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Water-assisted Solvent System
Part I: Dyeing with
Monochlorotriazine Dyes

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DYEING OF COTTON FROM A WATER-ASSISTED SOLVENT SYSTEM

PART I: DYEING WITH MONOCHLOROTRIAZINE DYES

by H. M. SILVER

ABSTRACT

A method of dyeing cotton with the monochlorotriazine class of reactive dyes from a charged perchloroethylene solution is described. The charge consisted of water, sodium dodecylbenzenesulphonate, lauryl monoethanolamide and trisodium phosphate. Dyeings were successfully carried out up to a level of 5% dye. The cloths were then soaped in an aqueous medium. The fastness properties of the solvent-dyed cloths were as good as those of aqueous-dyed cloths.

KEY WORDS

Cotton — monochlorotriazine dyes — perchloroethylene — dyeing — charged system.

INTRODUCTION

The sudden and great concern about the polluting of man's environment has sparked off tremendous interest in the development of new methods which may assist in combating the threat to the environment. Increasing demands by an exploding population and an expanding industry are being placed on the limited water resources. Solvent processing of textile fibres has been employed for a number of years. Most developments have been centred around solvent finishing processes but in recent years more research has been carried out on the solvent dyeing of textile fibres[1, 2, 3].

The lack of published work on the dyeing of cellulose, from a solvent medium, is remarkable for the consumption of cotton is still greater than the combined consumption of all other textile fibres[4]. Solvent systems have been described for the dyeing of polyester[5] and polyamide[6] and these will lose much of their effectiveness if cotton, in a blend with these fibres, cannot be dyed from the same solvent or in the same machine as this would entail drying the material between the two processes. Some success in the dyeing of cellulose from a solvent-based system has been achieved by the Delaware Valley Research Committee[7]. Direct and soluble vat dyes were used. The system, however, proved to be sensitive to moisture which resulted in spotty dyeings.

At this stage it would appear that a "charged" system is necessary to dye cellulose in order to swell the fibres and to dissolve the dyestuff. This is the system

which Mecheels[8] referred to as the solvent/tenside/water system. The author explained that, if the water content is low, the system would be a true, homogeneous solution, but as more water is required for the dyeing of cotton than is possible for a true solution of water and perchloroethylene to exist, the system becomes an emulsion with typical colloidal properties. A tenside is necessary to emulsify the water in the perchloroethylene. Dyeing from such a system is similar to normal aqueous dyeing[1]. The solvent acts as a carrier in the dyeing process and as the amount of water used is much less than that used in conventional dyeing processes, the solvent process is equivalent to dyeing in a very short liquor.

The first of this series of publications deals with the dyeing of cellulose with the monochlorotriazine class of reactive dyes. Subsequent publications will deal with the use of acryloylamide and trichloropyrimidine classes of reactive dyes.

A solvent, which is to be used in such a process must comply with certain requirements in its physical and chemical properties viz. appropriate boiling point, low latent heat of evaporation, non-flammability, non-toxicity etc.[9, 10].

EXPERIMENTAL

The work of Swanepoel and Roesstorff[11] and Van der Merwe and Van Rooyen[1] indicated that wool could be dyed in a charged solvent system up to a 5% depth of shade. The dyeing recipe of the latter team was:

 35% o.m.f. water
 15% o.m.f. glycerol
 7,5% o.m.f. sodium dodecylbenzenesulphonate (SDBS)
 5,0% o.m.f. lauryl monoethanolamide (LEA)
 The liquor (perchloroethylene) to goods ratio was 10:1.
 SDBS is an emulsifier and LEA is a foam stabilizer

Van der Merwe and Van Rooyen found that the addition of glycerol improved the dyeing efficiency significantly but could not replace water entirely. It was decided to use the above recipe as a basis for this work.

A commercially processed pure cotton fabric was used throughout. According to the manufacturers, the cloth was caustic scoured, bleached first with hypochlorite and then with peroxide and finally mercerised. The specifications of the fabric used are the following:

Density	:	139 g/m²
Ends/cm	:	28,4
Picks/cm	:	22,4
Tex — warp	:	24,3
— weft	:	22,6
Crimp (%) — warp	:	5
— weft	:	20
Yarn twist — warp	:	687 t.p.m.
— weft	:	764 t.p.m.

The initial conditions under which the solvent dyeings took place such as temperature, alkali concentration and time of dyeing, were taken from the pattern cards for aqueous dyeings of the various manufacturers. The details for the aqueous dyeings were based on the instructions in the pattern cards.

The following should be noted:
1. Unless otherwise stated —
   (i) all percentages of dyes and chemicals are calculated on the mass of fabric;
   (ii) all dyeings were carried out at a level of 5% dye.
2. The term, absorption, refers to the amount of dye still attached to the fibres after they had been soaped and rinsed.

Ten gramme samples of this fabric were dyed in the stainless steel capsules of a Hansau Linitest Apparatus. The dye was dissolved in the required amount of water and the solution emulsified in 100 ml of perchloroethylene, in which the SDBS and LEA had been dissolved, by stirring with a magnetic stirrer. Unless otherwise stated, glycerol was not used as a co-polar solvent.

The material was entered and the dyeing commenced at 50°C. The temperature was then raised to 80°C over 40 minutes and the dyeing continued at this temperature for the appropriate time. The required amounts of alkali were dissolved in the minimum amount of water and were added to the dye bath at the appropriate stage of dyeing. Where urea, electrolytes or other additives were used, these were dissolved with the dye in the water.
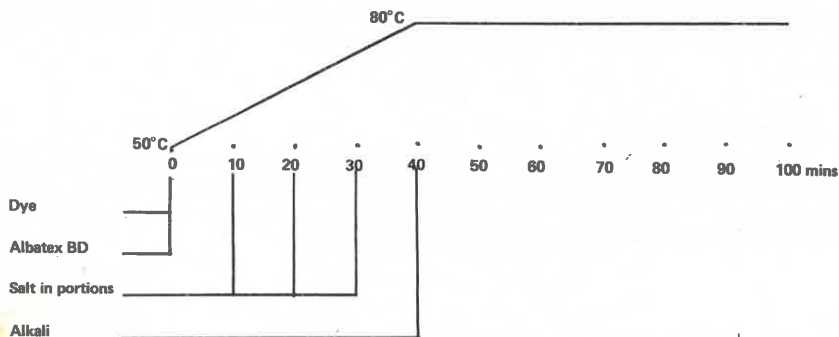
For comparative purposes, samples were dyed in the conventional manner. Ten gramme samples of the cotton fabric were dyed in the stainless steel capsules of a Hansau Linitest Apparatus. The following recipe was used:

      80 g/l Na$_2$SO$_4$
      20 g/l Na$_3$PO$_4$
      2 g/l Albatex BD (Ciba-Geigy)

The liquor to goods ratio was 10:1, and the following procedure was used —

Upon completion of a dyeing, the dyed cloths were dried and then soaped in an aqueous solution of 1 g/l Eriopon HD (Ciba-Geigy) for 20 minutes at the boil. The cloths were then rinsed in cold water. This water was added to the rinse liquor.

Three different procedures were followed to determine the colour yield. These were:

(i)   to determine the percentage dyestuff on the cloth;
(ii)   to determine the K/S values*;
(iii) to determine the chromaticity co-ordinates and tristimulus values of the cloth.

The procedure used depended on what was being measured. The results presented later refer to the amount of dye that was on the cloth after soaping and rinsing, unless otherwise stated.

DETERMINATION OF PERCENTAGE DYE ON CLOTH

A. Solvent Dyeing:

Upon the completion of a dyeing, the emulsion in the capsules was collected and the emulsion was broken with the aid of ethyl alcohol. The aqueous portion was separated and made up to 500 ml with water. To determine the mass of dye, which was not taken up by the cloth, three 25 ml samples of the extracted solution, · which contained all the dyestuff, were centrifuged at 17 000 r.p.m. for 60 minutes and the absorbance of the clear supernatent solution read on a Beckman BD Spectrophotometer at the wavelength of maximum absorbance for the particular dye. The solution had been sufficiently diluted so that Beer's Law was obeyed.

The rinse liquors were cooled and made up to 1l with water. Where necessary, it was diluted and three samples were taken so as to read the absorbance of the rinse liquor.

From the average values of the absorbance of the dye and rinse liquors, the amount of dye not absorbed and that was rinsed off was calculated. As the amount of dye, which was initially added to the capsules was known, the percentage dye which remained on the cloth was calculated.

B. Aqueous Dyeings:

After the completion of the dyeing, the dye liquor was made up to a known volume. Three aliquots were taken and the absorbance measured on the Spectrophotometer. The absorbance of the rinse liquor was determined as described above. The percentage dye on the cloth was then calculated.

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*K/S = A function of the dye concentration on the fibre; derived from reflectance data and calculated from the Kubelka-Munk-equation[12].

DETERMINATION OF K/S VALUES

After the dyed cloth had been soaped, rinsed and dried, the reflectance of the cloth was measured at 10 different places on the cloth at the wavelength of maximum reflectance on the Beckman DB Spectrophotometer, to which a reflectance attachment had been fitted. From these values the K/S values were determined. The average value and coefficient of variation were calculated.

DETERMINATION OF CHROMATICITY CO-ORDINATES AND TRISTIMULUS VALUES

The chromaticity co-ordinates (x and y), the tristimulus value (Y) and the tristimulus ratios (X/Y and Z/Y) were measured on a Harrison-Shirley Digital Colorimeter. The readings were taken at 10 different places on the dyed cloth after it had been soaped, rinsed and dried and the average values calculated.

FASTNESS DETERMINATIONS

A number of cloths were tested to determine their fastness properties. The cloths were tested after they had been soaped and rinsed. These tests were conducted, to determine the fastness of the dyed cloth to light, washing and alkali perspiration.

RESULTS AND DISCUSSION

In the case of the monochlorotriazine class, the temperature of maximum fixation varies for the different dyes as well as for the different alkalis that may be used. When trisodium phosphate or sodium hydroxide was used, the temperature of fixation is the lowest. As the former alkali is the weaker, it was felt that, as the alkali would be concentrated in a small volume of water, it would be preferable to use a weaker alkali as the rate of the reaction would be slower resulting in more level dyeings and better penetration.

The first step was to establish the amount of water required to dissolve the dye and produce a level dyeing. The dyes used in this part of the investigation were C.I. Reactive Red 42 and C.I. Reactive Blue 39.

The influence of the different amounts of emulsified water on the absorption of C.I. Reactive Blue 39 and C.I. Reactive Red 42 dyed on cotton from a solvent emulsion system is shown in Table I.

The absorption decreased with increasing water concentration. It was decided to use 60% water for the rest of this investigation with this class of dye as the decrease in absorption by increasing the amount of emulsified water (from 50 to 60%) was compensated for by the better levelness and general appearance of the cloths.

Having established the optimum amount of water that is necessary, a series of dyeings was conducted to establish the effect of urea, the time of alkali addition, electrolyte and glycerol on the absorption of the dye on cotton. From the results obtained with direct dyes, on the concentration of SDBS and LEA, it was felt that these concentrations need not be changed from those established previously[13].

Table II lists the influence of the various additives on the absorption of the two reactive dyes when dyed from a solvent system.

TABLE I
% ABSORPTION OF C.I. REACTIVE BLUE 39 AND C.I. REACTIVE RED 42 DYED ON COTTON FROM A SOLVENT SYSTEM WITH DIFFERENT AMOUNTS OF EMULSIFIED WATER

% WATER	PERCENTAGE ABSORPTION	
	C.I. REACTIVE RED 42	C.I. REACTIVE BLUE 39
50	57,1	38,4
60	54,6	33,0
70	43,0	28,9
80	42,9	21,3

TABLE II
EFFECT OF VARIOUS ADDITIVES ON THE ABSORPTION OF C.I. REACTIVE BLUE 39 AND C.I. REACTIVE RED 42 DYED ON COTTON FROM A SOLVENT SYSTEM

Dyeing No.	3% Alkali Added After	Urea (%)	Electrolyte (g/l Na$_2$SO$_4$)	PERCENTAGE ABSORPTION	
				Blue 39	Red 42
1A	—	0	0	24,3	8,5
1B	—	10	0	16,5	14,9
2	—	0	80	29,1	49,5
3A	40 mins	0	0	43,0	50,9
3B	40 mins	10	0	26,1	39,2
4A	60 mins	0	0	24,9	45,7
4B	60 mins	10	0	23,4	36,5
5A	80 mins	0	0	17,6	36,7
5B	80 mins	10	0	15,9	19,9

The Dyeing Cycle used to obtain the results in Table II:

80°C

50°C

0   40   60   80   100 mins
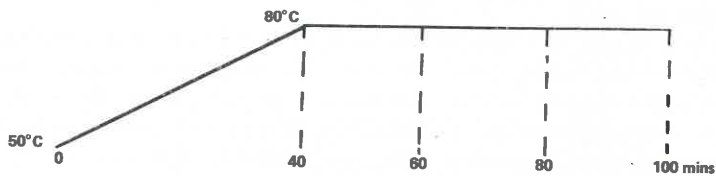
It seemed that the addition of an electrolyte (Na_2SO_4) to the dye bath resulted in an increase in the percentage absorption of the dye on the cloth but the appearance of the cloth showed that most of the dye had not diffused into the fibres but had been fixed on the surface only. In addition, the dye molecules had not spread evenly over the cloth, resulting in an extremely unlevel dyeing. It was therefore decided that no electrolytes would be used for any of the remaining dyeings from a solvent system. This unlevel dyeing might have been caused by the decrease in the electrokinetic potential with the addition of the electrolyte[14] or by a breakdown of the stability of the emulsion. The adsorption of the dye on the cotton fibres was thus much faster. As this occurred while the temperature was still low, and increasing to that temperature at which the dyeing took place, the diffusion of the dye into the fibre was slow. Upon addition of alkali, the dyestuff was fixed to the surface of the fibre and no further levelling or diffusion could take place. This process could have been assisted by the dyestuff being highly concentrated in the water globules of the emulsion and as the cotton is hydrophilic, these globules could have been nearest to the fibres, increasing the adsorption rate. With aqueous dyeings, the dyestuff is evenly distributed throughout the liquor and the adsorption would then be much slower.

The addition of urea to the dye bath caused a decrease in the adsorption of both dyes with the exception of C.I. Reactive Red 42 in dyeings 1A and 1B. A further dyeing was carried out in which 42% water and 18% glycerol was used as the polar solvent in place of 60% water. The percentage absorptions for C.I. Reactive Blue 39 and C.I. Reactive Red 42 were 23,75% and 17,23%, respectively. These values do not compare favourably with the results obtained in dyeing number 3A. It appeared that both urea and glycerol might have caused the polar phase of the emulsion to be more attractive to the dye molecules resulting in less adsorption, and thus fixation, to occur. A second consideration was that the rate of hydrolysis might have increased due to the additions of urea and glycerol. However, there was no experimental evidence to support this supposition.

The influence of the time of alkali addition on the absorption is more clearly shown in Table III, as the percentage dye from each phase is shown. This influence could have been ascribed to the alkali not having sufficient time to allow more dye to react with the fibres.

The optimum concentration of alkali was then sought and the effect of increasing concentrations of alkali on the absorption is shown in Figure 1. The figure shows that 5% Na_3PO_4 was sufficient. This mass of alkali was dissolved in

twice its mass of water.

Figure 2 shows the rate of absorption of the two reactive dyes from aqueous and solvent media over a period of 160 minutes. The main information, that was required, was to determine at what stage the solvent dyeing should be terminated. It was decided that a total dyeing time of 120 minutes was adequate and any increase in the absorption, which occurred after this, was of little significance. The rate of absorption of C.I. Reactive Red 42 was much faster from a solvent than from an aqueous medium. The converse applied for the rate of adsorption of C.I. Reactive Blue 39 as the rate was much faster from an aqueous medium than from a solvent system and the rate decreases much sooner with the former process.

A process from the above results for the dyeing of monochlorotriazine dyes from a solvent emulsion system on cotton could be established. The following recipe was employed —

x%   Dye
60%   Water
7,5%   SDBS
5,0%   LEA
5,0% Na_3PO_4
Liquor to goods ratio = 10:1

The emulsion was prepared as has been described above. The alkali was added to the dyeing after 40 minutes after which the dyeing was continued for a further 80 minutes.

TABLE III

DISTRIBUTION OF C.I. REACTIVE BLUE 39 AND C.I. REACTIVE RED 42 AFTER DYEING FROM A SOLVENT SYSTEM, SOAPING AND RINSING

Alkali added at	Urea (%)	Dye not Absorbed during Dyeing (%)		Dye removed by soaping and rinsing (%)		Absorption (%)	
		Blue	Red	Blue	Red	Blue	Red
Point 1+	0	9,0	6,0	47,9	43,0	43,0	50,9
Point 1	10	9,7	15,0	64,2	45,8	26,1	39,2
Point 2	0	4,7	9,6	70,3	44,7	24,9	45,7
Point 2	10	8,8	19,1	67,8	44,4	23,3	36,4
Point 3	0	4,6	9,2	77,8	54,0	17,6	36,6
Point 3	10	10,7	23,3	73,3	56,9	15,9	19,8

(+ = See dyeing cycle at foot of Table II for times of alkali addition)

A comparison of the increasing absorption of C.I. Reactive Blue 39 at increasing concentrations of dye from both media is shown in Figure 3. It will be noted that the solvent dyeings, dyed according to the above recipe, had been dyed to a deeper shade than the aqueous dyeings, dyed according to the same recipe. From Figure 2 it can be seen that the absorption of the solvent-dyed cloths at 120 minutes was slightly higher than the absorption of the aqueous-dyed cloths at 100 minutes (which is the time recommended by the manufacturer for the dyeing to end). This could be one of the reasons for the apparent discrepancy in the results shown in Figures 2 and 3. Unknown and uncontrollable factors might have influenced these dyeings.
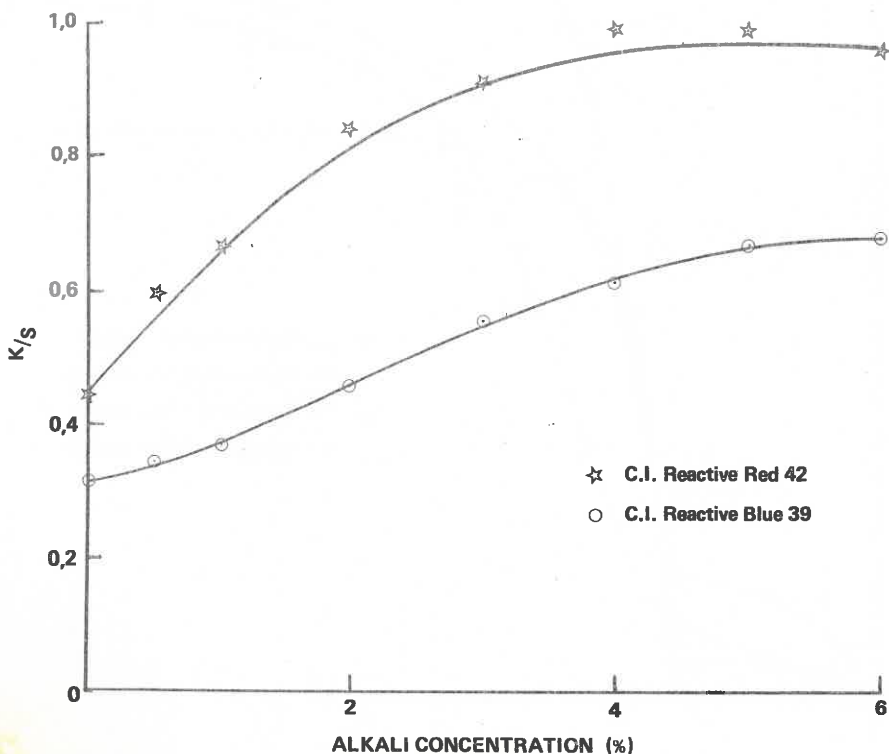
FIGURE 1

Influence of different concentrations of alkali on the absorption of C.I. Reactive Red 42 and C.I. Reactive Blue 39 on cotton from a solvent system
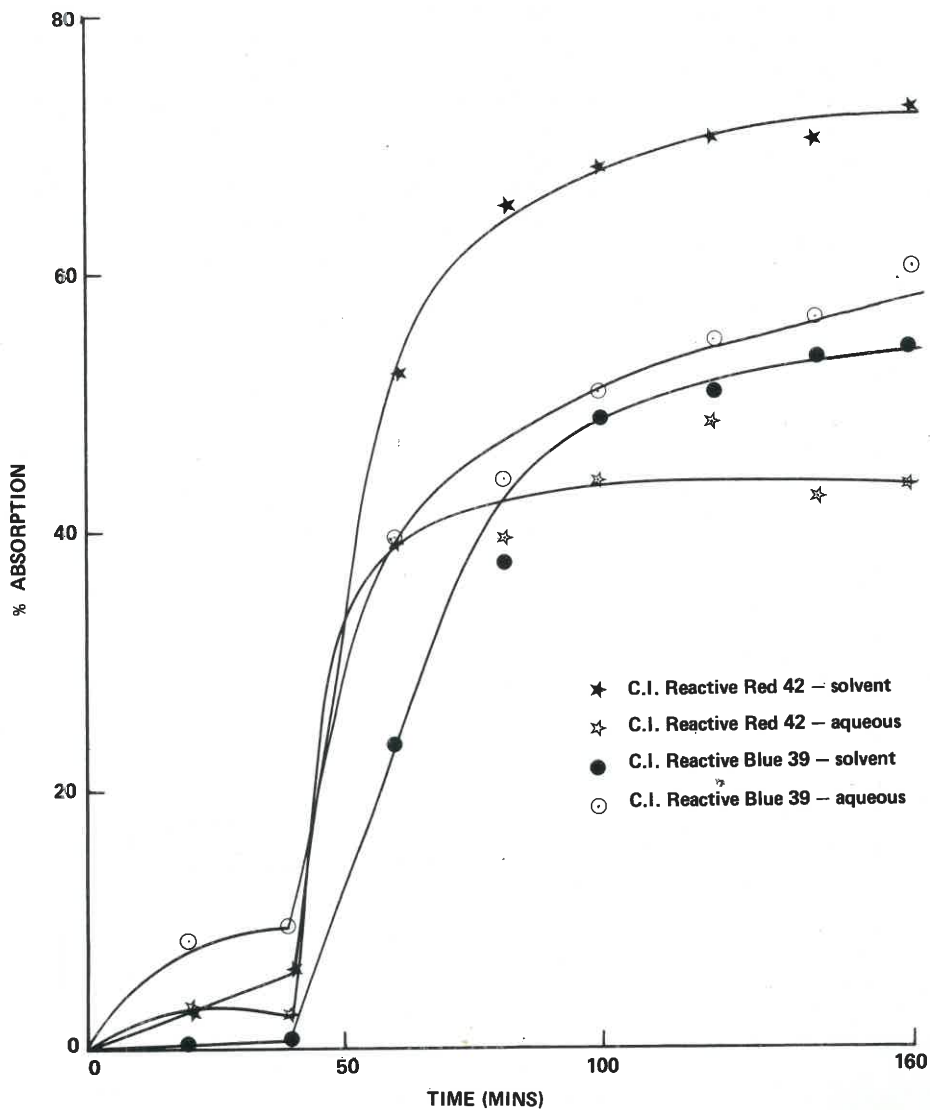
FIGURE 2

Rate of Absorption of C.I. Reactive Blue 39 and C.I. Reactive Red 42 on cotton from a solvent and aqueous system

Figure 4 shows the absorption of C.I. Reactive Red 42 at increasing concentrations of dye from both media. The results were as expected after their respective rates of absorption had been established with the solvent-dyed cloths adsorbing more dye than the aqueous-dyed cloths.

Six other dyes, of the same class, were then chosen in order to test the above recipe. In Table IV the results of these dyeings are listed.

With the exception of C.I. Reactive Blue 39, all the solvent dyeings showed a marked increase in the absorption of the dye on the cloths over the aqueous dyeings. Dyeing the cloths from a solvent system had no adverse effect on their fastness properties. It was found that there was a slight change in the hue of C.I. Reactive Red 43.

FIGURE 3

Absorption of C.I. Reactive Blue 39 on Cotton at increasing concentrations of Dye from a solvent and aqueous system

Finally, a few dichromatic dyeings from both media were attempted to see if there was any marked variation in shade due to the solvent method. The dichromatic dyeings showed that a similar hue was obtained from both media. These results are listed in Table V. The solvent-dyed cloths appeared to have dyed to a deeper shade, judging from the value of Y. The fastness properties of the solvent-dyed cloths were, again, not affected owing to the particular process employed. The light fastness of the cloths i.e. both aqueous and solvent dyed samples was, however, very low.

SUMMARY AND CONCLUSIONS

Dyeings were successfully carried out up to a depth of shade of 5% dye. The fastness properties of the solvent-dyed cloths were not affected by the dyeing process as their properties proved to be as good as the fastness properties of the aqueous-dyed cloths.

Results showed that it was inadvisable to add any electrolyte to the dye bath. Furthermore the addition of urea to the dye bath was not of any significance.

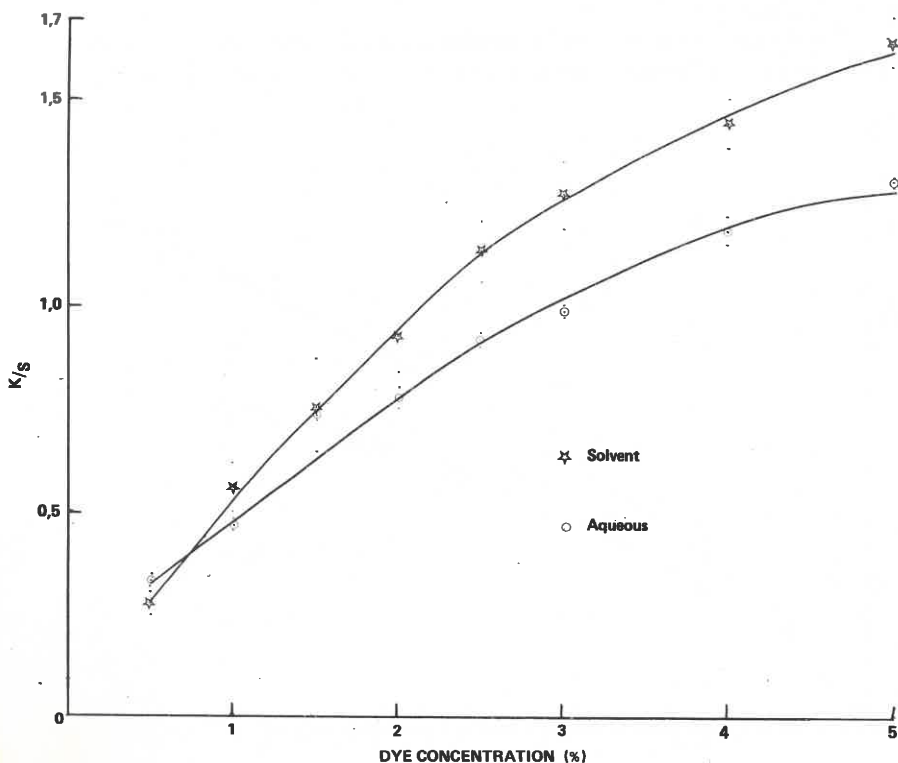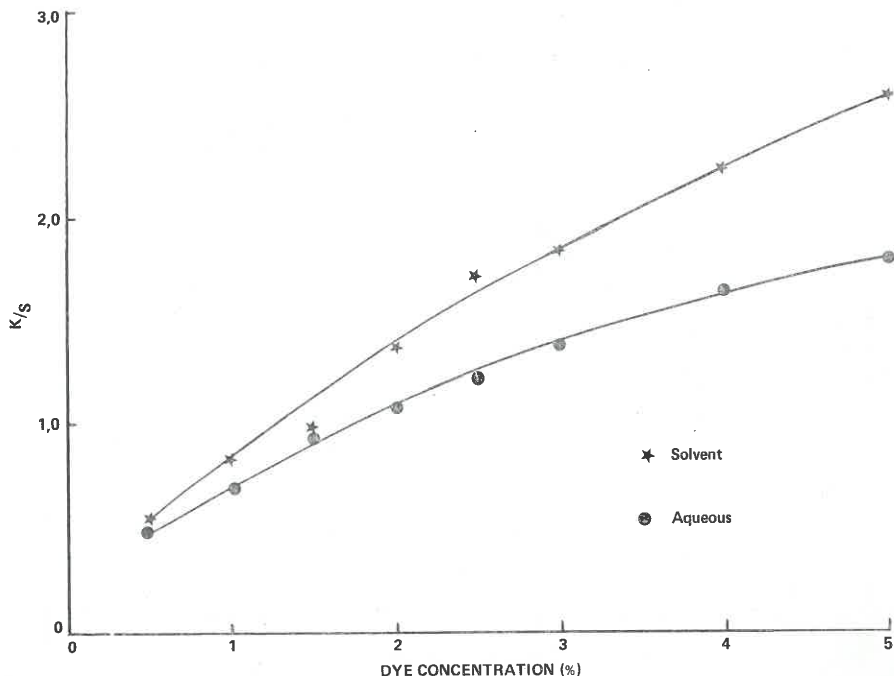**FIGURE 4**

Absorption of C.I. Reactive Red 42 on cotton at increasing concentrations of dye from a solvent and aqueous system

ACKNOWLEDGEMENT

THE USE OF PROPRIETARY NAMES

The fact that chemicals with proprietary names have been mentioned in this investigation in no way implies that there are not other as good or even better.

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TABLE V

CHROMATICITY CO-ORDINATES AND FASTNESS VALUES OF DICHROMATIC DYEINGS OF MONOCHLOROBIAZINE DYES DYED ON COTTON FROM AQUEOUS AND SOLVENT SYSTEMS

DYE MIXTURE	SOLVENT x	SOLVENT y	SOLVENT Y	SOLVENT X/Y	SOLVENT Z/Y	AQUEOUS x	AQUEOUS y	AQUEOUS Y	AQUEOUS X/Y	AQUEOUS Z/Y	WASH SOLVENT Change in Shade	WASH SOLVENT Staining Wool	WASH SOLVENT Staining Cotton	WASH AQUEOUS Change in Shade	WASH AQUEOUS Staining Wool	WASH AQUEOUS Staining Cotton	PERSP. SOLVENT Change in Shade	PERSP. SOLVENT Staining Wool	PERSP. SOLVENT Staining Cotton	PERSP. AQUEOUS Change in Shade	PERSP. AQUEOUS Staining Wool	PERSP. AQUEOUS Staining Cotton	LIGHT Solv.	LIGHT Aqu
2% C.I. Reactive Red 42 + 0,5% C.I. Reactive Blue 39	0,338	0,215	7,19	1,56	2,03	0,335	0,217	8,21	1,54	2,03	4-5	5	5	4-5	5	4-5	4-5	5	5	4	5	4-5	3	3-4
1% C.I. Reactive Yellow 35 + 0,5% C.I. Reactive Red 43	0,537	0,354	30,03	1,51	0,30	0,511	0,350	32,41	1,45	0,38	5	5	5	5	5	5	5	5	5	5	5	5	3	3
2% C.I. Reactive Yellow 35 + 1% C.I. Reactive Blue 7	0,258	0,499	29,08	0,52	0,48	0,269	0,470	33,93	0,57	0,54	4	5	5	4	5	5	5	5	5	4	5	4-5	3	3
0,8% C.I. Reactive Orange 2 + 0,2% C.I. Reactive Blue 40	0,369	0,339	15,95	1,08	0,84	0,358	0,334	18,08	1,06	0,90	5	5	5	5	5	5	5	5	5	4	5	5	2	2

Header groupings: CHROMATICITY CO-ORDINATES AND TRISTIMULUS VALUES AND RATIOS (SOLVENT, AQUEOUS) · FASTNESS TO WASHING (SOLVENT, AQUEOUS — Change in Shade, Staining: Wool, Cotton) · FASTNESS TO ALKALI PERSPIRATION (SOLVENT, AQUEOUS — Change in Shade, Staining: Wool, Cotton) · FASTNESS TO LIGHT (Solv., Aqu).

TABLE IV

CHROMATICITY CO-ORDINATES, FIXATION VALUES AND FASTNESS VALUES OF COTTON SAMPLES DYED WITH MONOCHLOROTRIAZINE DYES FROM AQUEOUS AND SOLVENT DYES

DYESTUFF	% ABSORPTION OF DYE		CHROMATICITY CO-ORDINATES AND TRISTIMULUS VALUES AND RATIOS										FASTNESS TO WASHING						FASTNESS TO ALKALI PERSPIRATION						FASTNESS TO LIGHT	
			SOLVENT					AQUEOUS					SOLVENT			AQUEOUS			SOLVENT			AQUEOUS				
													Change in Shade	Staining (Wool)	Staining (Cotton)	Change in Shade	Staining (Wool)	Staining (Cotton)	Change in Shade	Staining (Wool)	Staining (Cotton)	Change in Shade	Staining (Wool)	Staining (Cotton)		
	Sol.	Aqu.	x	y	Y	X/Y	Z/Y	x	y	Y	X/Y	Z/Y													Sol.	Aqu.
C.I. Reactive Blue 39	53,9	55,6	0,210	0,185	6,29	1,13	3,23	0,208	0,185	6,38	1,12	3,23	4-5	5	4	5	5	4	4	5	5	3-4	5	5	6-7	5-6
C.I. Reactive Red 42	72,8	53,6	0,531	0,272	12,61	1,94	0,70	0,510	0,265	14,43	1,90	0,83	3-4	5	5	4-5	5	5	4	4-5	4-5	3-4	5	4-5	3-4	3
C.I. Reactive Orange 2	57,1	39,1	0,566	0,383	31,43	1,47	0,12	0,545	0,386	34,86	1,40	0,16	5	5	5	5	5	5	5	5	5	4-5	5	5	4	4
C.I. Reactive Yellow 35	27,8	20,7	0,441	0,490	73,89	0,90	0,13	0,433	0,483	74,60	0,89	0,16	5	5	5	5	5	5	5	5	5	5	5	5	6-7	6
C.I. Reactive Blue 40	42,5	37,0	0,244	0,206	2,73	1,18	2,61	0,241	0,204	3,15	1,18	2,67	5	5	5	5	5	4-5	5	4	4-5	4-5	5	4-5	6	5-6
C.I. Reactive Red 43	51,3	28,6	0,600	0,325	14,81	1,84	0,21	0,535	0,325	24,62	1,63	0,41	5	5	4-5	5	5	4-5	5	5	4	5	5	4-5	3-4	3-4
C.I. Reactive Blue 7	62,8	39,0	0,164	0,212	14,41	0,77	2,29	0,177	0,229	20,29	0,77	2,55	4	5	5	3-4	5	4-5	4-5	5	4	4-5	5	4	6-7	6-7
C.I. Reactive Brown 2	44,1	28,5	0,532	0,381	18,72	1,39	0,21	0,523	0,385	20,65	1,35	0,23	5	5	5	5	5	5	5	5	5	5	5	5	7	6-7