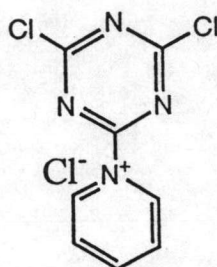


## CHAPTER IV

### Results and Discussion

#### 4.1 Synthesis of Modifying Agent, 2,4-dichloro-6-pyridino-s-triazine (DCPT)

The 2,4-dichloro-6-pyridino-s-triazine (DCPT) which was used in this experiment has the following formula:



This compound was prepared by the method described in section 3.1.

#### 4.2 FT-IR study of the modifying agent

Fourier transform infrared spectroscopy (FT-IR) was carried out to confirm the chemical structure of the modifying agent. The FT-IR spectrum of the DCPT is shown in Figure 4.1 and some of absorption bands are summarized in Table 4.1. The main characteristic absorption bands of the DCPT are found at 1640 and 1620<sup>(22)</sup>, respectively, which are attributable to the stretching vibrations of N-H and C=N bands, respectively.

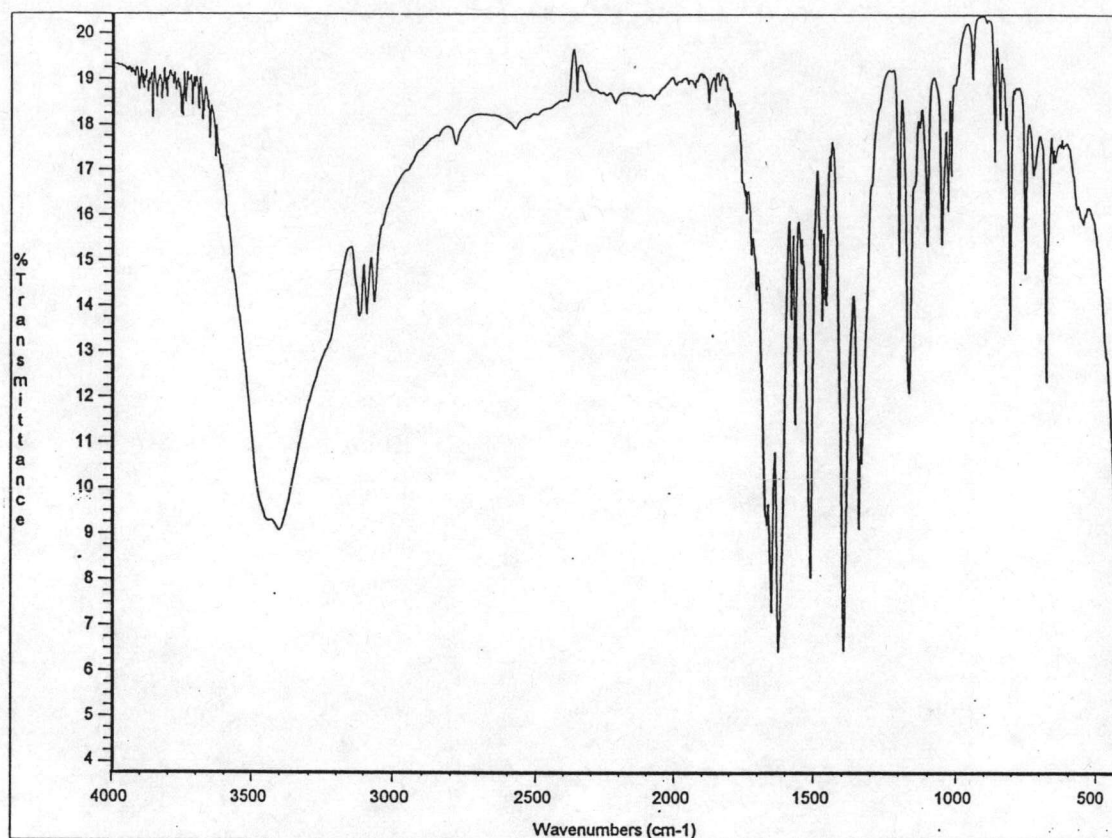


Figure 4.1 FT-IR spectrum of DCPT

Table 4.1 FT-IR absorption bands of DCPT

Wave number (cm <sup>-1</sup> )	Assignments
3100	=C-H stretching (pyridine ring)
1680-1640	C=C stretching (pyridine ring)
1640	N-H stretching (from the pyridinium cation)
1620	C=N stretching (aromatic heterocycle)
1615-1500	N-H bending (from the pyridinium cation)
1400-1300	C-N stretching (aromatic heterocycle)
1300-1000	C-N stretching (pyridine ring)
795	C-Cl stretching (triazine ring)
750-670	=C-H out of plane bending

### 4.3 HPLC study of the modifying agent

HPLC technique was used to follow the reaction between cyanuric chloride and pyridine.

Figure 4.2 and 4.3 show HPLC chromatograms of cyanuric chloride and pyridine alone, respectively. From the chromatogram of cyanuric chloride, it can be observed that a single peak at the retention time of 2 min. represents the presence of cyanuric chloride. From Figure 4.3, the chromatogram shows that pyridine reaches the detector in 2.51 min. which is slower than a cyanuric chloride. This is because pyridine is relatively more polar when compared to cyanuric chloride. Its ability to dissolve in 65:35 of acetonitrile and water mobile phase is therefore less than cyanuric chloride and so it diffuses relatively slower, as a result.

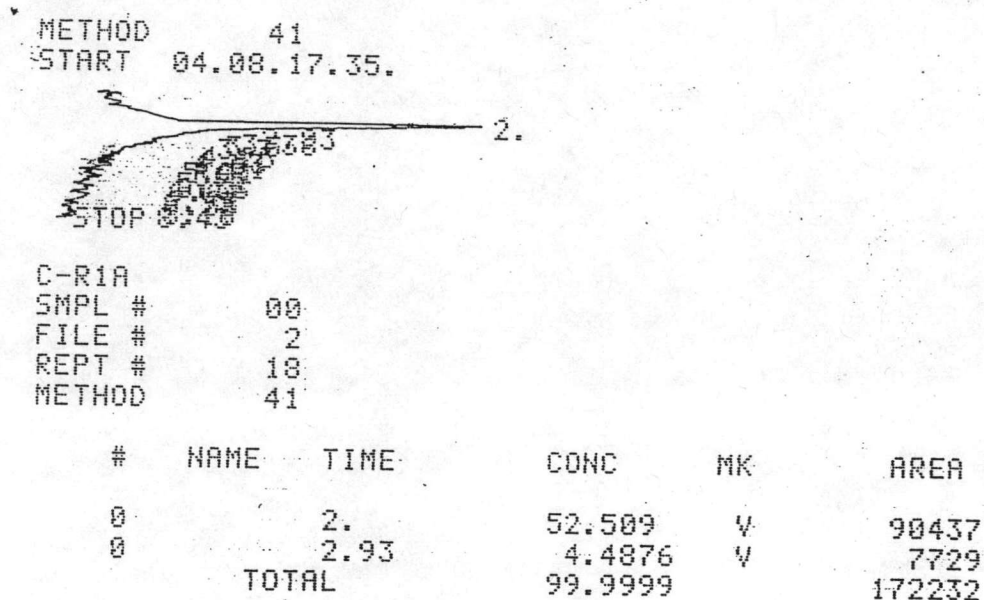
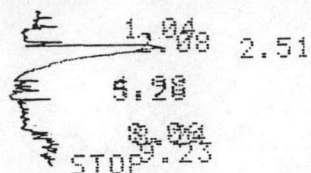


Figure 4.2 HPLC chromatogram of cyanuric chloride

METHOD 41  
 START 04.08.17.24.

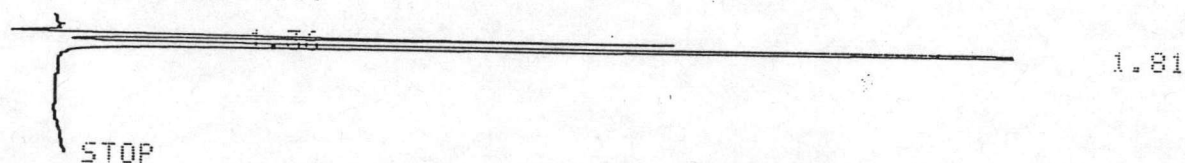


C-R1A  
 METHOD 41  
 00  
 FILE # 2  
 REPT # 17  
 METHOD 41

#	NAME	TIME	CONC	MK	AREA
0		2.51	99.9999		43304
			99.9999		43304

Figure 4.3 HPLC chromatogram of pyridine.

METHOD 41  
 START 12.09.15.33.



C-R1A  
 SMPL # 00  
 FILE # 2  
 REPT # 22  
 METHOD 41

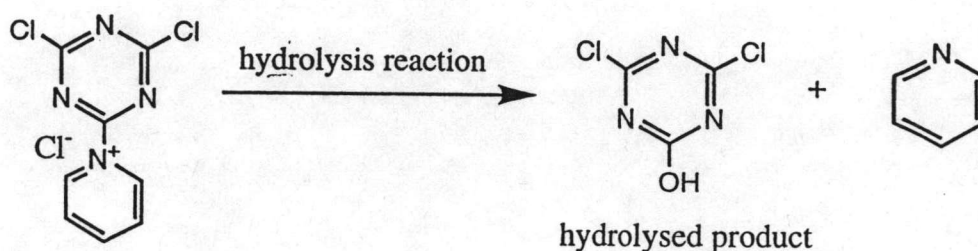
#	NAME	TIME	CONC	MK	AREA
0		1.36	19.6443		26240
0		1.81	80.3556	V	107335
	TOTAL		99.9999		133575

Figure 4.4 HPLC chromatogram of DCPT

Mobile phase ACN/water 65:35  
 Column C<sub>18</sub> bonded phase  
 Flow rate 1.6 ml/min  
 Detector UV absorption, 325 nm.



From Figure 4.4, the chromatogram indicates that reaction mixture taken at the end of reaction has two components. The peak at a retention time of 1.81 min. is believed to represent the peak of DCPT. The another peak at retention time of 1.36 min. is thought to be the peak of hydrolysis product of DCPT, the dichloro-5-hydroxyl-s-triazine derivative. This conclusion is based on the fact that the ionic compound migrates slower than non-ionic compound in ACN/water (65:35) mobile phase. The involved reactions may be written as follows:



Scheme 4.1 Hydrolysis reaction of the 2,4-dichloro-6-pyridino-s-triazine derivative.

The presence of dichloro-5-hydroxyl-s-triazine derivative suggests that the DCPT is unstable and susceptible to hydrolysis reaction. The instability of the DCPT in solution was also investigated. The DCPT solution was kept at room temperature for 1 hour and then subjected to HPLC analysis. The result from Figure 4.5 illustrates that the intensity ratio between the peak at a retention time of 1.55 min. (dichloro-5-hydroxyl-s-triazine derivative) and 2.09 min. (DCPT) increases significantly, confirming that the DCPT can be easily hydrolysed. The result from hydrolysis reaction gives hydrolysed product and a pyridine molecule. So that in Figure 4.5, the peak at a retention time of 2.59 is correspondent the presence of pyridine. The unstable characteristic of the DCPT therefore causes the drawback to exploit the compound commercially. In this project, the application of the DCPT to cotton fabric was carried

out immediately without isolation from reaction mixture to prevent the hydrolysis of the DCPT.

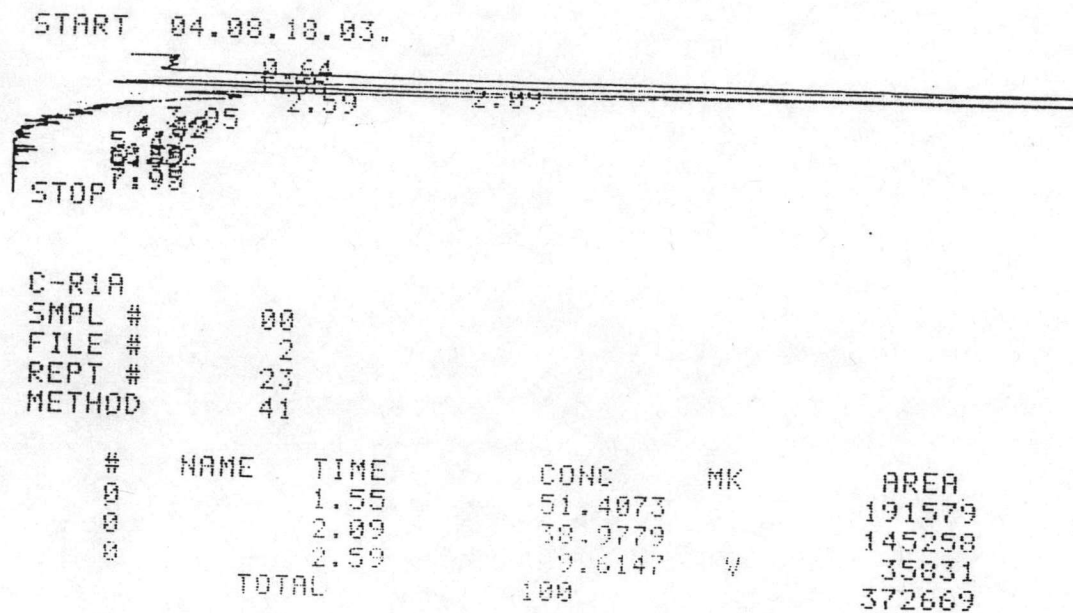


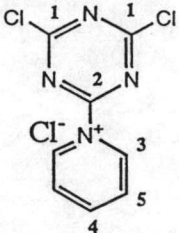
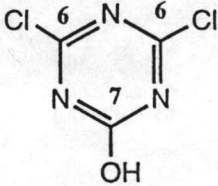
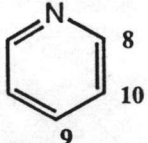
Figure 4.5 HPLC chromatogram of DCPT solution (kept at room temperature for 1 hour)

#### 4.4 NMR study of the modifying agent

The  $^{13}\text{C}$ -NMR spectroscopy was used to confirm the chemical structure of the DCPT compound. NMR peaks of all carbon atoms were shown in Table 4.2.

From the HPLC chromatogram of DCPT solution indicates that the reaction mixture taken at the end of reaction has three components, DCPT compound, hydrolysed product and pyridine was released when hydrolysis reaction occurred.

Table 4.2 Liquid state  $^{13}\text{C}$ - NMR chemical shift of DCPT.

Compound	Chemical shift ( $\delta$ )	Assignments
<b>DCPT</b>	171.537	1
	170.287	2
	154.493	3
	143.323	4
	130.738	5
<b>Hydrolysed product</b>	171.718	6
	173.495	7
<b>Pyridine</b>	154.016	8
	143.208	9
	130.623	10

#### 4.5 Total nitrogen determination of modified cotton fabric

To confirm that the DCPT was covalently fixed to cellulose, the total nitrogen content on treated fabric was determined. The results are shown in Table 4.3. Total nitrogen content of DCPT cotton is shown in column 2 while the total nitrogen content

of DCPT cotton further treated with ethylenediamine is illustrated on column 3. It can be seen that total nitrogen content is increased with an increase in the concentration of DCPT. Furthermore, these data indicate that pretreatment of cotton in a liquor containing 15 g/l of DCPT solution giving a maximum total nitrogen content ratio which resulting in the maximum reactive sites to react with dye molecules.

Table 4.3 Total nitrogen content of modified cotton fabrics at various concentrations of DCPT

Concentrations of DCPT (g/l)	Total nitrogen content (%)		Actual total Nitrogen content ratio
	Before adding ethylenediamine	After adding ethylenediamine	
5	0.257	0.261	1.016
10	0.369	0.393	1.065
15	0.464	0.624	1.345
20	0.559	0.697	1.247
25	0.758	0.828	1.092
30	0.841	0.891	1.059

#### 4.6 Application of DCPT

The application of the DCPT to cotton fabric was carried out using both exhaustion and padding method as described in section 3.6.1 and 3.6.2 respectively.

##### 4.6.1 Application of commercial reactive dye to the modified cotton fabric obtained from exhaustion method

###### 4.6.1.1 Dyeings by exhaustion method

###### 4.6.1.1.1 Effect of DCPT concentrations on dye fixation

The dyeing of modified cotton fabric in the absence of alkali was investigated. The aminated cotton fabrics with various concentrations of the DCPT were dyed with commercial reactive dye, Cibacron F (2% o.w.f.) at 85°C for 1 hour in the presence of



20 g/l of NaCl. The dyebath exhaustion was measured at the end of dyeing before the measurement of the extent of dye fixation on treated fabrics after soaping was carried out. This investigation was classified into two processes, one- step and two- step methods, the results were shown in Table 4.4 and 4.5 respectively.

Table 4.4 Color yield, % dye exhaustion and % fixation of exhaust dyeings of modified cotton at various concentrations of DCPT in one-step treatment process.

Concentrations of DCPT (g/l)	K/S		%Exhaustion	%Fixation
	Before soaping	After soaping		
0	6.446	2.179	15.63	5.28
5	22.386	20.898	74.05	69.13
10	23.677	22.078	75.52	70.42
15	22.364	20.880	76.90	71.80
20	23.066	20.114	75.34	65.70
25	20.965	18.363	74.54	65.29
30	19.496	16.663	74.87	63.99

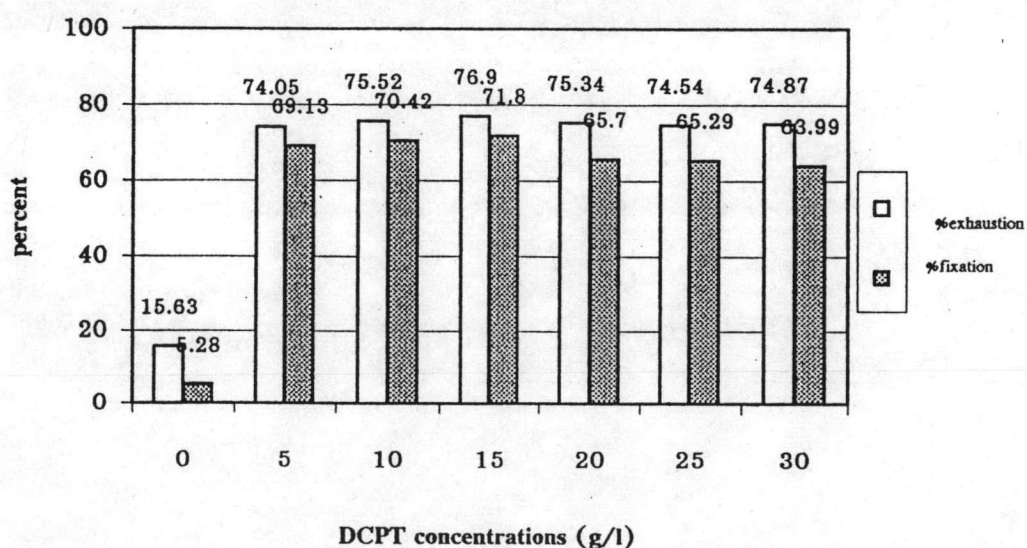


Figure 4.6 Chart of the exhaust dyeing of modified cotton obtained from one-step treatment process.

Table 4.5 Color yield, % dye exhaustion and % fixation of exhaust dyeings of modified cotton at various concentrations of DCPT in two-step treatment process.

Concentrations Of DCPT (g/l)	K/S		%Exhaustion	%Fixation
	Before soaping	After soaping		
0	6.446	2.179	15.63	5.28
5	18.533	17.660	63.89	60.88
10	20.703	19.162	68.63	63.52
15	23.323	20.612	78.80	69.64
20	24.468	21.428	79.13	69.30
25	22.831	20.197	78.63	69.56
30	23.562	21.247	79.29	71.50

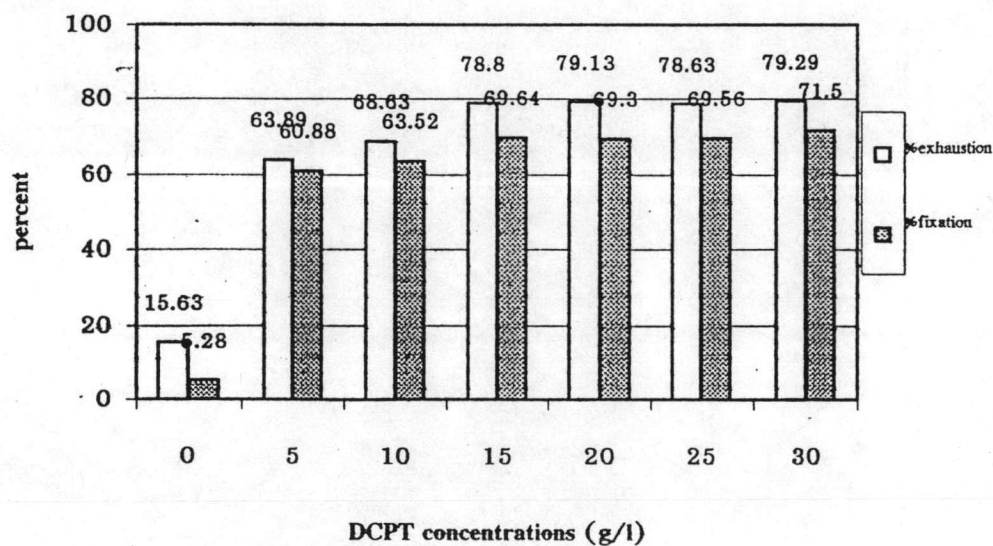


Figure 4.7 Chart of the exhaust dyeing of modified cotton obtained from two-step treatment process.

The percentage of dye exhaustion and the total dye fixation were shown in the above Tables. It can be seen that the covalent fixation of reactive dye on cellulosic fiber can be achieved without the requirement of alkali. The percentage of dye exhaustion in all case of the DCPT concentration applied is about 75% while the degree of dye fixation is slightly lower. It is important to point out that most dye molecules which can diffuse inside the fiber can react with the reactive dye site. This conclusion is confirmed by the slightly change in dye color yield before and after soaping. The significant difference in K/S values between before and after soaping samples indicate that almost dye molecules being absorbed into the fiber can react with the reactive dye sites. It can be said that this dyeing system, by comparison, provides an advantage over conventional method in terms of reproducibility, since the degree of dye hydrolysis is relatively much smaller.

#### 4.6.1.1.2 Effect of dye concentration on dye fixation

Cotton fabrics were pretreated using 15 g/l DCPT and 10 g/l  $\text{Na}_2\text{CO}_3$  and were then dyed using various dye concentrations ranging from 1 to 4% o.w.f. in the presence of 20 g/l NaCl. The results of the color yield and the degree of dye fixation are given in Table 4.6.

Table 4.6 Color yield, % dye exhaustion and % fixation of exhaust dyeings of modified cotton at various dye concentrations.

Concentrations of dye(%o.w.f)	K/S		%Exhaustion	%Fixation
	Before soaping	After soaping		
1	15.374	14.724	72.67	69.60
2	23.556	21.595	69.42	63.64
3	26.942	24.684	68.90	63.13
4	28.257	26.132	63.17	58.42

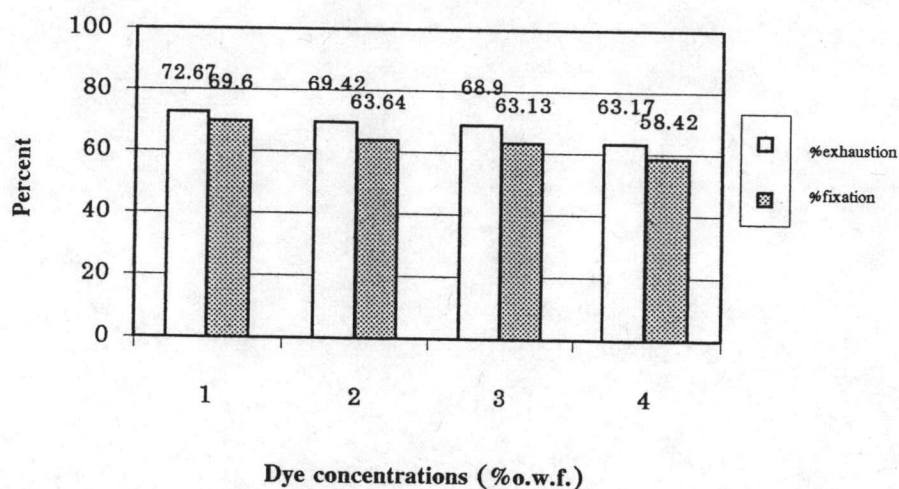


Figure 4.8 Chart of the exhaust dyeing of modified cotton at various dye concentrations.

Comparison between dye color yield of the dyed fabric before and after soaping is also shown in the table. From data, it should be noted that a deep depth of shade has been already achieved when a concentration of 2% o.w.f. was used. Above 2% o.w.f. of dye concentration only a small increase in K/S value was observed. From the results of dye exhaustion and the degree of dye fixation, it can be seen that the trend of dye uptake onto the fiber as well as the degree of dye fixation gradually decreases when the dye concentration increases. These results reflect the buildup of fixed dye onto cellulose retards the further absorption of dye molecule from dyebath due to the effect of negative repulsion.



#### 4.6.1.1.3 Effect of electrolyte concentration on dye fixation

Generally, cotton cellulose in an aqueous solution has slightly negative charges due to the accumulation of anionic ions on the fiber surface. These slightly negative charges on cotton cellulose usually cause the repulsive barrier to prevent the absorption ability of anionic dyestuffs such as reactive dyes into the fiber. So, dyeing of cotton with a reactive dye requires electrolytes such as sodium chloride or sodium sulfate to suppress the negative charges on the cotton surface and then lead to the increase in dye absorption. Therefore, an attempt to obtain high dye exhaustion was investigated by varying the concentrations of sodium chloride concentration. In this study, the cotton fabric was pretreated with 15 g/l DCPT and 10 g/l of  $\text{Na}_2\text{CO}_3$  and was then dyed (1% o.w.f.) at different salt concentrations in a range of 10-40 g/l. The results of dye exhaustion and the degree of dye fixation are illustrated in Table 4.7.

Table 4.7 Color yield, % dye exhaustion and % fixation of exhaust dyeings of modified cotton at various NaCl concentrations.

Concentrations of salt (g/l)	K/S		%Exhaustion	%Fixation
	Before soaping	After soaping		
10	15.419	12.803	64.90	53.89
20	17.708	16.588	74.14	69.45
30	19.251	18.236	75.70	71.71
40	20.248	18.960	78.62	73.62

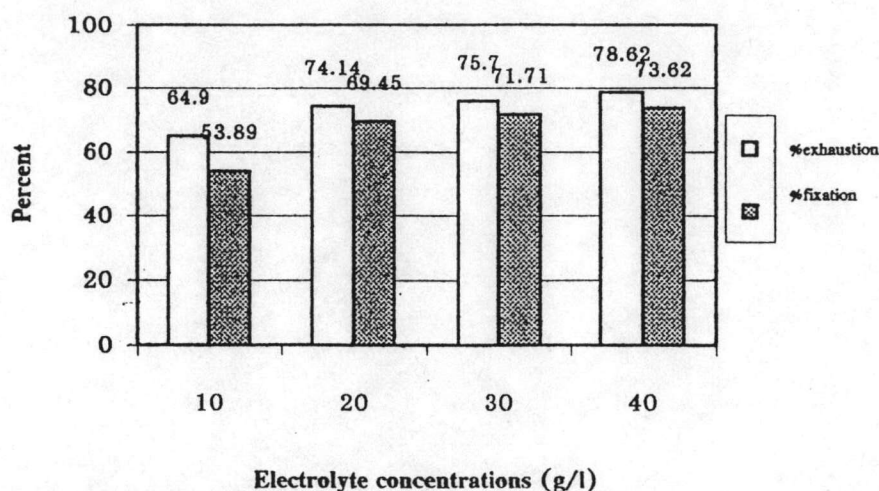


Figure 4.9 Chart of exhaust dyeing of modified cotton at various electrolyte concentrations.

The results show that the percentage of dye exhaustion as well as dye fixation increases as the concentration of salt increases. However, the addition of salt from 20 to 40 g/l yields a moderate increase in dye uptake. An attempt to achieve the dye uptake more than 90% exhaustion was unsuccessful. It is believed that the build up of anionic dye molecules inside the fiber acts as a negative shield to prevent a further increasing in dye absorption. Conclusion which can be drawn from this experiment is that concentration of NaCl of 20 g/l is the most appropriate. The use of high salt concentration, eventhough bringing about a slight increase in dye utilization, may cause the environmental problems.

#### 4.6.1.2 Dyeings by thermofixation method

For comparison, dyeing of modified cotton by thermofixation method was studied. Cellulose fabric previously modified with 15 g/l DCPT was chosen in the study. Modified cotton fabrics were padded with two sets of pad- liquors containing 10, 20, 30 and 40 g/l dye respectively, and 1 g/l wetting agent; the first set was used without adding urea by the addition of 100 g/l urea to each pad-liquor of the second set

was made before padding. All paddings were carried out using a pad mangle set a pressure to give 80 % wet pick-up. The padded fabrics were dried at 100°C for 10 min. and were then baked at 160°C for 3 min. The dyed cotton were soaped in a solution containing non-ionic detergent (5 g/l) at boil for 15 min. The measurement of the absorbance of the diluted soaped-off dye solution was carried out and the dye fixation value was then calculated. The results of dye fixation values were shown in Table 4.8.

Table 4.8 % Dye fixation of thermofix dyeings of modified cotton at various dye concentrations.

Concentrations of dye (g/l)	%Fixation	
	Pad-liquor With urea	Pad-liquor without urea
10	38.32	34.91
20	53.65	44.88
30	57.39	47.08
40	60.02	50.21

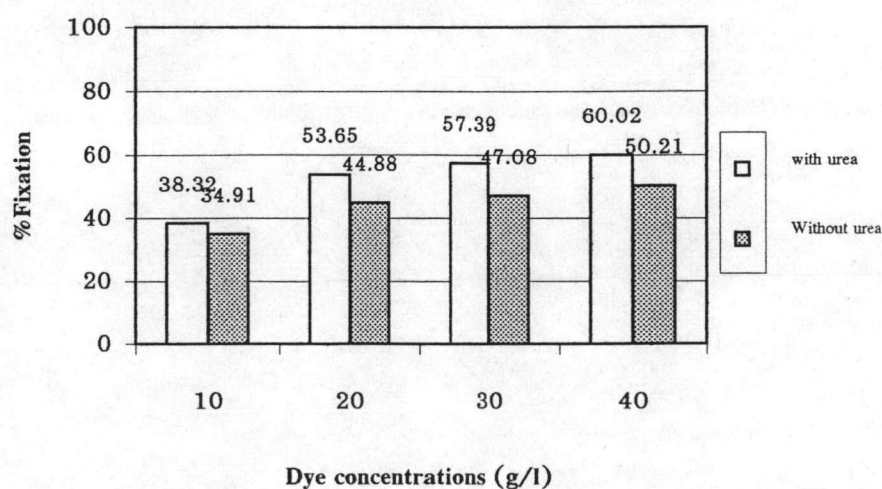


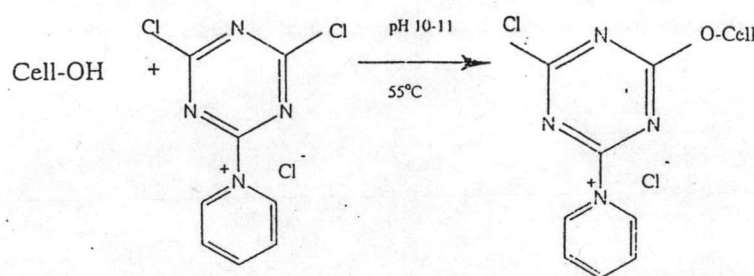
Figure 4.10 Chart of thermofix dyeing of modified cotton at various dye concentrations.



In general, dyeing by thermofixation method gives relatively lower dye fixation when compared to those results obtained from exhaustion method. The relatively low dye fixation in this case may be partly due to the short reaction time and the inadequate contact between dye molecules on reactive sites on the fiber. The later problem can be overcome by adding urea to enhance dye diffusion. The results as indicated in the second column on Table 4.8, confirm that adding urea into dye bath markedly improves the degree of dye fixation. Surprisingly, trend of the degree of dye fixation implies that higher dye concentration gives the higher degree of dye fixation, indicating that the effect of negative repulsion can not be applied to the thermofixation method.

#### 4.6.2 Application of commercial reactive dye to the modified cotton fabric obtained from a pad-batch method

Another process commercially practiced in dyeing industry is a pad-batch method. So, it is worthwhile that an investigation of application of the modifying agent by this method should be included in this project. Cotton fabric was padded with 15 g/l DCPT solution to give 80% wet pick-up and then re-padded again with 10 g/l  $\text{Na}_2\text{CO}_3$ . The padded fabric was wrapped-up in polyethylene sheet and then batched at room temperature for 24 hours. In this stage, the following reaction may occur ;



After being batched for 24 hours, the batched fabric was rinsed and then re-padded with the solution containing 10 g/l ethylenediamine and 100 g/l urea, Finally, this cotton fabric was steamed for 4 min. at  $100^\circ\text{C}$  and rinsed thoroughly in running



water. The reactive fiber of which structure is shown in Figure 4.11 was expected to be obtainable.

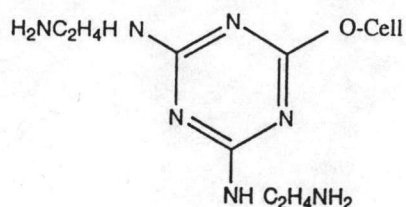


Figure 4.11 Chemical structure of the reactive fiber.

The modified cotton was dyed in the absence of alkali using the following methods:

#### 4.6.2.1 Dyeings by exhaustion method

The dyeing was carried out by using the method described in section 3.7.1. A concentration of dye used in this study was varied in the range of 1 to 4% o.w.f. The resultant percentage of exhaustion and fixation values are shown in Table 4.9.

Table 4.9 Color yield, % dye exhaustion and % fixation of exhaust dyeing of cotton modified by a pad-batch method.

Concentrations of dye(% o.w.f.)	K/S		%Exhaustion	%Fixation
	Before soaping	After soaping		
1	14.150	7.913	60.54	33.86
2	15.419	7.590	63.60	31.31
3	14.228	6.759	59.58	28.30
4	14.829	6.523	61.49	27.05

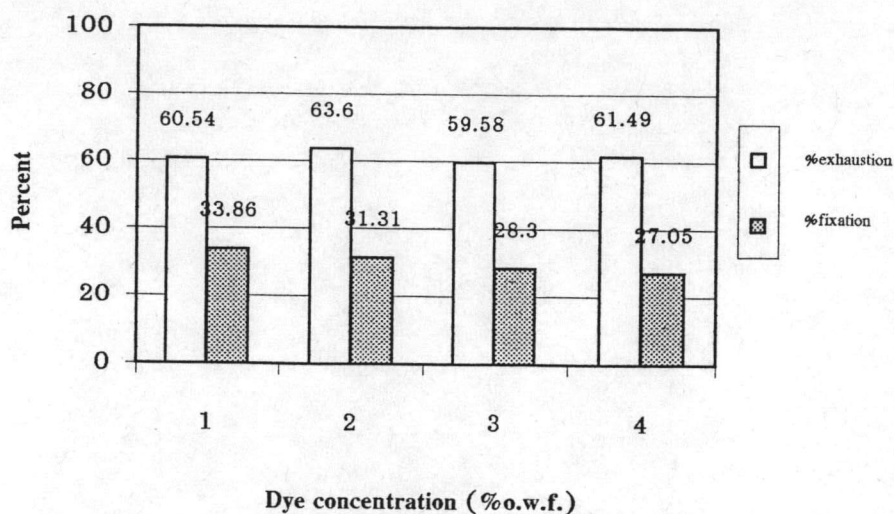


Figure 4.12 Chart of the exhaust dyeing of cotton modified by a pad-batch method.

From above results, it can be seen that the percentage of dye exhaustion in all cases of the dye concentration applied is about 60% which is lower than those obtained from the dyeing of modified cotton by an exhaustion method. Disappointingly, the degree of dye fixation is greatly low. These can be explained that the availability of reactive sites on the modified cotton by a pad-batch method is too low which reflects that an attempt to introduce a reactive group into cotton by this method yields an unsatisfactory result. Therefore, it may be concluded that application of the modifying agent by a pad-batch method is inefficient when compared to the exhaustion method. The reason behind this may be partly due to the fact that ethylenediamine has a low boiling point ( $108^{\circ}\text{C}$ ) which is likely to be evaporated at the steaming temperature ( $100^{\circ}\text{C}$ ) before undergoing the reaction with the DCPT-cotton.

#### 4.6.2.2 Dyeings by thermofixation method

The modified cotton fabrics were dyed in a pad-liquor containing 10-40 g/l dye, 100 g/l urea and 1 g/l wetting agent as described in section 3.7.3. The results are shown in Table 4.10.

Table 4.10 % Dye fixation of thermofix dyeing of cotton modified by a pad-batch method.

Concentrations of dye (g/l)	%Fixation
10	22.99
20	23.98
30	24.74
40	29.32

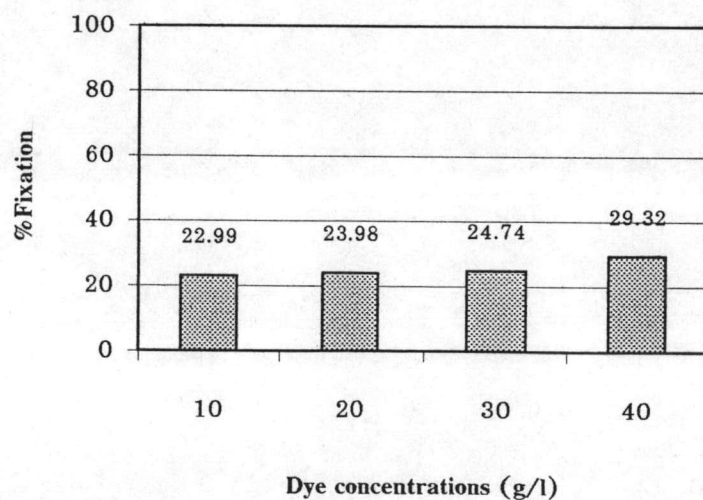


Figure 4.13 Chart of thermofix dyeing of cotton modified by a pad-batch method.

The degree of dye fixation as indicated in the second column in Table 4.10 shows that thermofixation dyeing of cotton fabric modified by a pad-batch process gives slightly lower dye fixation when compared to those results obtained from exhaustion method (as shown in Table 4.9). These results (Table 4.9 and 4.10) suggest that cotton fabric modified by a pad-batch method is not recommended. In summary, the most suitable method for applying the modifying agent to cotton fabric should be carried out using an exhaustion method.

#### 4.6.3 The study on conventional dyeing procedures.

##### 4.6.3.1 Dyeing of unmodified cotton fabric by exhaustion method under alkaline conditions.

All dyeings were carried out as described in section 3.7.2. The resultant exhaustion and fixation values are shown in Table 4.11

Table 4.11 Color yield, % dye exhaustion and % fixation of exhaust dyeing of unmodified cotton.

Concentrations of dye (%o.w.f)	K/S		%Exhaustion	%Fixation
	Before soaping	After soaping		
1	7.722	6.632	54.34	46.67
2	17.229	13.721	53.09	42.28
3	22.634	17.242	53.76	40.95
4	24.205	21.145	44.54	38.91



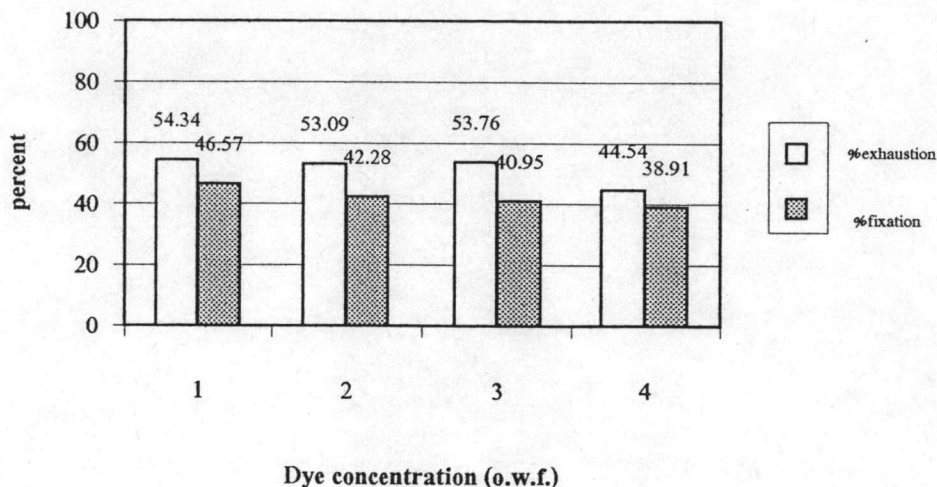


Figure 4.14 Chart of the exhaust dyeing of unmodified cotton.

#### 4.6.3.2 Dyeing of unmodified cotton fabric by thermofixation method under alkaline conditions.

All dyeings were carried out as also described in section 3.7.4. The resultant fixation values are shown in Table 4.12.

Table 4.12 Dye fixation values of thermofix dyeing of unmodified cotton fabric

Concentrations of dye (g/l)	%F
10	47.41
20	59.96
30	63.39
40	65.08

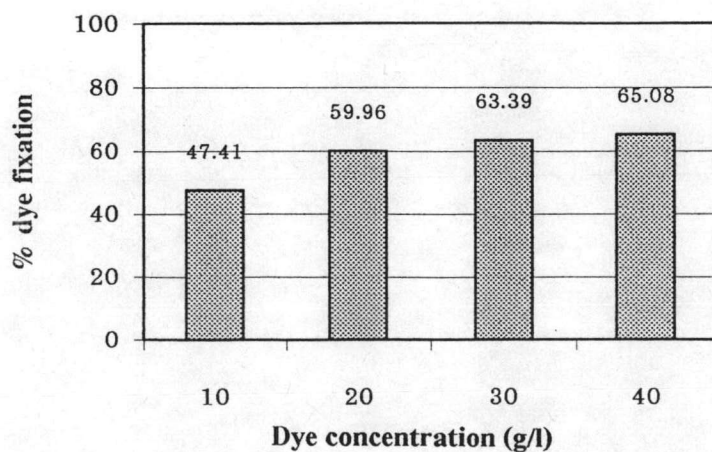
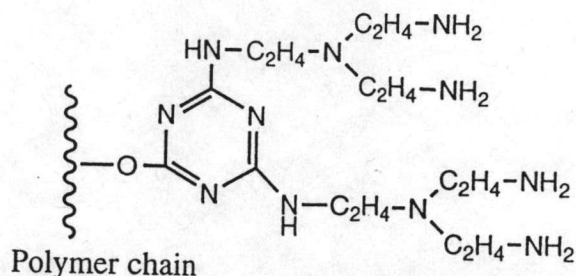


Figure 4.15 Chart of thermofix dyeing of unmodified cotton.

It is obvious that percentage of dye fixation on unmodified cotton fabric in the presence of alkali obtained either from exhaustion dyeing method or thermofixation method is rather low. This problem has been well addressed that the main contributing factor that makes the dye fixation rather poor is the hydrolysis reaction of dye under alkali dyeing condition. As in the case of dyeing the modified cotton where no alkali involved during dyeing of reactive dye, it may be said that dyeing cotton fabric by the new dyeing system may offer superior results in terms of dye color yields, dye waste discharge and good reproducibility. This dyeing system is quite suitable in the case of heavy shade dyeing.

### An attempt to improve the dyeability of DCPT treated cotton fabric

In this study, tris-(2-aminoethyl)amine was used to replace the ethylenediamine. As a result, the modified cellulose which has the following structure may be obtainable:



It can be seen that one moiety of tris-(2-aminoethyl)amine can provide twice as much as reactive groups than ethylenediamine, hence resulting in high dye fixation expectable. The resultant dyeings of tris-2-(aminoethyl)amine treated DCPT cotton are shown in Table 4.13. As expected, a noticeable improvement in the dyeability of DCPT cotton fabric was achieved when tris-(2-aminoethyl)amine was used instead of ethylenediamine (see Table 4.13). Since, tris-(2-aminoethyl)amine moiety reacts with the DCPT cotton cellulose giving more reactive dye sites, it means that the concentration of the DCPT required to be incorporated into the fiber could be reduced. It is true, from Table 4.13 that DCPT concentration at 5 g/l is enough to obtain maximum dye fixation. On the other hand, 15 g/l of DCPT was used and yet dye fixation was still lower, in the case of ethylenediamine treated DCPT cotton cellulose. Therefore, tris-(2-aminoethyl)amine is recommended as an alternative aminizing agent for this dyeing system.

Table 4.13\_ Colour yield, % dye exhaustion and % fixation of dyeings of tris-(2-aminoethyl)amine treated DCPT cotton at various concentrations of DCPT.

Concentrations of DCPT(g/l)	K/S		% Exhaustion	% Fixation
	Before soaping	After soaping		
5	26.035	22.403	87.38	75.19
10	25.500	23.124	83.38	75.61
15	25.920	23.635	84.97	77.48
20	26.257	23.917	86.41	78.71
25	25.099	22.512	86.56	77.64
30	25.360	22.579	86.82	77.64

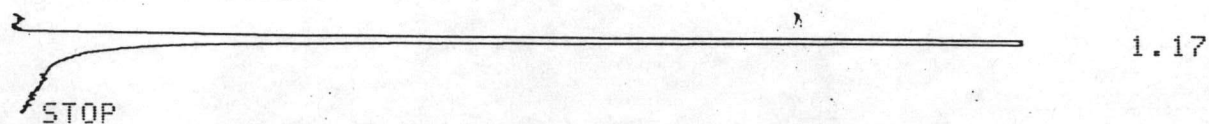


### The studying on stability of reactive dye in the dye bath

Stability of reactive dye to hydrolysis reaction can be investigated by HPLC and TLC techniques. HPLC study was carried out by sampling the dyebath before and after the dyeing process and was then run into the HPLC instrument to compare the retention time ( $t_R$ ) values of both dye samples. The HPLC chromatogram was shown in Figure 4.16.

(a)

START 01.10.14.57.

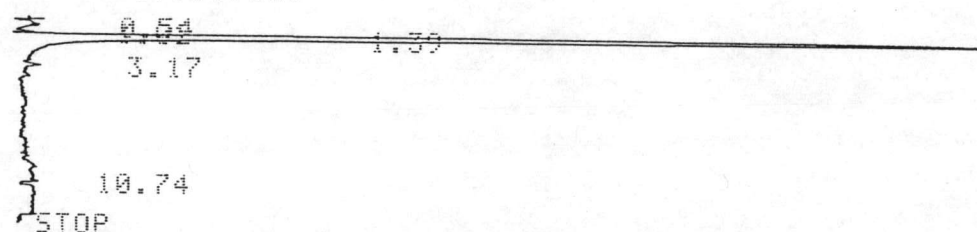


C-R1A  
 SMPL # 00  
 FILE # 1  
 REPT # 5912  
 METHOD 41

#	NAME	TIME	CONC	MK	AREA
0		1.17	99.9999		170112
	TOTAL		99.9999		170112

(b)

START 12.09.13.30.



C-R1A  
 SMPL # 00  
 FILE # 2  
 REPT # 15  
 METHOD 41

#	NAME	TIME	CONC	MK	AREA
0		0.54	2.374		1637
0		1.02	0.8456		583
0		1.39	95.8824		66123
0		3.17	0.8978		619
	TOTAL		99.9999		68962

(c)

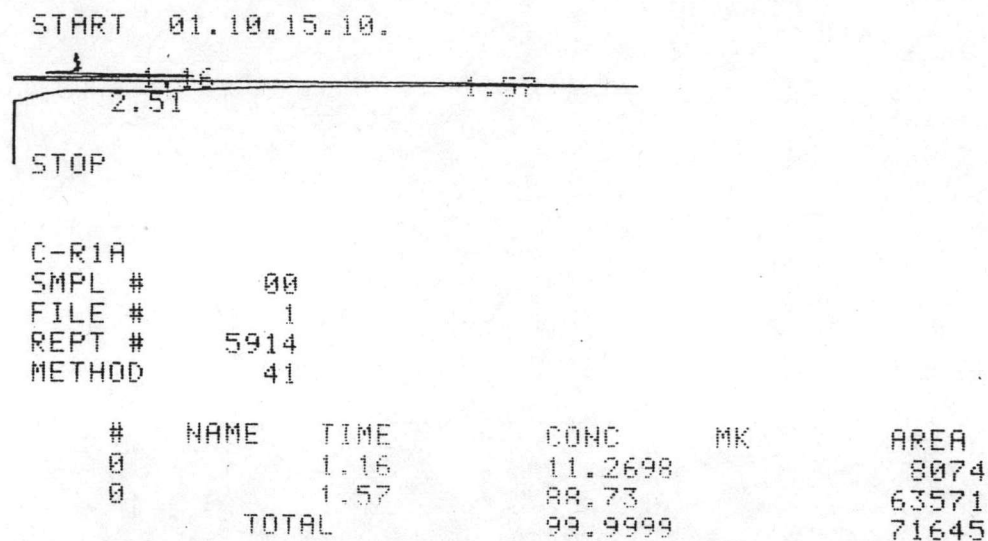


Figure 4.16 HPLC chromatogram of dye bath sampling.

(a) Before dyeing process.

(b) After dyeing process.

(c) Mixture of (a) and (b)

Mobile phase ACN/ water 55:45

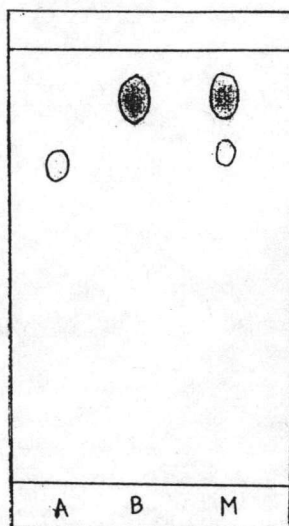
Column C<sub>18</sub> bonded phase

Flow rate 1 ml/min.

Detector UV absorption, 580 nm.

From the above chromatograms indicate that the peak at retention time around 1.08 min. was attributed to the non-hydrolysed dye molecules and the last peak at retention time 1.39 min. correspond to the hydrolysed dye molecules because they had more polarity and were absorbed strongly onto the surface of solid stationary phase. So, they will travel down the column slower than the non-hydrolysed dye molecules that were more weakly absorbed. Furthermore, the result from Figure 4.16 (C) can be seen that the mixture of dye bath sampling before and after dyeing process has two compounds. This can be concluded that hydrolysis reaction of dye was occurred.

For TLC technique, the  $R_f$ -value of hydrolysed dye was lower than that obtained from original dye since it was absorbed more strongly onto the surface of silica gel. The thin layer chromatogram as shown in Figure 4.17.



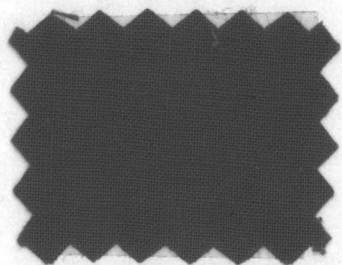
A = Dye sample from dye bath at the beginning of dyeing.

B = Dye sample from dye bath at the end of dyeing.

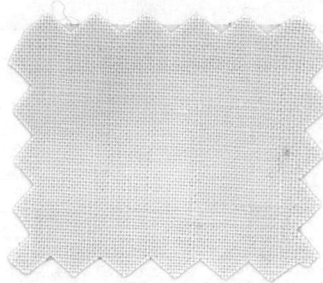
M = Mixture of A and B

Figure 4.17 TLC chromatogram of dye sample taken from the dye bath at the beginning and at the end of dyeing process.

In order to confirm that the dye sample after dyeing process was obviously hydrolysed, the dye solution after dyeing process was reused to dye a modified cotton fabric. The result of dyeing showed poor color yield which indicated that hydrolysed dye could not react with the reactive site on the fiber. It can be concluded that even though dyeing is carried out in the absence of alkali but some of dye molecules still undergo hydrolysis reaction due to high reaction temperature involved. The sample of dyed cotton as shown below (Figure 4.18):



The first dyeing  
of modified cotton fabric



The second dyeing  
of modified cotton fabric

Figure 4.18 Examples of dyed cotton from studying on stability of reactive dye.