CHAPTER II

MATERIALS AND EXPERIMENTAL PROCEDURES

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Fabric: A cotton poplin with following construction was used:

$38^s/38^s$ (15 tex/15 tex); 128 ends and 72 picks per inch.

Before the laboratory treatments the fabric was desized, kier boiled, bleached and mercerized in a mill.

Chemicals: - The cross-linking agents used were commercial products with about 50% solid content and purity of 98-99% as found from their nitrogen and formaldehyde analysis. These were DMEU ("Ahuramine KETU"); DMDHEU ("Hicofor DMH").

The metal salts e.g. $MgCl_2.6H_2O$, $ZnCl_2.2H_2O$ were of B.D.H. analytical grade. Organic acids used were also of B.D.H analytical reagent grade. From the organic acids partially or fully neutralized salts were prepared as follows.

Citric acid (95.4 g.) was dissolved in approximately 500 ml. of water. To this solution weighed quantity of $MgCO_3$ (38.1 g.) and Na_2CO_3 (24.00 g.) was slowly added with constant stirring. The solution was finally made upto 1000 ml. This gives 10% (w/v) solution of NaMgCit. The solutions of NaZnCit, ZnHCit, ZnH_2Cit, MgHCit, NaH Tart and other salts were similarly

prepared by neutralizing the acid with stochiometric quantities of Na_2CO_3, $MgCO_3$, $ZnCO_3$.

pH Measurement:- pH was measured on a "Systronics"(model 321) pH meter.

TEST METHODS

Fabric properties:- Dry and Wet Crease Recovery were measured according to Monsanto method (ASTM D1295-60T). Each result given is the average of 10 samples (five samples with warp (W) threads creased and five samples with weft (F) threads creased).

Tensile Strength: - Tensile strength of the fabric was measured on 1" x 8" ravelled strips with the constant rate of traverse machine ($2\frac{5"}{8}$ per minute (ASTM, D39-49). The distance between the jaws was 3". Each result given is the average of 5 samples.

CHEMICAL ANALYSIS

Estimation of formaldehyde: - Formaldehyde content of the fabric was determined spectrophotometrically by the chromotropic acid method[1,2]. About 0.1 g. sample was extracted with 100 ml. of 12N sulphuric acid for 24 h. 2 ml. of the extract was used for the estimation.

Nitrogen Content: - Nitrogen content of the sample was determined by the Kjeldahl macro method.

Polarographic Estimation of Zinc: - Bound zinc in the fabric was determined by polarographic method. The fabric was wet ashed, extracted with 2 ml. of concentrated hydrochloric acid, buffered with NH_4Cl & NH_4OH and then, 1 ml. of 5% gelatin and 5 ml. of 5% Na_2SO_3 were added. The solution was made upto 100 ml. Zinc content in the solution was estimated polarographically.

Concentration vs. pH: - The standard solutions of metal salts were prepared by dissolving the approximately weighed amount of salt in distilled water and estimating zinc by oxine[3] method and magnesium by EDTA method[4].

Zinc nitrate stock solution 50 percent (w/v) was diluted to give 100 ml. each from 1 to 10 percent zinc nitrate. Similarly ZnHCit (10%) was diluted to give 1 to 9% solutions of ZnHCit. The pH of these solutions was measured. Solutions of different concentrations (1 to 10%) were mixed with equimolar solutions of Zinc nitrate and finally made upto 100 ml. The pH of these mixed solutions was also measured. For increasing the concentrations, the mixed solutions were concentrated on a water bath at 70 - 80°C. pH was measured at different stages of concentration after cooling to room temperature. Similarly pH measurements

were also made for solutions of organic acid salts NaZnCit, ZnHCit, ZnH_2Cit, NaMg Tart, MgH Tart alone and in the presence of $Zn(NO_3)_2$ and $ZnCl_2$.

Acid-Alkali Titration: - Citric acid (100 ml., 0.01 M) was titrated with N/10 NaOH at room temperature with adequate stirring and pH changes were measured after each addition. The titrations were also carried out after mixing citric acid (100 ml., 0.01 M) with $ZnCl_2$ (1,2,20 ml. 1M). Similarly solutions of tartaric, oxalic and succinic acids were also treated. Such titrations were also carried out in the presence of cellophane, glucose and dimethylol ethylene urea.

Resin-Catalyst Titrations: - 160 ml. of DMEU (50% w/v) were dissolved in 600 ml. of water. To this solution organic acid solution was slowly added with constant stirring. The pH of the resin solution was measured after each addition. Such titrations of resin with partially neutralized organic acids alone and in the presence of $Zn(NO_3)_2$ and $ZnCl_2$ were also carried out.

Infra-red Spectra: - The infra-red spectra of untreated and treated fabrics were obtained by KBr disc method[5]. Cotton fabric was powdered in a small wiley mill through a 20 mesh screen. About 2 mg. of this sample (accurately weighed) was mixed with 200 mg. of potassium bromide thoroughly and transparent KBr

pellets were prepared. Spectra were obtained on Perkin-Elmer 137 double-beam spectrophotometer.

Fabric Treatments: - The cross-linking treatments were given to one metre long samples. The samples were padded with resin solution containing required catalyst to 75% wet pick-up and the fabrics were dried and cured in a Monfort single compartment stenter. Temperature control was possible within $\pm$ 2.5°C. The cured samples were rinsed in running tap water and washed with 2 g/l. soda ash solution at 50°C for 10 - 15 minutes. The samples were then thoroughly washed with tap water followed by distilled water, dried in air and conditioned at 65% RH.

Rate Measurements: - Rate of reaction of cotton with dimethylol ethylene urea in the presence of different catalysts was measured as follows:

The desired amount of the resin and catalyst was applied to the fabric by padding (about 75% wet pick up). The padded fabrics were dried at 30°C for 3 minutes in the stenter using only air draft and then placed at a temperature selected for the reaction in a special cabinet made for the kinetic study.

The size of the cabinet was 3' x 2' x 2', and at bottom of it six electric bulbs of 60 Watt each were fitted for temperature regulation. The temperature fluctuation in the cabinet was $\pm$ 2°C.

The fabric samples were withdrawn from the cabinet at various intervals, rinsed in cold water, air dried and conditioned. Wet and dry crease recovery, nitrogen content and bound formaldehyde were estimated by the methods discussed earlier.

Heating of Fabric with Catalyst Only: - The fabric samples were padded with catalyst solution, dried at 80°, 100° or 120°C for one minute and cured at 120°, 140° and 160°C for four minutes using the stenter. The samples were then washed air dried and conditioned. Tensile strength of the so treated samples was measured.

Distillation Experiments: - Oxalic acid (5 g.) was mixed with sodium chloride (5 g.) and dissolved in water to make 25 ml. This solution was distilled on an oil bath and about 16 - 17 ml. distillate was collected and titrated against 0.1 N NaOH. Similar experiments were carried out with tartaric, malic and succinic acid. The set was repeated using magnesium chloride in place of sodium chloride.

Thermogravimetric Analysis: - The thermogravimetrical analysis was carried out in an apparatus supplied by Lenseis Co., West Germany. One arm of the T.G.A. balance consisted of the boat carrying the sample while the other one was connected to the

electromagnetic weight compensator. The output of the electromagnetic weight compensation system, which is proportional to the difference of weight, was fed to a normal sensitive potentiometric recording unit. The change of weight was continuously recorded during heating. The temperature of reaction was measured with the help of Pt/Rh thermocouple.

The rate of heating was maintained at $5^{\circ}C$/min. T.G.A of 50 mg. of NaCl and 50 mg. succinic acids were carried out. T.G.A of a mixture of 50 mg. NaCl with 50 mg. succinic acid was also carried out. Measurements were carried out in air.

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Abbreviations

% w/v	=	% Weight/volume in the recipe
WCR	=	Wet crease recovery angle (Monsanto°)
DCR	=	Dry Crease recovery angle (Monsanto°)
Ten.str.	=	Tensile strength (Kg)
DMEU	=	Dimethylol ethylene urea
Cat X	=	Partially neutralized organic acid salts
Cat M	=	Mixture of metal salts with organic and inorganic acids
Metal salts	=	Latent acid type salts viz. zinc chloride, zinc nitrate
DMDHEU	=	Dimethylol dihydroxy ethylene urea
W	=	Warp
F	=	Weft