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154

SHRINK-RESIST TREATMENT OF WOOL FABRICS WITH AN ALKYLATED METHYLOLMELAMINE RESIN

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# SHRINK-RESIST TREATMENT OF WOOL FABRICS WITH AN ALKYLATED METHYLOLMELAMINE RESIN

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## **ABSTRACT**

Adequate shrink-resist properties were imparted to woven and single and double jersey wool fabrics by the application of methylolmelamine — polyethylene — and acid colloid methylolmelamine — polyethylene solutions to the fabrics. Chlorinatión of the fabrics prior to the application of the resin mixtures was found to be a prerequisite for obtaining the required results. Fabrics treated with the acid colloid resin — polyethylene mixtures gave the best shrink-resist results. Good shrink-resist results were obtained with a resin add-on as low as 0,5% (o.w.f.). The handle of the acid colloid treated fabrics did not deteriorate to any significant extent.

### **KEY WORDS**

Wool fabrics; alkylated methylolmelamine; lactic acid; sodium dichloroisocyanurate; polyethylene; felting shrinkage; relaxation shrinkage; shrink-resist treatment; acid colloid; chlorination; curing; washing test.

# INTRODUCTION

The application of polymers to wool for the prevention of felting shrinkage has been the subject of extensive investigations during the last decade. The treatment of wool fabrics with melamine-formaldehyde condensates was one of the earliest methods which was used to impart shrink-resist properties to wool<sup>(1-6)</sup>. A considerable amount of work was done on this type of resin which led to the patenting of several processes, for example the "Lanaset" process. The use of aminoplast resins for the shrinkproofing of wool has, however, never been accepted to any large extent commercially, mostly because of the adverse effect the treatment had on the handle of the wool fabrics.

The present investigation was undertaken to establish whether wool fabrics could effectively be rendered shrink-resistant by the application of an alkylated methylolmelamine resin without harshening the handle of the fabrics to any significant extent.

## **EXPERIMENTAL**

## **Materials**

The following unfinished wool worsted fabrics were used in this study: IWS Standard woven material (density  $150 \text{ g/m}^2$ ), Punto-di-Roma double jersey (density

419 g/m²) made from R32 tex yarn, 1 × 1 rib double jersey (density 561 g/m²) and plain single jersey (density 294 g/m²) knitted from R72 tex/2 yarn. The knitted fabrics treated with the acid colloid solutions were Punto-di-Roma (density 384 g/m²), 1 × 1 rib (density 524 g/m²) and single jersey (density 274 g/m²) made from R30 tex yarn. Samples from these fabrics (approximately 30 × 30 cm) were used in the experiments. The sample fabrics were dry cleaned with perchlorethylene before they were treated.

Aerotex M-3 resin, a methylated melamine — formaldehyde condensate, was supplied by Cyanamid as a 80% aqueous solution. The catalyst used was Aerotex Accelerator No. 5 (Cyanamid), a zinc nitrate solution. Sodium dichloroisocyanurate, Fichlor 60S (Fisons), was used as the chlorinating agent. Two surfactants were used, the nonionic surfactant Tergitol Speedwet (Union Carbide) and the anionic surfactant Nansa HS 80 (Marchon Products). The polyethylene emulsion used in the experiments was Mystolube S (30% solids) from Catomance. Laboratory grade lactic acid (87,5%) was used in the preparation of the acid colloid solutions.

Chlorination of sample fabrics

The fabrics were treated at room temperature in a solution containing 10% sodium sulphate, on weight of fabric (o.w.f.), wetting agent (Nansa HS 80) and the required amount of chlorinating agent (Fichlor 60S) to chlorinate the fabrics to a 1% (o.w.f.) level. The pH of the solution was adjusted to 4,5–5,0 by the addition of acetic acid. A liquor to goods ratio of 13:1 was used. After 55 minutes the fabrics were dechlorinated for 30 minutes in a 3% sodium bisulphite solution at room temperature, rinsed and dried.

# Preparation of Acid Colloid Resin Solution

A 10% resin colloid stock solution was prepared by thoroughly mixing Aerotex M-3 resin solution (125 g) with water (835 g) and then slowly adding lactic acid (40 g) whilst stirring. The mixture was then left to age for 16 hours at room temperature. The acid colloid solutions have a characteristic bluish-white translucent appearance. Acid colloid solutions were also prepared by heating the mixture in a water bath for 40 minutes at 40°C instead of aging for 16 hours.

# Shrink-resist treatments of the fabrics

# (a) With resin solutions

The fabric samples were padded to a wet pick-up of 75–100% in the various solutions containing 2–8% Aerotex M-3 resin, 0–4% softener (Mystolube S), Accelerator No. 5 (27% of weight of Aerotex M-3 resin, based on "as received" material) and 0,2% wetting agent (Tergitol Speedwet). The fabric samples were partially dried in the air, oven dried for 5–10 minutes at 90–100°C, and finally cured at 130°C for 10–15 minutes.

# (b) With acid colloid resin solutions

The fabric samples were padded to a wet pick-up of 75-100% in solutions containing 0.5-3% resin colloid, 0-1.5% softener (Mystolube S) and 0.2% wetting agent (Tergitol Speedwet). After air drying, the fabrics were cured for 12 minutes at  $120^{\circ}$ C.

# Measurement of felting shrinkage

The percentage decrease in length and width of a square approximately 21 × 21 cm marked on each fabric sample was measured after washing in a 50 litre Cubex machine according to the Australian Wool Board's specifications for machine washability<sup>(7)</sup>.

## RESULTS AND DISCUSSION

It is well known that most resins are relatively ineffective in preventing wool from felting unless the wool is given some form of pretreatment<sup>(1, 2)</sup>. As a result of

TABLE I
SHRINKAGE OF STANDARD MATERIAL TREATED WITH AEROTEX M-3
RESIN MIXTURES

%Polymer on Fabric (o.w.f.)	Chlorination Pretreatment	% Shrini 3 minute	kage after s washing	% Shrinkage after 48 minutes washing	
		Length	Width	Length	Width
0	_	3,8	4,3	44,0	46,2
0	+	2,4	3,8	22,7	25,4
4 A	_ ,	1,0	0,5	31,6	33,0
8 A	<u>-</u>	1,0	1,4	21,1	41,2
4 A	+	1,0	1,4	3,3	3,8
8 A	+	1,0	1,4	2,9	2,9
4 A + 1 M	+	_	_	2,3	2,3
4 A + 2 M	+	_	****	3,3	1,9
4 A + 3 M	+	_	<u> </u>	2,3	2,3
4 A + 4 M	+	_		1,4	1,9
3 A + 1 M	+	_		2,3	1,9
3 A + 2 M	+	_	_	2,8	2,8
2 A + 1 M	+		_	6,1	7,5

A – Aerotex M-3 resin M – Mystolube S

the pretreatment the critical surface tension of the wool fibres is increased, thereby allowing the resin to spread more evenly on the fibres. This means that pretreated wool requires considerably less resin than untreated wool to impart acceptable shrink-resist properties to wool fabrics.

It was found that 1% chlorination (o.w.f.) of the wool with sodium dichloroisocyanurate was quite sufficient to ensure good shrink-resist results from fabrics treated with the resin mixtures. Wool chlorinated at relatively low pH values (4-4.5) generally gave better results than wool chlorinated at higher pH values (5-6.5)

Several commercially available polymers such as a polyacrylate, polyvinyl acetate, polyvinyl alcohol and polyethylene were used as softeners in conjunction with Aerotex M-3 resin in an attempt to improve the handle of resin treated fabrics. Treatment of fabrics with these resin — polymer mixtures gave good shrink-resist

TABLE II
SHRINKAGE OF KNITTED FABRICS TREATED WITH AEROTEX M-3
RESIN MIXTURES

% Polymer	Fabric	Chlorination	% Shri after 3 i wasl	minutes	% Shrinkage after 48 minutes washing	
(o.w.f.)	structure	structure Pretreatment		Width	Length	Width
0	Punto-di Roma	_	1,4	1,9	15,4	9,5
0	22	+	1,9	1,9	12,9	5,2
4A + 2M	,,	+	2,4	0,5	4,3	4,8
3 A + 1 M	,,	+	3,4	0,5	8,6	0,5
3 A + 2 M	,,,	+	2,9	1,0	6,7	1,4
0	Single Jersey	-	1,0	5,7	13,9	18,1
4A + 2M	22	+	-1,4	1,9	1,0	1,9
3 A + 1 M	22	+	1,0	1,4	8,1	1,4
3 A + 2 M	>>	+	-1,4	2,4	7,2	4,3
4 A + 2 M	1 X 1 Rib	+	5,2	1,0	5,6	2,0
3 A + 1 M	>>	+	5,6	1,5	7,3	3,5
3 A + 2 M	22	+	2,6	2,5	13,5	0

Negative shrinkage values indicate expansion

A – Aerotex M-3 Resin

M – Mystolube S

results and also improved the handle of the fabrics. The best overall results, however, were obtained from fabrics treated with resin-polyethylene mixtures.

Optimization experiments showed that fabrics treated with a mixture of 3% resin and 2% polyethylene (o.w.f.) had the best handle and also possessed adequate shrink-resist properties (Tables I and II). On further processing, such as washing for 15 minutes and tumble drying, the handle of the fabrics was further improved.

The use of acid colloid solutions of melamine — formaldehyde condensates for the shrink-resist treatment of wool was described as early as 1957<sup>(4)</sup>. It was claimed that wool fabrics treated with acid colloid solutions had a softer handle than fabrics treated with the conventional resin solutions. Although some work was done on wool fabrics treated with hydrogen peroxide prior to the application of the acid colloid resin solutions, most of the previous work was done on wool which had received no pretreatment.

In this investigation very good shrink-resist results were obtained by treating wool fabrics with acid colloid resin mixtures, even with a resin add-on of as low as 0,5% (o.w.f.) (Tables III and IV). Treatment of the fabrics with resin colloid — polyethylene mixtures (2:1 ratio) at relatively low add-on levels (up to 2%) caused very little deterioration in the handle of the fabrics. Further processing such as washing for 15 minutes and tumble drying virtually restored the original handle of the fabrics.

TABLE III
SHRINKAGE OF STANDARD MATERIAL TREATED WITH AEROTEX M-3
ACID COLLOID RESIN MIXTURES

% Polymer on Fabric (o.w.f.)	Chlorination Pretreatment	after	rinkage 3 minutes hing	% Shrinkage after 48 minutes washing	
(0. W.1.)		Length	Width	Length	Width
0	_	5,7	5,3	47,8	50,7
0	+	2,9	1,4	30,1	34,8
0,5 A	+	_	_	2,4	2,9
1,0 A	+	_	_	1,7	3,8
2,0 A	, <b>+</b>	_	_	3,4	0
3,0 A	+			1,0	1,4
0,5 A + 0,25 M	+	_		·0,7	1,7
1,0 A + 0,5 M	+	_	_	2,6	1,7
2,0 A + 1,0 M	+	_	_	1,0	1,7
3,0 A + 1,5 M	+	_	_	1,2	0,5

A - Aerotex M-3 acid colloid resin

M – Mystolube S

By comparing the shrinkage values of the standard woven material in Tables I and III it can clearly be seen that acid colloid resin treated fabrics gave considerably better results than fabrics treated with conventional resin solutions.

**TABLE IV** SHRINKAGE OF KNITTED FABRICS TREATED WITH AEROTEX M-3 ACID COLLOID RESIN MIXTURES

% Polymer on Fabric	Fabric Structure	Chlorination Pretreatment	% Shrinkage after 3 minutes washing		% Shrinkage after 48 minutes washing	
(o.w.f.)			Length	Width	Length	Width
0	Punto-di- Roma	_	10,0	2,9	25,8	8,1
0	,,	+	10,0	-1,4	13,9	4,2
0.5  A + 0.25  M	,,	+	8,4	-2,8	13,4	2,8
1,0 A + 0,5 M	"	+	5,7	-1,9	8,8	1,4
2,0 A + 1,0 M	,,	+	6,7	-1,4	9,5	1,2
3,0 A + 1,5 M	,,	+	4,3	-0,5	6,7	1,4
0	1 X 1 Rib	_	11,0	-3,4	19,6	0
0	,,	+	6,2	-4,8	8,6	1,4
0.5  A + 0.25  M	"	+	6,5	-3,1	10,3	2,0
1,0 A + 0,5 M	,,	+	7;2	-3,3	9,1	0,7
2.0  A + 1.0  M	,,	+	5,7	-2,6	8,4	-0,3
3,0 A + 1,5 M	,,	+	4,3	-1,6	7,4	0,5
0	Single Jersey	_	7,2	8,1	19,6	13,9
0	,,	+	-1,5	- 3,8	15,5	11,5
0.5  A + 0.25  M	,,	+	-4,3	-2,6	6,0	2,1
1,0 A + 0,5 M	,,	+	-1,2	-2,1	7,3	0,2
2,0 A + 1,0 M	,,	+	-3,8	-2,9	2,0	2,8
3,0 A + 1,5 M	,,	+	-7,2	-1,2	4,5	1,0

The values given are average values from several experiments Negative shrinkage values indicate expansion

A - Aerotex M-3 acid colloid resin

M - Mystolube S

Extended washing trials (Table V) further substantiated the superiority of acid colloid treated fabrics (especially fabrics treated with acid colloid — polyethylene mixtures) over fabrics treated with conventional resin solutions.

Although it is very difficult to differentiate exactly between relaxation shrinkage and felting shrinkage, examination of a large number of fabrics showed that very little felting shrinkage took place during the first 3 minutes of the wash test. It is, therefore, reasonable to ascribe the changes in the dimensions of fabrics during 3 minutes washing principally to relaxation shrinkage or expansion. The relatively high shrinkage values of knitted fabrics after 3 minutes washing (Table IV) show that the treatment of knitted fabrics with acid colloid mixtures does not contain the relaxation shrinkage or expansion of the fabrics to any significant

TABLE V
SHRINKAGE BEHAVIOUR OF STANDARD MATERIAL TREATED WITH VARIOUS AEROTEX M-3 RESIN MIXTURES AFTER EXTENDED WASHING TRIALS

%Polymer on Fabric (o.w.f.)	Type of solution	% Shrinkage after 48 minutes washing		% Shrinkage after 192 minutes washing	
1 2010 (0: 11:)		Length	Width	Length '	Width
0,5 A	С	2,4	2,9	12,0	14,6
1,0 A	C	1,7	3,8	7,7	11,5
1,5 A	C	1,4	3,1	4,8	8,4
2,0 A	C	3,4	0	5,7	2,9
2,5 A	C	3,1	1,0	5,7	2,4
3,0 A	C	1,0	1,4	1,9	2,9
0,5 A + 0,25 M	С	0,7	1,7	4,0	5,2
1,0 A + 0,5 M	C	2,6	1,7	6,2	6,9
1,5 A + 0,75 M	C	0,7	2,4	3,1	5,7
2,0 A + 1,0 M	C	1,0	1,7	2,9	4,3
2,5 A + 1,25 M	C.	2,4	0,5	4,0	1,7
3,0 A + 1,5 M	С	1,2	0,5	3,1	1,9
3 A + 2 M	N	1,5	2,0	16,6	20,0

The values given are average values from several experiments

A - Aerotex M-3 resin

M - Mystolube S

C - Aerotex M-3 acid colloid resin solution

N - Aerotex M-3 resin solution

extent. Taking this fact into consideration it can be seen from the shrinkage results (Table IV) that the treatment of knitted fabrics with acid colloid mixtures is, however, an effective means of imparting adequate shrink-resist properties to the fabrics.

It is interesting to note that the shrinkage results of the Punto-di-Roma and  $1 \times 1$  rib chlorinated control fabrics compare favourably with some of the results of the corresponding acid colloid treated fabrics (Table IV). The chlorinated control fabrics, however, showed extensive surface felting whereas the acid colloid treated fabrics showed very little surface felting.

A further advantage of the acid colloid resin treatment of fabrics is the relatively low curing temperature which is required. At this temperature very little yellowing of the wool was noticeable.

#### **CONCLUSIONS**

Adequate shrink-resist properties can be imparted to woven and knitted wool fabrics by the application of methylolmelamine resin-polyethylene mixtures without significantly affecting the handle of the fabrics. Pretreatment of the wool fabrics is, however, essential. Acid colloid resin treatment of the fabrics proved to be especially effective. Results from this investigation indicate that resin add-on levels of 1% to 2% are sufficient to impart the required resistance to felting shrinkage to most woven and knitted wool fabrics.

#### THE USE OF PROPRIETARY NAMES

The fact that chemicals with proprietary names have been used in this investigation in no way implies that they are recommended by SAWTRI or that there are not others as good or even better.

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