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Liquid Ammonia Mercerisation of Cotton

Part VII: Liquid Ammonia Treatment of 67/33 Cotton/Wool Blended Fabrics

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LIQUID AMMONIA MERCERISATION OF COTTON PART VII: LIQUID AMMONIA TREATMENT OF 67/33 COTTON/WOOL BLENDED FABRICS

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ABSTRACT

The effect of a liquid ammonia treatment on certain properties of 67/33 cotton/wool, all-cotton and all-wool fabrics was investigated. It was found that the treatment reduced the shrinkage of all-cotton fabrics during washing, but this effect was less significant in the case of cotton/wool blends, while no effect was observed on the all-wool fabric. A similar trend was observed in the case of the physical properties of the treated fabrics, viz. a decrease in the improvement of certain properties from all-cotton to cotton/wool and all-wool fabrics. Finally, a cost analysis for a caustic soda and a liquid ammonia mercerisation process is appended.

INTRODUCTION

The utilisation of anhydrous liquid ammonia as a processing solvent in the textile field is becoming more popular[1]. Liquid ammonia is not only used as a swelling agent for mercerisation treatments, but also as a solvent for dyeing[2-4]. Much has been reported about its beneficial effect on the properties of cotton in enhancing strength, lustre and in decreasing shrinkage during washing. The well-known flat and convoluted shape of a mature and dry cotton fibre lends itself ideally to the primary action of a swelling agent such as liquid ammonia.

A wool fibre, on the other hand, is also affected by liquid ammonia but in a different way than is the case with a cotton fibre. The bulking of wool fibres with liquid ammonia, first disclosed by Omnium de Prospective Industriel (OPI)[5], and the treatment of wool sliver in liquid ammonia forms the basis of a new process (developed by Sitralaine of Tourcoing, France) for bulking of carpet wool. Pitts[6] investigated the effect of liquid ammonia on the wool fibre and suggested that the differential contraction of the cortical components in the fibre was responsible for the bulking phenomenon. When a wool sliver was immersed in liquid ammonia for a few minutes and then removed, a contraction and consolidation of the fibres occurred upon evaporation of the ammonia. Pitts furthermore found that the phenomenon of supercontraction of wool in liquid ammonia was characterised by a crimp reversal with the ortho- and paracortices changing position as a consequence of their differential contraction. This reversal was found to be accompanied by a substantial increase in the fibre crimp frequency and associated bulk compressibility. It was suggested that the treatment of wool with liquid ammonia resulted in the replacement of free and bound water within the fibre structure by ammonia. When the fibres were removed from the ammonia, the reverse process occurred and the fibres supercontracted. The extent of this supercontraction was limited by the disulphide bonds which were found to be relatively unaffected by liquid ammonia. It was shown that the reduction of wool either before or during the liquid ammonia treatment increased the degree of supercontraction of the wool. It was also found that fine wool contracted more in liquid ammonia than coarse wool, most likely due to the more pronounced bilateral structure of fine merino fibres[6].

The purpose of this study was to investigate the effect of a liquid ammonia treatment on certain properties of a cotton/wool blended fabric and an all-wool fabric. Furthermore the economics of a liquid ammonia treatment is compared with that of a conventional sodium hydroxide mercerisation treatment.

EXPERIMENTAL

A lightweight 67/33 cotton/wool 2/2 twill weave fabric (mass/unit area 140 g/m^2), a heavyweight 67/33 cotton/wool 2/2 twill weave fabric (280 g/m^2), an all-wool 3/2 twill step-2 wool worsted fabric (268 g/m^2) and a plain weave all-cotton fabric (185 g/m^2) were used in this investigation. The lightweight blended fabric was divided into two lots. One lot was crabbed and scoured to remove the size from the fabric before the liquid ammonia treatment. The fabric shrank about 10 *per cent* in area during crabbing and scouring. The other lot as well as the heavyweight blended fabric, which had not been sized, were treated in the loomstate form. The cotton fabric was desized commercially. The wool fabric was crabbed, scoured and decatised. The various fabrics were treated with liquid ammonia on the SAWTRI liquid ammonia merceriser as described previously[7]. The fabrics were stretched either four or eight *per cent* in the warp direction, the contact time in liquid ammonia was 15 seconds and the ammonia was removed from the fabrics by infra-red heat or hot water (at approximately 75°C). The effect of the liquid ammonia treatment on the shrinkage and dimensional stability of the fabrics during the liquid ammonia treatment and after washing in a Cubex apparatus, as well as the various physical properties, were determined in the usual manner[8, 9].

TABLE I

AREA SHRINKAGE OF COTTON/WOOL, COTTON AND WOOL FABRICS AFTER LIQUID AMMONIA AND SUBSEQUENT WASHING TREATMENTS

Fabric	Stretch (%)	NH_3 Removed by	Area Shrinkage + (%) After			
			NH_3	Relaxation	60' Wash	120' Wash
100% Cotton (185 g/m²)	Control			10,0	12,8	12,4
	4	Heat	- 2,2	5,0	6,0	7,0
	4	Hot water	0,9	5,5	4,8	6,2
	8	Heat	- 3,6	6,5	6,7	7,2
	8	Hot water	- 1,5	2,9	5,3	5,3
Lightweight 67/33 Cotton/Wool (140 g/m²) (loomstate)	Control			20,2	18,8	18,6
	4	Heat	5,7	13,8	13,3	12,0
	4	Hot water	6,0	10,1	9,2	9,7
	8	Heat	6,3	14,6	12,1	12,9
	8	Hot water	6,7	15,2	9,0	9,7
Lightweight 67/33 Cotton/Wool (Crabbed and Scoured)	Control			16,1	16,3	16,4
	4	Heat	12,0	14,6	14,1	12,8
	4	Hot water	12,2	12,7	12,5	13,0
	8	Heat	10,6	14,8	14,0	15,7
	8	Hot water	11,7	11,7	12,2	14,9
Heavyweight 67/33 Cotton/Wool (280 g/m²) (loomstate)	Control			13,8	15,2	15,6
	4	Heat	5,0	8,8	10,3	10,6
	4	Hot water	5,1	5,5	6,5	7,0
	8	Heat	0,7	3,4	9,9	10,7
	8	Hot water	3,5	5,0	4,8	4,8
100% Wool (268 g/m²)	Control			3,8	18,4	24,8
	4	Heat	0,3	4,3	18,0	26,8
	4	Hot water	2,9	2,4	15,9	26,7
	8	Heat	- 1,9	4,3	18,6	28,0
	8	Hot water	6,0	4,1	16,5	26,1

+ Based on the original dimensions of the fabric.

RESULTS AND DISCUSSION

The area shrinkage values of the fabrics after the liquid ammonia treatment and subsequent washing tests are given in Table I. Once again it can be seen that the liquid ammonia reduced the shrinkage of the all-cotton fabric during washing, as found before[8]. The degree of shrinkage of the lightweight cotton/wool fabric was also reduced by the liquid ammonia treatment. The average shrinkage values, however, were higher than that of the all-cotton fabric, probably due to the presence of the wool fibres. It is furthermore clear that the total area shrinkage of the lightweight blended fabric treated with liquid ammonia in the loomstate form was lower than that of the crabbed and scoured fabric treated with liquid ammonia. The liquid ammonia treatment also considerably reduced the degree of shrinkage of the heavyweight blended fabric during washing when compared with the untreated, washed fabric. It is furthermore clear that the liquid ammonia treatment had no effect on the degree of shrinkage of the all-wool fabric. In general, removal of the ammonia from the treated fabrics by water resulted in lower shrinkage values during washing than removal of the ammonia by heat. The degree of stretch applied to the fabrics had no effect on the shrinkage of the fabrics during washing. It can therefore be stated that, in contrast to cotton fabrics, liquid ammonia appears to have no effect on the dimensional stability of all-wool fabrics and that the stabilisation effect of liquid ammonia would most likely decrease as the wool content of a cotton/wool blend increases.

Some physical properties of the untreated and liquid ammonia treated fabrics are given in Table II. The results were analysed statistically (by Analysis of Variance). The analysis showed that the liquid ammonia treatment had no effect on the breaking strength of any of the fabrics. The ammonia treatment furthermore had little effect on the breaking extension of the fabrics but did, however, tend to decrease the breaking extension of the all-cotton fabric, while it increased that of the lightweight blend (loomstate) slightly when compared with the untreated fabrics. It is also clear from Table II that the ammonia treatment increased the bursting strength of the all-cotton fabric, while it decreased that of the lightweight blended fabrics. The ammonia treatment generally had a smaller effect on the bursting strength of the heavyweight blend and the all-wool fabrics. The analysis furthermore showed that the all-cotton and cotton/wool fabrics treated with liquid ammonia had a *higher tear strength* than the untreated fabrics, while no effect was observed in the case of the all-wool fabric. Removal of the ammonia by heat resulted in higher tear strength values than removal by water in the case of the all-cotton and lightweight (crabbed and scoured) blended fabrics. The opposite was found for the lightweight blend (loomstate) while no differences were observed in the case of the heavyweight blend and all-wool fabrics.

TABLE II
PHYSICAL PROPERTIES OF COTTON/WOOL, COTTON AND WOOL FABRICS AFTER A TREATMENT WITH LIQUID AMMONIA

Fabric	Stretch (%)	NH_3 Removed By	Physical Properties								
			Breaking Strength (N)	Breaking Extension (%)	Bursting Strength (kN/m²)	Tear Strength (N)	Flex Abrasion (cycles to rupture)	Flat Abrasion % Mass Loss After 5000 Cycles	Flat Abrasion % Mass Loss After 10 000 Cycles	Bending Length (cm) (Stiffness)	Monsanto Crease Recovery Angle (°) (W + F)
100% Cotton (185 g/m²)	Control		642	12,4	1170	20,7	941	2,6	4,4	2,25	162
	4	Heat	622	10,8	1316	28,8	2105	2,5	4,2	2,43	206
	4	Hot water	641	11,8	1308	20,6	1141	3,1	4,7	2,71	185
	8	Heat	613	10,5	1334	28,6	1964	2,1	4,3	2,18	202
	8	Hot water	654	11,0	1438	25,5	1310	2,3	3,7	2,13	164
Lightweight 67/33 Cotton/Wool (140 g/m²) (loomstate)	Control		346	9,4	951	35,7	714	7,2	10,7	2,07	236
	4	Heat	390	11,0	814	32,8	829	4,6	7,7	2,48	245
	4	Hot water	322	10,1	888	41,8	839	7,0	9,2	1,55	245
	8	Heat	402	12,6	799	37,5	984	5,0	7,7	2,32	243
	8	Hot water	344	10,6	878	43,0	782	5,9	10,1	1,50	258
Lightweight 67/33 Cotton/Wool (Crabbed and Scoured)	Control		346	12,1	974	35,4	1103	5,9	9,0	1,45	236
	4	Heat	359	12,4	727	41,2	980	5,6	8,6	1,50	262
	4	Hot water	339	11,5	778	36,4	832	6,4	9,2	1,56	230
	8	Heat	335	12,1	672	41,4	848	6,6	11,3	1,48	248
	8	Hot water	325	11,4	721	41,9	921	8,0	12,0	1,55	247
Heavyweight 67/33 Cotton/Wool (280 g/m²) (loomstate)	Control		760	19,2	1669	33,1	4352	1,7	2,6	2,49	247
	4	Heat	732	17,0	1947	38,1	4768	1,7	2,3	1,93	246
	4	Hot water	809	18,3	1888	37,7	4574	1,9	2,3	2,07	248
	8	Heat	783	16,7	1621	37,1	4186	2,0	2,5	2,02	252
	8	Hot water	786	17,0	1731	38,7	5180	2,0	2,4	1,94	247
100% Wool (268 g/m²)	Control		414	37,3	971	25,8	3229	2,0	3,2	1,69	313
	4	Heat	433	36,0	997	25,8	3102	2,4	2,8	1,73	288
	4	Hot water	432	38,1	984	26,4	3733	2,6	3,3	1,74	311
	8	Heat	443	36,5	1022	27,5	2546	1,9	2,8	1,75	315
	8	Hot water	418	37,1	979	27,5	3151	2,5	3,1	1,69	311

The resistance to flex abrasion of the all-cotton, lightweight blended fabric (loomstate) and the heavyweight blended fabric was improved by the ammonia treatment while that of the crabbed and scoured lightweight blend was decreased. The ammonia treatment had no significant effect on the resistance to flex abrasion of the all-wool fabric. The different degrees of stretch which were applied to the fabrics had no effect on their flex abrasion resistance, but the manner in which the ammonia was removed influenced this fabric property. Removal of the ammonia by heat resulted in a significantly higher resistance to flex abrasion than removal by hot water on the all-cotton fabric, but in lower values in the case of the heavyweight blend and all-wool fabrics. No differences were observed, however, in the case of the two lightweight blended fabrics. The analysis showed that the ammonia treatment had no significant effect on the resistance of the fabrics to flat abrasion. The ammonia treatment furthermore tended to increase the bending length of the fabrics, but this increase was found to be less than five *per cent* The lower degree of stretch produced slightly higher bending length values than the higher degree of stretch.

Ammonia treatment increased the crease recovery angles of the all-cotton and the lightweight blended fabrics, but had little effect on that of the heavyweight blend and all-wool fabrics. Removal of the ammonia by heat generally gave higher crease recovery angles than removal by hot water in the case of the all-cotton and cotton/wool blends, whereas the opposite was found for the all-wool fabric. The lower degree of stretch also produced higher crease recovery angles than the higher degree of stretch in the case of the all-cotton and lightweight blended fabrics, and in lower angles in the case of the all-wool and heavyweight blended fabrics.

SUMMARY

Light- and heavyweight 67/33 cotton/wool blended fabrics, an all-cotton and an all-wool fabric were treated with liquid ammonia on the SAWTRI liquid ammonia merceriser to establish the effect of liquid ammonia on the dimensional stability and certain physical properties of these fabrics.

As far as the dimensional stability of the fabrics was concerned the best results were obtained on the all-cotton fabrics. When the cotton content of the fabric was reduced by substitution with wool, the effectiveness of liquid ammonia in reducing the shrinkage of the fabrics during washing was reduced. The liquid ammonia treatment had no effect on the degree of shrinkage of the all-wool fabric during washing.

A statistical analysis of certain physical properties of the fabrics showed that the liquid ammonia treatment did not affect the breaking strength of the fabrics. Furthermore it had a relatively small effect on the breaking extension of the fabrics. The bursting strength of the all-cotton fabric was increased

significantly by the liquid ammonia treatment, whereas no improvement was observed on the fabrics containing wool. The tear strength of the all-cotton and the cotton/wool fabrics was improved by the liquid ammonia treatment, but that of the all-wool fabric was unaffected. The liquid ammonia treatment considerably increased the resistance of the all-cotton fabric to flex abrasion and that of the lightweight blended (loomstate) fabric and heavyweight blended fabric to a lesser extent, while it decreased that of the crabbed and scoured lightweight blend slightly. No effect was observed in the case of the all-wool fabric.

Ammonia treatment had no significant effect on the resistance of the fabrics to flat abrasion. Treatment, in general, increased the stiffness of the fabrics but this increase was found to be less than five *per cent*. Crease recovery angles of the all-cotton and lightweight blended fabrics were increased by the liquid ammonia treatment while it had no effect on the crease recovery angles of the heavyweight blend and all-wool fabrics.

In conclusion it can be stated that liquid ammonia had a beneficial effect on the properties of a fabric composed of cotton fibres only, whereas this effect was not so marked on a cotton/wool blended fabric. As far as dimensional stability and certain physical properties were concerned, no advantage was gained by treating an all-wool fabric with liquid ammonia.

Cost analyses are appended for caustic soda and liquid ammonia mercerisation processes. It was found that liquid ammonia mercerisation of fabrics can be economically viable compared to the caustic soda process when the ammonia is recovered and recycled for re-use.

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REFERENCES

1. Lewin, M., Rau, R.O. and Sello, S.B., The Role of Liquid Ammonia in Functional Textile Finishes. *Text. Res. J.*, **44**, No. 1, 680 (Sept., 1974).
2. Anon, The RAM System. *Colourage,* **22**, No. 19, 25 (Sept., 1975).
3. Kane, S.M., Liquid Ammonia — New Solvent for Dyeing. *Am. Dyestuff Rep.,* **62**, No. 5, 27 (1973).

4. Wade, R.H. and Creely, J.J., Application of Non-Traditional Dyes for Cotton from Liquid Ammonia. Proceedings of the 14th Textile Chemistry and Processing Conference (U.S. Department of Agriculture), New Orleans, 71-72 (1975).
5. Omnium de Prospective Industriel S.A. French P 72 08875 (1972).
6. Pitts, J.M.D., The Treatment of Wool in Liquid Ammonia. *J. Text. Inst.*, **67**, No. 1, 12 (1976).
7. Hanekom, E.C. and Barkhuysen, F.A., Liquid Ammonia Mercerisation of Cotton. Part I: Construction of a Pilot Plant Chainless Merceriser. *S. African Wool & Text. Res. Inst.* Techn. Rep. No. 277 (Dec., 1975).
8. Barkhuysen, F.A., Liquid Ammonia Mercerisation of Cotton. Part II: The Influence of Anhydrous Liquid Ammonia on the Dimensional Stability of Cotton Fabrics. *S. African Wool & Text. Res. Inst.* Techn. Rep. No. 286 (March, 1976).
9. Barkhuysen, F.A., Liquid Ammonia Mercerisation of Cotton. Part III: The Influence of Anhydrous Liquid Ammonia on the Physical Properties of Cotton Fabrics. *S. African Wool & Text. Res. Inst.* Techn. Rep. No. 289 (March, 1976).

APPENDIX

Cost analyses have been carried out to compare a conventional caustic soda mercerisation process with several variations of a liquid ammonia process. By calculating the selling price per metre for the treated fabric in order to achieve a "break-even" price it is possible to estimate the relative economic viability of the several variants. Because many assumptions have been made for various items, the break-even values must be used for illustrative purposes only.

The basis of the analysis was to calculate a break-even price for the treated fabric to give a 10 *per cent* return on capital during a five year period (the effects of inflation were not taken into account) and to permit full depreciation over five years. A tax rate of 40 *per cent* has been assumed.

The following assumptions are made:

1. The fabric is one metre wide, costing one Rand per metre and weighing 250 g/m^2.
2. The processing speed is constant at 35 m/min.
3. Working time per year is 2 x 8 hour shifts per day, 250 days per year.
4. From the above it follows that 8 400 metres of fabric are processed per year.
5. One kilogram of a 19 *per cent* caustic soda solution or of liquid ammonia is taken up by one kilogram of fabric (i.e. 100 *per cent* wet pick-up).

6. Caustic soda in bulk costs 39 c/kg and liquid ammonia 28 c/kg.
7. Water consumption for (a) rinsing out the caustic soda after impregnation or (b) water removal of the ammonia from the fabric is assumed to be 6,5 ℓ/kg of fabric. The effect of half this consumption for the ammonia process is also examined since liquid ammonia is much easier to remove than caustic soda.
8. Water consumption to prepare the caustic soda solution is taken as 0,81 ℓ/kg of solution.
9. Water cost at 0,014 c/ℓ, is assumed.
10. Power consumption is assumed equal and constant for each process and is therefore neglected.
11. Manpower is assumed to be two operators for each process at a labour rate of R2,00 per hour.

The accompanying table (all values expressed as kilo Rand unless otherwise indicated) shows the analyses for the caustic soda process and for twelve variants of the ammonia process. These are:

I	: A caustic soda process with no recovery of caustic soda.
II	: A caustic soda process with 90 *per cent* recovery of caustic soda for comparison with 90 *per cent* recovery of liquid ammonia.
A — F	: Water removal of residual ammonia from the fabric with no recovery of ammonia from the water.
B, D & F	: Water used at the same rate as for the caustic soda process for rinsing the fabric.
A, C & E	: Water used at half the rate as for the caustic process.
A & B	: Capital outlay equal to the caustic soda process (R500 000).
C & D and E & F	: Capital outlay of R200 000 and R100 000, respectively.
G, H & I	: Heat removal of the residual ammonia with no recovery of this ammonia. G allows for R500 000, H allows for R200 000 and I allows for R100 000 for the ammonia plant.
J, K & L	: Heat removal of the residual ammonia with 90 *per cent* recovery of ammonia. All three allow for R500 000 for recovery plant. J allows for R500 000, K allows for R200 000 and L allows for R100 000 for the ammonia plant.

Note: In the cases where the ammonia is not recovered, the cost of an effluent plant has been included in the cost of the ammonia plant. Furthermore, the lay-out of the Table is such that the influence of changing any of the costs can be made easily and quickly.

COSTING ANALYSIS OF CAUSTIC SODA AND LIQUID AMMONIA MERCERISATION PROCESSES

Investments	Mercerisation Process													
	Caustic Soda		Liquid Ammonia											
	I	II	A	B	C	D	E	F	G	H	I	J	K	L
Capital (C)	500	500+100	500	500	200	200	100	100	500	200	100	500+500	200+500	100+500
Raw Fabric	8400	8400	8400	8400	8400	8400	8400	8400	8400	8400	8400	8400	8400	8400
Chemicals	155,6	15,56	588	588	588	588	588	588	588	588	588	58,8	58,8	58,8
Water	214,9	214,9	95,5	191,1	95,5	191,1	95,5	191,1	0	0	0	0	0	0
Labour	16	16	16	16	16	16	16	16	16	16	16	16	16	16
Value Added	8786,5	8646,46	9099,5	9195,1	9099,5	9195,1	9099,5	9195,1	9004	9004	9004	8474,8	8474,8	8474,8
0,573 x C*	286,5	343,8	286,5	286,5	114,6	114,6	57,3	57,3	286,5	114,6	57,3	573	401,1	343,8
Break Even before Tax	9073,0	8990,26	9386	9481,6	9214,1	9309,7	9156,8	9252,4	9290,5	9118,6	9061,3	9047,8	8875,9	8818,6
Break Even before Tax per Metre (Rand/Metre)	1,080	1,070	1,118	1,129	1,097	1,108	1,090	1,101	1,106	1,086	1,078	1,077	1,057	1,049

*This allows for depreciation over 5 years and a return on capital of 10% for 10 years after tax at 40% is allowed.

Some conclusions that can be drawn from these analyses of process costs are:

1. The caustic soda process considered here costs 8 c/metre. The recovery of caustic soda reduces these costs by 1 c/metre.
2. The various liquid ammonia processes increase the processing costs by 4,9 c/metre in the lowest case of ammonia recovery and by 12,9 c/metre in the worst case of non recovery of ammonia.
   the ammonia process or higher possible production speeds, then the ammonia process only shows to advantage when the ammonia is *recovered* and recycled for re-use.
4. The influence of capital costs can be assessed: a reduction in capital outlay from R500 000 to R200 000 makes an approximate difference of 2 c/metre.
5. It is clear from Columns II and L, considering the same capital outlay and the same percentage recovery of the two chemicals, that the liquid ammonia process is about 2,1 c/metre cheaper than the caustic soda process.
6. In the cases where the ammonia is not recovered (A — I) it can be calculated that the destruction of the evaporated ammonia gas by absorption in water, as in the scrubbing tower used at SAWTRI using water at a rate of 28 000 ℓ/hour, will add another 0,1 c/metre onto the costs due to the extra water consumption and not taking recycling of this water into account.
7. It is believed from these analyses that, based as they are on certain assumptions and estimates, liquid ammonia mercerisation of fabric could be economically viable compared to the caustic soda process.

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page 8: Under appendix, assumption no. 4:

For 8 400 metres, please read 8 400 000 metres.