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A Preliminary Investigation into Dyeing by Low Add-On Techniques: Reactive Dyes on Cotton by Loop-Transfer

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A PRELIMINARY INVESTIGATION INTO DYEING BY LOW ADD-ON TECHNIQUES : REACTIVE DYES ON COTTON BY LOOP TRANSFER

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ABSTRACT

The loop-transfer technique was used to dye a cotton fabric with reactive dyes. Depending on the dyestuffs, level dyeings were obtained at pick-up levels of only 35% to 40% (omf) using a transfer to both sides of the fabric. A suitable way to avoid any shade differences between face and back of the fabrics was simultaneous transfer. A high transfer pressure and a suitable non-ionic surfactant were essential to reduce the pick-up as far as possible without sacrificing levelness. The tendency of the dyestuffs to migrate was considerably reduced. Maximum fixation values were between 79% and 90% and were in close agreement with the values obtained with conventional padding. Transfer speeds between 0,75 m/min and 5 m/min were used.

INTRODUCTION

The finishing and dyeing of textiles by padding techniques is well established. However the minimum pick-up which can be obtained by padding is only 60% to 70%. This high wet pick-up is not considered to be necessary for the finishing process[1], but is merely a result of the basic principle involved, namely oversaturation of the fabric in a trough or nip followed by squeezing.

A high wet pick-up exhibits several disadvantages:

An unnecessary amount of water is consumed for the process of finishing or dyeing and additional energy is required for the drying process. The drying stage is one of the most expensive operations in the entire finishing sequence[1]. The importance of this cost factor will undoubtedly increase in the future due to the increasing cost of energy. Furthermore, during the drying process, migration of the finishing chemicals or dyestuffs is possible, resulting in an increased concentration at the surface and selvedges of the fabric. In the case of easy-care resins this results in a reduction in crease recovery and mechanical strength of the fabric[1, 2]. The excess of water may also require greater quantities of auxiliaries, or even extra auxiliaries, e.g. migration inhibitors. During the passing of the fabric through the trough or nip, chemicals of low fibre affinity previously applied are leached. For that reason the curing of THPOH treated cotton for example requires a gaseous ammonia fixation and the technically simpler padding with an alkali solution is not possible[3].

Recently, some new application methods have been developed which allow lower wet pick-up values to be attained in order to overcome some of the abovementioned problems. These include the pad-transfer technique, the kiss-roll technique and the loop technique.

In the *pad-transfer technique*, the padded fabric, after squeezing, comes into contact with fabric which is still dry and transfers, during a second squeezing, a certain amount of liquor to the dry fabric[4,5]. Due to the initial oversaturation, the remaining pick-up is relatively high and is still in the pick-up range obtainable with a good pad mangle[5].

The *kiss-roll* and *loop-transfer techniques* offer much lower pick-ups, as only a limited amount of liquor is available. In the kiss-roll technique, the pick-up is controlled either by a different speed of kiss-roll and fabric[1], a method already used in industry for finishing (Triatex MA technique), or by adjusting the viscosity of the liquor[6]. The 3-roll loop-transfer technique does not only allow a variation of the transfer-pressure, but also a variation of the squeeze-pressure, this resulting in a controlled pick-up of liquor at the transferring loop. In this way, a 3-roll loop transfer offers a wide range of pick-ups by simple mechanical adjustments.

The techniques mentioned have been mainly used for the application of finishing agents. The possibility of dyeing using low add-on techniques is mentioned[6,7], but systematic investigations do not seem to have been reported.

This study reports on the use of a 3-roll loop transfer system or the dyeing of cotton with reactive dyes. The investigation was designed to obtain some information about the influence of transfer conditions and liquor composition on pick-up, as well as on levelling and fixation.

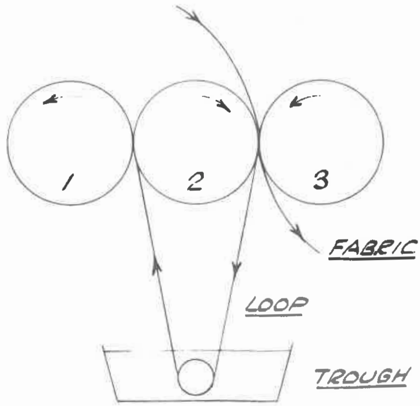
EXPERIMENTAL

Fabric:

Except where specified otherwise, an all cotton double jersey fabric of 300 g/m², scoured and bleached, was used throughout this study.

Padder:

Benz laboratory 3-roll horizontal padder, type KLFH "K" (see Diagram)



Loop-transfer with 3-roll-padder

Note: The squeeze pressure between rolls 1 and 2 controls the effective pick-up of the loop from the trough while adjustment of the pressure between rolls 2 and 3 (transfer pressure) controls the transfer of liquor to the fabric. (Arbitrary scale from 0 to 10 for both squeeze and transfer pressure).

Loop:

An all-cotton terry towelling material of 380 g/m² was used.

Transfer liquors (Percentages based on m/v solution):

The standard recipe for ®Procion MX dyes comprising 6% Procion MX dyestuff, 0,5% urea, 2% $NaHCO_3$, 1% Na_2CO_3 and 0,2% ®Nonidet P40 was used throughout. The alkali was diluted separately and added to the dyestuff solution immediately before use to bring the solution to the above composition.

The standard recipe for ®Procion H dyes comprises: 6% Procion H dyestuff, 20% urea, 2% Na_2CO_3 and 1,5% ®Lissapol NX (non-ionic detergent). Alterations in concentrations or composition made to determine the influence of changing conditions on pick-up, levelling and fixation are mentioned under "Results and Discussion".

The variations cover the following range/
Procoin H dye from 1% to 6%,
Lissapol NX from 0,75% to 5%,
replacement of Lissapol NX ®Aerosol OT or ®Nonidet P40,
urea from 10% to 20%

Padding liquor[8] (Percentage m/v)

3% Procion H dye, 20% urea, 2% Na_2CO_3 and 1% Aerosol OT.

Transferring

Transfer was carried out by passing the fabric through the loop-padder twice with each side of the fabric having contact with the loop once.

Drying and curing:

The loop-transfer-dyed or padded fabrics were stored for 60 to 90 min at room temperature, then dried at 50° C for 15 min and baked at 155—160° C for a further 15 min.

Determination of covalent fixation

Representative samples of about 1 g were soxhlet-extracted with 80 mℓ of water/pyridine (80/20) for at least 90 min. The extract was then made up to volume and the concentration of the removed dyestuff determined photometrically.

RESULTS AND DISCUSSION

Preliminary results

Preliminary tests indicated that a transfer-pressure lower than the squeeze-pressure should be employed to avoid a build-up of liquor in the transfer nip. When working discontinuously, the liquor in the nip causes a higher pick-up on the first part of the fabric as well as a continuous flow of liquor to the selvedges of the fabric (provided that the loop is wider than the fabric). When working continuously, the liquor flows towards the selvedges of the fabric, thus resulting in deeper dyed selvedges. The same width for both the loop and the fabric which is being dyed should, therefore, avoid the above mentioned difficulties. The use of an overlapping loop seam for transfer causes a higher pick-up. For this reason the loop should be endless or straight stitched.

Low levels of pick-up reduced migration of dye-stuff considerably. In comparison to padded fabrics it was frequently not necessary to suspend loop-transfer dyed fabrics during drying in order to avoid migration.

Transfer of the dye-liquor to only one side of the fabric resulted in an uneven distribution of dye throughout the fabric. Even pick-ups higher than 40% and storing the fabrics for several hours did not effect an even penetration. This was unexpected judging from publications concerned with the application of finishing agents by means of low add-on systems[1,2,7]. All these publications claim an even distribution of finishing agents. Unfortunately, the way in which the distribution was determined is nowhere described. Provided the finishing agents were really distributed evenly, the different behaviour found for dyestuffs may be attributed to the fibre affinity of dyestuffs, which is considerably higher than that for finishing agents.

Loop-transfer applied to both sides of the fabric, allowed total pick-ups of less than 25%, and therefore it was possible to transfer dye liquor to both sides of the fabric in the following experiments without sacrificing the desired low pick-up. However, the loop-transfer technique posed some mechanical problems. The loop material tended to stretch, thus causing creases and folds which resulted in an uneven application of the transfer liquor. Furthermore, there was a certain tendency for the loop to move to one side of the padder. The choice of a mechanically more stable loop material (for example man-made fibre reinforced towelling material) together with some additional tensioning devices to the padder itself would probably have reduced these problems. A replacement of the loop and the loop roll by a non-woven covered roll might have improved the system even more.

1. Variation of mechanical conditions

The first experiments were designed to determine the influence of different transfer conditions on loop-transfer at a *constant liquor composition.*

Influence of transfer-pressure on penetration and levelling

The same level of pick-up can be obtained by either using a low squeeze pressure (resulting in a high pick-up of the loop) and a low transfer-pressure or by employing a high squeeze pressure (giving a low loop pick-up) and a high transfer-pressure. To protect the structure of a pressure-sensitive fabric, a transfer-pressure as low as possible would, therefore, be desirable. However, experiments performed using different squeeze-and transfer-pressures, which were chosen to give the same pick-up, indicated that the fabrics, which were dyed using a higher transfer-pressure, showed better penetration (resulting in a slightly brighter shade) and were more level. Since penetration and levelling are considered to be the limiting factors for a further reduction of pick-up, a high transfer-pressure is inevitable to reduce the pick-up as far as possible.

Influence of transfer speed on pick-up and fixation

The influence of transfer speed on pick-up in the range of 0,75 to 5 m/min is shown in Fig 1. An increase in pick-up from about 35% to 46% (omf) results from increasing speed in this range. This result obtained for the relatively low speed range contrasts significantly with the results published for the speed range from 5 m/min to 30 m/min, in which no essential increase in wet pick-up was found[5]. Further experiments clearly showed that the increase in pick-up with increasing speed was due to the presence of Lissapol NX.

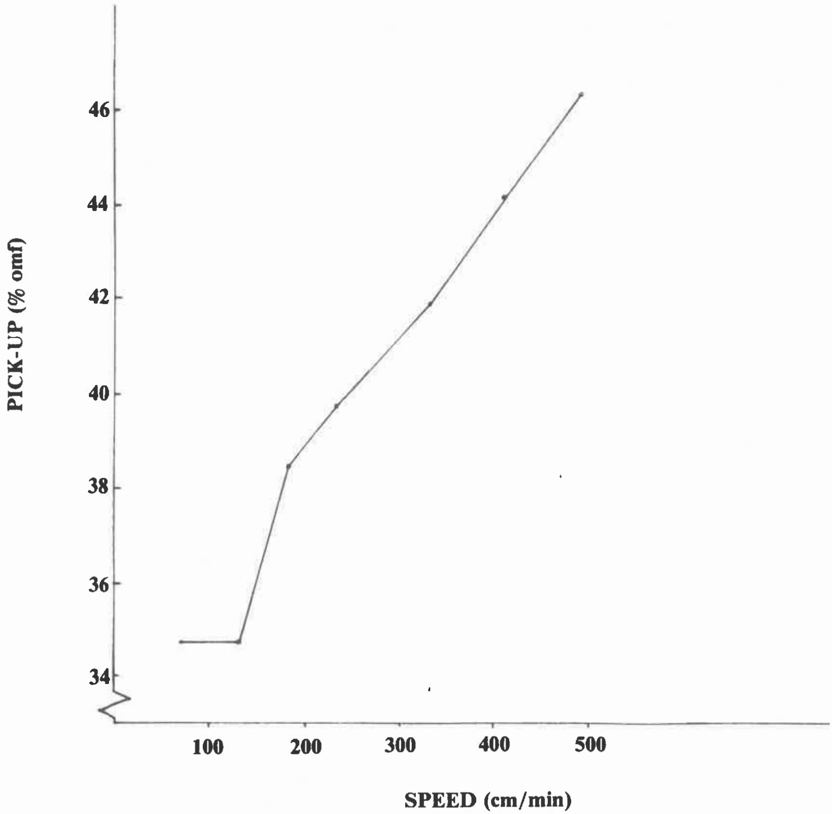


Fig. 1 Influence of transfer speed on pick-up
6% Procion Yellow H-3R, 20% urea, 2% Na_2CO_3, 1,5% Lissapol NX, loop pressure = 10, transfer pressure = 4

To avoid the possible influence of different pick-up levels on the degree of fixation, the influence of speed was investigated using fabrics having the same pick-up. The transfer speed had no practical effect on the fixation values for Procion Yellow H-3R (Fig 2).

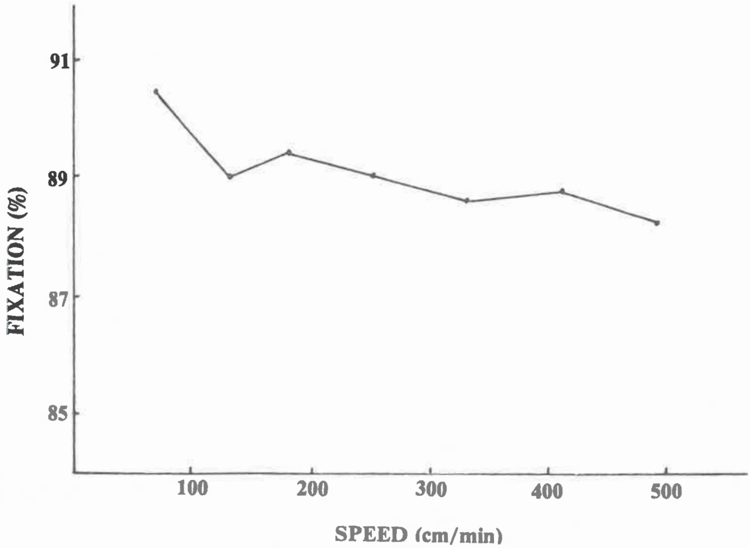


Fig. 2 Influence of transfer speed on fixation
6% Procion Yellow H-3R, 20% urea, 2% Na_2CO_3, 1,5% Lissapol NX pick-up range 36 – 41% (omf)

Reproducibility of pick-up

The reproducibility of pick-up under different conditions is shown in Tables I and II. For low squeeze- and transfer- pressures, pick-up differences up to 6,6% from the average pick-up were found (Table I). This means that tolerances in the low transfer-pressure range are too large for practical purposes of dyeing, since it would be noticeable in different depths of shades in most cases. Better reproducibility was obtained when higher loop and transfer-pressures were employed (Table II). Visible shade differences are not expected when working in the higher loop-pressure range. Due to the pressure, to which the loop material is exposed, the pick-up ability of the loop itself deteriorated with time, giving lower pick-ups under the same pressure conditions. This means that even in the higher pressure range, a pressure adjustment is necessary from time to time to ensure a uniform pick-up. Therefore, for purposes of this investigation it was considered to be necessary to determine the pick-up of each experiment by weighing.

TABLE I

REPRODUCIBILITY OF PICK-UP USING LOW LOOP- AND TRANSFER-PRESSURES

Speed (m/min)	Loop pressure	Transfer pressure	Pick-up (%) omf Sample:				Average pick-up (%) omf	% difference relative to average pick-up			
			1	2	3	4		1	2	3	4
0,75	4	1	20,7	30,5	29,5	27,0	28,9	-0,7	+ 5,5	+ 2,1	+ 6,6
0,75	4	2	40,7	39,5	36,0	37,5	38,4	+6,0	+ 2,9	-6,3	-2,6
0,75	4	3	46,3	44,4	42,0	44,4	44,4	+4,3	0	-4,5	0

TABLE II

REPRODUCIBILITY OF PICK-UP USING HIGHER LOOP- AND TRANSFER-PRESSURES

Wetting agent	Speed (m/min)	Loop Pressure	Transfer Pressure	Pick-up (%) omf	Average pick-up (%) omf	% difference relative to average pick-up
1,5% Lissapol NX	0,75	10	5	36,4		0,7
				36,3		0,4
				36,4	36,15	0,7
				35,6		-1,5
				36,5		1,0
				35,7		-1,2
1,5% Aerosol OT	0,75	10	5	39,4		-3,0
				40,2		-1,0
				40,9	40,62	0,7
				41,6		2,4
				40,0		1,5
				41,6		2,4

NOTE: Transfer and loop transfer pressures are given in arbitrary units (existing on the padder used)

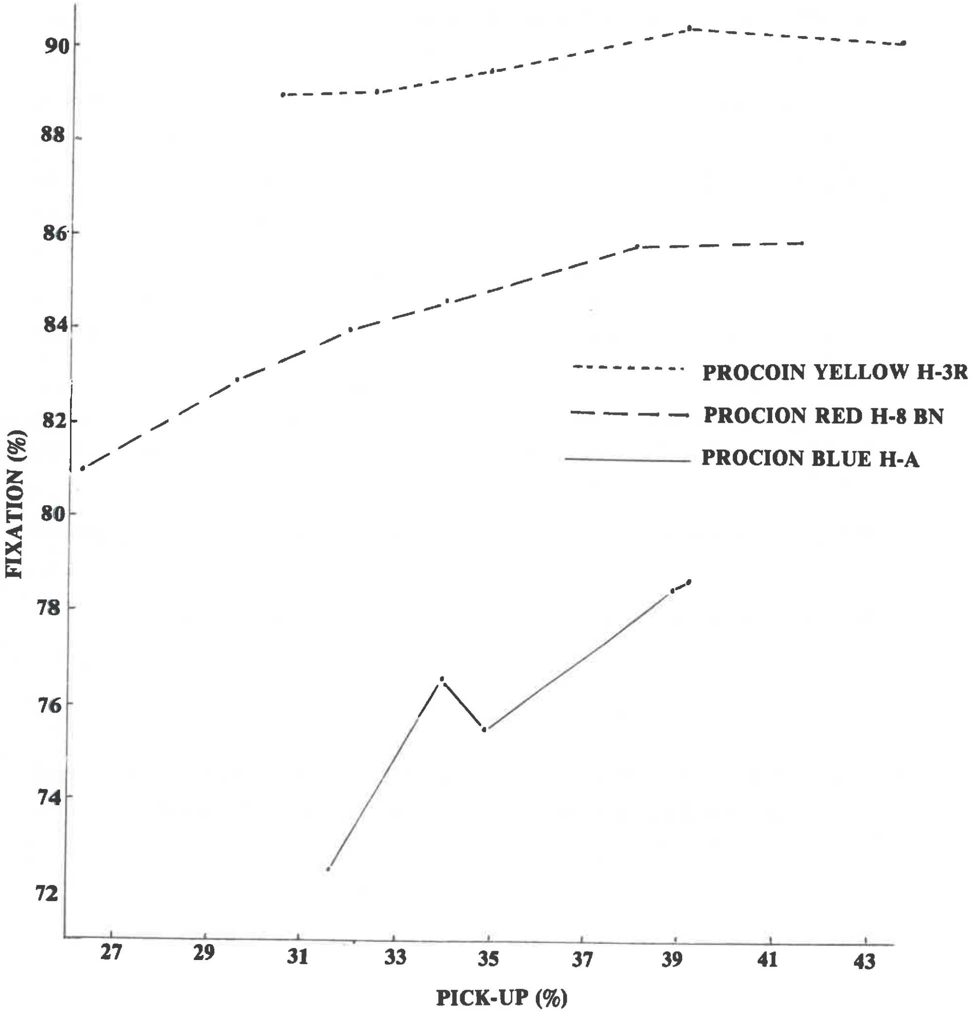


Fig. 3 Influence of pick-up of loop-transfer liquor on fixation. 6% Procion H dye, 20% urea, 2% Na_2CO_3, 1,5% Lissapol NX

Influence of pick-up on degree of fixation

The influence of pick-up on fixation for Procion Red H-8BN, Blue H-A and Yellow H-3R is shown in Fig 3. The curves for the different dyestuffs suggest that a certain level of pick-up is necessary to ensure the highest possible fixation. Fixation values of 78% were obtained for Blue H-A, 86% for Red H-8BN and 90% for Yellow H-3R. The pick-up necessary to obtain maximum fixation was similar to the pick-up necessary to obtain level dyeings. Procion Yellow H-3R, showing the best levelling, showed fixation values close to the maximum already at only 35% pick-up. The maximum fixation of Blue H-A, on the other hand, which showed the poorest levelling, required a pick-up of about 40% (omf). Furthermore, the maximum fixation of dyestuffs showing a poorer levelling depended strongly on the level of pick-up. These observations suggest that a minimum pick-up, depending on the levelling behaviour of the dyestuff used, is necessary to distribute the dyestuff within the fibre in such a manner that maximum fixation can occur.

2. **Influence of variations in the transfer liquor composition on pick-up, levelling and degree of fixation**

Influence of different wetting agents on pick-up and levelling

From Table III it can be seen that for the same transfer conditions, Aerosol OT (anionic wetting agent) effected a higher pick-up than Nonidet

TABLE III
INFLUENCE OF WETTING AGENT ON PICK-UP FOR DIFFERENT LOOP-TRANSFER PRESSURES

Wetting agent	Speed (m/min)	Loop Pressure	Transfer Pressure	Pick-up (%) omf
1% Nonidet P40	0,75	4	1	24,7
	0,75	4	2	30,2
	0,75	4	3	34,7
1% Aerosol OT	0,75	4	1	28,7
	0,75	4	2	40,7
	0,75	4	3	46,3
1% Nonidet P40	0,75	10	3	27,5
	0,75	10	4	31,5
	0,75	10	5	35,5
	0,75	10	6	37,5
1% Aerosol OT	0,75	10	3	29,2
	0,75	10	4	37,0
	0,75	10	5	35,4
	0,75	10	6	39,8

P40 (non-ionic wetting agent). The influence of the different wetting agents on pick-up was more evident in the low transfer-pressure range (using a high loop liquor content) and diminished with higher transfer-pressures (using a low loop liquor content). Even when working with higher transfer-pressures, the amount of wetting agent used influenced the pick-up (Table IV). The results shown in Tables I to III were obtained with a highly reactive Procion MX dyestuff. Because different levels of pick-up influence the levelling, the influence of the different wetting agents on levelling could not be assessed.

TABLE IV

INFLUENCE OF AMOUNT OF WETTING AGENT ON PICK-UP

Wetting agent	Speed (m/min)	Loop pressure	Transfer pressure	Pick-up (%) omf
0,2% Nonidet P40	0,75	10	4	27,4
	0,75	10	6	35,5
	0,75	10	10	38,6
1% Nonidet P40	0,75	10	4	31,5
	0,75	10	6	37,5
	0,75	10	10	41,5

Assuming that the use of a dye of lower affinity should result in a more level dyeing, the influence of a non-ionic detergent on the levelling of a dyestuff with low reactivity was investigated. Non-ionic surfactants can form a complex with reactive dyes, thus retarding the diffusion of dye into the fibre. Normally, they are to be avoided for padding liquors[8]. It was hoped that the levelling would be improved by the retarding effect of Lissapol NX, a non-ionic detergent. Therefore, the levelness of dyeings obtained from transferring liquors containing Lissapol NX or Aerosol was compared. Knowing that different transfer-pressures as well as different pick-up levels can cause differences in levelling, the same transfer pressure was used, thus resulting in higher pick-ups for the liquor containing Aerosol OT. After loop-transfer some of the samples were baked immediately while others only after a storage period of 90 min to allow levelling to take place. It was found that storage prior to fixation improved levelling for samples treated with both surfactants. However, the samples treated with Lissapol NX showed a much better levelling at only 36% pick-up than the samples treated with Aerosol OT at a pick-up of 41%. This clearly indicated the positive effect of a non-ionic surfactant on the levelling of dyes with low reactivity in the case of loop-transfer. The dyeings obtained with Lissapol

NX in the liquor were commercially acceptable in the case of Procion Red H-8BN, and even better for Yellow H-3R, at a pick-up of about 36%. Blue H-A showed slightly inferior levelling, but this could have been improved by increasing the pick-up to about 40%.

Influence of dye concentration on degrees of fixation

The influence of dye concentration in the loop-transfer liquor was investigated using the standard recipe for Procion H dyes but increasing the dye concentration from 1% to 6%. This means that for a pick-up of from 37% to 39% the dyestuff concentration increased from about 0,4 to 2,4% (omf). Fig 4 shows fixation values from about 75% for Blue H-A to about 89% for Red H-8BN.

The difference in fixation found for Blue H-A could have been caused by a certain instability of the dye in the pyridine/water mixture used for spectrophotometric determination. (This was also observed when establishing the calibration curves). The dye concentration within the investigated concentration range was found to have no influence on the degree of fixation.

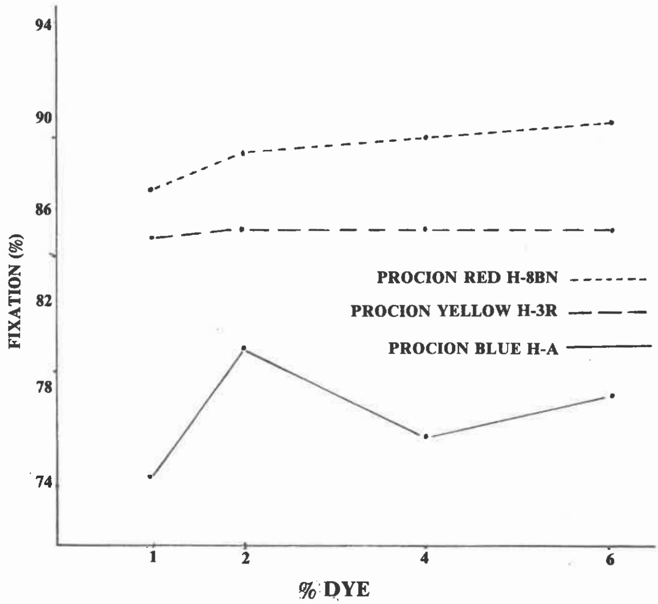


Fig. 4 Influence of dyestuff concentration in the loop-transfer liquor on fixation X% Procion H dye, 20% urea, 2% Na_2CO_3, 1,5% Lissapol NX, pick-up range 37 – 39% (omf)

Influence of non-ionic surfactant concentration on degree of fixation

The standard recipe for Procion H dyes was used with alterations in the concentration of Lissapol NX from 0,75 to 5% with a pick-up range of from 36,5 to 39%, this resulting in a surfactant add-on of about 0,4 to 2% (omf). No influence of the surfactant concentration on the fixation values was found within this range (Fig 5). The differences in fixation values for Blue H-A were probably due to the reasons mentioned above.

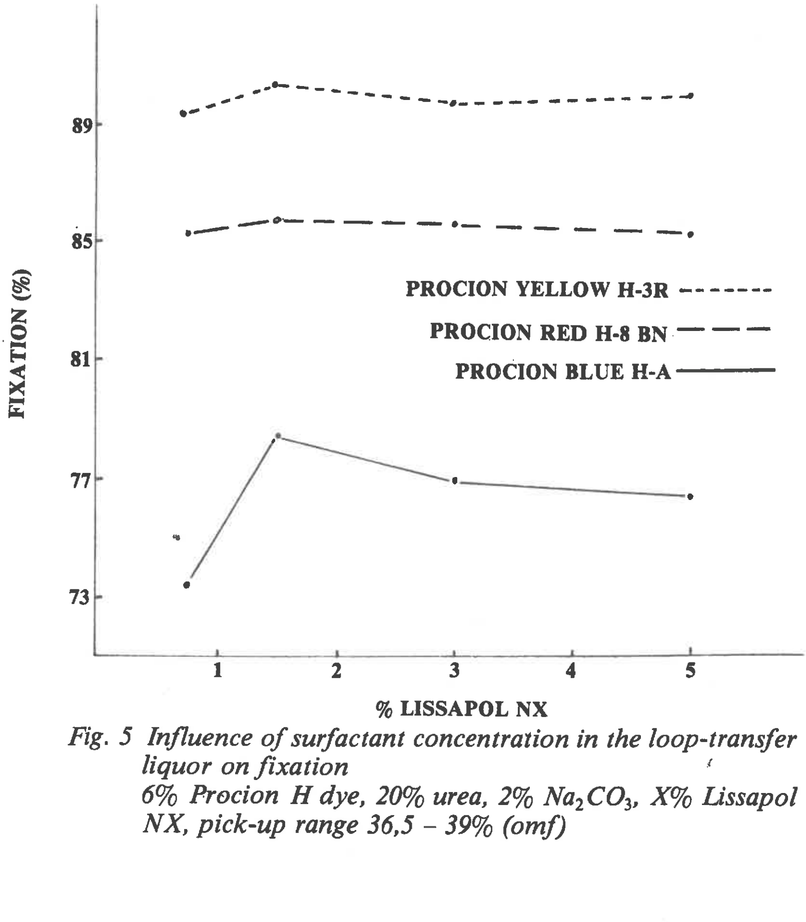


Fig. 5 Influence of surfactant concentration in the loop-transfer liquor on fixation
6% Procion H dye, 20% urea, 2% Na_2CO_3, X% Lissapol NX, pick-up range 36,5 – 39% (omf)

Influence of urea concentration on degree of fixation

Using the standard recipe for Procion H dyes, the concentration of urea in the Loop-transfer liquor was changed from 10% to 20% and a pick-up of from 36% to 38% was used which resulted in a urea add-on of about 4 to 8% (omf). Fig 6 shows that an increase from 10% to 20% urea in the liquor was accompanied by an increase in the degree of fixation from 86,5% to 90,5%.

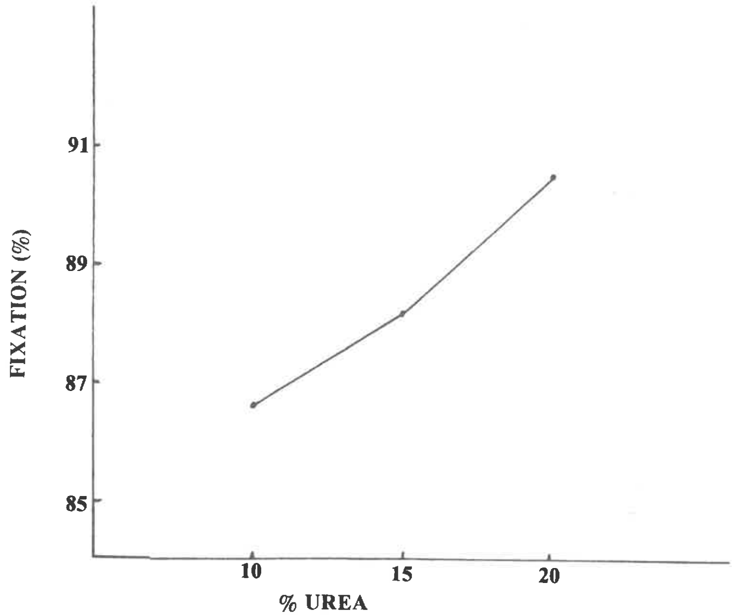


Fig. 6 Influence of amount of urea in transfer-liquor on fixation. 6% Procion Yellow H-3R, 2% Na_2CO_3, 1,5% Lissapol NX

Shade differences between face and back of the fabric

The Benz 3-roll padder used for loop-transfer throughout this investigation allows only the transfer of liquor to one side of the fabric at a time. The subsequent transfer of dye liquor to both sides of the fabric resulted in a slight shade difference with the side to which the dye liquor was transferred first always showing a slightly deeper shade. Different attempts were made to overcome this problem:

(a) After finding that the use of the same transfer pressure for both transfers resulted in a 10 to 20% lower second pick-up, the transfer pressure for the second transfer was increased in order to avoid any differences between the first and second pick-up. However, the shade difference remained.

(b) Drying of the fabric after the first transfer resulted in very unlevel dyeings caused by "strikes-through".

(c) Padding of fabrics with 0,5 to 4,0% aqueous solutions of wetting agents (Nonidet P40, Aerosol OT) as well as drying previous to loop-transfer did not reduce the shade differences either.

(d) The shade difference was not eliminated when using a plain-weave fabric instead of the double jersey fabric normally used, thus indicating that the shade difference was not caused by the structure of the fabric.

(e) The use of dyestuff with high reactivity without alkali in the loop-transfer liquor and the use of dyestuffs with low reactivity did not avoid the shade difference either. This clearly indicated that the shade difference was not a result of premature fixation. Accoring to the manufacturer's information[8] the reactive dyestuffs used had either medium or high fibre affinity. Differences in fibre affinity had no visible influence on the shade differences.

(f) The only way found to avoid any differences in shade was a modified transfer technique allowing a simultaneous transfer to the front and back of the fabric with a 3-roll padder. For convenience the simultaneous loop-transfer requires a 4-roll padder with two loops and two troughs. For continuous working a 4-roll padder is necessary.

Comparison of degree of fixation for loop-transfer and padding

The influence of the dyeing technique used on fixation was investigated for various Procion H dyes. Using a pick-up for loop-transfer from 38% to 39% and for padding from 78% to 79%, the resulting add-on of dyestuffs for both techniques was identical (about 2,4% omf). No significant differences in fixation were found for both techniques (Table V).

TABLE V.

COMPARISON OF FIXATION BETWEEN LOOP-TRANSFER AND PADDING

Procion Dyestuff	% Fixation by loop-transfer (maximum values)	% Fixation by normal padding
Red H-8BN	85,8	85,7
Blue H-A	78,7	73,9
Yellow H-3R	90,5	88,1

SUMMARY AND CONCLUSIONS

The three-roll loop-transfer technique was investigated for the dyeing of cotton fabric with reactive dyestuffs. Due to the low level of pick-up, migration of dyestuff during drying was reduced considerably. To obtain good penetration and level dyeings, a transfer to both sides of the fabric was necessary together with a minimum pick-up and the use of a non-ionic surfactant. The latter is thought to act as a retarder.

A pick-up lower than 25% was possible when transferring to both sides of the fabric, but pick-up levels from 35% to 40% were necessary to obtain level dyeings, the amount of pick-up depending on the dyestuff used. Levelness at a given pick-up level was found to improve in the order: Blue H-A. Red H-8BN and Yellow H-3R. Furthermore, it was possible to improve levelness by the use of higher transfer pressures.

The subsequent transfer to both sides of the fabric resulted in slight shade differences with the side transfer-dyed first slightly darker. A possible way to eliminate shade differences was to have a simultaneous transfer to both sides of the fabric. This method requires a 4-roll padder for practical reasons. The short-term reproducibility of pick-up was better in the higher pressure range, but the structure of the loop material may change with time due to the pressure, and lower pick-up levels can result.

Increasing transfer speed in the range of 0,75 to 5 m/min increased the pick-up, for example from 35% to 46% under certain circumstances, but the degree of fixation was independent of the transfer speed. Furthermore, the degree of fixation was independent of the concentration of dyestuff in the liquor used in the range of 1,0% to 6,0% and also independent of the concentration of non-ionic surfactant (in the range of 0,75% to 5,0%).

The pick-up and the amount of urea in the liquor was found to influence the degree of fixation. The influence of pick-up on fixation was particularly evident in the lower pick-up range with lower pick-up levels resulting in lower degrees of fixation. With an increase in pick-up, the influence on fixation decreased and did not seem to exist for pick-up levels higher than 38%. Furthermore, dyestuffs with a better levelling power seem to show a smaller dependence of fixation on pick-up. It may be concluded that a minimum level of pick-up is necessary to distribute the dye in the fibre in such a manner that maximum fixation is possible. The degree of fixation increased with increasing amounts of urea within the investigated range of 10% to 20% urea in the loop-transfer liquor. However, it should be appreciated that the differences in degree of fixation caused by the above mentioned factors are very small, particularly as the maximum fixation values were found to be between 79% and 90%. The maximum difference in fixation within the pick-up range investigated was about 3% (absolute) and the maximum difference caused by different urea concentration about 4% (absolute).

Fixation values for fabrics dyed with the same percentage of dyestuff either by loop-transfer or conventional padding, showed very close agreement. Despite the good results obtained for loop-transfer, the principle showed some mechanical weaknesses. However, technical modifications to the equipment could eliminate most of these weaknesses.

In conclusion it can, therefore, be stated that the use of loop-transfer for purposes of dyeing is possible, showing good results in respect of levelling and fixation. Judging from publications concerned with the application of finishing agents by low add-on techniques, the application of dyestuffs is, however, more difficult due to the requirements of minimum pick-up and the use of a suitable surfactant in order to obtain level dyeings and maximum fixation.

ACKNOWLEDGEMENT

The author wishes to thank Dr M.B. Roberts for valuable discussions and proposals throughout this investigation.

THE USE OF PROPRIETARY NAMES

The product names ®Procion and ®Lissapol are registered trade names of Messrs ICI Ltd., ®Aerosol is a registered trade mark of Messrs Cyanamid Ltd., and ®Nonidet is a registered trade mark of Messrs Shell Chemicals Ltd. The fact that chemicals with proprietary names have been used in this report in no way implies that SAWTRI recommends them or that there are not others which may be of equal or even better value.

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ERRATUM

Page 12 - second paragraph, 6th line : for Red H-8BN, please read Yellow H-3R.

Pafe 12 - Fig 4: Please invert legend for Procion Red H-8BN and Procion Yellow H-3R.