The role of an enzyme in reducing wool shrinkage

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A proteolytic enzyme derived from the bacterium *Streptomyces fradiae* has been applied on to wool in an attempt to improve its shrink-resistance properties. The influence of enzyme concentration and treatment time in the improvement of felting shrinkage in different washing cycles has been determined. The extent of attack on the fibre was also measured.

INTRODUCTION

A great deal of work has been undertaken over many years in an attempt to develop treatments for wool that will overcome the fibre's the natural tendency to shrink during washing. The numerous procedures now available on the market bear testimony to this effort. Yet however effective these procedures may be, some of them do pose environmental problems. Enzymatic processes based on the action of papain are environmentally friendly but not as yet very successful in conferring worthwhile degrees of shrink resistance. Therefore workers have tended to concentrate on alternative non-polluting enzymatic processes.

A proteolytic enzyme derived from the bacterium *Streptomyces fradiae* (known as SFP) is capable of attacking natural keratin, hydrolysing some peptide linkages [1]. In the present study the SFP enzyme has been applied to wool as a follow-up to some results found by Compañia Española de Penicilina y Antibióticos (CEPA) [2–6]. The conditions necessary to improve the shrink resistance of wool without causing an excessive alteration of the fibre have been examined.

EXPERIMENTAL

Materials

Fabric was specially knitted from 2/24 wool yarn, fabric cover factor 1.2. Before treatment the fabric was fully relaxed under IWS TM 31 conditions (ISO 6330 7A). The SFP enzyme was supplied by CEPA; its proteolytic activity was 800 Anson units per milligram [7].

Treatments

Prior to the application of enzyme to wool, the optimum application conditions were determined on the basis of data supplied by CEPA about temperature and pH of maximum activity [8]. These conditions were found to be 50°C at pH 7.5. The enzyme was applied to wool in a static bath (liquor ratio 20:1) without any liquor movement or agitation of the sample. Enzyme concentrations of 1–5 g/l and treatment times between 30 and 240 min were used.

Some trials were also made in the presence of sodium sulphite, which has been shown to promote enzymatic activity [8]. The concentration of sodium sulphite was varied between 0 and 10 g/l. The action of enzyme on wool was assessed in terms of a number of different parameters, outlined below.

Shrinkage

Trials were carried out to assess the degree of wool shrinkage in a Cubex apparatus for 30 min (5 min in each direction) at 25°C, liquor ratio 25:1 with 3 g/l ECE reference detergent. Tests were also carried out according to IWS TM 31, which can be used to determine either relaxation or felting shrinkage. In order to determine felting shrinkage all the samples were shrunk by relaxation before enzyme application according to the ISO 6330 7A procedure, with the exception that the total load was reduced to 1 kg. Main conditions of ISO 6330 7A are a time of 3 min and a temperature of 40°C, with gentle action, 1 g/l ECE detergent (without fluorescent brightener), and three rinses.

Shrinkage was determined by measuring the samples before and after being submitted to severe agitation in accordance with the ISO 6330 7A and 5A programmes (washing time 12 min, temp. 40°C, normal action, 0.3 g/l ECE detergent, four rinses). The number of cycles of the programme to which the samples are submitted depends on the intended end use of the material. In the present study, examining fabrics for use in sweaters, the 7A and two 5A tests were adopted. The device used was a special Wascator laboratory washing machine, model FOM 71. As indicated in the ISO 6330 standard, samples are conditioned at 20°C and 65% RH before any measurements were taken. The linear felting shrinkage is defined by Eqn 1:

$$S_l = \frac{D_r - D_s}{D_r} \times 100 \qquad (1)$$

where S_l = linear felting shrinkage, %
D_r = relaxed sample dimension
D_s = shrunk sample dimension

The percentage area shrinkage is defined by Eqn 2:

$$S_a = S_{l1} + S_{l2} - \frac{S_{l1} S_{l2}}{100} \qquad (2)$$

where S_a = area shrinkage, %
S_{l1} = breadthwise linear shrinkage, %
S_{l2} = lengthwise linear shrinkage, %

Weight loss

The weight loss caused by enzymatic treatment and after each shrinkage trial was evaluated, since degradation of the polypeptide chains may give rise to breakage of the fibres, which work loose during treatment in a wet agitation process. Determination of loss weight was made on samples conditioned at 20°C and 65% RH.

Physical and chemical parameters

The following parameters were also considered:

(a) Variation in strength and elongation of fibres, as measured on an Instron tensile tester
(b) Alkali solubility, urea–bisulphite solubility, cystine, cysteine, cysteic acid and amine end group content
(c) Microscopic examination of the fibres through a scanning electronic microscope.

RESULTS AND DISCUSSION

The results relating to shrinkage and weight loss are shown in Table 1, indicating that enzymatic treatment caused a reduction in wool shrinkage. This reduction was perceptible at low enzyme concentrations and short treatment times, and increased slightly on increasing the enzyme concentration. At a specific enzyme concentration the effect also increased with longer treatment times. When the enzymatic treatment caused a substantial reduction of shrinkage, the losses of weight in the shrinkage test were high, which means that the wool fibre had been damaged to a considerable extent. Hence we concluded that treatment at the longest time used (240 min) was not appropriate, as excessive losses of weight occurred in all the enzyme concentrations tried.

Table 1 Influence of enzyme on shrinkage and weight loss in wool (Cubex machine)

Enzyme concn (g/l)	Time (min)	Shrinkage (%)	Weight loss (%)
0	0	20.4	0.7
1	30	15.6	1.6
	60	15.5	2.1
	120	14.5	2.7
	240	12.2	6.2
2	30	14.8	2.3
	60	13.5	2.6
	120	11.5	4.1
	240	12.0	9.3
3	30	13.9	2.6
	60	13.5	3.0
	120	12.5	5.1
	240	11.5	14.7
4	30	12.5	2.7
	60	12.1	3.7
	120	11.9	5.3
	240	8.7	12.5

Table 2 Influence of enzyme on shrinkage in wool after washing tests

Enzyme (g/l)	Area shrinkage (%) 7A	7A + 2 5A
0	0	50.0
Treatment time 30 min		
1	4.0	40.8
2	3.9	40.7
3	3.7	37.9
4	1.4	37.4
5	1.2	37.3
Treatment time 60 min		
1	2.3	38.4
2	2.0	38.4
3	2.0	38.0
4	0.6	38.0
5	0.6	36.2

Table 2 shows the shrinkage results of the washing treatments in the Wascator machine, according to ISO 6330 (7A and two 5A tests). Comparing the results of Tables 1 and 2 shows that the total shrinkage of untreated wool in the Wascator machine was much higher than the corresponding test in the Cubex, demonstrating that the treatment intensities of the two methods were different.

The shrinkage reduction was more significant in the less severe tests. For example, a shrinkage reduction of up to 85% was attained in the case of an enzymatic treatment at 5 g/l over 60 min using cycle 7A. When the 7A cycle was combined with the 5A cycle reductions up to 45% were achieved. In the most drastic shrinkage test (one 7A cycle followed by two 5A cycles) the reduction of the surface loss was only 28% for the same enzymatic treatment. Hence it was concluded that, although this enzymatic treatment was effective in improving reduction in wool shrinkage, it did not confer the desirable shrink-resistance levels for washing wool in a washing machine under severe conditions.

Table 3 shows the losses of weight after enzymatic treatments in the shrinkage trials indicated (in a Wascator machine according to ISO 6330 standard). Again, losses in weight increased on increasing the enzyme concentration and time, as expected. In the shrinkage tests the losses of weight also increased as concentration and time were increased, but this effect was not considered excessive (except at 5 g/l and 60 min) in view of the severity of the trial.

Experiments were also conducted to verify that enzymatic attack was more serious at high temperatures, as would be experienced during dyeing with reactive dyes. The highest dyeing temperature used was 100°C over a time of 60 min. It was found that the enzymatic treatment under these conditions did not cause an excessive further loss in weight.

Table 3 Influence of enzyme on weight loss in wool after washing tests

Enzyme (g/l)	Weight loss (%) 7A	7A + 2 5A
0	0	2.3
Treatment time 30 min		
1	0.8	1.7
2	0.9	1.8
3	0.9	2.2
4	1.8	2.2
5	1.6	2.0
Treatment time 60 min		
1	0.9	1.8
2	1.0	2.1
3	2.4	2.5
4	2.5	2.8
5	3.5	4.5

Enzymatic treatments were also carried out in the presence of sodium sulphite, which tended to act as as an activator, the enzymatic action increasing on increasing the sodium sulphite concentration over the range from 2 to 5 g/l. The losses in weight experienced would not be acceptable in commercial wool processing. Table 4 shows the values obtained for area shrinkage, and clearly the presence of sodium sulphite, despite causing an increase in enzyme action on wool, did not materially improve the shrinkage results.

Table 5 shows the physical and chemical parameters of the wool fibres both before and after enzymatic treatment. The various physical and chemical parameters did not undergo any significant changes and were therefore considered acceptable after treatment.

Micrographs of wool treated under different conditions show the effect of the enzymatic treatment on the fibre scales (Figure 1). Some fibres appear very little affected by the treatment while others show a severe effect on the scale structure and even on the fibre cortex. This means that the enzymatic treatment did not produce an even action on the wool, which was probably the main reason why the expected values for the shrinkage reduction were not obtained.

CONCLUSIONS

Treating wool with the SFP enzyme causes a reduction in shrinkage even at low enzyme concentrations; the effect increases with increasing treatment concentration and time. Shrinkage reduction is greater with less intense washing treatments. In severe washing methods, although there is a certain amount of shrinkage reduction, the effect is not sufficient for practical purposes.

The enzymatic treatment attacks the fibre, causing weight losses in the treatment or in further severe wet processes. These losses of weight are of little importance at low or medium enzyme concentrations and times, but are unacceptable at higher enzyme concentrations. No substantial changes in the physical or chemical properties of wool were detected under any but the most severe treatment conditions. Microscopic observation shows that the enzymatic action is not uniform; some fibres are practically intact while others are considerably damaged. The achievement of a better uniformity of treatment would probably give rise to better shrinkage results. This will be the subject of further work.

Table 4 Influence of sodium sulphite on enzymatic action (shrinkage results)

Enzyme (g/l)	Sodium sulphite concn (g/l)	Area shrinkage (%) 7A	7A + 2 5A
Treatment time 60 min			
1	0	0.7	40.6
	1	0.7	40.8
	2	0.5	39.8
	5	0.2	40.2
	10	1.8	39.3
2	0	3.3	40.3
	1	4.2	39.0
	2	6.2	38.4
	5	6.2	38.6
	10	7.0	39.0
3	0	3.3	40.2
	1	4.5	41.1
	2	4.3	39.1
	5	5.0	36.0
	10	6.3	38.4
Treatment time 60 min			
1	0	5.0	40.0
	1	3.9	39.0
	2	6.2	38.1
	5	6.4	33.0
	10	6.7	30.6
3	0	3.9	35.7
	1	−1.7	30.3
	2	−3.5	
	5	6.6	
	10	3.1	

Table 5 Properties of wool before and after enzymatic treatment (enzyme concn 3 g/l)

Parameter	Untreated wool	Treatment time 30 min	60 min
Strength, g	9.3	8.5	7.6
Elongation, %	43.4	39.8	25.4
Alkali solubility, %	10.8	11.5	12.2
Urea–bisulphite solubility, %	32	41	43
Cystine content, %	12	12	12
Cysteine content, %	0.26	0.23	0.23
Amine end group content	206	213	220

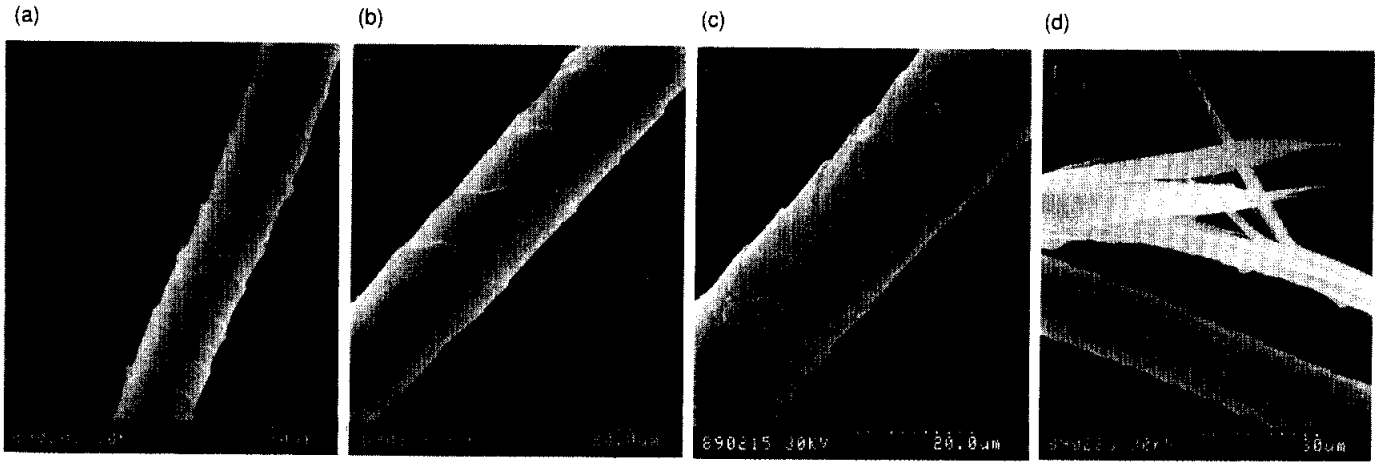

Figure 1 Micrographs of wool fibres treated with SFP enzyme at different concentrations and treatment times: (a) untreated, (b) 1 g/l enzyme/60 min, (c) 3 g/l/60 min, (d) 5 g/l 120 min

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