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Chrome Dyes on Wool: The Influence of Some Shrink-Resist Treatments on Residual Chromium Values

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CHROME DYES ON WOOL : THE INFLUENCE OF SOME SHRINK-RESIST TREATMENTS ON RESIDUAL CHROMIUM VALUES

by M.B. ROBERTS

ABSTRACT

Residual chromium values were determined after dyeing untreated, chlorinated and chlorine/Hercosett treated wools with chrome dyes using various levels of dichromate. At relatively low levels of dichromate usage little difference in residual chromium was found between substrates. As the level of dichromate was increased, residual chromium values for chlorine/Hercosett treated wool increased at a much greater rate than did those for untreated wool, chlorinated wool giving results intermediate between the other substrates.

Superwash standards with respect to wet fastness could be achieved on shrink-resist treated wool with little or no increase in dichromate compared with that required to achieve adequate fastness on untreated wool. Some dyes failed to meet these standards irrespective of the level of dichromate employed.

INTRODUCTION

Recent legislation concerning the maximum chromium concentration permissible in effluents has led to much research into methods of reducing the residual chromium content to acceptable limits. Two fundamental systems have been shown capable of satisfying these limits. The first is the treatment of the effluent, either by classical chemical methods involving reduction of hexavalent chromium to the trivalent state followed by precipitation of trivalent chromium as the hydroxide on additio of alkali, or by passage through a chelating resin[1]. There is little doubt that this method produces the lowest residual chromium but this advantage is offset by costs of processing and disposal of the precipitate.

The second approach is the modification of the chroming technique. Several workers [2-4] have shown that the quantity of chromium relative to the quantity of dye, used until recently, was considerably in excess of that required and that by decreasing the ratio of chromium to dye, it is possible to satisfy present legal requirements while still achieving the necessary fastness ratings. The only slight disadvantage associated with this technique is that the final shade is sometimes slightly different to that of dyeings carried out with greater amounts of chromium. Benisek[5] has shown that residual chromium in the bath may be significantly reduced by the addition of lactic acid and that, by so doing, the normal levels of chromium may be employed and the legal requirements regarding residual chromium are satisfied after the usual dilution of the spent

dye liquor.

So far as can be ascertained, there appears to have been no study of the effect of fibre pretreatments, such as shrinkproofing, when used in conjunction with the high dye to chromium ratio, upon the levels of residual chromium and the resultant fastness characteristics. This report is intended to clarify the position.

EXPERIMENTAL

Materials

The fabric used throughout the investigation was an all-wool worsted material of fabric density 140 g/m^2. It was divided into three portions, two of which were chlorinated with ®Basolan DC by padding on a solution adjusted to pH 2,3 with sulphuric and acetic acids and containing 1,5% active chlorine and 0,2 g/ℓ ®Nonidet P40 to a pick-up of 100%. After a dwell period of 5 minutes the fabric was neutralised in a solution containing 30 g/ℓ each of sodium bisulphite and sodium bicarbonate and finally rinsed in a 1% acetic acid solution. One of the chlorinated fabrics was then treated with ®Hercosett 125 by padding on a solution containing 10 g/ℓ Hercosett 125 and sufficient sodium bicarbonate to give a pH of 7 to a pick-up of 100%. The fabric was dried at 60° C and cured for 15 minutes at 90° C.

Dyeing

The dyeing method employed in this investigation was the afterchrome process. The dyes used were commercial samples from the ®Eriochrome range. The depth of shade was 3% omf based on the standard strength dye and 8% omf for the black. Samples of 10 g were used for each dyeing. Dyeings were carried out in an Ahiba Turbomat TM6 laboratory dyeing machine. Dyebaths were set at 50° C with the following (all percentages expressed omf):

1,0% ®Albegal W
3,0% dye (or 8% for the black)
Acetic acid to pH 5

The liquor-to-goods ratio was 25 : 1.

Demineralised water was used in all cases.

After entering the fabric sample the bath was run for 5 minutes and then heated to the boil in 30 minutes. After a further 30 minutes at the boil, the pH

was adjusted to 4 with formic acid to complete the exhaustion. After a further 30 mins at the boil, the baths were cooled to 80°C and the required amount of potassium dichromate added. The bath was reheated to the boil and boiling was continued for 45 minutes. After removal of the dyed samples, the liquors were retained for residual chromium determination.

Testing

The method used for the determination of chromium was a well established one based upon the spectrophotometric estimation of a coloured chromium complex[6]. This method permitted the separate determinations of total chromium and hexavalent chromium.

Dyeings on chlorinated and chlorine-Hercosett treated wool were tested for fastness to washing and alkaline perspiration by IWS test method 193[7] and 174[8] respectively and to potting[9]. Dyeings on untreated wool were tested for fastness to the same agencies by the appropriate methods[9-11].

RESULTS AND DISCUSSION

The results of this investigation are conveniently displayed in a single table for each dye; each table illustrating the effect of fibre treatment upon residual chromium values and wet fastness characteristics at different levels of applied dichromate.

Discussion of the results may be divided into two parts: the first being simply concerned with the effects of fibre treatment upon residual chromium content and the second with the more complex relationship between fibre treatment, residual chromium levels and minimum wet fastness requirements.

At the lower levels of applied dichromate irrrespective of the fibre treatment, residual hexavalent and total chromium values are either zero or so small that they are not likely to cause difficulties in satisfying current legislation. With increasing levels of applied dichromate, the residual chromium figures increase sharply and it is apparent that the highest values are exhibited with chlorine-Hercosett treated wool and the lowest with untreated wool, the figures for chlorinated wool lying between these extremes. It is also apparent that the use of the greater quantities of dichromate will result in residual chromium levels of such magnitude that it is unlikely that mere dilution of the spent liquor would reduce them to an acceptable level. Clearly a treatment such as that proposed by Benisek[5] or one based upon a precipitation technique would be required.

With respect to wet fastness characteristics almost all the dyes surveyed exhibit optimum wet fastness characteristics on untreated wool at such low levels of applied dichromate that treatment of the effluent appears largely unnecessary. Even the potting fastness, which may be regarded as a severe wet test, is perfectly satisfactory at these low levels of applied dichromate. The one

TABLE I

INFLUENCE OF SHRINK-RESIST TREATMENTS ON WET FASTNESS PROPERTIES AND RESIDUAL CHROMIUM VALUES FOR CI MORDANT RED 17 ON WOOL AT VARIOUS LEVELS OF APPLIED DICHROMATE

Applied Chromate Concn. (% omf)	Residual Chromium (mg/ℓ)		Fastness to:								
			Washing			Alkaline Perspiration			Potting		
	Cr^{6+}	Total Cr	Effect on Shade	Staining on Wool	Staining on Cotton	Effect on Shade	Staining on Wool	Staining on Cotton	Effect on Shade	Staining on Wool	Staining on Cotton
UNTREATED FABRIC											
0,2	0	0	5	5	5	5	5	5	5	4—5	5
0,4	0	0,5	5	5	5	5	5	5	5	4—5	5
0,8	0	1,75	5	5	5	5	5	5	5	4—5	5
1,2	2,7	4,5	5	5	5	5	5	5	5	4—5	5
1,5	9,0	12,5	5	5	5	5	5	5	5	4—5	5
CHLORINATED FABRIC											
0,2	0	0	5	4—5	4—5	4—5	4	5	4—5	3—4	5
0,4	0	0,1	5	4—5	5	5	4	5	4—5	3—4	5
0,8	0,1	0,3	5	5	5	5	4	5	5	4	5
1,2	8,5	10,0	5	5	5	5	4—5	5	5	4	5
1,5	17,5	21,3	5	5	5	5	4—5	5	5	4—5	5
CHLORINE/HERCOSETT TREATED FABRIC											
0,2	0	0,1	5	4—5	5	5	3	4—5	4B	3—4	5
0,4	0	0,3	5	4—5	5	5	3	4—5	4B	3—4	5
0,8	1,0	2,8	5	4—5	5	5	4	5	4B	4	5
1,2	5,0	15,0	5	4—5	5	5	4—5	5	5	4	5
1,5	25,0	75,0	5	4—5	5	5	4—5	5	5	4	5

TABLE II

INFLUENCE OF SHRINK-RESIST TREATMENTS ON WET FASTNESS PROPERTIES AND RESIDUAL CHROMIUM VALUES FOR CI MORDANT ORANGE 44 ON WOOL AT VARIOUS LEVELS OF APPLIED DICHROMATE

Applied Chromate Conc$^{n.}$ (% omf)	Residual Chromium (mg/ℓ)		Fastness to:								
			Washing			Alkaline Perspiration			Potting		
	Cr^{6+}	Total Cr	Effect on Shade	Staining on Wool	Staining on Cotton	Effect on Shade	Staining on Wool	Staining on Cotton	Effect on Shade	Staining on Wool	Staining on Cotton
UNTREATED FABRIC											
0,2	0	0,2	5	4	4	5	2	3	3—4	2—3	4—5
0,4	0	0,4	5	4—5	4—5	5	3	4	3—4	3	4—5
0,8	0,	1,2	5	5	4—5	5	3	4—5	3—4	4	5
1,2	2,5	15,0	5	5	5	5	3—4	4—5	4	4—5	5
1,5	17,5	32,5	5	5	5	5	4	4—5	4	4—5	5
CHLORINATED FABRIC											
0,2	0,	0,1	4—5	3—4	3—4	5	3	3—4	3—4	2—3	4—5
0,4	0	0,1	4—5	3—4	3—4	5	3	3—4	3—4	3	4—5
0,8	0,1	0,6	4—5	4	4	5	3—4	4—5	3—4	3—4	5
1,2	9,4	13,8	4—5	4		5	4	4—5	4	4	5
1,5	26,3	28,8	4—5	4	4	5	4	4—5	4	4	5
CHLORINE/HERCOSETT TREATED FABRIC											
0,2	0	0,2	4	3—4	3—4	4	2	3	3—4	2—3	4—5
0,4	0	0,3	4	4	4	4	2—3	3—4	3—4	3	4—5
0,8	3,8	4,1	5	4—5	4—5	5	3	4	3—4	3—4	5
1,2	13,8	17,5	5	4—5	4—5	5	3	4	4	3—4	5
1,5	30,0	35,0	5	4—5	4—5	5	3	4	4	3—4	5

dye which does not conform to this general pattern is Mordant Orange 44 (Table II). The weakness of this dye is its tendency to stain adjacent fabrics, particularly wool, under conditions designed to simulate potting and alkaline perspiration. It will be observed that the higher levels of chromate addition selected are required to produce a satisfactory performance in these respects with a high level of residual chromium as a consequence.

One of the major objectives of this investigation was to establish whether or not the more stringent demands regarding fastness to washing and to alkaline perspiration for *Superwash* wool would necessitate the use of greater quantities of dichromate than would be necessary for non-shrinkproofed wool. If this were so then the situation could arise in which no special treatment would be required after dyeing untreated wool but a treatment might be required after dyeing shrinkproofed wool.

Superwash specifications demand that the following levels of fastness are satisfied in respect of washing (TM 193) and alkaline perspiration (TM 174).

Change of shade	3 — 4
Hercosett staining	4
Cotton staining	3 — 4

Inspection of the fastness ratings indicate that two dyes, namely Mordant Orange 44 (Table II) and Mordant Black 11 (Table VII) fail to satisfy these requirements irrespective of the level of applied dichromate, the Orange 44 failing TM 174 and the Black 11 failing TM 193, respectively. The remaining dyes satisfy the requirements without the use of the higher levels of dichromate, although in some cases, the level required is higher than that necessary for untreated wool.

On chlorinated wool the fastness ratings are a little improved compared with results on chlorine-Hercosett treated wool in respect of the fact that those dyes failing the *Superwash* specification on the latter substrate, irrespective of the level of applied dichromate, were found to satisfy these criteria, although one of the dyes, namely Mordant Orange 44 only just met the specification by using the higher levels of dichromate.

An overall view of the results indicates that with the exception of those dyes which reveal a weakness in wet fastness characteristics, irrespective of any previous treatment, wet fastness properties can be obtained on untreated, chlorinated and chlorine/Hercosett treated wool by using not more than 0,8% dichromate for a 3% depth of shade. This value is in close agreement with that indicated by Ciba Geigy[4]. Residual chromium levels resulting from this ratio of dye to dichromate are such that, in many cases, no effluent treatment appears necessary but, in few cases, the normal dilution resulting from rinsing, etc. should be sufficient to meet current legislation.

TABLE III

INFLUENCE OF SHRINK-RESIST TREATMENTS ON WET FASTNESS PROPERTIES AND RESIDUAL CHROMIUM VALUES FOR CI MORDANT YELLOW 5 ON WOOL AT VARIOUS LEVELS OF APPLIED DICHROMATE

Applied Chromate Conc$^{n.}$ (% omf)	Residual Chromium (mg/ℓ)		Fastness to:								
			Washing			Alkaline Perspiration			Potting		
	Cr^{6+}	Total Cr	Effect on Shade	Staining on		Effect on Shade	Staining on		Effect on Shade	Staining on	
				Wool	Cotton		Wool	Cotton		Wool	Cotton
UNTREATED FABRIC											
0,2	0	0	4—5R	5	5	5	3	4	4R	3	5
0,4	0	0,5	4—5R	5	5	5	3—4	4—5	5SR	3—4	5
0,8	0	2,8	4—5R	5	5	5	5	5	5	3—4	5
1,2	4,1	5,0	4—5R	5	5	5	5	5	5	3—4	5
1,5	11,3	17,5	4—5	5	5	5	5	5	5	3—4	5
CHLORINATED FABRIC											
0,2	0	0	4—5	4—5	4—5	4—5	3—4	4	4R	3	5
0,4	0	0,1	4—5	5	4—5	5	4	4	4—5	3—4	5
0,8	0	0,2	4—5	5	4—5	5	4—5	4—5	5	3—4	5
1,2	7,6	10,0	4—5	5	5	5	5	5	5	4	5
1,5	20,0	23,8	4—5	5	5	5	5	5	5	4	5
CHLORINE/HERCOSETT TREATED FABRIC											
0,2	0	0	4R	4—5	3	5	2	3—4	4R	3	5
0,4	0	0,5	5	4—5	3—4	5	3—4	4—5	4R	3—4	5
0,8	1,0	3,5	5	4—5	4—5	5	4—5	5	5	4—5	5
1,2	12,5	15,0	5	4—5	4—5	5	5	5	5	4—5	5
1,5	26,8	28,8	5	4—5	4—5	5	5	5	5	4—5	5

TABLE IV

INFLUENCE OF SHRINK-RESIST TREATMENTS ON WET FASTNESS PROPERTIES AND RESIDUAL CHROMIUM VALUES FOR CI MORDANT BLUE 3 ON WOOL AT VARIOUS LEVELS OF APPLIED DICHROMATE

Applied Chromate Conc$^{n.}$ (% omf)	Residual Chromium (mg/ℓ)		Fastness to:								
			Washing			Alkaline Perspiration			Potting		
	Cr^{6+}	Total Cr	Effect on Shade	Staining on Wool	Staining on Cotton	Effect on Shade	Staining on Wool	Staining on Cotton	Effect on Shade	Staining on Wool	Staining on Cotton
UNTREATED FABRIC											
0,2	0	0,2	4	5	5	5	5	5	5G	5	5
0,4	0	0,4	4—5G	5	5	5	5	5	5	5	5
0,8	0	1,2	4—5G	5	5	5	5	5	5	5	5
1,2	7,5	11,3	4—5G	5	5	5	5	5	5	5	5
1,5	21,3	26,3	4—5G	5	5	5	5	5	5	5	5
CHLORINATED FABRIC											
0,2	0	0,1	4	5	5	5	5	5	4—5G	4	5
0,4	0	0,1	3—4	5	5	5	5	5	5	4—5	5
0,8	0,6	1,5	3—4	5	5	5	5	5	5	5	5
1,2	11,3	15,0	3—4	5	5	5	5	5	5	5	5
1,5	26,3	30,0	3	5	5	5	5	5	5	5	5
CHLORINE/HERCOSETT TREATED FABRIC											
0,2	0	0,1	4	5	5	5	5	5	4—5G	4	5
0,4	0	0,3	4	5	5	5	5	5	5	4—5	5
0,8	2,1	5,9	4	5	5	5	5	5	5	5	5
1,2	20,0	23,8	4	5	5	5	5	5	5	5	5
1,5	35,0	38,8	4	5	5	5	5	5	5	5	5

TABLE V

INFLUENCE OF SHRINK-RESIST TREATMENTS ON WET FASTNESS PROPERTIES AND RESIDUAL CHROMIUM VALUES FOR CI MORDANT BLUE 1 ON WOOL AT VARIOUS LEVELS OF APPLIED DICHROMATE

Applied Chromate conc n. (% omf)	Residual Chromium (mg/ℓ)		Fastness to:								
			Washing			Alkaline Perspiration			Potting		
	Cr^{6+}	Total Cr	Effect on Shade	Staining on		Effect on Shade	Staining on		Effect on Shade	Staining on	
				Wool	Cotton		Wool	Cotton		Wool	Cotton
UNTREATED FABRIC											
0,2	0	0	3	5	5	5	5	5	5	4	5
0,4	0	0,5	4	5	5	5	5	5	5	5	5
0,8	0	0,5	4—5	5	5	5	5	5	5	5	5
1,2	2,3	5,5	4—5	5	5	5	5	5	5	5	5
1,5	7,5	22,5	4—5	5	5	5	5	5	5	5	5
CHLORINATED FABRIC											
0,2	0	0,1	3—4	5	5	4—5	4—5	5	5	4—5	5
0,4	0	0,1	3—4	5	5	5	5	5	5	5	5
0,8	0	0,2	3—4	5	5	5	5	5	5	5	5
1,2	3,9	5,0	3—4	5	5	5	5	5	5	5	5
1,5	13,8	33,8	3—4	5	5	5	5	5	5	5	5
CHLORINE/HERCOSETT TREATED FABRIC											
0,2	0	0	3—4D	5	5	5	5	5	5	4—5	5
0.4	0	1,5	3D	5	5	5	5	5	5	5	5
0,8	1,1	2,5	3D	5	5	5	5	5	5	5	5
1,2	11,3	38,8	3D	5	5	5	5	5	5	5	5
1,5	20,0	55,0	3D	5	5	5	5	5	5	5	5

TABLE VI

INFLUENCE OF SHRINK-RESIST TREATMENTS ON WET FASTNESS PROPERTIES AND RESIDUAL CHROMIUM VALUES FOR CI MORDANT BLACK 40 ON WOOL AT VARIOUS LEVELS OF APPLIED DICHROMATE

Applied Chromate Conc n. (% omf)	Residual Chromium (mg/ℓ)		Fastness to:								
			Washing			Alkaline Perspiration			Potting		
	Cr^{6+}	Total Cr	Effect on Shade	Staining on		Effect on Shade	Staining on		Effect on Shade	Staining on	
				Wool	Cotton		Wool	Cotton		Wool	Cotton
UNTREATED FABRIC											
0,2	0	0,2	4—5	5	5	5	5	5	5	4	5
0,4	0	0,3	4—5	5	5	5	5	5	5	4	5
0,8	0,1	1,1	4—5	5	5	5	5	5	5	4—5	5
1,2	7,5	11,3	5	5	5	5	5	5	5	4—5	5
1,5	17,5	20,0	5	5	5	5	5	5	5	4—5	5
CHLORINATED FABRIC											
0,2	0	0,1	4	4	4	5	4	5	4G	3—4	5
0,4	0	0,6	4R	5	4—5	5	4—5	5	4	4	5
0,8	0	0,9	4R	5	4—5	5	5	5	4	4	5
1,2	9,0	11,5	4R	5	5	5	5	5	4—5	4	5
1,5	20,0	23,8	3—4R	5	5	5	5	5	5	4	5
CHLORINE/HERCOSETT TREATED FABRIC											
0,2	0	0,2	4D	5	5	5	4	5	4G	3—4	5
0,4	0	0,3	4D	5	5	5	4	5	4G	3—4	5
0,8	0,3	3,5	4D	5	5	5	4	5	4	4	5
1,2	9,3	15,0	4D	5	5	5	4—5	5	4	4	5
1,5	26,3	28,9	4D	5	5	5	5	5	5	4	5

TABLE VII

INFLUENCE OF SHRINK-RESIST TREATMENTS ON WET FASTNESS PROPERTIES AND RESIDUAL CHROMIUM VALUES FOR CI MORDANT BLACK 11 ON WOOL AT VARIOUS LEVELS OF APPLIED DICHROMATE

Applied Chromate Conc^{n}. (% omf)	Residual Chromium (mg/ℓ)		Fastness to:								
			Washing			Alkaline Perspiration					
	Cr^{6+}	Total Cr	Effect on Shade	Staining on Wool	Staining on Cotton	Effect on Shade	Staining on Wool	Staining on Cotton	Effect on Shade	Staining on Wool	Staining on Cotton
UNTREATED FABRIC											
0,2	0	0	5	5	5	5	4—5	5	5	3	5
0,4	0	0	5	5	5	5	5	5	5	4—5	5
0,8	0	0,1	5	5	5	5	5	5	5	4—5	5
1,2	0,3	0,7	5	5	5	5	5	5	5	4—5	5
1,5	1,0	1,9	5	5	5	5	5	5	5	4—5	5
CHLORINATED FABRIC											
0,2	0	0	4—5	3	3	5	4	4	5	3—4	5
0,4	0	0	5	3—4	4	5	4—5	5	5	3—4	5
0,8	0	0,2	5	4	4	5	4—5	5	5	4—5	5
1,2	0,3	0,9	5	4	4—5	5	4—5	5	5	4—5	5
1,5	2,5	9,4	5	4—5	4—5	5	4—5	5	5	4—5	5
CHLORINE/HERCOSETT TREATED FABRIC											
0,2	0	0	3	4—5	5	5	3	3—4	5	4	5
0,4	0	0	3	4—5	5	5	4	4—5	5	4	5
0,8	0	0,1	3	4—5	5	5	4—5	5	5	4—5	5
1,2	0,5	2,1	3	4—5	5	5	4—5	5	5	4—5	5
1,5	7,5	11,3	3	4—5	5	5	4—5	5	5	4—5	5

No account has been taken in this investigation of the influence of the depth of shade of each dye. It remains a possibility that with greater depths the need for increased amounts of dichromate may create a different picture to that observed here.

One interesting feature observed with some dyes on chlorinated and chlorine-Hercosett treated wool is the slight increase in the change of shade after washing, with increase in dichromate. This may be observed with Mordant Black 40 (Table VI) and Mordant Blues 1 (Table V) and 3 (Table IV). It is possible that the effect is the result of oxidation during the dyeing stage or during the washing operation. Some chrome dyes are known to be susceptible to the perborate component in the detergent used in the TM 193 test.

SUMMARY AND CONCLUSIONS

Shrink resist treatments on wool give rise to significantly increased residual chromium contents in the spent liquor compared to untreated wool only when relatively high levels of dichromate are employed. When the recently proposed reduced levels of dichromate are used on untreated, chlorinted and chlorine/Hercosett treated wool the residual chromium is at such a level that no special treatment appears necessary except in a few cases where the normal dilution of the bath with rinse water should suffice.

Wet fastness properties of dyed shrink-resist treated wools are capable of meeting *Superwash* standards when low additions of dichromate are employed, the only exceptions being those dyes which show poor wet fastness whether or not the substrate has been treated.

Some shade changes are associated with the use of lower additions of dichromate. If it is desired to obtain the conventional shade by addition of more dichromate rather than by shading then a treatment to reduce the chromium content of the spent liquor would be required.

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THE USE OF PROPRIETARY NAMES

The product names ®Eriochrome and ®Albegal are registered trade marks of Ciba Geigy Ltd, ®Basolan is a registered trade mark of BASF Ltd., ®Nonidet is a registered trade mark of Shell Chemical Ltd. The fact that chemicals with proprietary names have been mentioned in this report in no way implies that there are not others of equal or greater merit.

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