Rec: 139364

SAWTRI TECHNICAL REPORT

NO 459 WU4/F/1/4

Resin Treatment of Cotton Fabrics in the Slack State in Liquid Ammonia

by

F.A. Barkhuysen and N.J.J. van Rensburg

SOUTH AFRICAN
WOOL AND TEXTILE RESEARCH
INSTITUTE OF THE CSIR

P.O. BOX 1124
PORT ELIZABETH
REPUBLIC OF SOUTH AFRICA

ISBN 0 7988 1538 8

RESIN TREATMENT OF COTTON FABRICS IN THE SLACK STATE IN LIQUID AMMONIA

by F. A. BARKHUYSEN and N. J. J. VAN RENSBURG

ABSTRACT

Lightweight and heavyweight cotton fabrics were treated in the slack state with a solution of DMDHEU resin and NH_4NO_3 catalyst in liquid ammonia. The physical properties of the fabrics generally compared favourably with those of fabrics which had been pretreated with liquid ammonia or sodium hydroxide, followed by aqueous resin treatment or resin treatment in liquid ammonia. The effect of different concentrations of water in the liquid ammonia on the physical properties of the fabrics was also investigated. In the case of the lightweight fabric the addition of water to the liquid ammonia had practically no effect, whereas for the heavyweight fabric, an increase in the amount of water added to the liquid ammonia resulted in an increased crease recovery angle and a decrease in strength.

INTRODUCTION

It is well known that liquid ammonia pretreatment beneficially affects the physical properties of cotton fabrics which are subsequently treated with aqueous resin solutions[1–7]. It has been reported, for example, that the strength loss induced by conventional resin treatment could be reduced by as much as 30% after pretreatment with liquid ammonia[8]. This effect is due mainly to the ability of liquid ammonia to release stresses in the fibre prior to becoming permanently fixed by chemical cross-linking treatment. Furthermore, liquid ammonia treatment improves the uniformity and regularity of the fibrillar order and packing density of the cellulose structure. This rearrangement improves the cross-link formation between adjacent cellulose fibrils[9].

The possibility of applying a durable press resin to cotton from a liquid ammonia medium has been investigated by some research workers[10–12]. In general, however, relatively little information is available about this aspect. Furthermore, it appears that various investigators did not always agree on the advantages of such a treatment. For example, Bredereck and Pfundtner[10] claimed that they obtained satisfactory results, whereas Lewin *et al*[11] stated that less reactive aminated products of the resin were formed when methylol based resins were applied to cotton from liquid ammonia.

Barkhuysen[12] studied the simultaneous dyeing and resin treatment of cotton in liquid ammonia. He found that various reactive dyes could be fixed to cotton by the use of a DMDHEU resin and ammonium nitrate as catalyst.

Furthermore, the K/S values and the crease recovery angles of the fabrics increased when the concentration of resin in the solution was increased. In the abovementioned study the emphasis, however, was on the dyeing behaviour of the cotton, and several aspects of the resin treatment of cotton in liquid ammonia remain to be investigated.

It was decided, therefore, to study the treatment of cotton fabrics with resin from a liquid ammonia medium in more detail. Untreated fabrics as well as fabrics which had been pretreated with liquid ammonia or sodium hydroxide were treated with DMDHEU resin in liquid ammonia. For purposes of comparison some fabrics were treated with aqueous solutions of DMDHEU resin. Some physical properties of the fabrics were determined, and the results obtained were analysed statistically.

EXPERIMENTAL

Fabrics

Scoured, bleached and desized lightweight (145 g/m^2) and heavyweight (185 g/m^2) cotton fabrics were used.

Chemicals and Treatments

In all cases 5% and 10% (m/v) solutions of a DMDHEU type resin (®Fixapret CP) were prepared in liquid ammonia as well as in water. For all the resin treatments, 10% NH_4NO_3 (on mass resin) was used as catalyst. In addition to the treatments where anhydrous liquid ammonia was used, other treatments were carried out in liquid ammonia containing from 5 to 15% (v/v) water. (During dyeing of cotton in liquid ammonia, it was found that the presence of water in the dyebath resulted in more even dyeing[12]. It was decided therefore to investigate the effect of water added to the liquid ammonia resin solution on the distribution of resin through the fabric). Untreated control fabrics as well as fabrics which had been pretreated with liquid ammonia or sodium hydroxide were treated with resin. Some control samples were treated with aqueous resin solutions. The preparation of the resin solution and the treatment conditions were the same as those described previously[12].

The nitrogen content of the resin treated samples was determined on a Perkin Elmer 240 Elemental Analyser. The various physical properties of the fabrics were obtained employing standard test methods.

RESULTS AND DISCUSSION

Some physical properties of the different cotton fabrics which were treated under various conditions with DMDHEU resin are given in Appendix Tables I and II for the lightweight (LW) and heavyweight (HW) fabrics, respectively. These results were analysed statistically, applying multiple linear regression analyses. In these analyses the various fabric properties were considered as the dependent variables.

The independent variables were:

X_1 : % Resin (5% and 10%)
X_2 : % Water added to the liquid ammonia (0, 5, 10, 15%)
X_3, X_4, X_5 : Dummy variables

The dummy variables (X_3—X_5) used to describe the differences in the various treatment conditions, were coded as follows:

No.	Treatment Conditions	X_3	X_4	X_5
1.	Control + resin (aqueous)	0	0	0
2.	NH_3 pretreated + resin (aqueous)	0	0	1
3.	NaOH pretreated + resin (aqueous)	0	1	0
4.	Resin in NH_3	0	1	1
5.	NH_3 pretreated + resin in NH_3	1	0	0
6.	NaOH pretreated + resin in NH_3	1	0	1

To allow for the shrinkage of the fabrics which occurred during either the pretreatment or the resin treatment, another variable, X_6, was incorporated in the analysis. This was accomplished by multiplying the dependent variables by a factor, $\frac{100-X_6}{100}$, where X_6 is the percentage area shrinkage of the fabrics after the various treatments. The results obtained on the untreated control fabric which had not been resin treated, were omitted from these analyses and the untreated control fabrics which were resin treated were used as the control.

A linear model was chosen which included all the independent variables and their interactions with each other. The equation was of the form:

$$Y_j = A_o + A_1.X_k + A_2.X_k.X_l \quad \ldots\ldots \quad (1)$$

(j = 1-11, k and l have values between 1 and 5 and A_1 and A_2 are the coefficients of the independent variables) and was fitted to the results to obtain the "best fit" equation. The various dependent variables were regressed separately against the independent variables. All equations given are significant at the 95% level.

The analysis of the breaking strength results of the two fabrics produced the following equations:

LW Fabric

$Y_4 = 293 - 12{,}6\,X_1$ ....(5)

$r = 0{,}66$

HW Fabric

$Y_4 = 353 - 14{,}12\,X_2.X_5$ ....(6)

$r = 0{,}76$

For the *lightweight* fabric only the resin concentration (X_1) was found to have a significant effect on the breaking strength of the fabrics. An increase in the resin concentration resulted in a decrease in the breaking strength of the fabrics.

For the *heavyweight* fabric, the breaking strength followed a similar trend to that observed for its flat abrasion resistance. In other words, the breaking strength of the heavyweight fabric was independent of the amount of resin or the variation in the treatment conditions, but only depended on the concentration of water in the liquid ammonia, decreasing with increasing concentration of water.

Breaking Extension (Y_5)

The following best fit equations were obtained when the breaking extension results were analysed:

LW Fabric

$Y_5 = 16{,}1 - 0{,}563\,X_1$ ....(7)

$r = 0{,}56$

HW Fabric

$Y_5 = 8{,}02 - 0{,}285\,X_2.X_5$ ....(8)

$r = 0{,}80$

The same conclusion arrived at for the breaking strength of these two fabrics, is valid for the breaking extension results.

Crease Recovery Angle (Y_6)

The analysis of the crease recovery angle results yielded the following best fit equations for the two fabrics:

LW Fabric

$$Y_6 = 225 + 5{,}5\,X_1 - 15{,}3\,X_3 \quad \text{(9)}$$

$$r = 0{,}89$$

HW Fabric

$$Y_6 = 255{,}3 + 2{,}93\,X_2.X_5 \quad \text{(10)}$$

$$r = 0{,}85$$

An increase in the resin concentration resulted in an increase in the crease recovery angle of the lightweight fabric, while the fabrics that were pretreated with either liquid ammonia or sodium hydroxide prior to resin treatment in liquid ammonia, had lower crease recovery angles than the fabrics pretreated with liquid ammonia or sodium hydroxide followed by conventional resin treatment, as well as the fabrics treated with resin in liquid ammonia.

In the case of the heavyweight fabric, an increase in the percentage water in the liquid ammonia resulted in an increase in the crease recovery angle of the fabrics.

Flex Abrasion Resistance (Y_7)

The following best fit equations were obtained in the case of the flex abrasion resistance:

LW Fabric

$$Y_7 = 197 - 11{,}398\,X_1 \quad \text{(11)}$$

$$r = 0{,}59$$

HW Fabric

$$Y_7 = 179{,}8 - 24{,}51\,X_2.X_5 \quad \text{(12)}$$

$$r = 0{,}89$$

The equation for the lightweight fabric shows that the flex abrasion of the treated fabrics only depended on the resin concentration, decreasing with an increase in resin concentration.

The flex abrasion resistance of the heavyweight fabric, as was found for its flat abrasion, was affected by the amount of water present in the liquid ammonia. An increase in the amount of water resulted in a decrease in the resistance of the fabrics to flex abrasion.

Tear Strength (Y_8)

The best fit equations for the two fabrics were as follows:

LW Fabric

$Y_8 = 7{,}992 - 0{,}380\,X_1$ ....(13)

$r = 0{,}61$

HW Fabric

$Y_8 = 10{,}24 - 0{,}307\,X_1 - 0{,}312\,X_2.X_5$ ....(14)

$r = 0{,}89$

The tear strength of the *lightweight* fabric was affected only by the resin concentration, and decreased with increasing resin concentration.

In the case of the *heavyweight* fabric an increase in the amount of resin (X_1) and an increase in the amount of water in the liquid ammonia resin bath decreased its tear strength.

Mass/Unit Area (Y_9)

For the *lightweight* fabric, no significant equation was obtained, indicating that none of the various treatment affected the density of the fabrics.

The best fit equation for the *heavyweight* fabric was as follows:

$Y_9 = 0{,}427 - 0{,}00196\,X_2.X_5$ ....(15)

$r = 0{,}63$

This equation indicates that an increase in the amount of water in the liquid ammonia reduced the mass/unit area of the fabric slightly.

Thickness (Y_{10})

The analyses showed that the various treatments had no effect on the thickness of the *lightweight* fabric.

HW Fabric

The regression equation for the *heavyweight* fabric was:

$Y_{10} = 185{,}66 + 1{,}45\,X_1.X_4 + 7{,}577\,X_3 - 0{,}728\,X_2.X_5$ ....(16)

$r = 0{,}84$

This equation shows an interaction between a number of independent variables. Firstly, the thickness of the heavyweight fabric increased with an increase in the concentration of the resin, in the case of certain treatments. Secondly, the fabrics that were pretreated with liquid ammonia and sodium hydroxide before resin treatment in liquid ammonia, were thicker than the others. Thirdly, the addition of increasing amounts of water to the liquid ammonia resin bath, decreased the fabric thickness.

Nitrogen Content (Y_{11})

The X_6 variable was omitted in this case since the shrinkage of the fabrics should have no effect on the nitrogen content of the samples. The best fit equations were as follows:

LW Fabric

$$Y_{11} = -0{,}078 + 0{,}134\,X_1 + 0{,}054\,X_2.X_5 + 0{,}217\,X_3 \quad \ldots\ldots(17)$$

$$r = 0{,}98$$

HW Fabric

$$Y_{11} = 0{,}174 + 0{,}105\,X_1 + 0{,}036\,X_2.X_5 \quad \ldots\ldots(18)$$

Equation (17) shows that in the case of the lightweight fabric, the nitrogen content, i.e. the amount of resin on the cotton, increased with an increase in the resin concentration and with an increase in the amount of water in the liquid ammonia resin solution. The fabrics that were pretreated with liquid ammonia or sodium hydroxide prior to a resin treatment in liquid ammonia, had a higher nitrogen content than the fabrics which were not pretreated.

In the case of the heavyweight fabric (Equation 18), an increase in the resin concentration as well as an increase in the water content during the liquid ammonia resin treatment, resulted in fabrics with higher nitrogen contents.

SUMMARY AND CONCLUSIONS

The treatment of lightweight and heavyweight cotton fabrics with a DMDHEU resin and ammonium nitrate as catalyst in liquid ammonia, was investigated. Untreated fabrics as well as fabrics which had been pretreated with liquid ammonia or sodium hydroxide were treated in the slack state with the resin in a liquid ammonia solution. Two concentrations of resin (5% and 10%) and four levels of water added to the liquid ammonia bath (0, 5, 10 and 15%) were used. For purposes of comparison some fabrics were treated with aqueous resin solutions in the conventional manner. Certain physical properties of the fabrics were determined. The results obtained were subjected to a statistical analysis which showed that, as far as the effect of the different independent variables (resin concentration, percentage water in liquid ammonia and different treatments) was concerned, an increase in resin concentration generally resulted in an increase in crease recovery angle and a decrease in the strength of the fabrics. The amount of water in the liquid ammonia did not always affect the physical properties of the fabrics. In fact, in the case of the lightweight fabrics it has no effect on the physical properties tested. In the case of the heavyweight fabrics on the other hand, the presence of water in the liquid ammonia bath affected all the properties studied, with the exception of bending length and bursting strength. In general an increase in the amount of water in the liquid ammonia resulted in an increase in the crease recovery angle, and a decrease in the tear strength, breaking strength and extension, and the resistance to flat and flex abrasion.

The statistical analysis showed that, in the cases where no water was added to the liquid ammonia, there was no difference between the physical properties of the fabric treated in a slack state with resin in liquid ammonia and those of fabrics which had been pretreated in liquid ammonia or sodium hydroxide, followed by an aqueous resin treatment or a resin treatment in liquid ammonia. Under these conditions of resin treatment, a swelling pretreatment with liquid ammonia or sodium hydroxide is obviously not necessary. When water was added to the liquid ammonia bath there was, for all practical purposes, no difference between the results obtained from the various treatments on the lightweight fabric. (Only in the case of the crease recovery angle was a small effect noticed.) In the case of the heavyweight fabric, however, the addition of water to the liquid ammonia resulted in some differences between the various treatments. For example, the crease recovery angles of the control resin treated fabric, and the fabrics pretreated with sodium hydroxide followed by an aqueous resin treatment and the fabrics pretreated with liquid ammonia followed by a resin treatment in liquid ammonia, were normally higher than those of fabrics treated by the other methods. Similarly the former treatments resulted in greater strength losses than the latter. The effect (differences between the two treatments) generally became more pronounced as the concentration of water in the liquid ammonia was increased.

ACKNOWLEDGEMENTS

The authors wish to thank Mrs C. Dorfling for carrying out the resin treatments; the Textile Physics and Statistics Department for the physical testing of the fabrics and statistical analysis of the results, respectively.

THE USE OF PROPRIETARY NAMES

The fact that products with proprietary names (® denotes registered trade marks) have been used in this report does not imply that SAWTRI recommends them and that there are not substitutes which may be of equal value or even better.

REFERENCES

1. Anon., *Text. Asia* **9**, (1), 118 (1978).
2. Heap, S. A., *Int. Dyer and Text. Print.*, **160**, (10), 486 (1978).
3. Heap, S. A., *Text. Inst. Ind.*, **16**, (12), 387 (1978).
4. Pandey, S. N., *Colour.*, **23**, (12A), 22 (1976).
5. Vail, S. L., Verburg, G. B. and Calamari, T. A., *Text. Chem. Color.*, **7**, (10), 26 (1975).
6. Barkhuysen, F. A., *SAWTRI Techn. Rep.* No. 293 (April, 1976).
7. Kelly, I. W., *SAWTRI Techn. Rep.* No. 442 (Feb., 1979).
8. Anon., *Indian Text. J.*, **88**, (3), 134 (1977).
9. Heap, S. A., *Text. Manuf.*, **97**, (1155), 91 (1971).
10. Bredereck, K. and Pfundtner, R., *Textveredl.*, **10**, (3), 92 (1975).
11. Lewin, M., Rau, R. O. and Sello, S. B., *Text. Res. J.*, **44**, (1), 686 (1974).
12. Barkhuysen, F. A., *SAWTRI Techn. Rep.* No. 427 (Sept. 1978).
13. Heap, S. A., *Text. Manuf.*, **100**, (1184), 27 (1973).
14. Barkhuysen, F. A., Ph.D. Thesis University of Port Elizabeth (Dec., 1979).

APPENDIX

TABLE I

SOME PHYSICAL PROPERTIES OF LIGHTWEIGHT COTTON FABRICS TREATED WITH RESIN UNDER VARIOUS CONDITIONS IN LIQUID AMMONIA AND IN WATER

FABRIC TREATMENT	Bending Length (cm)	Bursting Strength (kN/m^2)	Martindale Abrasion (% mass loss at 500 cycles)	Breaking Strength (N)	Breaking Extension (%)	Crease Recovery Angle (W+F)°	Flex Abrasion (cycles to rupture)	Tear Strength (N)	Mass/Unit area (g/m^2)	Nitrogen Content (%)	Thickness (mm)
Untreated control	1,67	700	0,29	485	13,7	176	344	12,9	119	0	0,30
Untreated control + 5% DMDHEU	1,49	314	5,83	192	10,2	275	28	3,9	125	0,70	0,34
Untreated control + 10% DMDHEU	1,59	248	15,35	132	8,9	294	16	3,3	127	1,24	0,35
NH_3 Pretreated + 5% DMDHEU	1,65	407	2,82	249	12,2	265	82	8,2	130	0,60	0,35
NH_3 Pretreated + 10% DMDHEU	1,51	337	2,76	193	10,8	289	31	6,0	130	1,19	0,36
NaOH Pretreated + 5% DMDHEU	1,56	446	1,88	247	15,7	271	128	5,6	153	0,47	0,39
NaOH Pretreated + 10% DMDHEU	1,60	302	4,77	192	13,1	279	22	4,2	160	1,16	0,35
NH_3 + 5% DMDHEU	1,52	397	2,07	225	13,9	261	55	5,7	143	0,64	0,37
NH_3 + 10% DMDHEU	1,50	291	4,34	183	11,6	297	31	4,2	141	1,47	0,34
NH_3 + 5% H_2O + 10% DMDHEU	1,55	265	7,15	170	10,3	289	21	3,9	136	1,50	0,31
NH_3 + 10% H_2O + 10% DMDHEU	1,58	288	8,49	170	9,9	297	21	3,8	132	1,66	0,36
NH_3 + 15% H_2O + 10% DMDHEU	1,59	257	3,43	144	9,0	297	13	3,4	134	2,18	0,31
NH_3 Pretreated + (NH_3 + 5% DMDHEU)	1,55	405	2,57	202	13,9	265	84	6,6	142	0,92	0,36
NH_3 Pretreated + (NH_3 + 10% DMDHEU)	1,55	319	4,34	201	11,9	287	34	5,9	139	1,58	0,36
NaOH Pretreated + (NH_3 + 5% DMDHEU)	1,57	589	1,58	335	18,3	235	313	8,6	161	0,65	0,37
NaOH Pretreated + (NH_3 + 10% DMDHEU)	1,54	347	3,64	180	13,1	281	30	4,7	159	1,42	0,34

APPENDIX

TABLE II

SOME PHYSICAL PROPERTIES OF HEAVYWEIGHT COTTON FABRICS TREATED WITH RESIN UNDER VARIOUS CONDITIONS IN LIQUID AMMONIA AND IN WATER

FABRIC TREATMENT	Bending Length (cm)	Bursting Strength (kN/m^2)	Martindale Abrasion (% mass loss at 500 cycles)	Breaking Strength (N)	Breaking Extension (%)	Crease Recovery Angle (W + F)°	Flex Abrasion (cycles to rupture)	Tear Strength (N)	Mass/Unit area (g/m^2)	Nitrogen Content (%)	Thickness (mm)
Untreated control	2,06	1290	0,41	761	13,0	179	2797	21,9	179	0	0,44
Untreated control + 5% DMDHEU	1,62	572	1,92	315	7,4	265	135	7,5	184	0,67	0,44
Untreated control + 10% DMDHEU	1,77	423	1,85	245	5,5	280	28	5,5	194	1,25	0,44
NH_3 Pretreated + 5% DMDHEU	1,52	927	1,57	475	8,5	252	276	—	190	0,51	0,44
NH_3 Pretreated + 10% DMDHEU	1,60	729	2,38	374	7,4	282	43	—	195	0,98	0,41
NaOH Pretreated + 5% DMDHEU	1,74	909	1,34	458	8,8	249	266	8,5	193	0,57	0,43
NaOH Pretreated + 10% DMDHEU	1,76	675	4,54	304	7,0	272	48	5,2	205	1,02	0,42
NH_3 + 5% DMDHEU	1,52	791	1,03	442	12,5	249	390	10,0	215	0,81	0,49
NH_3 + 10% DMDHEU	1,69	387	3,28	242	7,3	282	48	5,3	215	1,47	0,47
NH_3 + 5% H_2O + 10% DMDHEU	1,65	404	7,59	231	6,1	291	29	5,3	206	1,77	0,44
NH_3 + 10% H_2O + 10% DMDHEU	1,68	391	10,2	229	6,4	285	20	5,2	204	1,80	0,45
NH_3 + 15% H_2O + 10% DMDHEU	1,72	396	26,5	235	6,0	287	24	5,3	194	1,49	0,42
NH_3 Pretreated + (NH_3 + 5% DMDHEU)	1,52	829	1,88	411	9,9	257	383	10,6	205	0,81	0,46
NH_3 Pretreated + (NH_3 + 10% DMDHEU)	1,55	765	2,76	334	7,3	280	71	8,1	197	1,31	0,43
NaOH Pretreated + (NH_3 + 5% DMDHEU)	1,66	856	1,16	419	9,6	268	209	9,4	204	0,82	0,44
NaOH Pretreated + (NH_3 + 10% DMDHEU)	1,70	940	1,83	368	7,1	281	88	7,1	201	0,99	0,41

Published by
The South African Wool and Textile Research Institute,
P.O. Box 1124, Port Elizabeth, South Africa
and printed in the Republic of South Africa
by Nasionale Koerante Beperk, P.O. Box 525, Port Elizabeth.

ISBN 0 7988 1538 8