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Experiments to Promote Colour Changes in Wool

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Abstract: Wool colour is not stable and it changes during storage. This study developed wool challenge methods by using a one-week water bath incubation at 40°C or incubation overnight at a higher temperature to promote colour changes in wool. The study showed that the bacteria present in raw wool play a significant role in colour biodegradation during challenge (storage). A stable wool colour could be obtained for wool specification after a challenge treatment before colour measurement.

Key words: wool colour, incubation, bacteria, storage.

INTRODUCTION

Changes in wool colour during growth, storage and processing are well known in the industry.[1] The colour test certification for New Zealand wool is valid for three months only,[2] after which no guarantees of colour stability are given. Three-year storage trials with commercial quantities of New Zealand wool have resulted in the detection of colour changes.[3] Similar studies on Australian wool are under way. Identification of factors believed to cause colour changes and the 'challenging' of wool samples to change colour by exacerbating these factors should assist in predicting which wools are likely to deteriorate in colour. Such factors include moisture and elevated temperature. 'Challenge' in this paper refers to subjecting wool to conditions which promote deterioration in colour if the wool is susceptible to such colour change. Such an approach has the potential to lead to a standard test in which colour changes can be brought about more rapidly than during storage. Some samples of wool will change colour as a result of being challenged and others, resistant to colour deterioration, will not.

Experiments challenging wool for one week at 40°C and saturated humidity[4] identified the following colours:

(i) Resistant colours, for wool samples not responding to the challenge.

(ii) Stable colours, for wool samples susceptible to the colour changing factors. The stable colour is the colour beyond which little further change occurs in the wool if the challenging conditions continue.

There is a need to reduce the challenge time from one week to less than one working day if the procedure is to be the basis for commercial certification of colour. The experiments described herein were therefore designed to

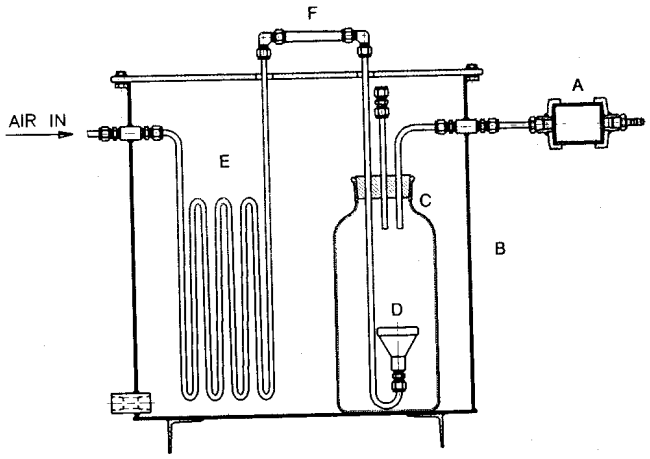

Fig. 1. Apparatus for wool challenging. A, sample cell; B, tank; C, humidifier flask; D, gas disperser; E, pre-heating coil; F, by-pass.

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TABLE 1
Colour Parameters of Wool A after Different Challenges at 40°C ($Y/Y-Z$)

Colour	*Base colour*[c]	*Incubated colour*[d]	*Moist N_2, o/night*	*Moist air, o/night*	*Moist air, 5 days*
Y	$56{\cdot}3^{a}$	$57{\cdot}0^{a}$	$62{\cdot}4^{b}$	$56{\cdot}2^{a}$	$58{\cdot}2^{a,b}$
$Y-Z$	$5{\cdot}9^{a}$	$7{\cdot}4^{b}$	$5{\cdot}2^{a}$	$6{\cdot}2^{a,b}$	$5{\cdot}7^{a}$

[a,b] Means with the same superscripts are not significantly different at a confidence level of 0·05.
[c] Unchallenged.
[d] Enclosed water bath for one week at 40°C.

TABLE 2
Colour Parameters of Wool B after Different Challenges at 40°C

Colour	*Base colour*	*Incubated colour*	*Moist air (5 h)*	*Moist air (18 h)*	*Moist air (50 h)*	*Moist air (4 h recycled)*	*Moist air (16 h recycled)*
Y	$62{\cdot}9^{a}$	$55{\cdot}0^{b}$	$63{\cdot}3^{a}$	$60{\cdot}7^{a,b}$	$56{\cdot}2^{b}$	$63{\cdot}9^{a}$	$62{\cdot}9^{a}$
$Y-Z$	$1{\cdot}0^{a}$	$2{\cdot}8^{c}$	$1{\cdot}6^{a,b}$	$1{\cdot}7^{a,b}$	$2{\cdot}2^{b,c}$	$1{\cdot}1^{a}$	$1{\cdot}3^{a,b}$

accelerate wool colour changes under laboratory conditions.

APPARATUS AND MATERIALS

The apparatus developed to challenge wool colour change is shown in Fig. 1. The wool sample cell was cylindrical with length 56 mm and diameter 54 mm. The wool was packed inside in 30 g quantities, resulting in densities equivalent to those found in pressed farm bales. The cell was connected to a gas handling and humidifying system. Gas from a cylinder (air or nitrogen) was admitted and passed through copper coils, enabling it to pre-heat to the temperature of the bath. The influx gas then passed via a sintered metal disc through a vessel of water, and from there into the sample cell.

The following factors could be varied between successive experiments.

(i) The composition of the influx gas. Its flow rate was always 1000 $cm^3\ min^{-1}$.
(ii) The bath temperature, and hence the humidity of the gas entering the sample cell.
(iii) Duration of exposure of the wool to influx gas.

It was also possible to admit dry gas by by-passing the vessel containing water. In a number of experiments nitrogen–5% ammonia was used as the influx gas. In most experiments the efflux gas was discharged from the cell into the atmosphere. However, in some cases the efflux gas was recirculated by means of a pump. For comparative purposes a number of experiments were performed using a closed water bath containing the wool sample in vapour generated in the bath. This apparatus had been previously used in week-long tests.[4]

Wool was obtained from the Australian Wool Testing Authority Ltd. Twelve core reference samples, denoted A–L inclusive, were used in the colour experiments. Duplicate samples were challenged and examined. The results presented are means of the duplicate samples tested. Significance of differences was determined by analysis of variance. For each wool used, a washed, dried and carded sample was measured for yellowness according to Australian Standard 4536.[5] The method is outlined in Appendix 1, and the effects of different treatments were examined by analysis of variance.

RESULTS AND DISCUSSION

Table 1 shows results for wool A under various conditions in the apparatus shown in Fig. 1 compared with wool incubated in a water bath for one week or not challenged at all.

Wool A has quite a yellow base colour as indicated by its $(Y-Z)$ value of 5·9. Whereas a week of incubation brings the yellowness to 7·4, none of the overnight tests raised the $(Y-Z)$ value to that achieved by incubation for a week. Results for wool B, a relatively white, resistant wool, are given in Table 2.

Tests of wool B in the apparatus also brought the yellowness towards its stable value. However, only the 50 h exposure to air, too long for a routine test, brought the yellowness close to its stable value. Recycling the air did not make the process any faster.

TABLE 3
Colour Parameters of Wools C–F Challenged with Nitrogen–5% Ammonia at 40°C ($Y/Y-Z$)

Wool sample	*C*	*D*	*E*	*F*
Base colour	63·0/2·2	62·4/1·2	65·5/1·5	61·7/2·0
Incubated[a]	51·5/4·8	53·5/4·2	60·0/5·5	49·8/4·3
NH_3 treated[b]	61·7/1·9	61·7/0·9	64·9/3·0	60·3/2·5

[a] Incubated in the water bath for one week.
[b] 5% ammonia, 1 h with apparatus in Fig. 1.

TABLE 4
Colour Parameters Obtained with Air at Temperatures over 40°C ($Y/Y-Z$)

Wool	*Base Colour*	*Incubated colour (40°C)*	*5 h flowing moist air*		
			45°C	*55°C*	*65°C*
E	65·5/1·5	60·0/5·5	64·0/2·2	63·5/2·4	63·2/3·5
G	65·5/1·8	59·8/3·2	63·9/1·7	62·5/2·8	60·9/3·7
H	62·2/2·5	58·6/5·7	62·1/3·1	61·5/3·5	59·5/5·3
Mean Y	64·4[a]	59·4[b]	63·2[c]	62·5[c]	61·2[d]
Y−Z	1·9[a]	4·8[b]	2·3[a]	2·9[c]	4·2[d]

TABLE 5
Bacterial Counts for Challenged Wool (number g wool^{-1})

Wool	*Raw wool*	*Incubated*	*5 h flowing moist air*		
			45°C	*55°C*	*65°C*
G	45×10^3	30×10^5	50×10^3	60×10^6	50×10^2
H	17×10^3	18×10^5	20×10^4	25×10^5	12×10^3

TABLE 6
Yellowness Change after Incubation ($Y-Z$)

Wool	*C*	*G*	*H*	*F*	*J*	*Mean*
Greasy wool	2·4	1·6	3·2	2·3	5·6	3·0[a]
Sterilised wool	1·0	1·1	1·0	1·1	0·9	1·0[b]
Bacterial effect	1·4	0·5	2·2	1·2	4·7	2·0

The change is relative to the colour of a non-incubated sample: the calculation is based on results for sterilised wool with incubation minus those for sterilised wool without incubation, to remove the effect of sterilisation itself.

Wools C–F exposed to 5% (molar basis) ammonia in nitrogen as the influx gas for 1 h resulted in the colours shown in Table 3. During storage of wool, ammonia is believed to be generated by biodegradation.[6] Alkaline conditions are expected to accelerate yellowing. The ammonia appears not to affect colour change markedly even at 5%, which is much higher than wool would experience naturally. Unpublished work by the present group indicates that the natural level of ammonia in wool bales is up to 5 ppm after one year of storage.

Moist air or ammonia at 40°C did not exacerbate colour change to a degree sufficient to form the basis of a routine test. Higher temperatures were therefore used, and Table 4 shows results obtained at 45°C, 55°C and 65°C.

Challenging at 65°C brings the wool colour closer to that obtained with the one-week incubation colour, and therefore provides a more promising basis for a routine test. At 45°C the colour was not significantly different to the base colour. The colour change process is clearly very sensitive to temperature. For wools G and H bacterial counts were also performed at 45°C, 55°C and 65°C and are listed in Table 5.

The figures in Table 5 show that after one week of water bath incubation at 40°C the bacterial counts for wools G and H increased 100-fold. After the wools were challenged at 55°C for 5 h the bacterial counts were even higher. A challenge temperature of 65°C caused the bacterial count to return to a value closer to that for untreated wool. The higher temperature (around 55°C) appears not to have had a lethal effect on the micro-organisms present, but in fact, on the evidence available, to have promoted bacterial multiplication.

In order to study the effect, if any, of bacteria on colour change, further experiments were performed in which the wool had been sterilised by gamma radiation. Table 6 shows results of exposure for one week of sterilised wool to water vapour at 40°C in the water bath. Wool samples not having been sterilised were also examined.

About one unit change of ($Y-Z$) is attributable to the water vapour exposure; this is the change observed with sterilised wool. Much larger effects due to bacteria (average 2 units) are observed with greasy wool. The bacteria play a significant part in colour change and this is most evident at higher temperatures (around 55°C). Subsequent experiments were therefore carried out at 50–60°C, the highest temperature range compatible with bacterial survival. This has the potential to lead to a short routine test for a stable colour. Table 7 lists results obtained with both pieces of apparatus in this temperature range employing exposure times not exceeding one day.

Experiment 1 was for unchallenged wool. Experiment 2 used the water bath at 50°C overnight. Experiment 3 used 55°C for 5 h with the water bath. Experiment 4 used 55°C with the water bath for one day. Experiment 5 used the novel apparatus (Fig. 1) at 55°C (gas temperature) and flowing moist air for 5 h. Experiment 6 used 60°C overnight with the water bath. Experiment 7 used one week with the water bath at 40°C.

TABLE 7
Wool Yellowness ($Y-Z$) after High-Temperature, Short Duration Experiments

Experiment[a]	*Wools*									
	C	*D*	*E*	*F*	*G*	*H*	*I*	*J*	*K*	*L*
1	2·2	1·2	1·5	2·0	1·8	2·5	4·2	5·6	12·7	8·6
2	2·1	1·2	2·7	2·0	2·3	—	4·8	5·5	12·9	7·4
3	2·0	1·5	2·1	3·0	1·8	2·9	5·3	5·6	12·3	9·2
4	3·7	3·6	3·3	4·8	4·3	4·3	6·0	7·6	13·2	9·6
5	3·2	3·7	2·4	—	2·8	3·5	—	—	—	—
6	5·0	5·6	5·8	5·1	—	—	6·8	7·5	13·0	11·0
7	4·8	4·2	5·5	4·3	3·3	5·7	4·6	11·2	13·0	8·8

[a] See details in main text.
1 = base colour, 7 = incubated colour.

The conditions in Experiment 6 gave values of ($Y-Z$) slightly higher than those in Experiment 7, yet it involves only overnight exposure, whereas Experiment 7 using 40°C involved exposure for one week. Overnight water bath treatment at 60°C (Experiment 6) is therefore a sufficient basis for a short-term test and there was a high degree of correlation with incubated colour results ($r^2 = 0{\cdot}84$). Therefore, wools with different incubation colour were differentiated at this temperature. The colour change process displays a marked temperature sensitivity, and 50°C is not high enough for an overnight test. Hence careful temperature control will be necessary in the performance of a standard test.

CONCLUSIONS

A bacterial population and a relatively high (55–60°C) temperature and 100% humidity have been identified as factors exacerbating colour changes in wool. It has been found possible to exploit these factors in such a way that a stable colour can be achieved by overnight exposure. This can form the principle of a short-term commercial test to certify the stable colour of different samples.

ACKNOWLEDGMENT

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REFERENCES

1. Roberts, M. B., *Some effects of common processing conditions upon the mohair fibre*. Technical Report 351, South African Wool and Textile Research Institute, 1977.
2. Carnaby, G. A. & Ranford, S. L., *Meeting scoured wool delivery specifications*. Report R188, Wool Research Organisation of New Zealand, 1991.
3. Ranford, S. L., Hammersley, M. J. & Corrigan, M. K., *Colour changes in New Zealand wool under storage conditions*. 10th Report to International Wool Textile Organisation, Wool Research Organisation of New Zealand, 1991.
4. Cottle, D. & Zhao, W., Incubation of wool to promote a stable wool colour. *Journal of the Textile Institute* (submitted).
5. Average yellowness of wool. Australian Standard AS 4536.
6. Gochel, M., Belly, M. & Knott, J., Biodegradation of wool during storage. *Proceedings, 8th International Wool Textile Research Conference*, 1990, Vol 3, pp. 235–45.

APPENDIX 1: METHOD OF COLOUR DETERMINATION

Wool colour measurement is based on the electronic measurement of intensity of various colours reflected from the surface of scoured wool by a colorimeter. The colorimeter measures the amounts of light reflected in the red, green and blue spectra. The values obtained were approximate CIE XYZ tristimulus values. The light source used was CIE Illuminant C (simulated overcast-sky daylight) with the 2-degree observer. The Y value describes the lightness of the wool (50 to 70, mostly 58–66 for Australian wool). The greater the value the lighter the wool. Yellowness is associated with a reduction in the reflected blue component relative to the lightness and is expressed as ($Y-Z$) which can vary in the range -2 to 12 and is usually 1–4 for Australian wools. The smaller the value of ($Y-Z$), the less yellow the wool.