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**Dyeing of Cotton from a Water-
Assisted Solvent System**

**Part II—Dyeing with Acryloylamide
and Trichloropyrimidine Dyes**

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DYEING OF COTTON FROM A WATER-ASSISTED SOLVENT SYSTEM

PART II – DYEING WITH ACRYLOYLAMIDE AND TRICHLOROPYRIMIDINE DYES

by H.M. SILVER

ABSTRACT

Methods of dyeing cotton with the acryloylamide and trichloropyrimidine classes of reactive dyes from a charged perchloroethylene solution are described. The charge consisted of water, sodium dodecylbenzene sulphonate, lauryl monoethanolamide and trisodium phosphate. With the acryloylamide dyes, urea was added to the charge. Dyeings were successfully carried out to a depth of shade of 5%. After dyeing, the cloths were soaped in an aqueous medium. The fastness properties of the solvent-dyed cloths were as good as those of the aqueous-dyed cloths.

KEY WORDS

Cotton – acryloylamide dye – trichloropyrimidine dyes – perchloroethylene – charged system – water assisted dyeings.

INTRODUCTION

In Part I of this series,⁽¹⁾ in which the dyeing of cotton with monochlorotriazine dyes was investigated, the reasons for the development of new dyeing systems were briefly stated. As systems are available for the solvent dyeing of polyester and polyamide, the necessity of developing a solvent system to dye cotton became evident. Before the feasibility of dyeing blends of cotton and polyester or blends of cotton and polyamide from solvent system can be investigated, a system for the dyeing of cotton should be established.

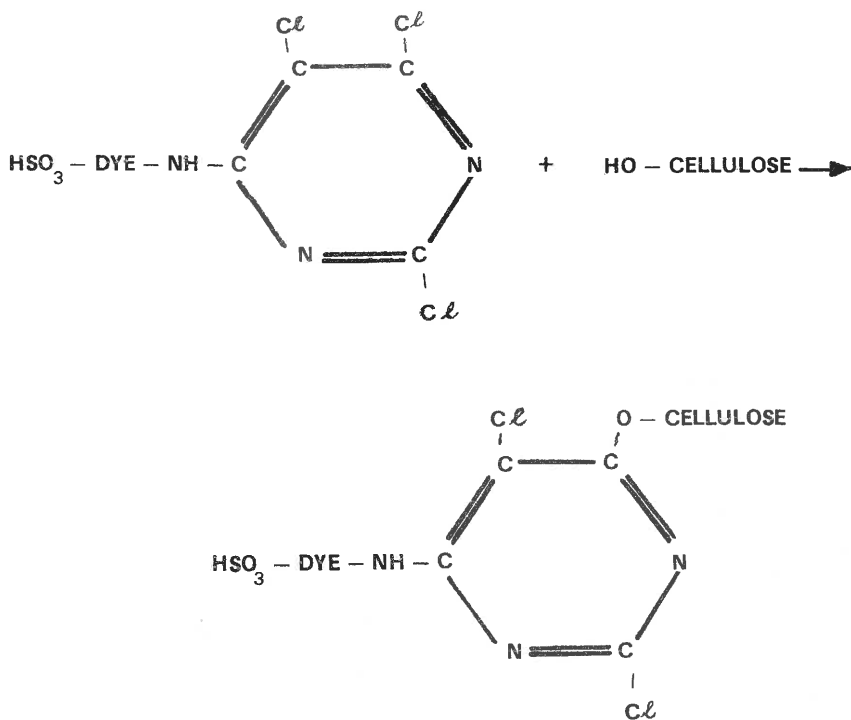
As a further step in the solvent dyeing of cotton with reactive dyes, the use of acryloylamide and trichloropyrimidine dyes has been investigated. The acryloylamide dye contains an activated vinyl group, which combines with the hydroxyl groups in the cellulose by means of an addition reaction, to form ether linkages. The bonds are frequently unstable under alkaline conditions.

An example of this type of reaction is shown below:

- (i) $\text{DYE} - \text{NH} - \text{CO} - \text{CH}_2 - \text{CH}_2 - \text{OSO}_3\text{Na} + \text{NaOH} \longrightarrow$
 $\text{DYE} - \text{NH} - \text{CO} - \text{CH} = \text{CH}_2 + \text{Na}_2\text{SO}_4 + \text{H}_2\text{O}$
- (ii) $\text{DYE} - \text{NH} - \text{CO} - \text{CH} - \text{CH}_2 - \text{HO} - \text{Cellulose} \longrightarrow$
 $\text{DYE} - \text{NH} - \text{CO} - \text{CH}_2 - \text{CH}_2 - \text{O} - \text{Cellulose}$

The fixation of the trichloropyrimidine class of reactive dyes on cotton occurs in a similar manner to the monochlorotriazine class. Both classes contain an amide chlorine reactive group which is fixed by a substitution reaction to the hydroxyl groups in the cellulose to give ester bonds. These bonds are more stable under alkaline than under acidic conditions.

The reaction between the dye molecule and cellulose is illustrated below.



EXPERIMENTAL

The initial conditions, under which the solvent dyeings were carried out, were adapted from the pattern cards supplied by the various manufacturers, for aqueous dyeings.

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.

The same cotton fabric was used in this investigation, as was described in PART I⁽¹⁾.

A. Acryloylamide Dyes

The dye was dissolved in the required volume of water and the liquor then emulsified in a solution comprising 100 ml perchloroethylene, 7.5% sodium dodecylbenzene-sulphonate (SDBS) and 5.0% lauryl monoethanolamide (LEA) by stirring with a magnetic stirrer. Where urea, or other additives were used, these were dissolved with the dye in the water. The dyeings were conducted in the stainless steel capsules of a Hansau Linitest Apparatus.

Ten gram samples of the cotton fabric were then entered and the dyeing commenced at 30° C. The temperature was then raised to 60° C over 30 minutes and the dyeing was continued at this temperature for the appropriate period of 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. Upon the completion of the dyeing, the 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 soaping liquor.

For comparative purposes, samples were dyed in the conventional manner in an aqueous medium.

The following recipe was used:

50 g/l	Na ₂ SO ₄
10 g/l	Na ₃ PO ₄

The liquor to goods ratio was 10 : 1.

The bath was set at 30° C with x% dye, salt and alkali and heated to 60° C over 30 minutes. The dyeing was continued for a further 60 minutes after which the cloths were soaped and rinsed as described above.

The same procedures were followed to determine the colour yield as was described for the monochlorotriazine dyes (1).

B. Trichloropyrimidine Dyes

Ten gram samples of the cotton fabric were dyed in the stainless steel capsules of a Hansau Linitest Apparatus. The emulsion was prepared as was described before.

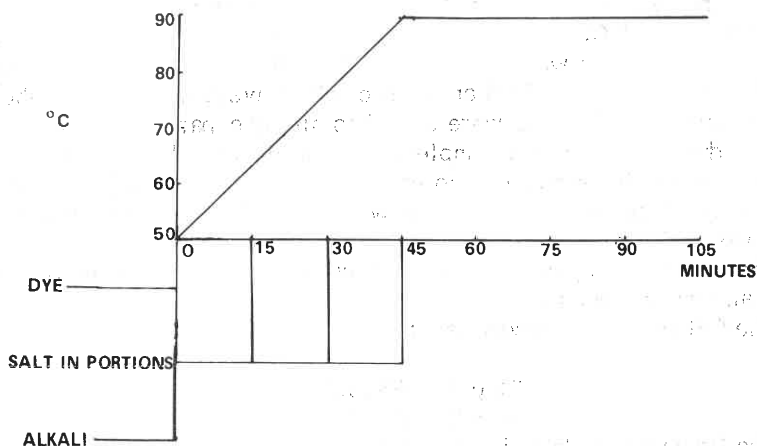
The dyeings were commenced at 50°C after which the temperature was raised to 90°C over a period of 45 minutes and the dyeing continued at this temperature for the appropriate period of time. The requisite amounts of alkali were dissolved in a minimum amount of water and were added to the dyebath at the appropriate stage of dyeing. Where urea, electrolytes or other additives were used, these were dissolved with the dye in the water.

At the completion of the dyeing, the cloths were soaped and rinsed as described before.

Ten gram samples of cotton fabric were also dyed according to the following recipe:

x% dye
80 g/l Na_2SO_4
20 g/l Na_3PO_4

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



At the completion of the dyeing, the cloths were soaped and rinsed as described before.

The procedures which were followed to determine the colour yield were the same as those described in PART I.

RESULTS AND DISCUSSION

A. Acryloylamide Dyes

From the results obtained on the influence of electrolytes on the dyeing with monochlorotriazine dyes from a solvent system (1), it was decided that the effect of electrolyte would not be considered for these dyes.

The influence of different concentrations of emulsified water on the absorption of C.I. Reactive Yellow 37 and C.I. Reactive Red 63, the two dyes used for the trials on the acryloylamide class of dyes is shown in Table I.

TABLE I

ABSORPTION OF C.I. REACTIVE YELLOW 37 AND C.I. REACTIVE RED 63 DYED ON COTTON FROM A SOLVENT SYSTEM WITH DIFFERENT AMOUNTS OF WATER

Water (%)	PERCENTAGE ABSORPTION	
	C.I. Reactive Yellow 37	C.I. Reactive Red 63
50	30,2	55,6
60	35,2	53,0
70	30,4	49,3
80	32,5	47,9

When C.I. Reactive Red 63 was used the percentage absorption had a tendency to decrease as the percentage of emulsified water was increased but in the case of C.I. Reactive Yellow 37 no clearly defined tendency was evident from the results. The range of absorption values in both cases was small and in view of the fact that 60% emulsified water appeared to give more even dyeings, it was decided to use this percentage for future experiments.

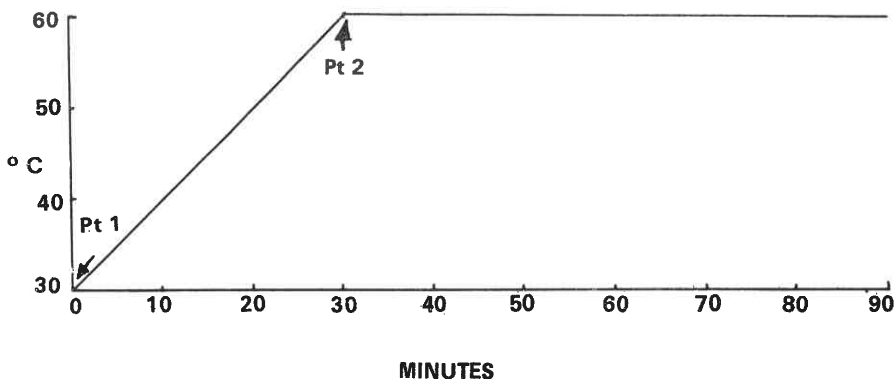
The influence of a number of different parameters on the absorption of the two reactive dyes onto cotton was next investigated. These include the time of alkali addition, the influence of urea and the influence of using a mixture of water and glycerol in place of pure water as the polar solvent.

The discrepancies between the results in Tables I & II for similar dyeings are due to the different concentrations of alkali used. As the optimum concentration of alkali had not yet been determined, the amount of alkali for the different series of dyeings were varied. The amount of alkali added within a series was however kept constant.

TABLE II

EFFECT OF VARIOUS ADDITIVES ON THE ABSORPTION OF C.I. REACTIVE YELLOW 37 AND C.I. REACTIVE RED 63 DYED ON COTTON FROM A SOLVENT SYSTEM

Water (%)	Glycerol (%)	Alkali added at	Urea (%)	Percentage Absorption C.I. Reactive	
				Yellow 37	Red 63
60	0	Pt 1	0	42,8	39,8
60	0	Pt 2	0	43,6	48,4
60	0	Pt 1	10	47,9	43,8
60	0	Pt 2	10	51,7	42,8
42	18	Pt 1	0	—	21,4
42	18	Pt 2	0	—	22,3
54	6	Pt 1	0	—	33,8
54	6	Pt 2	0	—	34,9



The dyeing cycle used to obtain the results on Table II

The manufacturers of the above dyes recommended that the alkali should be added at the start of the aqueous dyeing.

As it was considered that better dyeings might be obtained if the alkali was added at a later stage, the time of alkali addition was varied as shown in Table 11.

From the results in Table 11, the addition of alkali at Point 2 was beneficial towards the absorption. These results were supported by the appearance of the cloths which were dyed more level.

The influence of urea on the absorption differed for each dye. With C.I. Reactive Yellow 37, the addition of urea increased the absorption on the fibre but with C.I. Reactive Red 63 the effect was not consistent. Therefore the influence of different concentrations of urea on the absorption of both dyes was studied. These results are illustrated in Figure 1. The detrimental effect of increasing concentrations of urea on the absorption of C.I. Reactive Red 63 is obvious. It would appear that there is a linear relationship between the concentration of urea and the percentage absorption. It could be that as more urea was added to the dye bath, the swelling of the cellulose fibres was suppressed and fewer molecules of the dye could penetrate the fibres. The absorption of C.I. Reactive Yellow 37 reached a maximum when about 10% urea had been added to the bath. It was, therefore, decided that urea would only be used for the rest of this investigation with C.I. Reactive Yellow 37. The difference in behaviour was difficult to explain as the chemical constitution of the dyes was not available.

The influence of additives, such as urea, formamide, alcohols, etc., on the structure and properties of dye solutions has recently been studied⁽²⁾. Kissa⁽³⁾ found that urea increases the dye fixation by keeping the reactive dye in solution during the diffusion of the dye to its reaction site. But urea can also decrease the fixation of the dye. In combination with alkali and heat, urea enhances the cleavage of an ether-type dye-cellulose bond but has only a slight effect on an ester-type bond. Cockett *et al*⁽⁴⁾, in studying the effect of urea on the dyeing behaviour of wool at low temperatures, commented that the increase in the rate of dyeing, when urea is added to the bath, is due to the disaggregation of the dye in solution and reduction in dye-protein hydrophobic interactions. The action of urea in promoting fibre swelling is also likely, according to the authors, to be a contributing factor. Asquith and Booth⁽⁵⁾, who also studied the influence of urea on wool, found that the swelling of wool was suppressed by the action of urea. With reactive dyes, the disaggregation causes an increase in the number of reactive groups which can react with wool. It has been shown that in a large aggregate of dyestuff molecules only one reactive group in the aggregate will react with the fibre.

The above conclusions, which have been reached on the action of urea

on wool, could very well apply to the action of urea on cellulose fibres. Urea is known to be very effective in breaking hydrogen bonds. There is a distinct possibility of intra-molecular hydrogen bonds occurring in the cellulose molecule⁽⁶⁾.

Asquith and Booth also stated that, with low molecular weight dyes, the overall effect of the addition of urea, is to increase the rate and extent of dyeing, but, with higher molecular weight dyes, the size of the monomolecular particles are unable to enter the fibre due to its suppressed state. It appeared that the views of Asquith and Booth applied. Had the addition of urea had the same effect with both reactive dyes, it might have been conceivable that the action of the urea and alkali caused the covalent bonds to be severed, but, as the effect is different for the two reactive dyes, it seemed, to the author, that the molecular size of the yellow dye might have been small so that it was still possible for the disaggregated molecules to diffuse into the fibre while the molecules of C.I. Reactive Red 63 were too large for many of the molecules to diffuse into the fibres, whose swelling had been suppressed owing to the action of urea.

Replacing pure water with a water-glycerol mixture decreased the absorption of C.I. reactive Red 63 considerably. This was probably caused by the water-glycerol mixture being more favourable to the dye molecules than a pure water solution, thus shifting the equilibrium to the polar phase.

Figure 2 shows the effect of increasing concentrations of alkali on the absorption of the two reactive dyes. It would seem that 6% Na_3PO_4 was sufficient for both dyes.

The rate of absorption of the two reactive dyes from solvent and aqueous media is shown in Figure 3. As the alkali was added at the start of the aqueous dyeings, the initial rate of fixation was much faster for these dyeings. It would seem that the acryloylamide dyes have low reactivity in a neutral bath as almost no absorption occurred before the alkali was added to the solvent dyeing. Thereafter, the rate of absorption of the solvent dyeings increased rapidly, exceeding the rate of the aqueous dyeings. It was decided that no significant increase in the absorption of the dye from the solvent dyeings occurred after 120 minutes.

From the above results a recipe for the dyeing of acryloylamide dyes from a solvent system could therefore be formulated. The recipe consisted of:

x %	Dye
60%	Water
7,5%	SDBS
5,0%	LEA
6,0%	Na_3PO_4
Liquor to goods ratio = 10:1	

The preparation of the emulsion and the procedure were as described above. The alkali was added after 30 minutes (i.e. at 60°C) and the dyeing continued for a further 90 minutes.

Using the above recipe, the absorption of the two reactive dyes, at increasing concentrations of dyestuff, was determined. In the case of C.I. Reactive Yellow 37, dyeings from the solvent medium were conducted with and without urea. The results are shown in figures 4 & 5. The difference in the coefficient of variation between the two solvent-dyed cloths in Figure 5 was significant. The cloths, which were dyed from baths to which urea had been added, had a slight increase in yield for each concentration of dye over those with no urea. The major influence of the urea was to improve the levelness of the cloth. This levelling action of urea might be a result of the ability of the chemical to disaggregate the dye molecules allowing a more even diffusion into the fibres. The coefficients of variation of the aqueous-dyed cloths was so small that it was decided not to show them.

As was expected, the absorption of C.I. Reactive Red 63 from a solvent system was greater than the absorption from an aqueous medium (see Figure 4) after their respective rates of absorption had been determined.

A number of additional dyes in this class were then dyed from aqueous and solvent media. With the latter system, dyeings were conducted in which urea was either added or omitted from the recipe. These results are listed in Table III.

The addition of urea to the dye baths improved the levelness of the dyed cloths in all cases. This could be due to the action of urea in disaggregating the dye molecules thereby allowing a more even adsorption on and diffusion into the fibres. As might have been expected, its influence on the absorption of the dye varied. The reasons for this variation have been elucidated above. The absorption of the dyes showed no constant trend when dyed from a solvent system when compared with the absorption of the cloths dyed from an aqueous medium. This can be seen if C.I. Reactive Yellow 37 and C.I. Reactive Yellow 24 are compared. With the former more dye was absorbed from a solvent than aqueous medium and with urea, in the dye bath, the absorption was further increased. But with C.I. Reactive Yellow 24, similar depths were produced for all three dyeings. With all the dyes tested above, there was no instance in which the fastness properties of the solvent-dyed cloths were adversely affected because of the dyeing process.

TABLE III

CHROMATICITY CO-ORDINATES, ABSORPTION VALUES AND FASTNESS VALUES OF COTTON SAMPLES DYED WITH ACRYLAMIDE DYES FROM AQUEOUS AND SOLVENT SYSTEMS

PROCESS	DYESTUFF	% ABSORPTION	x	y	Y	X / Y	Z / Y	FASTNESS TO WASHING			FASTNESS TO ALK. PERS.			FASTNESS TO LIGHT
								Change in Shade	Staining Wool	Staining Cotton	Change in Shade	Staining Wool	Staining Cotton	
Solvent + Aqueous	C.I. Reactive Red 63	57.8 40.4	0.577 0.521	0.305 0.297	14.20 19.59	1.88 1.75	0.37 0.69	5 5	5 5	5 5	4 4	5 5	4-5 4-5	5-6 4
Solvent + Aqueous	C.I. Reactive Yellow 37	51.3 55.5	0.459 0.459	0.453 0.452	70.97 69.76	0.92 0.93	0.09 0.09	5 5	5 5	5 5	5 4-5	5 5	5 5	7 7
Solvent + Aqueous	C.I. Reactive Blue 38	31.4 40.5	0.173 0.176	0.277 0.276	17.05 16.50	0.62 0.63	1.96 1.95	4-5 4-5	5 5	4 4-5	4 4-5	5 5	4-5 4-5	6 6
Solvent + Aqueous	C.I. Reactive Violet 5	51.9 50.8	0.329 0.327	0.185 0.195	4.31 4.76	1.77 1.76	2.58 2.61	4-5 4	5 5	5 4-5	4 4	5 5	4-5 4-5	6-7 6-7
Solvent + Aqueous	C.I. Reactive Blue 19	47.8	0.320	0.186	6.16	1.71	2.61	4-5	5	5	4	5	4-5	6-7
Solvent + Aqueous	C.I. Reactive Orange 25	55.6 52.4	0.186 0.185	0.130 0.129	5.09 5.17	1.43 1.44	5.21 5.26	5 4	5 4-5	5 5	4 4	5 5	5 5	7 6-7
Solvent + Aqueous	C.I. Reactive Red 49	60.7	0.187	0.129	4.90	1.44	5.22	5	5	5	4	5	5	7
Solvent + Aqueous	C.I. Reactive Yellow 24	48.8 48.8	0.483 0.480	0.377 0.377	47.20 47.09	1.28 1.27	0.36 0.37	4 4-5	5 5	4 4-5	4 4	5 5	3-4 4	5-6 5
Solvent + Aqueous	C.I. Reactive Yellow 24	49.4	0.471	0.373	47.94	1.25	0.40	3-4	5	4-5	3-4	5	3-4	4
Solvent + Aqueous	C.I. Reactive Yellow 24	46.8 40.6	0.420 0.419	0.239 0.239	5.21 5.40	1.75 1.75	1.39 1.40	4 5	5 5	4-5 5	5 5	5 5	5 5	6-7 6-7
Solvent + Aqueous	C.I. Reactive Yellow 24	54.3	0.406	0.233	6.22	1.74	1.53	5	5	5	5	5	5	7
Solvent + Aqueous	C.I. Reactive Yellow 24	28.9 26.4	0.491 0.485	0.440 0.440	31.65 32.55	1.11 1.10	0.15 0.16	5 5	5 5	5 5	5 4D	5 5	5 5	7 7
Solvent + Aqueous	C.I. Reactive Yellow 24	30.0	0.473	0.438	35.18	1.07	0.19	4-5	5	5	3-4D	6	5	6-7

In order to establish what effect this variation in absorption from the two systems would have on dichromatic dyeings, a few combinations were selected and the results obtained are given in Table IV.

It was only the mixture of C.I. Reactive Red 63 and C.I. Reactive Blue 19 that produced a dyeing in which the hue was noticeably different. The solvent-dyed cloths were much redder in appearance, indicating that more of the red component of the mixture was absorbed by the cloth. This is not altogether surprising when one considers the results in Table III. C.I. Reactive Red 63 adsorbed more dye from a solvent than aqueous dye bath while C.I. Reactive Blue 19 adsorbed more dye from the aqueous bath. It is such occurrences which can create problems in colour matching by changing from an aqueous to a solvent dyeing system.

Once again, the cloths, which were dyed from a solvent system to which urea had been added, produced the more level dyeings. In view of this leveling action of urea, it is considered that the recipe, which is given before, should be altered to include 10% urea. The urea should be dissolved with the dye in the preparation of the emulsion.

TABLE IV

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

DYESTUFF MIXTURE	PROCESS	x	y	Y	X ₁ /Y	Z ₁ /Y	FASTNESS TO WASHING				FASTNESS TO ALKALI PERSPIRATION			FASTNESS TO LIGHT	
							Change in Shade	Staining		Change in Shade	Wool	Cotton	Wool		Cotton
								Wool	Cotton						
1.5% C.I. Reactive Blue 19 + 1.5% C.I. Reactive Blue 38	Solvent + Urea	0.187 0.188	0.180 0.181	9.64 9.57	1.04 1.04	3.47 3.45	4-5 5	5 5	4-5 5	5 5	5 5	5 5	6-7 6-7		
	Aqueous	0.192	0.174	9.98	1.10	3.60	5	5	5	5	5	5	7		
2% C.I. Reactive Yellow 24 + 0.5% Reactive Blue 19	Solvent + Urea	0.396 0.393	0.438 0.436	17.87 18.29	0.90 0.89	0.36 0.37	5 5	5 5	5 5	5 5	5 5	5 5	6-7 6-7		
	Aqueous	0.396	0.479	18.68	0.87	0.49	5	5	5	5	5	5	7		
2% C.I. Reactive Yellow 19 + 1% C.I. Reactive Violet 5	Solvent + Urea	0.440 0.430	0.431 0.426	31.72 33.44	1.02 1.00	0.28 0.32	5 5	5 5	5 5	5 5	5 5	5 5	4-5 5		
	Aqueous	0.411	0.406	34.01	1.01	0.44	5	5	5	5	5	5	4		
2% C.I. Reactive Red 63 + 2% C.I. Reactive Blue 19	Solvent + Urea	0.305 0.298	0.220 0.215	4.60 4.75	1.38 1.38	2.12 2.23	5 5	5 5	5 5	5 5	5 5	5 5	3-4 3-4		
	Aqueous	0.235	0.183	6.42	1.30	3.14	4	5	4-5	5	5	5	6-7		

B. Trichloropyrimidine Dyes

The dyes used in this part of this investigation were C.I. Reactive Blue 52 and C.I. Reactive Yellow 29. As with the previous two classes of reactive dyes viz. the monochlorotriazines and acryloylamides, the first step was to find the optimum amount of water necessary for dissolving the dye and producing a level dyeing. This is shown in Table V.

TABLE V

PERCENTAGE ABSORPTION OF C.I. REACTIVE BLUE 52 AND C.I. REACTIVE YELLOW 29 DYED ON COTTON FROM A SOLVENT SYSTEM WITH DIFFERENT AMOUNTS OF EMULSIFIED WATER

% Water	PERCENTAGE ABSORPTION OF C.I. REACTIVE	
	Blue 52	Yellow 29
50	25,0	11,9
60	25,7	12,2
70	27,5	17,9
80	27,0	—

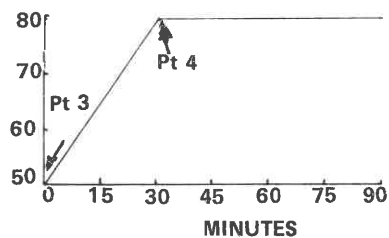
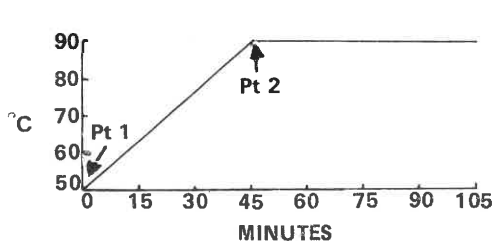
As it was not clear whether 70% or 80% water was the optimum concentration, a second set of dyeings was carried out. As it would be preferable to dye below the azeotropic boiling point, the temperature of the dyeings was varied, with one series being carried out at 90°C and the other at 80°C. The point at which alkali was added was also varied. These results are listed in Table VI.

TABLE VI

INFLUENCE OF DIFFERENT DYEING CONDITIONS ON THE ABSORPTION OF C.I. REACTIVE BLUE 52 AND C.I. REACTIVE YELLOW 29 DYED ON COTTON FROM A SOLVENT SYSTEM

PROCEDURE				PERCENTAGE ABSORPTION OF C.I. REACTIVE	
Dyeing Cycle	% Water	Temp. (°C)	Alkali added at	Blue 52	Yellow 29
				A	70
A	70	90	Point 2	59,7	24,6
A	80	90	Point 1	54,7	25,2
A	80	90	Point 2	57,0	24,4
B	70	80	Point 3	38,2	14,5
B	70	80	Point 4	42,1	11,2
B	80	80	Point 3	41,3	12,7
B	80	80	Point 4	41,5	12,8

The Dyeing Cycles used to obtain the results in Table VI



The decrease in absorption of the dyeings carried out at 80°C compared with those carried out at 90°C was too great and the remaining dyeings were carried out at the latter temperature. The stage at which alkali was to be added, that was used in the rest of this investigation was determined firstly, by the percentage absorption and secondly, by the appearance of the cloths. With the C.I. Reactive Blue 52, the absorption increased slightly if the alkali was added at a later stage, i.e. at either Points 2 or 4 of Dyeing

Cycles A & B respectively depending on whether the dyeing was carried out at 90 or 80°C respectively. In addition, the cloth had a more level appearance. With C.I. Reactive Yellow 29, a higher absorption was obtained if the alkali was added at the start of the dyeings, as is recommended for aqueous dyeings. It was felt, however, that this increase in absorption was not of any significance and that it would be preferable to add the alkali when the bath had attained its dyeing temperature.

There did not seem to be a great difference in the absorption between the two concentrations of water that were used in obtaining the above results. It was decided that 70% would therefore be used in the remaining dyeings as economical advantages would be greater with a lower percentage of emulsified water.

The influence of urea was next investigated and these results are shown in Table VII.

TABLE VII

INFLUENCE OF UREA ON THE ABSORPTION OF C.I. REACTIVE BLUE 52 AND C.I. REACTIVE YELLOW 29 DYED ON COTTON FROM A SOLVENT SYSTEM

PROCESS		PERCENTAGE ABSORPTION OF C.I. REACTIVE	
Alkali added at	Urea (%)	Blue 52	Yellow 29
Start of dyeing	0	48,6	22,5
90°C	0	49,5	19,0
Start of dyeing	10	47,8	23,0
90°C	10	33,1	11,1

No advantage was gained by adding urea to the dye bath and it was therefore not included in the recipe.

The increase in absorption with increasing concentrations of alkali is shown in Figure 6. An amount of 6% Na_3PO_4 was found to be sufficient and consequently this concentration was used henceforth.

The rates of absorption of the two reactive dyes from both media are shown in Figure 7. No appreciable fixation, from the solvent bath, occurred while the temperature of the bath was increasing. After 45 minutes, the alkali was added and the rate of absorption of C.I. Reactive Blue 52 increased rapidly. C.I. Reactive Yellow 29, however, showed extremely poor absorption properties from the solvent bath. The solvent dyeings seemed to be adequately completed after 120 minutes and any further continuation of

the dyeing did not seem to have any significant effect on the absorption.

A recipe for the dyeing of trichloropyrimidine dyes, from a solvent system, was formulated from the above results. It consisted of:

x% Dye
70% Water
7,5% SDBS
5,0% LEA
6,0% Na₃PO₄

Liquor to goods ratio = 10:1

The emulsion was prepared and the dyeing commenced as was described above. The alkali was added after 45 minutes, i.e. at 90°C, and the dyeing was continued for a further 75 minutes.

This recipe was then used to determine the absorption of the two dyes at increasing concentrations of dyestuff. The results are given in Figures 8 and 9. With C.I. Reactive Blue 52, the same depth of shade was produced from both media. The difference in yield, due to the dyeing processes, was clearly evident for C.I. Reactive Yellow 29. The solvent dyed cloths dyed to a much lower depth of shade than the aqueous-dyed cloths. This difference in the reactivity of the dye from the different media had become apparent after the rates of absorption had been obtained. The interaction of dyes with surfactants have been studied by a number of workers ^(7,8,9) As the chemical constitution of C.I. Reactive Yellow 29 was unknown, it was only possible to postulate the reasons for the low absorption that occurred with this dye. An interaction might have occurred between either the chromophore and the emulsifiers or the chromophore and the foam stabilizer, or both, forming a complex whose affinity for the cellulose fibres was low. It seemed unlikely that the reactive group of the dye had been involved in the formation of a complex as the absorption of C.I. Reactive Blue 52 was as good from a solvent system as from an aqueous medium.

A further six dyes were then dyed using the given recipe. The results are listed in Table VIII. The differences in Chromaticity co-ordinates and tristimulus ratios did not seem to be of any significance. No visual differences in hue were noticeable. In general, the fastness properties of all the cloths, i.e. both the solvent and aqueous dyed cloths, were the same.

It was only in the percentage absorption that a difference between the the two processes was found. Dyeing C.I. Reactive Yellow 29, C.I. Reactive Violet 6 and Reactone Golden Yellow S-2R from an aqueous medium produced a significantly deeper shade than when dyed from a solvent system. The reason for this significant decrease in absorption from a solvent bath could

be the formation of complexes, as described above. With the remaining dyes, a similar depth of shade was produced from both baths. This, however, did not seem to affect the tri- and dichromatic dyeings that were attempted, the results of which are listed in Table IX. The dyeing and fastness properties of the cloths from both media appeared to be the same.

TABLE VIII

CHROMATICITY CO-ORDINATES ABSORPTION VALUES AND FASTNESS VALUES OF COTTON SAMPLES DYED WITH TRICHLOROPYRIMIDINE DYES FROM SOLVENT AND AQUEOUS SYSTEMS

DYESTUFF	PERCENTAGE ABSORPTION OF DYE		CHROMATICITY CO-ORDINATES AND TRISTIMULUS VALUES AND RATIOS										FASTNESS TO WASHING						FASTNESS TO ALKALI PERSPIRATION						FASTNESS TO LIGHT	
	Sol.	Aq.	SOLVENT					AQUEOUS					SOLVENT			AQUEOUS			SOLVENT			AQUEOUS			Sol.	Aq.
			x	y	Y	A_x/Y	Z/Y	x	y	Y	A_x/Y	Z/Y	In Shade	Change	Staining	In Shade	Change	Staining	In Shade	Change	Staining	In Shade	Change	Staining		
			A_z/Z	A_y/Y	A_x/Z	A_z/Y	A_y/Z	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5		
C.I. Reactive Yellow 29	27.9	56.3	0.478	0.450	56.96	1.06	0.15	0.509	0.444	49.60	1.14	0.09	5	5	5	5	5	5	5	5	5	5	5	5	6-7	6-7
C.I. Reactive Blue 52	56.2	62.0	0.210	0.177	5.37	1.19	2.43	0.38	0.266	13.97	1.90	0.96	5	4-5	5	5	4-5	5	5	5	5	5	5	5	6-7	7
C.I. Reactive Red 56	63.7	57.9	0.517	0.266	12.93	1.93	0.79	0.501	0.282	13.97	1.14	3.41	5	4-5	5	5	4-5	5	5	5	5	5	5	5	4	4
C.I. Reactive Blue 8	65.5	57.6	0.204	0.168	3.95	1.20	3.69	0.205	0.179	5.29	1.14	0.22	5	5	5	5	5	5	5	5	5	5	5	5	5-6	5-6
C.I. Reactive Orange 34	63.3	63.3	0.571	0.350	14.63	1.62	0.21	0.567	0.356	14.24	1.61	2.35	5	4-5	5	5	4-5	5	5	5	5	5	5	5	6-7	6-7
C.I. Reactive Violet 6	35.6	58.6	0.338	0.195	6.16	1.73	2.36	0.340	0.195	5.38	1.73	2.35	5	5	5	5	4-5	5	5	5	5	5	5	5	5-6	5-6
Reactone B Green S-3G	35.5	33.9	0.224	0.479	15.48	0.47	0.61	0.237	0.465	18.23	0.51	0.62	5	5	5	5	4-5	5	5	5	5	5	5	4	4	4
Reactone Golden Yellow S-2R	46.5	61.6	0.542	0.418	40.27	1.29	0.08	0.548	0.412	36.73	1.33	0.08	4B	5	3	4-5B	5	4	5	5	5	5	5	5	4-5	4-5

TABLE IX

CHROMATICITY CO-ORDINATES AND FASTNESS VALUES OF TRI- AND DICHROMATIC DYEINGS OF TRICHLOROPYRIMIDINE DYES DYED ONTO COTTON FROM SOLVENT AND AQUEOUS SYSTEMS

DYE MIXTURE	CHROMATICITY CO-ORDINATES AND TRISTIMULUS VALUES AND RATIOS										FASTNESS TO WASHING						FASTNESS TO ALKALI PERSPIRATION						FASTNESS TO LIGHT	
	SOLVENT										SOLVENT			AQUEOUS			SOLVENT			AQUEOUS			Sol.	Aq.
	x	y	Y	X ₁ /Y ₁	Z ₁ /Y ₁	x	y	Y	X ₁ /Y ₁	Z ₁ /Y ₁	Change in Shade	Wool	Cotton	Change in Shade	Wool	Cotton	Change in Shade	Wool	Cotton	Change in Shade	Wool	Cotton		
3.5% C.I. React. Orange 34 + 1.2% C.I. React. Blue 62 + 0.7% C.O. React. Red 56	0.404	0.345	4.88	1.16	0.71	0.413	0.347	5.78	1.18	0.67	5	4-5	5	4	5	5	5	4	5	4-5	5	4-5	5	5-6
	2.5% C.I. React. Yell. 0.02% Reactive 81 Green 5 - 3G	0.231	0.217	11.85	1.06	2.51	0.234	0.211	12.21	1.06	2.42	5	5	4-5	5	5	5	4-5	5	5	4-5	5	5	5
1.0% C.I. React. Orange 34 + 1.2% C.I. React. Red 56	0.533	0.317	17.41	1.67	0.66	0.526	0.318	17.81	1.63	0.46	5	5	4-6	5	5	5	4-5	5	5	4-5	5	5	5	4
	0.1% C.I. React. Yellow 29 + 4% Reactive B Green 53G	0.229	0.482	16.51	0.48	0.56	0.248	0.472	19.61	0.52	0.56	4-5	5	4-5	5	5	4-5	5	5	4-5	5	5	5	4-5

SUMMARY AND CONCLUSIONS

A. Acryloylamide Dyes

It was found that it is possible to dye cotton fibre from a water-assisted solvent system using the acryloylamide class of reactive dyes. A final aqueous soaping and rinse was used for both solvent and aqueous systems in order to compare the two dyeing procedures.

The addition of urea to the solvent dye bath proved to be beneficial by acting as a levelling agent. With certain dyes the percentage absorption was increased by the addition of urea. The fastness properties of the solvent-dyed cloths were equal to the properties of the aqueous-dyed cloths.

B. Trichloropyrimidine Dyes

Of the three classes of reactive dyes which were used in this investigation, viz, the monochlorotriazines, acryloylamides and bichloropyrimidines the results obtained for the latter class were the least successful. With these dyes the absorption values of the aqueous dyeings were, in general better than those for solvent dyeings. The fastness properties of the solvent dyed cloths were not inferior to those of the aqueous dyed cloths.

Unfortunately, it proved necessary to dye at 90°C which is above the azeotropic boiling point (87,1° C). This would create further problems as, in any machine used, it would be necessary to ensure that the condensate is returned to the dye bath.

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THE USE OF PROPRIETARY NAMES

The fact that chemicals with proprietary names have been used in this investigation in no way implies that SAWTRI recommends them or that there are not substitutes which may be of equal value or even better.

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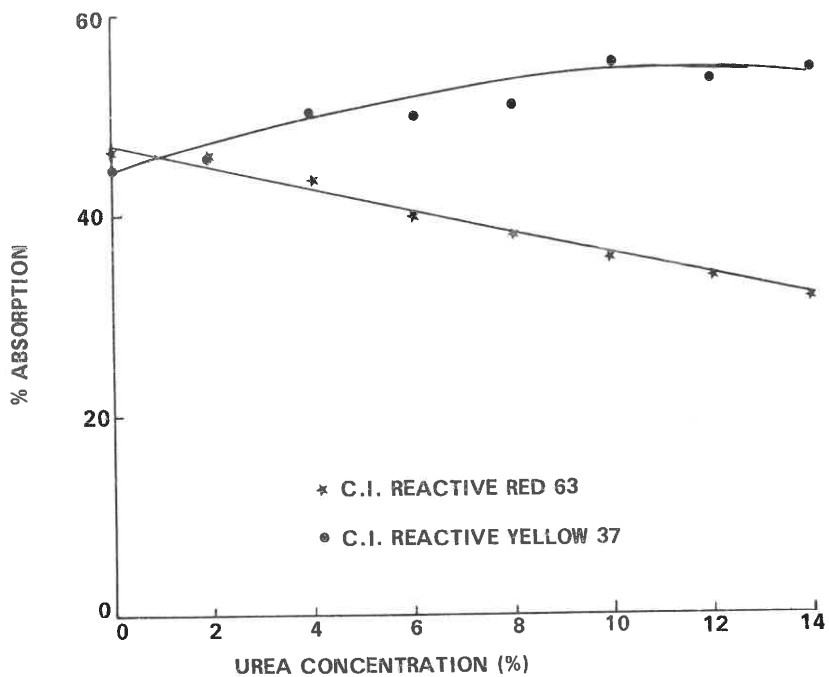


FIGURE 1
 Influence of different concentrations of Urea on the Absorption of C.I. Reactive Red 63 and C.I. Reactive Yellow 37 dyed from a Solvent System.

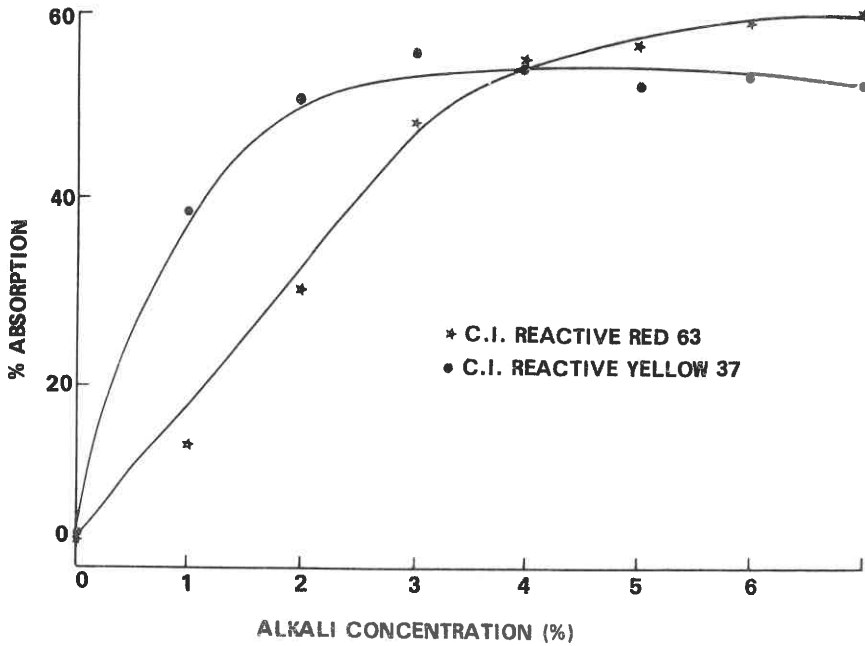


FIGURE 2

Influence of different concentrations of Alkali on the absorption of C.I. Reactive Red 63 and C.I. Reactive Yellow 37 dyes on Cotton from a Solvent System.

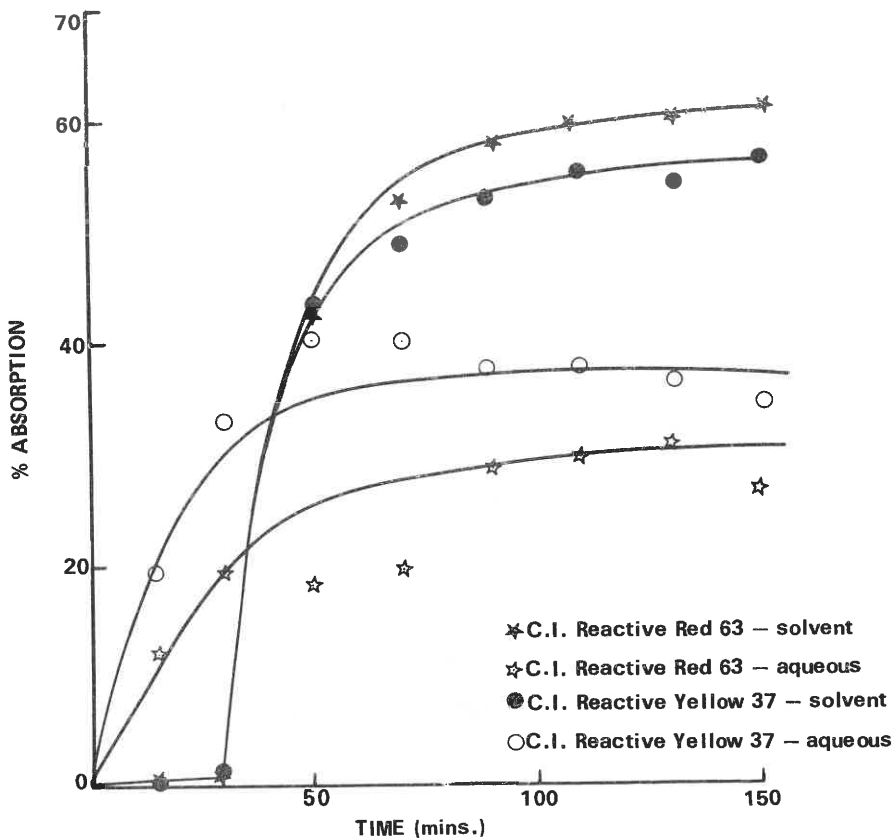


FIGURE 3
 Rate of Absorption of C.I. Reactive Red 63 and C.I. Reactive Yellow 37 on Cotton
 from a Solvent and Aqueous System.

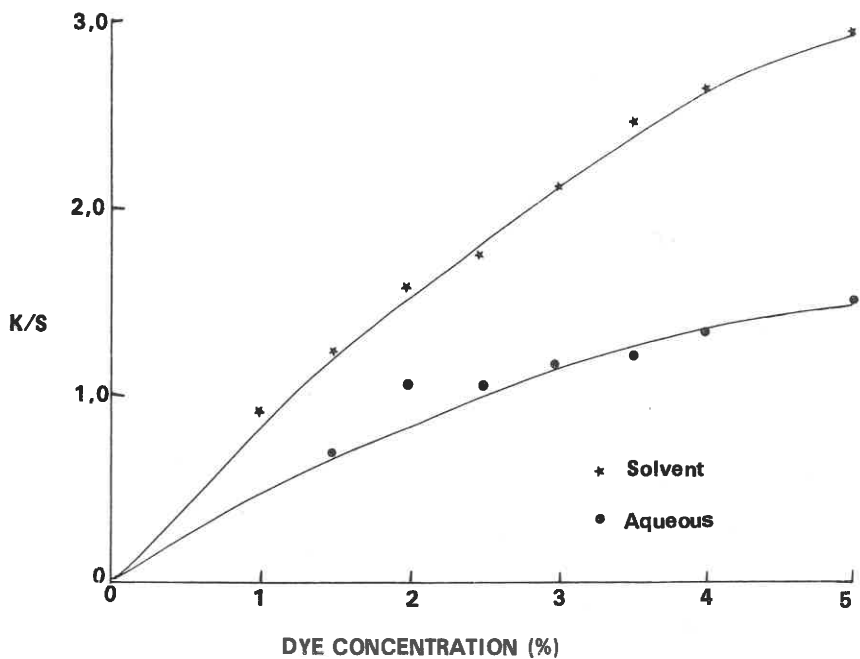


FIGURE 4

Absorption of C.I. Reactive Red 63 on Cotton at increasing concentrations of dye

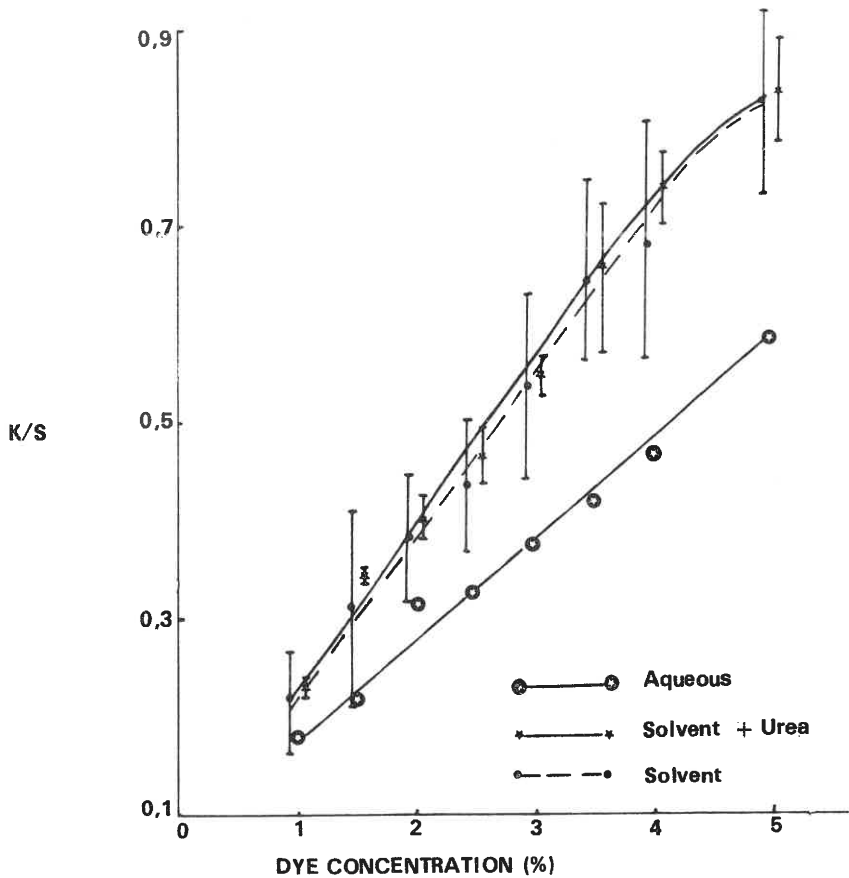


FIGURE 5

Absorption of C.I. Reactive Yellow 37 on Cotton at Increasing Concentrations of dye showing the standard deviation of the solvent-dyed cloths.

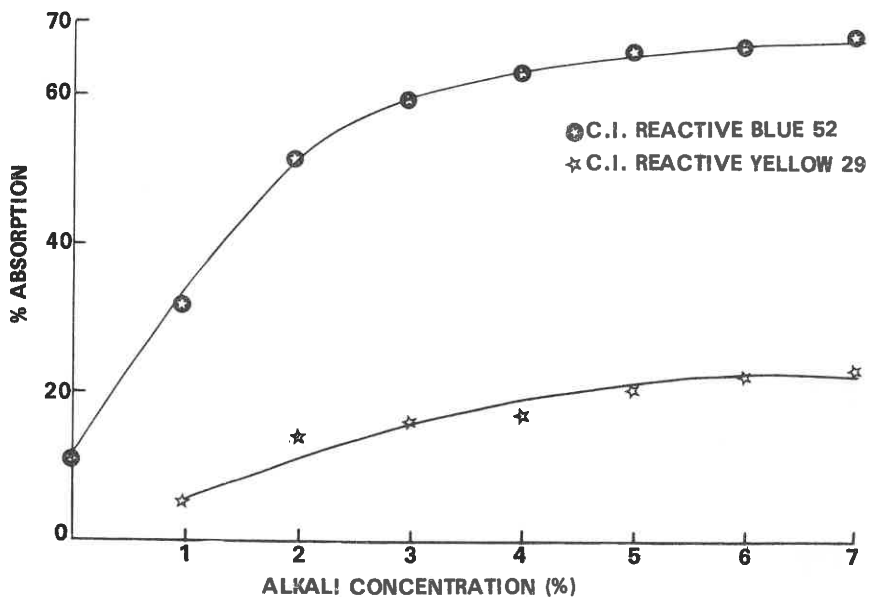


FIGURE 6

Influence of different concentrations of Alkali on the Absorption of C.I. Reactive Blue 52 and C.I. Reactive Yellow 29 on Cotton from a Solvent System.

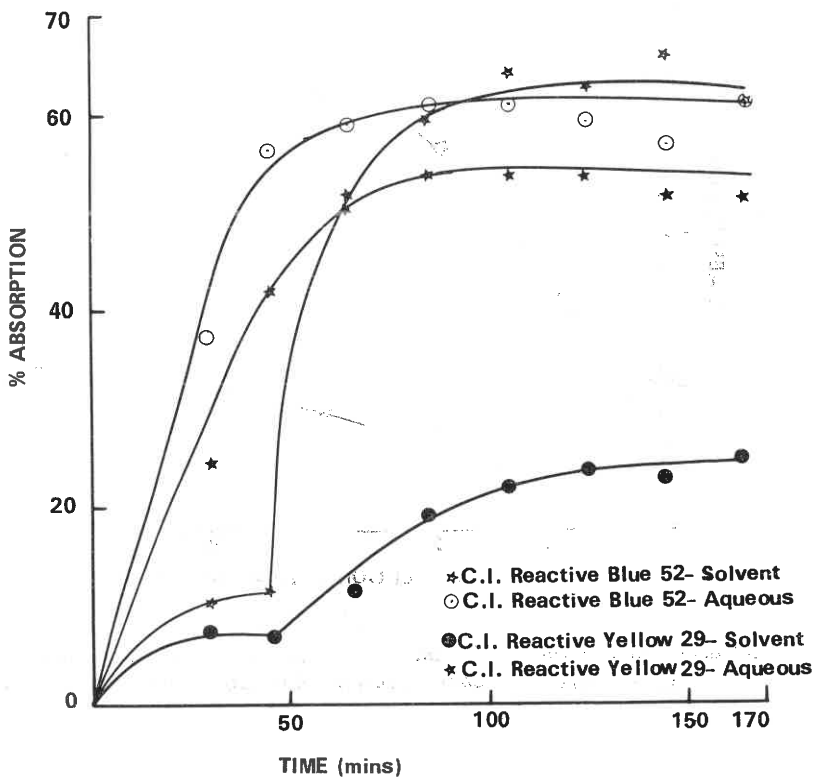


FIGURE 7

Rate of Absorption of C.I. Reactive Blue 52 and C.I. Reactive Yellow 29 on Cotton from Solvent and Aqueous Systems

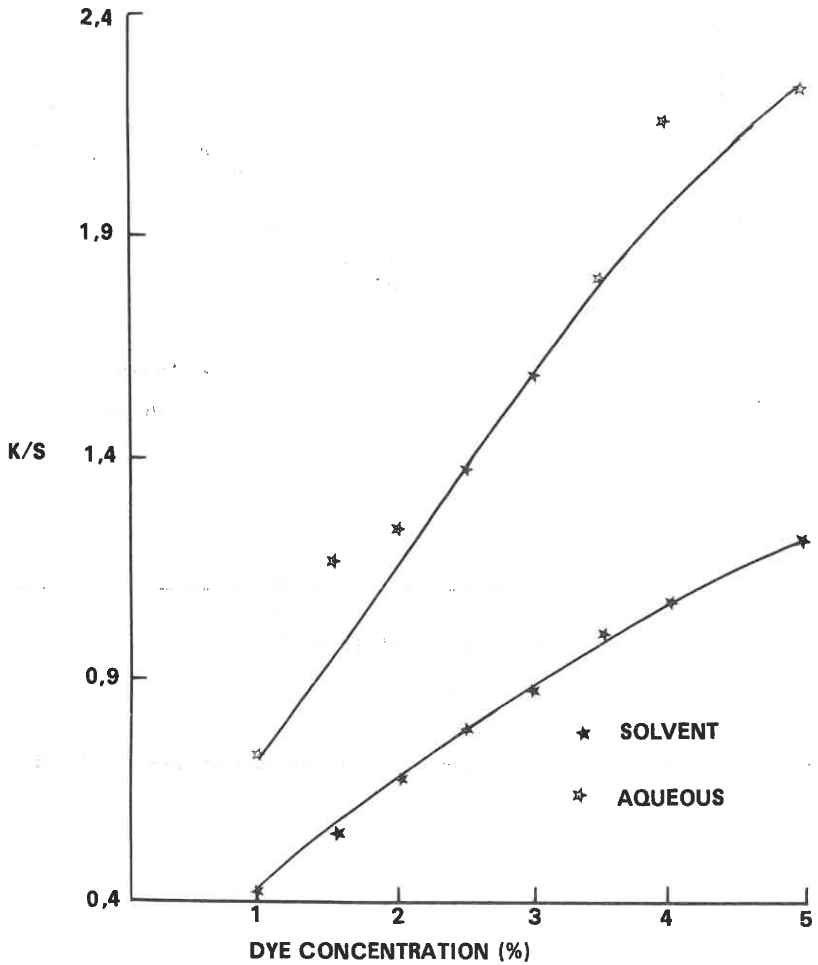


FIGURE 8

Absorption of C.I. Reactive Yellow 29, on cotton at increasing concentration of dye.

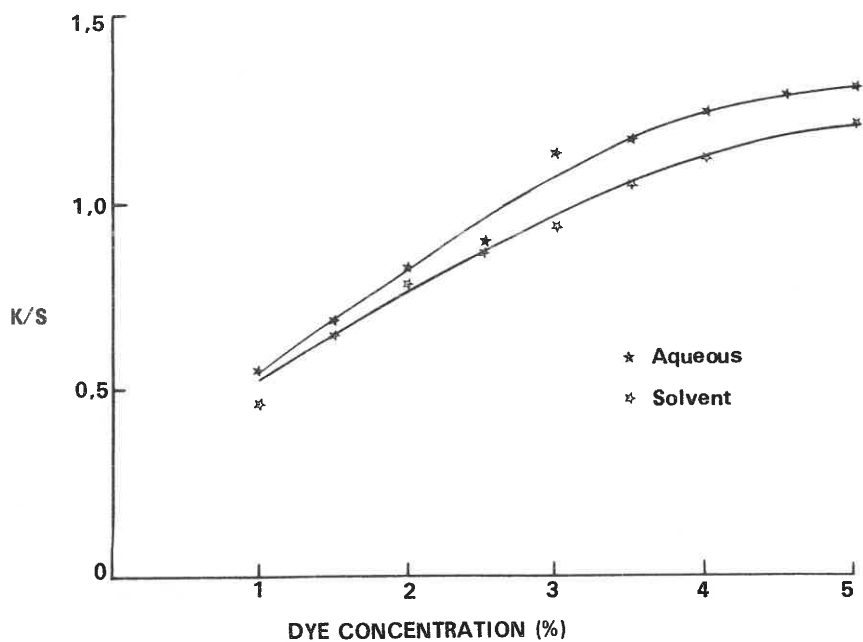


FIGURE 9

Adsorption of C.I. Reactive Blue 52 on Cotton at Increasing Concentrations of dye.

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