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The Application of a More Rapid Dyeing Technique to the Chrome Dyeing of Wool

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THE APPLICATION OF A MORE RAPID DYEING TECHNIQUE TO THE CHROME DYEING OF WOOL

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ABSTRACT

In the chrome dyeing of wool by the afterchrome process, a saving of time has been achieved during the dyeing phase by commencing the dyeing at a higher temperature than normal and controlling exhaustion by means of a steady reduction in pH. The final pH is that at which chroming occurs.

It has been found possible to avoid the necessity of reducing the temperature prior to addition of the dichromate by its gradual addition at the dyeing temperature over a period of 5 minutes.

INTRODUCTION

The rapdily escalating price of reactive dyestuffs has resulted in renewed interest in the use of chrome dyes as a means of producing dyeings of high wet fastness on wool. Recently, problems concerning residual chromium in spent dye liquor threatened the use of these dyestuffs but fortunately these have now been largely overcome, either by using considerably less dichromate or by using the lactic acid technique developed by IWS[1].

Chrome dyeing by the afterchrome process, the normal procedure for obtaining the optimum wet fastness, suffers from the disadvantage that it is extremely time consuming. An inspection of recommended methods of chrome dyeing indicates that the complete process, excluding rinsing, may take from 130 to 180 minutes, of which the dyeing stage, as distinct from the chroming stage, occupies from 90 to 125 minutes. At the present time, there appears to be no way of accelerating the chroming process apart from the use of elevated temperatures but this approach may lead to unacceptable fibre modification. Apart from this aspect the vast majority of wool dyeing machinery is designed to operate at temperatures at or below the boil. Hine and McPhee[2] claimed that chrome dyes could be applied to wool in about 200 minutes with a maximum temperature of 85°C but no mention was made of the applicability of the system to high twist yarns or to garments containing thick seams. Slinger *et al* [3] also examined the chrome dyeing of wool at 85°C but their investigation was confined to wool top dyed black.

It was, therefore, considered worthwhile exploring the possibility of speeding up the dyeing process, taking into account the temperature factor, particularly in the light of recent improvements in dye liquor circulation, and to establish whether such a process could successfully be applied to yarn, piece goods and garments.

Two fairly recent publications suggested that this approach might be fruitful. The first is the development by L.B. Holliday of a rapid dyeing method for Superwash wool[4]. This company has shown that it is possible to reduce the dyeing time for reactive dye and 1 : 2 metal complex dyes from 210 minutes to 150 minutes by commencing dyeing at 70° C and maintaining this temperature whilst adding first the auxiliaries followed by the dye. The temperature is then raised rapidly to the boil and maintained at the boil for from 30 to 90 minutes. They also recommend an ultra high production system with a starting temperature of 100° C which, it is claimed, reduces the conventional dyeing time by 100 minutes.

The second development, patented by Carpets International[5], concerns a method of applying, to wool and polyamide fibres, dyes whose exhaustion depends upon pH. The fibre is immersed in a dyebath at a pH of about 7,5 at a constant temperature and the pH is gradually reduced to promote exhaustion. Depending upon the final pH, the patentees claim that it is frequently possible to obtain 99% exhaustion and that the hot exhausted bath may be reused after recharging with dye and raising the pH.

The object of the present investigation was to establish whether the dyeing stage could be accelerated by omitting the heating-up phase and controlling the exhaustion by means of adjusting the pH to the extent that, at the end of the dyeing stage, exhaustion would be virtually complete and the pH would be of the order of 3,5, this being the optimum pH for the chroming stage so far as minimumresidual chromium is concerned.

EXPERIMENTAL

Materials

Laboratory scale experiments were conducted on a plain weave all-wool worsted material of fabric density 140 g/m². The fabric was scoured, semi-milled and decatised prior to the experimental work. Pilot scale trials were carried out using the above fabric and also a R60 tex/2 100% Botany wool yarn which had been previously scoured.

Dyeings

Dyeings were carried out on a laboratory scale using an Ahiba Turbomat TM6 machine with perforated beam attachments. Pilot scale dyeings were conducted on a small Longclose winch and on a single package Longclose machine.

Three dyes were employed from the ®Eriochrome range, namely Cyanine R (Mordant Blue 3), Flavine A (Mordant Yellow 5) and Black T (Mordant Black 11). The depth of dyeing was 3% (omf) for shades other than the black which was 8% (omf). Demineralised water was used throughout.

Various dyeing systems were employed as illustrated under "Results and Discussion". In all these systems formic acid was used to control the pH. Sodium sulphate was used in minimal quantities on account of its tendency to reduce the absorbtion of dichromate ions. To assist the levelling action, levelling agents other than anionic types were employed. A control dyeing using a conventional method was also carried out. In the experiments concerned with the simplification of the chroming procedure, the quantity of dichromate employed was 0,8% (omf).

Pilot scale dyeings were conducted using both the standard method and the optimum methods suggested after laboratory work.

Testing

Exhaustion values were determined at appropriate points in the dyeing cycles by taking spectrophotometric readings of aliquots of the dye liquor.

Levelling characteristics of the various dyeing systems were determined in the following manner. Dyeings were first carried out together with blank dyeings. At the end of the process, the dyed wool and blank dyed wool samples were cut in half and a portion of each was placed in a bath prepared by mixing half of each of the dyeing liquors. The pH of the bath was readjusted to its initial value and the dyeing cycle was repeated. Samples of dyed and undyed wool were removed at appropriate points in the cycle. The dye was extracted from the fibre with an aqueous pyridine solution (80/20) and determined spectrophotometrically. The degree of levelness is expressed percentagewise, higher values indicating the greater degree of levelling.

In the case of the pilot scale dyeings, penetration was checked while dyeing on the winch by sewing a 2 cm seam of three thicknesses of fabric across the width. Levelness was assessed in the single package machine by winding bands of dyed and undyed yarn on a single dye spring and repeating the dyeing cycle.

RESULTS AND DISCUSSION

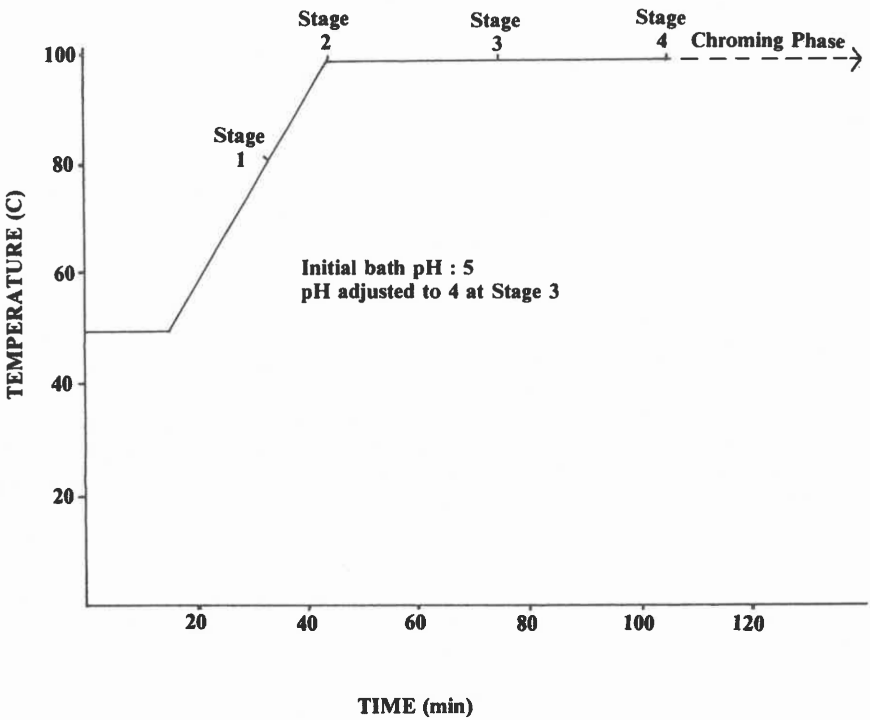
The characteristics of the standard method of afterchrome dyeing are shown in Table I. It will be observed that the rate of exhaustion is particularly high for Mordant Yellow 5 and Mordant Black 11, at least 75% of the dye being exhausted by the time the temperature has reached 80°C. The levelling characteristics indicate that the same two dyes show good migration but that the Mordant Blue 3 is markedly inferior in this respect.

An initial dyeing trial at 100°C with a commencing pH of 4 shows extremely rapid exhaustion for all three dyes and also very good migration characteristics, as shown in Table II. The rate of exhaustion may well be too

TABLE I

STANDARD METHOD OF AFTERCHROME DYEING

Auxiliaries: 1,5% Sodium sulphate
1% ®Albegal A

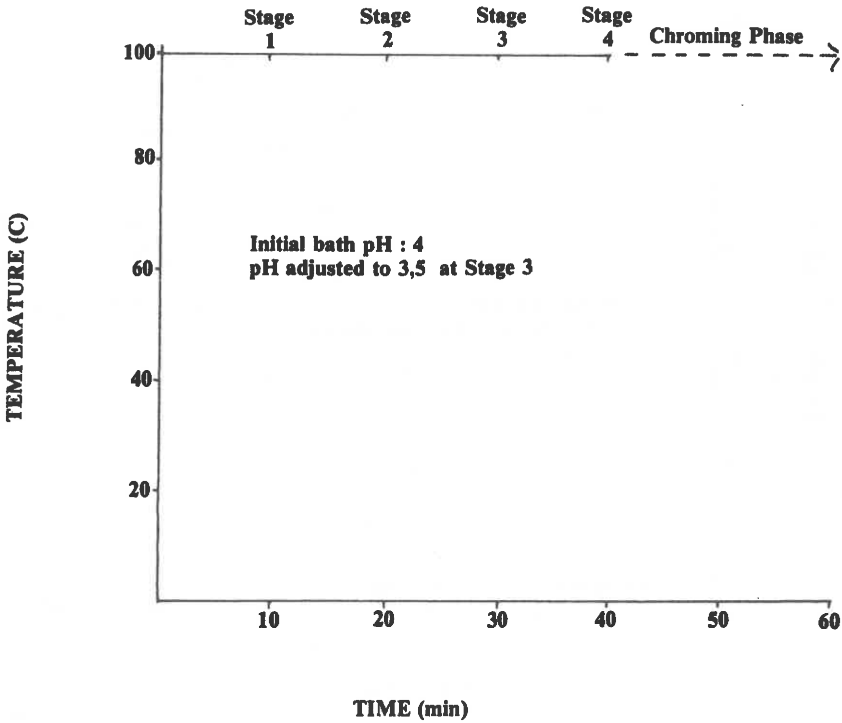


	Stage	Mordant Blue 3	Mordant Yellow 5	Mordant Black 11
Exhaustion (%)	1	48	84	77
	2	84	86	88
	3	93	85	90
	4	99	98	97
Levelling (%)	1	9	34	14
	2	10	41	15
	3	13	44	44
	4	24	46	46

TABLE II

DYEING AT 100°C WITH LOW pH AT COMMENCEMENT OF DYEING

Auxiliaries: 1,5% Sodium sulphate
1% ®Albegal A

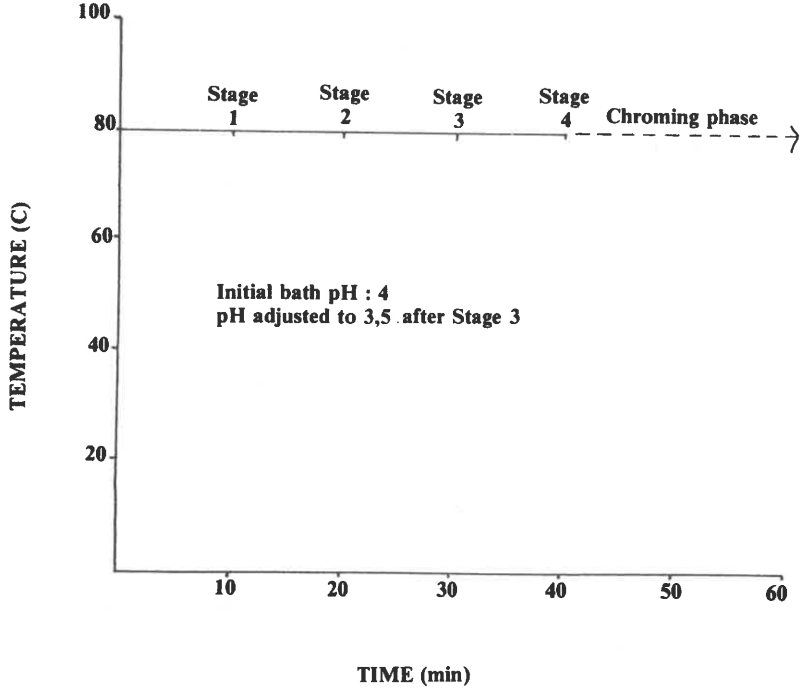


	Stage	Mordant Blue 3	Mordant Yellow 5	Mordant Black 11
Exhaustion (%)	1	91	81	74
	2	96	91	80
	3	97	92	83
	4	98	96	94
Levelling (%)	1	30	41	32
	2	37	44	40
	3	41	47	47
	4	44	48	49

TABLE III

DYEING AT 80°C WITH LOW pH AT COMMENCEMENT OF DYEING

Auxiliaries: 1,5% Sodium sulphate
1% ®Albegal A

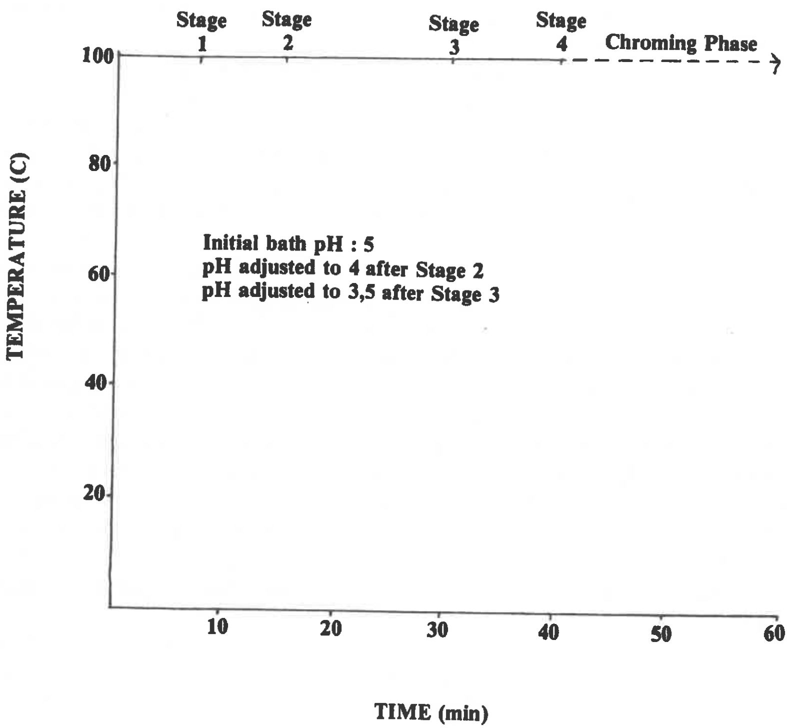


	Stage	Mordant Blue 3	Mordant Yellow 5	Mordant Black 11
Exhaustion (%)	1	66	86	74
	2	95	91	88
	3	98	87	95
	4	100	99	97
Levelling (%)	1	15	37	13
	2	18	39	17
	3	22	42	25
	4	25	43	28

TABLE IV

DYEING AT 100° C WITH STEADILY DECREASING pH

Auxiliaries: 1,5% Sodium sulphate
1% ®Albegal A



	Stage	Mordant Blue 3	Mordant Yellow 5	Mordant Black 11
Exhaustion (%)	1	32	21	43
	2	64	39	68
	3	90	90	87
	4	97	97	93
Levelling (%)	1	12	21	15
	2	22	41	35
	3	30	49	40
	4	39	49	48

great for many purposes and thus other methods were examined. Table III shows the results for a similar system carried out at 80°C. Although the exhaustion in the early stage is reduced the levelling potential is also much reduced with Mordant Blue 3 and Mordant Black 11.

Table IV shows the results of a system designed to decrease the rate of exhaustion while maintaining a high degree of migration. The use of an initial pH of 5 is clearly advantageous with respect to the rate of exhaustion. The final migration figures are similar to those of the standard method in two cases and much superior in the case of Mordant Blue 3.

Taking into account the fact that a considerable proportion of available chrome dyes may be chromed at 90°C, a dyeing trial at that temperature was also examined. The results are shown in Table V. The exhaustion proceeds at a fairly steady rate and the migration characteristics, although not as good as those obtained when dyeing at 100°C, are more uniform than those obtained by the standard method. Such a system should be adequate for dyeing in top or loose stock form.

At this stage various levelling agents and combinations thereof were examined in order to assess their suitability for use under these conditions. The optimum system appeared to be a combination of equal quantities of Albegals A and W, the total amount being 1% (omf) used in conjunction with 1,5% sodium sulphate. Although the latter component blocks sites in the fibre which would be taken up by dichromate anions during the chroming process its presence does promote the efficiency of these levelling agents. Addition of the auxiliaries to the system prior to the addition of the dye was found beneficial with respect to levelling and penetration. With the rapid circulation in the laboratory equipment a time interval of 5 minutes between addition of auxiliaries and dye was found adequate. Thus the optimum system is illustrated in Table VI. It results in a fairly steady exhaustion rate and gives rise to good levelling characteristics.

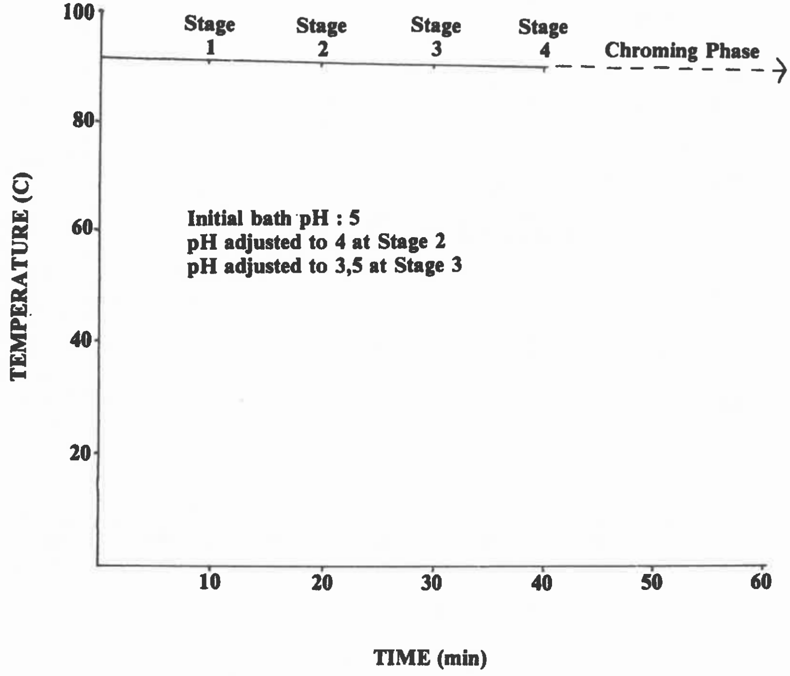
In most afterchrome processes, it is recommended that the bath temperature should be reduced to about 80°C before adding the dichromate in order to provide for a uniform uptake of the chromium salt. The temperature is then raised to the boil again to promote complex formation. This step is inconvenient and thus the possibility of adding the dichromate slowly at the boil was examined, particularly since from the results in Table VI, both exhaustion and levelling are essentially complete at Stage 3. It was found that by adding the dichromate over 5 minutes and continuing the boiling stage for 30 minutes an excellent dyeing could be achieved. A similarly good result was achieved by conducting the dyeing and chroming stage at 90°C although results shown in Table 5 indicate that the levelling characteristics are not quite so good.

Pilot scale winch dyeing at 100°C gave good results for all three dyes, penetration into the thick seam appearing identical to that of the standard

TABLE V

DYEING AT 90° C WITH STEADILY DECREASING pH

Auxiliaries: 1,5% Sodium sulphate
1% ®Albegal A

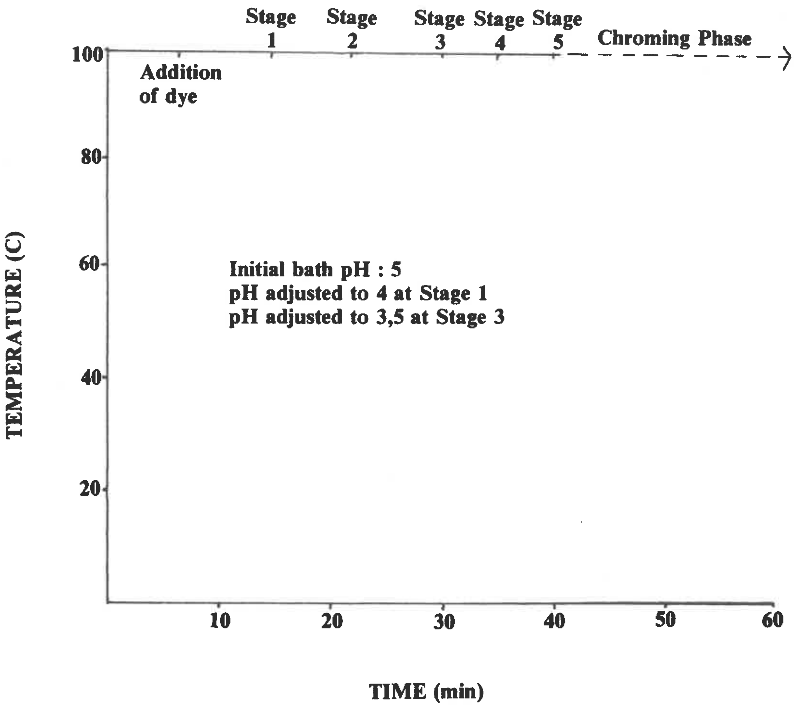


	Stage	Mordant Blue 3	Mordant Yellow 5	Mordant Black 11
Exhaustion (%)	1	27	20	38
	2	60	37	62
	3	86	83	80
	4	96	95	93
Levelling (%)	1	10	16	12
	2	17	37	22
	3	27	44	30
	4	30	45	38

TABLE VI

DYEING AT 100° C WITH STEADILY DECREASING pH

Auxiliaries: 1,5% Sodium sulphate
1% ®Albegal A



	Stage	Mordant Blue 3	Mordant Yellow 5	Mordant Black 11
Exhaustion (%)	1	34	24	48
	2	66	44	74
	3	91	92	90
	4	98	97	91
	5	98	98	99
Levelling (%)	1	22	22	19
	2	30	28	30
	3	38	42	46
	4	38	44	46
	5	40	44	48

method on the winch. Yarn dyeing in package form also gave good results. In the case of Mordant Blue 3, the dye showing the least propensity to migrate, results were significantly better than for the standard method. The migration value for the standard method was 24% and that for the proposed method 35%.

Similar trials at 90°C gave level dyeings on both the winch and the package machines but seam penetration on the winch was unsatisfactory and the migration value was only 27% for the Mordant Blue 3.

SUMMARY AND CONCLUSIONS

The purpose of this investigation was to examine the possibility of reducing the time necessary for the dyeing of wool by the afterchrome method. The results suggest that the use of a gradual reduction in pH, allied to the commencement of the dyeing at elevated temperatures, enables dyeings of good penetration and levelness to be achieved with a dyeing phase of only 40 minutes.

It is well known that chrome dyeing is one of the most damaging processes so far as the wool fibre is concerned and that reduction in both duration and temperature of process are desirable. For this reason dyeings were conducted at 80°C and 90°C as well as at the boil. The evidence suggests that although a maximum temperature of 90°C would be adequate for dyeing wool in loose stock or top form, it is unlikely to be adequate for yarns of high twist or garments with thick seams on account of inadequate penetration. A dyeing temperature of 80°C is even less satisfactory.

A simplification of the afterchroming phase has been achieved by adding the dichromate over a period of 5 minutes at the elevated temperature with the pH already adjusted to 3,5.

ACKNOWLEDGEMENTS

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THE USE OF PROPRIETARY NAMES

The product names ®Eriochrome and Albegal are registered trade marks of Ciba Geigy 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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