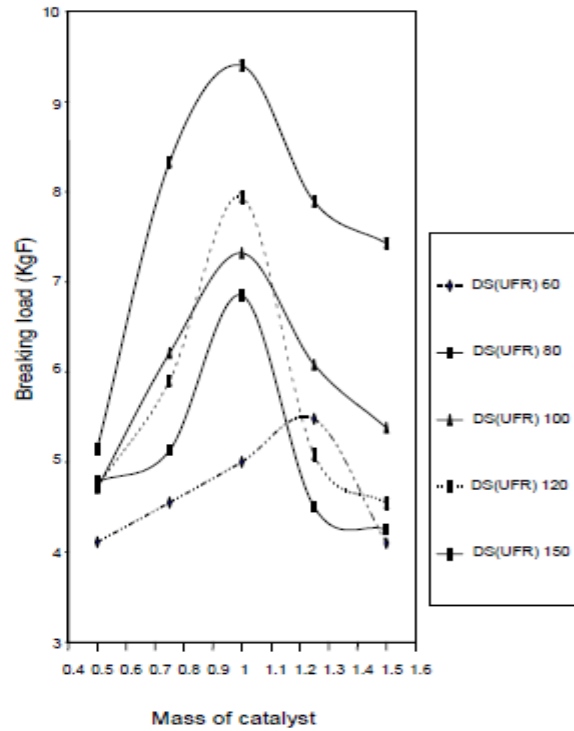


Fig. 30: Effect of temperature on breaking load of disubstituted resinated cotton fabric at pH Of 8 WEFT)

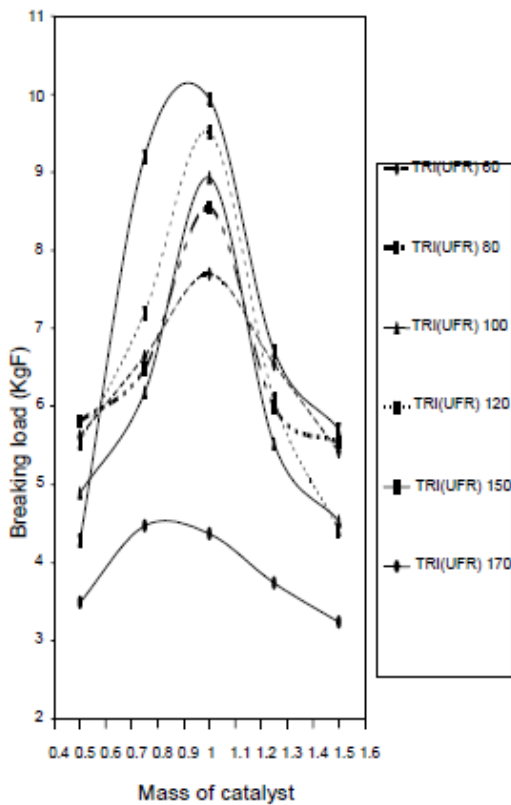


Fig. 31: Effect of temperature on breaking load of trisubstituted resinated cotton fabric at pH Of 8.0 (WEFT)

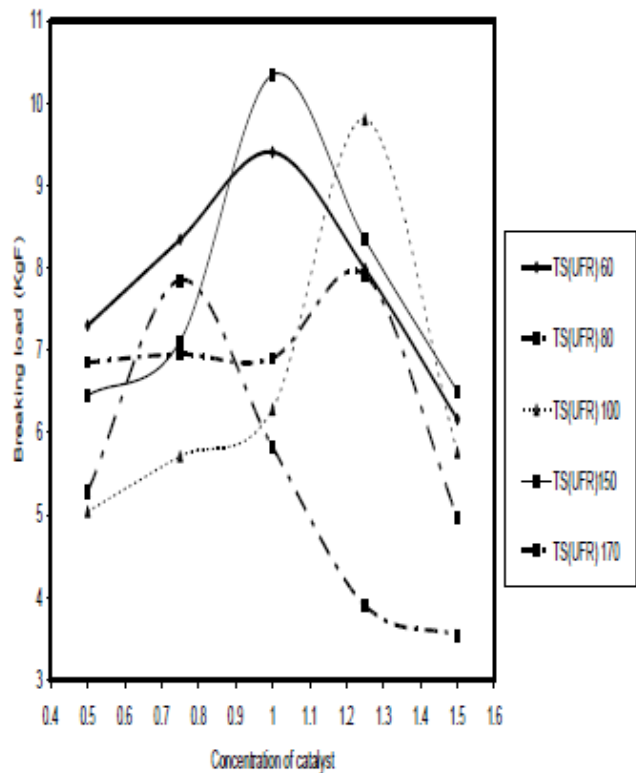


Fig. 32: Effect of temperature on breaking load of tetrasubstituted resinated cotton fabric at pH Of 8 (WEFT)

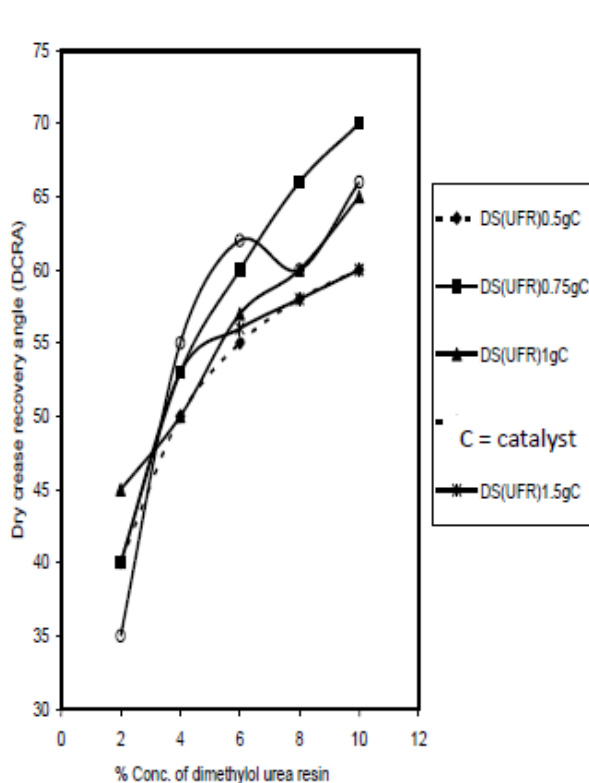


Fig. 14: Effect % concentration of dimethylol urea resin on dry crease recovery angle (DCRA) of cotton fabric (WARP)

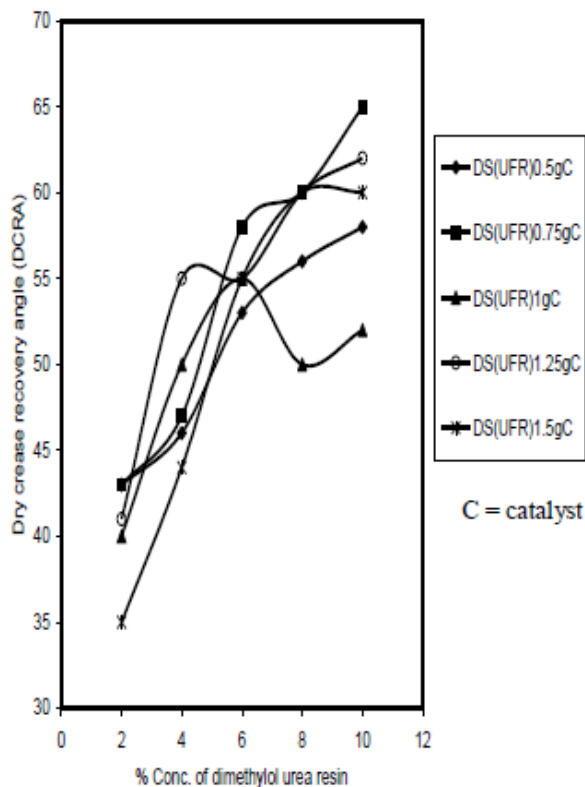


Fig. 17: Effect of % concentration of dimethylol urea resin on dry recovery angle (DCRA) of mercerized cotton fabric (WEFT)

V. DISCUSSION

Effect of Temperature and Catalyst Mass on the Breaking Load of Methylol Urea Resinated Cotton Fabric at pH of 8.0

When a polymer enthalpy changes, a corresponding change in physical and chemical properties takes place. This is because the dipole forces which cause attraction between polar groups on different chains of the polymers are temperature dependent (Moji, 2000). The lower the temperature, the more effective the dipole forces, but becomes weaker at higher temperature. Curing is the process of raising temperature to chemically react the resin with the cellulose (Express Indiana, 2002). In garment finishing, resin curing is accomplished through the use of batch or continuous oven. Proper curing is required to achieve maximum abrasion and tear resistance during service (Express Indiana, 2002). Heat distribution should be even from top to bottom and side to side with a variation not more than $\pm 2^{\circ}\text{C}$. Curing at 150°C to 155°C for 15 minutes gives desirable performance; curing time can be adjusted depending on the weight of the fabric. For heavy weight fabric the curing time can be increased or reduced for light weight fabrics. Allowing fabrics to over cure degrades the cotton due to excessive heat, distorting the attractive force between polar group of resin – cotton net work (Moji, 2000; Express Indiana, 2002). This degradation is particularly apparent when measuring the flex/abrasion, breaking load etc. As the temperature increases gradually to recommended curing temperature of 150°C , it can be seen from Figs. 23-30 that the effect of temperature on the breaking load temperature curve is significant and controls the overall characteristic tensile strength (Moji, 2000; Express Indiana, 2002). Beyond 170°C , the rigidity, yield strength and breaking load decreased while elongation increased (Cowie, 1991), this is evident in Figs 25-32 in both warp and weft directions.

Fig 25 shows the combine effects of catalyst mass and temperature on the breaking of mono substituted urea formaldehyde resinated cotton fabric sample (MSUFR 120). The sample gave a (BL) of 7.84kgf at 120°C , this is closely followed by MSUFR 100 sample cured at 100°C with a BL of 7.40 kgf. Fig 26, revealed the effect of temperature and catalyst mass on the breaking load of disubstituted methylol urea resinated fabric (warp), DSUFRI50 giving a breaking load of 10.01kgf at 150°C . This is followed by DSUFR120 sample cured at 120°C and DSUFR60 at 60°C with a breaking load of 9.26kgf and 8.21kgf respectively. Further elevation in the mass of catalyst and temperature above fabric curing temperature of 155°C (DSUFR170) gave a breaking load of 9.26kgf, this represents 8% reduction in tensile strength from the maximum breaking load of 10.01kgf.

Fig. 27: shows the fabric sample resinated with trisubstituted methylol urea (warp). This gave the maximum breaking load of 11.32kgf at 80°C, followed by 10.6kgf at 150°C and 10.32kgf at 100°C within the range 0.75g to 1.00g of catalyst mass. Also further increase in catalyst mass and temperature up to 170°C gave a breaking load of 7.62kgf, this represent 48% decrease in the tensile strength of the resinated fabric.

In fig. 28, Tetra substituted methylol urea resinated fabric (warp) cured at 150°C gave a B.L. of 11.05kgf and 10.40kgf at 100°C. The trend repeated itself within catalyst mass range of 0.7 g to 1.0g while elevation in temperature to 170°C was counter productive, and gave a breaking load of 8kgf, representing a reduction of 38% in tensile strength when compared with the maximum 11.32kgf.

Fig 29 and 30 captured the breaking load behavior of mono and disubstituted methylol urea resinated cotton fabric (weft) at various catalysts mass and temperature. The resination carried out at 120°C (MSUFR120) weft, gave the optimum breaking load of 7.20kgf at 1.0g catalyst mass, MSUFR40 sample cured at 40°C gave the least breaking load, this further confirms the importance of temperature in fabric treatment.

Fig. 30 revealed the behavior of disubstituted methylol urea resinated cotton fabric (weft). Sample DSUFR150 gave the highest BL of 10.01kgf, closely followed by sample DSUFR120, cured at 120°C, which failed at 9.26kgf. Subsequently, further elevation in curing temperature of samples up to 170°C, gave breaking load of 9.26kgf, representing 8% reduction in the tensile strength of sample. The trend replicated itself in fig. 31 and 32 which showed the effect of catalyst mass and temperature in the tetramethylol urea resinated cotton fabric in the weft direction. Sample TRIUFR 80°C and TSUFR 150 cured at 150°C gave a breaking load of 11.32kgf and 11.05kgf respectively between 0.5g to 1.0g catalyst mass. Subsequent elevation in temperature also reduce breaking load in both cases. Breaking load along the warp direction exceed that on the weft direction, reason being due to the amorphous regions of cellulose that 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, 2001).

Effects of Temperature and Catalyst Mass on Dry Crease Recovery Angle (DCRA) of Dimethylol Urea Resinated Cotton Fabrics(warp/weft)

Fabrics used to produce garments should resist wrinkle during wearing, have a smooth appearance after washing and tumble drying (Express Indiana, 2002). Creasing is chiefly due to the presence of weak Van Der Waal forces which characterized the amorphous regions; however when these regions are filled with low molecular weight resins, the crease recovery property is improved. Saunder (1973) and Ibrahim (1997) stated that when resins polymerize within the interstices of a material, the resistance of the material to creasing increases greatly. This is due to cross-link of resin and adjacent cellulose polymer chains, which gives cotton some elasticity, resiliency, and lower creasing propensity (Express Indiana, 2002). The recommended fibre wet pick up within 25 to 30 minutes of dipping and extraction is 65-70% (Express Indiana, 2002). Crease recovery of fabrics measures therefore the ability of fabrics to resist wrinkle that would otherwise distort aesthetic appeal to modern customers. It also gives information on the flexibility or recoverability of material from wrinkle. A good fabric should after deformation recover rapidly from deformation.

Fig. 14 and 17 show the result of DCRA of resinated cotton fabric at various mass of catalyst. The result revealed a gradual increase in the DCRA as the mass of catalyst, in the various substitutions increase. The 0.75g of catalyst samples gave the highest crease recovery angle in weft and warp direction. There was no marked difference in their respective DCRA in the two directions. This result is consistent with that of Ajayi (2001), Boyede, (1994) and Eribo, (2001). It is observed that as degree of substitution increases, the DCRA also increases. This increase is not infinite, since there are finite glucose residue; a unit of cellulose molecule. Each unit of glucose residue contains three (3) free – OH groups (Moji, 2000), but since there are 100-200 glucose residue in cellulose molecule, there will be 300-600-OH groups in the cellulose. Thus when these site are filled, no further cross-linking reaction would take place; hence there would be a saturation point. This point marks the turning point or decrease in the DCRA observed in the above figures. Finally the DCRA of the warp direction is slightly higher than in the weft direction. This is due to higher amorphosity in the warp direction, filled up during resination, thus strengthening the weak hydrogen bonds, between the cellulose and methylol group, which enhanced the resilience and springiness of the material (Ajayi, 1997)

VI. CONCLUSION

Acid damage of cotton-resin network was visible in all levels of the mechanical properties of the resinated samples tested. For optimum performance and profit maximization of methylol urea resins, should be prepared between 0.5g to 1.0g of catalyst.

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