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Liquid Ammonia Mercerisation of Cotton

**Part IV: Liquid Ammonia Mercerisation
as a Pretreatment for subsequent Durable
Press Treatments**

**by
F.A. Barkhuysen**

**SOUTH AFRICAN
WOOL AND TEXTILE RESEARCH
INSTITUTE OF THE CSIR**

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

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LIQUID AMMONIA MERCERISATION OF COTTON

PART IV: LIQUID AMMONIA MERCERISATION AS A PRETREATMENT FOR SUBSEQUENT DURABLE-PRESS TREATMENTS

by F.A. BARKHUYSEN

ABSTRACT

Cotton fabrics of different constructions and densities were pretreated with liquid ammonia before durable press finishing treatments. It was found that the strength losses encountered on the unmercerised resin-treated samples were reduced significantly by a pretreatment with liquid ammonia. It was also found that a pretreatment with liquid ammonia resulted in bigger strength retention after durable press finishing than a pretreatment with sodium hydroxide.

The removal of ammonia from the fabrics by heat during the liquid ammonia treatment resulted in the highest crease recovery angle of the fabrics but also in lower strength retention. No difference in the durable press properties of the fabrics was found whether the ammonia was removed from the fabrics by either cold or hot water during the pretreatment with liquid ammonia.

INTRODUCTION

The durable press treatment of cotton fabrics plays an important rôle in the finishing of cotton goods. Frequently, however, the level of durable press performance of the treated fabrics falls below that which is demanded by the consumer. An acceptable level can be produced easily, but this is, unfortunately, only achieved with major sacrifices in strength and durability. It has been stated that, apart from fibre damage caused by catalysts, heat and other conditions of treatment, the strength losses are proportional to the improvement in wrinkle recovery obtained¹. Durable press products account for about half of the cotton market and failure to find an answer to this problem of strength losses could have a serious effect on the long-term future of cotton as a textile fibre².

The response of the cotton fibre to durable press finishing treatments appears to be governed by:

- (i) the fibre structure
- (ii) the pretreatment with a swelling agent, and
- (iii) the cross-linking treatment.

Basically, durable press finishing involves chemical reactions at the

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surfaces of the fibrils, thereby forming cross-linkages between two adjacent fibrils. The chemicals used for these cross-linking reactions have relatively small molecules, and therefore cross-linkages can only be formed when the fibrils are sufficiently close together for the molecules to bridge the gap between them. It seems obvious, therefore, that the cotton would be strengthened considerably if the uniformity of fibrillar order and packing density could be improved, particularly if this improvement was uniform and regular across the whole fibre cross-section. It is generally accepted that the strength loss of resin-treated cotton is due to the presence of the cross-links which prevent the molecular chains of the cellulose from moving relative to one another, thus preventing a redistribution and equalisation of applied stress.

Another explanation for the reduction of tensile strength resulting from durable press finishing, is the accumulation of stresses by the cotton fibre from the initial drying out on the cotton boll, through spinning, weaving and subsequent processes. It is understandable that these stresses must be released before they are permanently fixed by chemical cross-linking treatments. Recently it was observed that a swelling treatment of cotton with liquid ammonia resulted in almost complete stress release.³

It has been stated that the development of improved durable press chemicals to obviate these deleterious effects seems to have reached a saturation point⁴. Other ways must be found, therefore, to eliminate or reduce the excessive strength losses which accompany a high level of durable press performance. One such approach is to utilise cotton varieties with high strength properties. This approach, however, is not always feasible because such varieties are not always readily available. Another alternative is to modify the cotton fibre structure prior to the cross-linking treatment. The best known method to achieve this objective is through mercerisation.

Improvements in the physical properties of cross-linked cotton as a result of fabric mercerisation by sodium hydroxide are small, possibly due to the unevenness of the treatment. The scope of this investigation, therefore, was to investigate the effect of liquid ammonia mercerisation of cotton fabrics as a pretreatment for subsequent cross-linking treatments, and to compare its potential in this regard with that of sodium hydroxide mercerisation.

EXPERIMENTAL

Materials:

The machine, materials, processing parameters and test methods used, were the same as those described previously^{5,6}.

A lightweight plain fabric, heavyweight plain fabric and twill fabric were treated with various aminoplast resins and catalysts with and without prior

mercerisation with liquid ammonia and sodium hydroxide. Mercerisation with sodium hydroxide was carried out industrially whereas all other treatments were carried out at SAWTRI.

Resin Application:

In the first series of experiments, fabrics were treated with Fixapret AH (BASF), a dimethylolethylene urea (DMEU) resin (50 per cent solids) or with Fixapret CP_{conc} (BASF), a dimethyloldihydroxyethylene urea (DMDHEU) resin (80 per cent solids), together with 0,2 per cent (on mass of solution) Tergitol Speedwet (Union Carbide), a non-ionic wetting agent. The catalysts used were Zn(NO₃)₂.6H₂O (10 per cent on mass of resin) or a mixture of MgCl₂/citric acid (10 per cent on mass of resin) in a 1:1 ratio. In this case 10 per cent resin was applied to the fabrics.

In the second series of experiments, fabrics were treated with a mixture of two resins, namely, Aerotex M3 (Cyanamid), and alkylated methylolmelamine resin (60 per cent solids), and Fixapret CP_{conc} in a 1:1 ratio. Only one catalyst was used in this case, namely, Zn(NO₃)₂.6H₂O (10 per cent on mass of resin). Two levels of resin application viz. 5 and 10 per cent (o.m.f.) were employed. Tergitol Speedwet (0,2 per cent on mass of solution) was again used as wetting agent.

In both series of experiments, the resin was applied by the conventional pad-dry-cure method. The fabrics were padded on a Benz laboratory continuous padding apparatus to approximately a 100 per cent wet pick-up. The samples were then air dried, after which they were cured for 3 minutes at 160°C. The fabrics treated with the MgCl₂/citric acid catalyst, however, were cured for 3 minutes at 120°C. Finally, the treated samples were washed in a domestic washing machine using a standard cotton washing cycle, and line dried before testing.

RESULTS AND DISCUSSION

The effect of resin treatment on the physical properties of the unmercerised and mercerised *lightweight* plain cotton fabric is shown in Table I. A comparison of the physical properties of the resin-treated unmercerised control fabric with the properties of the untreated control fabric, shows that the resin treatment reduced the tensile strength of the cotton significantly. The crease recovery angles of the fabrics were fairly high but the loss in tensile strength was approximately 40 to 50 per cent for the 4,5 per cent resin add-on and even higher for the 9,1 per cent resin application. The same applied to the unmercerised stretched control fabric. In this case, however, an even bigger loss in breaking strength was obtained after resin treatment. The percentage mass loss during flat abrasion was also significantly higher. It is clear from the table, however, that the tensile strength losses in the warp direction were generally

TABLE I
THE EFFECT OF RESIN TREATMENT ON THE PHYSICAL PROPERTIES OF
UNMERCERISED AND MERCERISED LIGHTWEIGHT PLAIN COTTON FABRICS

TREATMENT	PHYSICAL PROPERTIES																							
	CREASE RECOVERY ANGLE (degrees)				BURSTING STRENGTH (kN/m ²)				FLEX ABRASION (cycles to rupture)				FLAT ABRASION (% mass loss)				TEAR STRENGTH (N)				BREAKING STRENGTH (N)			
	A		B		A		B		A		B		A		B		A		B		A		B	
	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft
Untreated Control	192		823.2		423	369								9.4	10.6			462.6	407.7					
Untreated Control + Resin	261	291	470.4	401.8	215	167	87	74	4.5	15.0	5.6	5.9	4.5	3.8	283.2	257.7	253.8	214.6						
Untreated Mercerised Control + Resin	268	300	490.0	411.6	234	201	84	61	5.7	21.6	5.6	4.7	4.3	3.5	175.4	183.3	185.2	138.2						
Slack Mercerisation + Resin	278	299	568.4	431.2	289	369	200	200	3.0	4.1	6.8	6.8	4.9	4.5	227.4	202.9	178.4	129.4						
NH ₃ + Heat* Removal of Ammonia + Resin	281	306	411.6	323.4	292	207	161	105	3.6	4.6	9.1	6.7	7.3	4.5	291.1	254.8	246.0	206.8						
NH ₃ + Cold* Water Removal of Ammonia + Resin	262	298	460.6	392.0	305	263	129	124	3.4	7.3	8.0	6.8	8.1	4.4	352.8	211.7	267.5	196.0						
NH ₃ + Hot* Water Removal of Ammonia + Resin	265	289	450.8	392.0	332	207	154	179	3.4	6.1	9.1	5.8	7.4	3.9	355.8	246.0	310.7	182.3						
NaOH + Resin	249	283	548.8	441.0	362	272	73	49	3.2	14.2	8.6	6.0	6.2	3.2	418.5	227.4	327.3	165.6						

A = 4.5% resin / Aertotex M₁ + Fixapret CP conc + 10% Zn (NO₃)₂ · 6H₂O (on mass of resin)

B = 9.1% resin

* 6% stretch applied to the fabrics.

reduced significantly when the fabrics were mercerised with liquid ammonia or sodium hydroxide prior to the resin treatment. With respect to tear strength, flat abrasion and flex abrasion, liquid ammonia mercerisation generally seemed to give better results than sodium hydroxide mercerisation, especially at the higher level of resin application. In the case of bursting strength and breaking strength, on the other hand, sodium hydroxide mercerisation generally gave better results than liquid ammonia mercerisation. The crease recovery angles of the ammonia treated fabrics were significantly higher than those of the sodium hydroxide treated fabric, especially at the lower level of resin application. The lower retention of strength of the slack mercerised fabric after resin treatment, showed that the application of *stretch* during mercerisation is a prerequisite for improved retention of strength after resin treatment.

Removal of the ammonia by heat gave higher crease recovery angles than removal by water. The former treatment was, however, accompanied by slightly higher strength losses than the latter. It is also evident from Table I that cold or hot water removal of the ammonia at the slow speed at which the machine was running, did not produce significant differences in the performance of the fabrics after subsequent resin treatments.

Liquid ammonia mercerisation proved to be much better than sodium hydroxide mercerisation in reducing flat abrasion losses. Sodium hydroxide mercerisation, in fact, gave little improvement in resistance to flat abrasion when compared with the unmercerised resin-treated control fabric.

The percentage mass loss during flat abrasion of the unmercerised control fabric treated with 4,5 *per cent* resin, was about 80 *per cent* higher than that found for the untreated control fabric, and that of the fabric treated with 9,1 *per cent* resin was about 500 *per cent* higher. Mercerisation with liquid ammonia reduced the increase in flat abrasion losses to about 40 and 80 *per cent*, respectively.

Table II gives the results obtained on the *heavyweight plain* cotton fabric. A similar trend to that found for the *lightweight plain* fabric, was found in this case. Once again, it was found that resin treatment of the control fabrics led to significant tensile strength losses. Pretreatment of the fabrics with liquid ammonia or sodium hydroxide, however, reduced the strength losses to a large extent. In general, a pretreatment with liquid ammonia gave a higher strength retention and higher crease recovery angles than a pretreatment with sodium hydroxide. Hot water removal of ammonia during the mercerisation process seemed to produce slightly better bursting strength, tear strength and warp breaking strength results than the removal of ammonia by cold water. Cold water removal of ammonia, on the other hand, produced better weft breaking strength results than hot water removal.

The results obtained on the *twill fabric* are given in Table III. Once again, it can be seen that the treatment of unmercerised fabrics with aminoplast resins resulted in large reductions in strength of the cotton. Mercerisation of the

TABLE II
THE EFFECT OF RESIN TREATMENT ON THE PHYSICAL PROPERTIES OF UNMERCERISED AND MERCERISED HEAVYWEIGHT PLAIN FABRICS

Treatment	PHYSICAL PROPERTIES																			
	Crease Recovery Angle (degrees)		Bursting Strength (kN/m ²)		Flex Abrasion (cycles to rupture)				Flat Abrasion (% mass loss)		Tear Strength (N)				Breaking Strength (N)					
	A	B	A	B	A		B		A	B	A		B		A	B				
					Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft				
Untreated Control	180		1029.0		920	388					18.1	12.7			712.5	473.3				
Untreated Control + Resin	249	274	588.0	470.4	185	112	164	105			7.5	31.3	7.2	6.7	6.4	5.5	535.1	295.0	457.7	230.3
Untreated Stretched Control + Resin	258	290	607.6	519.4	319	175	141	121			5.8	19.4	7.5	7.1	6.0	5.6	519.4	309.7	454.7	229.3
Slack Mercerised + Resin	278	310	617.4	421.4	1196	566	312	266			2.2	4.1	10.0	9.2	6.1	6.5	585.1	327.3	394.0	214.6
NH ₃ + Heat* removal of Ammonia + Resin	266	306	744.8	568.4	931	314	73	67			3.7	5.5	12.0	8.8	9.3	6.1	554.7	188.2	547.8	132.3
NH ₃ + Cold* Water Removal of Ammonia + Resin	250	282	833.0	656.8	383	309	353	231			4.2	5.4	12.3	8.9	9.9	7.5	708.5	330.3	559.6	264.6
NH ₃ + Hot* Water Removal of Ammonia + Resin	254	288	911.4	793.8	484	305	305	192			3.1	6.2	12.7	9.5	10.3	8.2	752.6	273.4	674.2	198.9
NaOH + Resin	245	279	833.0	627.2	515	206	180	77			3.8	30.5	11.1	8.6	6.8	5.5	678.2	221.5	576.2	200.9

A = 4.5% resin / Aerotex M₃ + Fixapret Cp conc + 10% Zn (NO₃)₂·6H₂O (on mass of resin)
 B = 9.1% resin

* 8% Stretch applied to the fabrics

TABLE III
THE EFFECT OF RESIN TREATMENT ON THE PHYSICAL PROPERTIES OF
UNMERCERISED AND MERCERISED TWILL FABRICS

Treatment	PHYSICAL PROPERTIES																		
	Crease Recovery Angle (degrees)		Bursting Strength (kN/m ²)		Flex Abrasion (cycles to rupture)			Flat Abrasion (% mass loss)		Tear Strength (N)			Breaking Strength (N)						
	A	B	A	B	A	B	A	B	A	B	A	B	A	B	A	B			
			Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft			
Untreated Control	187		1362.3		1577	565				57.3	37.1			966.3	518.4				
Untreated Control + Resin	249	287	931.0	735.0	652	558	652	291	4.7	11.7	24.4	17.2	14.0	9.8	697.8	301.8	528.2	237.2	
Untreated Stretched Control + Resin	263	288	1029.0	842.0	-	-	-	-	8.3	18.6	15.2	10.1	13.4	9.4	665.4	358.7	591.9	282.2	
Slack Mercerised + Resin	263	295	980.0	784.0	1080	997	-	-	2.1	3.4	23.0	20.3	14.8	12.3	701.7	406.7	558.6	296.0	
NH ₃ + Heat* removal of Ammonia + Resin	256	281	1303.4	1009.4	1010	444	262	426	3.0	5.4	30.8	19.2	23.6	13.7	786.9	301.8	699.7	220.5	
NH ₃ + Cold* Water Removal of Ammonia + Resin	232	275	1362.2	1068.2	703	367	488	185	2.2	5.2	-	-	-	21.1	13.1	963.3	310.7	796.7	247.9
NH ₃ + Hot* Water Removal of Ammonia + Resin	243	274	-	-	536	653	506	547	3.1	5.4	31.3	27.0	24.1	14.5	906.5	297.9	800.7	246.0	
NaOH + Resin	253	281	1117.2	823.0	869	1221	180	56	4.8	19.9	24.6	19.1	14.4	10.5	985.0	360.6	694.8	233.2	

A = 4.5% resin / Aerotex M₁ + Fixapret CP_{conc} + 10% Zn (NO₂)₂·6H₂O (on mass of resin)
 B = 9.1% resin

* 8% Stretch applied to the fabrics

fabrics, however, generally reduced the strength losses significantly. Liquid ammonia mercerisation gave a significantly higher retention of fabric strength than sodium hydroxide mercerisation. In general, the crease recovery angles of the liquid ammonia mercerised fabrics were slightly lower than those of the sodium hydroxide treated fabrics. Little difference was observed between the physical properties of the samples whether cold or hot water was used to remove the ammonia from the fabrics. Heat removal of the ammonia gave slightly higher crease recovery angles than the removal of ammonia by cold or hot water, but it did, however, give slightly lower breaking strength values than the water removal treatments.

Recently Vail *et al*⁷ also found that the usual strength and abrasion losses of durable press cotton chambray can be partially reduced through the use of ammonia mercerisation prior to cross-linking. They found that the liquid ammonia mercerised fabrics had higher wet wrinkle recovery angles, higher resistance to abrasion as measured by the Stoll flex method, higher tear strength, higher moisture regain, lower stiffness but lower breaking strength. The DP appearance ratings of the chambray also were increased by the ammonia mercerisation.

In all the resin treatments employed so far, a mixture of a DMDHEU and a melamine type resin was used. It was also decided to investigate the effect of liquid ammonia mercerisation on the performance of other types of aminoplast resins. Fabrics were, therefore, treated with 10 per cent DMEU and DMDHEU resins, with $Zn(NO_3)_2 \cdot 6H_2O$ and a mixture of $MgCl_2$ /citric acid used as catalysts. The results obtained are given in Table IV.

It seemed that, for the lightweight plain fabric, the DMDHEU resin gave the highest crease recovery angles and the biggest losses in fabric bursting strength. With both resins the $MgCl_2$ /citric acid catalyst gave somewhat lower crease recovery angles, and slightly lower bursting strength losses than did the $Zn(NO_3)_2 \cdot 6H_2O$ catalyst. On average, liquid ammonia and sodium hydroxide mercerisation had little effect on the crease recovery angles and the bursting strength of the resin-treated fabrics when compared with the unmercerised resin-treated fabric. In the case of the heavyweight plain and the twill fabrics, however, mercerisation of the fabrics resulted in improved bursting strength retention after resin treatment, together with higher crease recovery angles. The pretreatment with liquid ammonia gave significantly better results than the sodium hydroxide pretreatment. The DMDHEU resin, once again, gave higher crease recovery angles than the DMEU resin, but also lower bursting strength values. Liquid ammonia mercerisation increased the crease recovery angle of the heavyweight plain fabric by about 5 per cent while sodium hydroxide mercerisation decreased it by about 2 per cent. For the twill fabric, liquid ammonia mercerisation did not increase the crease recovery angles when compared with the resin-treated control fabric, while sodium hydroxide mercerisation again resulted in lower crease recovery angles. The average

bursting strength of the liquid ammonia treated fabrics was about 50 *per cent* higher than that of the unmercerised resin-treated fabric for both the DMEU and DMDHEU resins. The bursting strength of the sodium hydroxide treated fabrics, however, was only about 10 *per cent* higher than that of the unmercerised resin-treated fabric for the DMEU resin and about 40 *per cent* higher for the DMDHEU resin.

SUMMARY AND CONCLUSIONS

Samples of unmercerised and mercerised cotton fabrics were treated with various resins and catalysts to study the effect of liquid ammonia mercerisation as a pretreatment on the properties of durable press treated cotton fabrics. Two durable press treatments were used. Similar fabrics were also mercerised with sodium hydroxide on an industrial scale for purposes of comparison.

Excessive strength losses were found for the unmercerised resin-treated fabrics but these were reduced significantly by mercerisation. Liquid ammonia mercerisation, however, reduced the strength losses to a greater extent than sodium hydroxide mercerisation, while maintaining a higher level of durable press performance.

The average bursting strength of the resin-treated fabrics was increased after a liquid ammonia pretreatment when compared with the unmercerised resin-treated fabrics. This was especially true for the two heavier fabrics. The same applies for the flex abrasion properties of these fabrics. The percentage mass loss of these fabrics during flat abrasion was reduced significantly by a liquid ammonia pretreatment, especially when a higher level of resin was applied to the fabrics. The tear strength and breaking strength of the resin-treated fabrics were increased after a liquid ammonia pretreatment when compared with the unmercerised resin-treated fabrics. The average strength loss found for the unmercerised resin-treated fabrics was about 47 *per cent*. A pretreatment with liquid ammonia and sodium hydroxide reduced this strength loss to about 34 and 39 *per cent*, respectively. The crease recovery angles of the ammonia pretreated fabrics were also found to be slightly higher than those of the sodium hydroxide pretreated fabrics.

Removal of the ammonia from the fabrics by heat resulted in the largest crease recovery angles after durable press treatment but resulted in a slightly lower strength retention than when the ammonia was removed by water. Little difference, as far as strength retention was concerned, was observed between cold and hot water removal of ammonia from the fabrics. Although the effect of cold or hot water removal of the ammonia from the fabrics was small, it must again be emphasised that this machine was running at a relatively slow speed. At higher production speeds hot water may be beneficial for the removal of the ammonia due to the better quenching action that can be obtained with hot water.

TABLE IV
THE EFFECT OF MERCERISATION ON THE BURSTING STRENGTH AND CREASE RECOVERY ANGLES
OF COTTON FABRICS TREATED WITH DIFFERENT AMINOPLAST RESINS AND CATALYSTS

Fabric	Treatment	DMEU				DMDHEU			
		Zn(NO ₃) ₂ ·6H ₂ O		MgCl ₂ /Citric Acid		Zn(NO ₃) ₂ ·6H ₂ O		MgCl ₂ /Citric Acid	
		Crease Recovery Angle (degrees)	Bursting Strength (kN/m ²)	Crease Recovery Angle (degrees)	Bursting Strength (kN/m ²)	Crease Recovery Angle (degrees)	Bursting Strength (kN/m ²)	Crease Recovery Angle (degrees)	Bursting Strength (kN/m ²)
Lightweight Plain Fabric	Untreated Control	184	852.6						
	Untreated Control + Resin	288	411.6	273	450.8	304	323.4	287	382.2
	**NH ₃ Mercerised + Resin	293	392.0	265	431.2	308	294.0	293	372.4
	NaOH Mercerised + Resin	283	441.0	259	450.8	301	313.6	286	411.6
Heavyweight Plain Fabric	Untreated Control	185	1499.4						
	Untreated Control + Resin	265	891.8	246	1087.8	286	705.6	271	862.4
	**NH ₃ Mercerised + Resin	272	1313.2	264	1489.6	293	1058.4	280	1274.0
	NaOH Mercerised + Resin	260	999.6	238	1176.0	283	735.0	265	1019.2
Twill Fabric	Untreated Control	195	999.6						
	Untreated Control + Resin	279	519.4	261	666.4	297	411.6	288	450.8
	**NH ₃ Mercerised + Resin	277	823.2	266	950.6	303	597.8	280	813.4
	NaOH Mercerised + Resin	269	754.6	243	833.0	299	519.4	—	725.2

** Ammonia removed by hot water

To summarise, it can be concluded that cotton fabrics pretreated with liquid ammonia retained more strength and a better durable press performance after durable press treatments than did fabrics pretreated with sodium hydroxide.

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THE USE OF PROPRIETARY NAMES

The fact that proprietary names have been mentioned in this report does not in any way imply that SAWTRI recommends them or that there are not substitutes which may be of equal value or even better.

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