

## The Wash-off of Reactive Dyes on Cellulosic Fibres. Part 3. Dichlorotriazinyl Dyes on Lyocell

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### ABSTRACT

*Lyocell fabric was dyed using six concentrations of three commercial dichlorotriazinyl reactive dyes using the dyeing method recommended by the dye maker. One surfactant-based and three sodium carbonate-based wash-off methods were used to remove unfixed dye from the dyeings and the fastness of the resulting dyed samples to the ISO C06/C2 wash test was determined. The results suggest that the use of the surfactant may be unnecessary in washing-off the dyeings and that an adequate level of both wash-off and wash fastness can be achieved using 2 g litre<sup>-1</sup> Na<sub>2</sub>CO<sub>3</sub>. The findings agree very closely with those previously secured using commercial monochloro-, dichloro- and bis-monochlorotriazinyl reactive dyes on cotton fabric. © 1997 Elsevier Science Ltd*

**Keywords:** cellulosic fibres, wash-off, reactive dyes, dichlorotriazinyl dyes.

### INTRODUCTION

It is well known that all commercial ranges of reactive dyes suffer the problem that during their exhaustive application to cellulosic fibres, the dyes undergo hydrolysis which severely reduces the efficiency of the dye-fibre reaction (fixation), resulting in dye wastage, the need to wash-off dyeings and major environmental problems. The first two parts of this paper<sup>1,2</sup> revealed that in the cases of commercial monochloro-, dichloro- and bis-monochlorotriazinyl reactive dyes on cotton fabric, wash-off using sodium carbonate as well as each of five surfactants was more effective in removing

unfixed dye than tap water alone, and that each of the surfactants when used in combination with sodium carbonate was more effective in removing dye than when used alone. From this work, it was proposed<sup>2</sup> that the use of surfactants in the wash-off of the three types of dye may be unnecessary and that an adequate level of wash-off and wash fastness could be achieved using 2 g litre<sup>-1</sup> aqueous sodium carbonate solution alone. Furthermore, it was suggested<sup>2</sup> that the avoidance of surfactants in wash-off and the alternative use of sodium carbonate may result in a dyeing process that was not only more cost-effective, but also more environmentally friendly.

Lyocell fibres, as exemplified by the first commercially available, solvent-spun, regenerated cellulosic fibre *Tencel* (Courtaulds), are claimed to offer environmental advantages over other regenerated fibres with regard to the recyclability of the solvent and the renewable source of cellulosic starting material.<sup>3</sup> Although preliminary results regarding the dyeability of lyocell fabric with reactive dyes have been published,<sup>3</sup> a detailed study has not appeared.

The aim of this work was to continue the study of the wash-off of various types of reactive dye on different cellulosic substrates by examining three commercial dichlorotriazinyl reactive dyes on woven lyocell fabric.

## EXPERIMENTAL

### Materials

#### *Fabric*

Scoured and bleached woven lyocell (123.5 g m<sup>-2</sup>) of 1.7 decitex was generously supplied by Courtaulds Research.

#### *Dyes*

Three reactive dyes, namely *Procion Blue MX-G* (CI Reactive Blue 163), *Procion Red MX-G* (CI Reactive Red 5) and *Procion Yellow MX-4G* (CI Reactive Yellow 2), were provided by Zeneca Colours and were not purified prior to use.

#### *Auxiliaries*

A commercial sample of *Lanapex R* (a mixture of modified phosphate esters with polyphosphates), which was used in the wash-off of the dyeings, was generously provided by ICI Surfactants.

All other chemicals were of Analar grade purity.

## Procedures

### Dyeing

All dyeings (0.1%, 0.5%, 1%, 2%, 4% and 6% omf) were carried out using lyocell fabric (4.0 g) in sealed stainless steel dyepots of 300 cm<sup>3</sup> capacity housed in a Zeltex Vistacolor laboratory scale dyeing machine using a liquor ratio of 20:1. The dyeing method used was the "Standard Method" recommended by Zeneca<sup>4</sup> and is shown in Fig. 1. At the end of dyeing, the dyed samples were rinsed in cold tap water at room temperature for 15 min and allowed to dry in the open air. Additions made to the various dyebaths are shown in Table 1.

### Wash-off

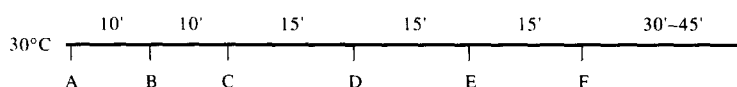
This process, to remove any unfixed dye from the dyed sample, was carried out by treating the dyeings with aqueous solutions of either 1 g litre<sup>-1</sup> *Lanapex R* or 1, 2 or 5 g litre<sup>-1</sup> Na<sub>2</sub>CO<sub>3</sub> at 98°C for 30 min using a liquor ratio of 50:1. At the end of wash-off, the dyed samples were rinsed in warm (50°C) water and then rinsed in cold water as recommended.<sup>5</sup>

### Wash fastness testing

The fastness of the dyeings was determined in accordance with the ISO C06/C2 method.<sup>4</sup>

### Colour measurement

The reflectance values of the dry, dyed unwashed-off and washed-off dyeings were measured using a Colorgen reflectance spectrophotometer interfaced to



**Fig. 1.** Standard dyeing method.<sup>4</sup> Having prepared the dyebath and adjusted the pH to below 7, the quantities specified in Table 1 were used as detailed: A, dye 0.1, 0.5, 1, 2, 4 or 6% omf; B, NaCl 2.5 g litre<sup>-1</sup>; C, NaCl 7.5 g litre<sup>-1</sup>; D, NaCl remaining amount; E Na<sub>2</sub>CO<sub>3</sub> 10% of the required amount; F, Na<sub>2</sub>CO<sub>3</sub> remaining amount.

**TABLE 1**  
NaCl and Na<sub>2</sub>CO<sub>3</sub> Requirements for Standard Dyeing Method<sup>2</sup>

Depth of shade (% omf)	NaCl (g litre <sup>-1</sup> )	Na <sub>2</sub> CO <sub>3</sub> (g litre <sup>-1</sup> )
up to 0.5	25	3
0.51 to 2.0	35	4
2.01 to 4.0	45	8
above 4.0	55	10

a personal computer, under illuminant D<sub>65</sub> using a 10° standard observer with specular component excluded and UV component included, from which the corresponding colour strength ( $K/S$  value) and CIE  $L^*$ ,  $a^*$ ,  $b^*$ ,  $C$  and  $h^\circ$  coordinates were calculated at the appropriate  $\lambda_{\max}$  of each dye. In addition, the difference in colour strength ( $\Delta K/S$ ) as well as the percentage difference in colour strength ( $\% \Delta K/S$ ) between dyeings that had not been washed-off and dyeings which had been washed-off were calculated as described previously.<sup>1</sup>

TABLE 2  
Colorimetric Data for CI Reactive Blue 163

Dye (% omf)	Wash-off	$\Delta K/S$	$\% \Delta K/S$	$L^*$	$a^*$	$b^*$	$C$	$h^\circ$
0.1	nil	—	—	71.58	-12.58	-16.12	20.45	232.04
	Lanapex R 1 g litre <sup>-1</sup>	0.17	17.5	73.22	-11.86	-15.99	20.45	233.45
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	0.31	31.9	74.43	-10.72	-12.54	19.91	229.47
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	0.38	39.1	76.01	-10.06	-10.68	16.50	226.74
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	0.43	44.3	76.56	-9.54	-8.87	14.67	222.91
0.5	nil	—	—	58.35	-13.74	-24.20	13.02	240.41
	Lanapex R 1 g litre <sup>-1</sup>	0.33	11.6	59.56	-13.25	-24.22	27.83	241.31
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	0.89	31.3	62.31	-12.83	-19.57	27.61	236.75
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	1.06	37.3	65.15	-12.11	-16.69	23.40	234.03
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	1.24	43.6	65.34	-11.93	-15.08	20.62	231.64
1.0	nil	—	—	51.62	-13.33	-27.07	19.23	243.78
	Lanapex R 1 g litre <sup>-1</sup>	0.77	15.3	53.72	-13.15	-26.81	30.18	243.87
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	1.71	34.0	55.13	-13.22	-22.64	29.86	239.72
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	1.83	36.4	58.35	-13.07	-20.04	26.22	236.88
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	2.49	49.6	59.66	-12.74	-16.94	23.92	233.12
2.0	nil	—	—	44.20	-12.08	-28.58	21.23	247.09
	Lanapex R 1 g litre <sup>-1</sup>	1.55	18.9	46.42	-12.11	-28.52	31.02	247.00
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	2.24	27.3	48.46	-12.75	-25.02	30.98	243.01
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	2.9	35.3	51.24	-13.09	-22.61	28.08	239.41
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	3.99	48.6	53.63	-13.07	-19.39	26.12	236.01
4.0	nil	—	—	40.05	-10.36	-29.60	23.38	250.72
	Lanapex R 1 g litre <sup>-1</sup>	1.52	12.9	41.87	-10.70	-29.91	31.36	250.31
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	2.50	21.3	43.81	-12.06	-26.52	29.13	245.54
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	3.40	29.0	44.51	-12.48	-25.38	28.28	243.80
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4.40	37.6	45.58	-12.86	-22.66	26.05	240.43
6.0	nil	—	—	38.82	-9.63	-29.89	31.41	252.15
	Lanapex R 1 g litre <sup>-1</sup>	1.46	11.5	41.04	-10.41	-30.42	32.15	251.10
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	1.26	9.9	40.33	-10.87	-27.40	29.48	248.36
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	2.92	23.0	41.49	-11.64	-25.36	27.41	245.34
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4.35	34.3	44.59	-12.79	-22.55	25.93	240.43

## RESULTS AND DISCUSSION

## CI Reactive Blue 163

Figure 2 shows the build-up profile achieved for the dye both before wash-off (i.e. after rinsing in cold water for 15 min) and after wash-off. It is evident that the colour strength ( $K/S$  values) of the washed-off dyeings was lower than that of the corresponding unwashed-off dyeings, indicating that unfixed reactive dye had been removed during wash-off. The  $\Delta K/S$  and  $\% \Delta K/S$  values displayed in Table 2 reveal that of the four wash-off methods used, that employing *Lanapex R* was less effective than the three  $\text{Na}_2\text{CO}_3$ -based methods in removing unfixed dye from the dyeings. It is also evident from Table 2 and Fig. 2 that the effectiveness of the sodium carbonate wash-off procedures increased with increasing concentration of alkali used. The colorimetric parameters of the rinsed and washed-off dyeings (Table 2) demonstrate that the four wash-off treatments caused a slight greening of shade and that this change in shade increased with increasing extent of dye removal.

Table 3 shows the effect of wash-off on the fastness of the dyeings to the ISO CO6/C2 wash test, from which it is clear that dyeings which had been washed-off using the surfactant generally exhibited slightly lower wash fastness than dyeings which had been washed-off using alkali; also, in the case of

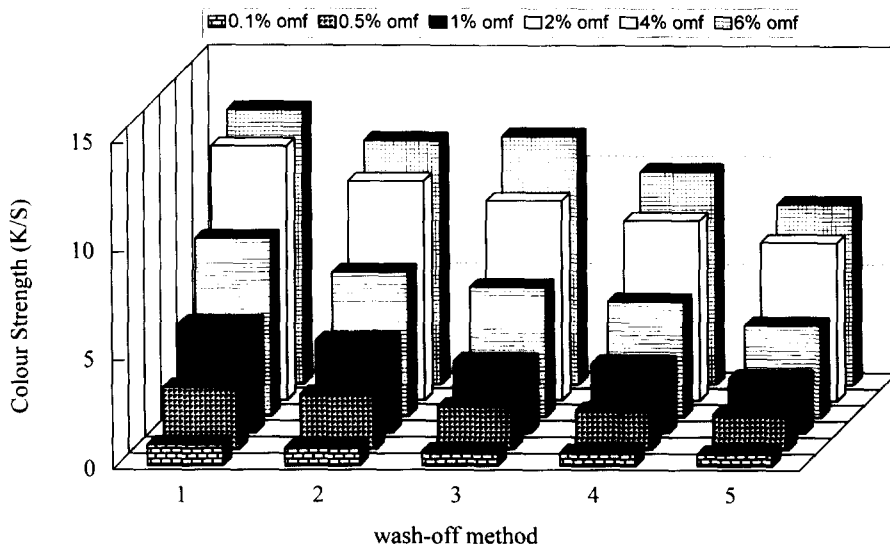


Fig. 2. 1, Cold water rinse, 15 min, room temperature; 2, *Lanapex R* 1 g litre<sup>-1</sup>, 98°C, 30 min; 3,  $\text{Na}_2\text{CO}_3$  1 g litre<sup>-1</sup>, 98°C, 30 min; 4,  $\text{Na}_2\text{CO}_3$  2 g litre<sup>-1</sup>, 98°C, 30 min; 5,  $\text{Na}_2\text{CO}_3$  5 g litre<sup>-1</sup>, 98°C, 30 min.

the sodium carbonate wash-off, wash fastness increased marginally as the concentration of alkali used increased from 1 to 5 g litre<sup>-1</sup>.

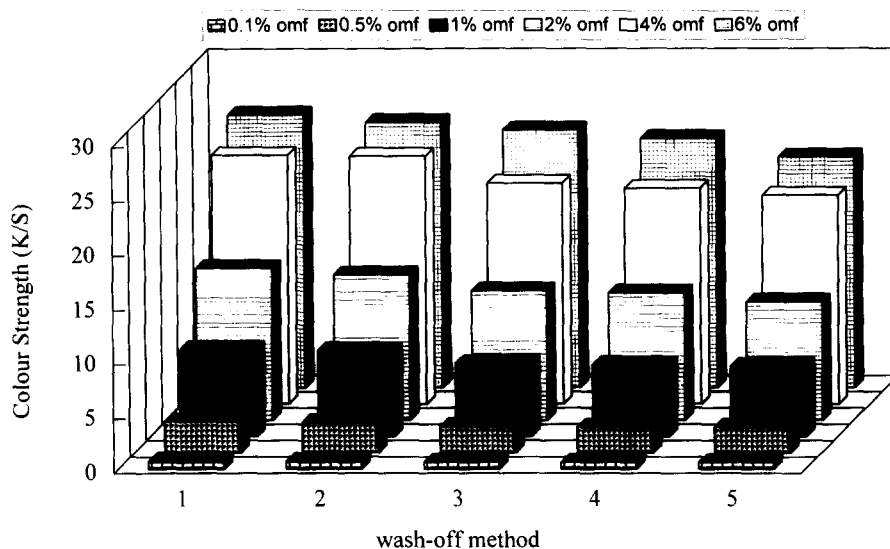
### CI Reactive Red 5 and CI Reactive Yellow 22

Figures 3 and 4 show the build-up profiles, respectively, for CI Reactive Red 5 and CI Reactive Yellow 22, both before and after wash-off. A comparison of these two build-up profiles with that obtained for CI Reactive Blue 163 (Fig. 2) reveals that whilst the colour strength of dyeings achieved with the blue and yellow dye were very similar, at each of the five depths of shade used, that of dyeings of the red dye was much higher.

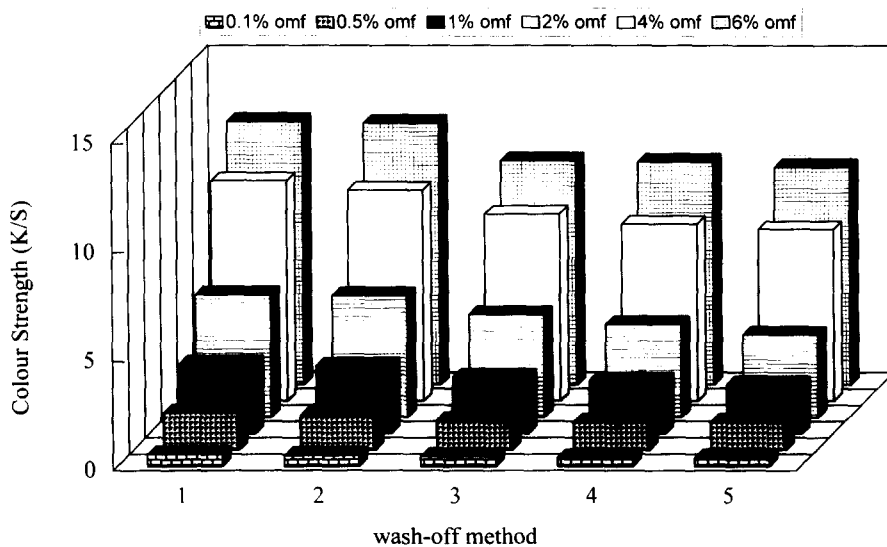
A further comparison of the results presented in Figs 2–4 clearly shows that the effect of wash-off on dyeings of the red and yellow dyes was very

**TABLE 3**  
Wash Fastness (ISO C06/C2) for Dyeings of CI Reactive Blue 163

Dye (% omf)	Wash-off	Change in shade	Staining	
			Lyocell	Cotton
0.1	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	3/4	4	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4/5
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	5	4/5	4/5
0.5	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	3	4	4/5
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4	4	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4	4	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4/5
1.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	3/4	4	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4/5
2.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	3	4	3/4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4
4.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	3/4	4	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4
6.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	3	4	3/4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	3/4	4	3/4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4	4	3/4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4	4/5	4



**Fig. 3.** 1, Cold water rinse, 15 min, room temperature; 2, *Lanapex R* 1 g litre<sup>-1</sup>, 98°C, 30 min; 3, Na<sub>2</sub>CO<sub>3</sub> 1 g litre<sup>-1</sup>, 98°C, 30 min; 4, Na<sub>2</sub>CO<sub>3</sub> 2 g litre<sup>-1</sup>, 98°C, 30 min; 5, Na<sub>2</sub>CO<sub>3</sub> 5 g litre<sup>-1</sup>, 98°C, 30 min.



**Fig. 4.** 1, Cold water rinse, 15 min, room temperature; 2, *Lanapex R* 1 g litre<sup>-1</sup>, 98°C, 30 min; 3, Na<sub>2</sub>CO<sub>3</sub> 1 g litre<sup>-1</sup>, 98°C, 30 min; 4, Na<sub>2</sub>CO<sub>3</sub> 2 g litre<sup>-1</sup>, 98°C, 30 min; 5, Na<sub>2</sub>CO<sub>3</sub> 5 g litre<sup>-1</sup>, 98°C, 30 min.

similar to that observed for the blue dye. The colour strength ( $K/S$  values) of the washed-off dyeings was lower than that of the corresponding unwashed-off dyeings, indicating that unfixed reactive dye had been removed during wash-off. The  $\Delta K/S$  and  $\% \Delta K/S$  values displayed in Tables 4 and 5 reveal that of the four wash-off methods used, that employing *Lanapex R* was less effective in removing unfixed dye from the dyeings than the three  $\text{Na}_2\text{CO}_3$ -based methods. In terms of the relative effectiveness of the three sodium carbonate wash-off methods, it is evident from Tables 4 and 5 and Figs 3 and 4 that, in general, the effectiveness increased with increasing concentration of

TABLE 4  
Colorimetric Data for CI Reactive Red 5

Dye (% omf)	Wash-off	$\Delta K/S$	$\% \Delta K/S$	$L^*$	$a^*$	$b^*$	$C$	$h^\circ$
0.1	nil	—	—	73.62	30.79	4.23		7.82
	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	0.04	5.12	74.28	31.31	3.95	31.08	7.19
	$\text{Na}_2\text{CO}_3$ 1 g litre <sup>-1</sup>	0.10	12.82	75.28	29.85	3.78	31.56	7.22
	$\text{Na}_2\text{CO}_3$ 2 g litre <sup>-1</sup>	0.15	19.2	74.64	30.56	3.71	30.09	6.93
	$\text{Na}_2\text{CO}_3$ 5 g litre <sup>-1</sup>	0.15	19.2	74.70	30.67	3.64	30.79	6.76
0.5	nil	—	—	62.33	45.59	8.80	30.08	10.93
	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	0.09	3.13	63.16	46.37	9.04	46.43	11.03
	$\text{Na}_2\text{CO}_3$ 1 g litre <sup>-1</sup>	0.49	17.07	64.70	43.47	8.15	47.24	10.62
	$\text{Na}_2\text{CO}_3$ 2 g litre <sup>-1</sup>	0.53	18.46	61.62	47.47	9.61	44.22	11.45
	$\text{Na}_2\text{CO}_3$ 5 g litre <sup>-1</sup>	0.53	18.46	62.09	46.65	9.05	48.43	10.98
1.0	nil	—	—	52.01	57.19	17.50	47.52	17.01
	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	0.15	1.86	52.68	58.15	18.07	59.81	17.26
	$\text{Na}_2\text{CO}_3$ 1 g litre <sup>-1</sup>	1.35	16.70	53.40	55.84	16.42	60.89	16.39
	$\text{Na}_2\text{CO}_3$ 2 g litre <sup>-1</sup>	1.47	18.20	53.72	55.21	15.36	58.20	15.55
	$\text{Na}_2\text{CO}_3$ 5 g litre <sup>-1</sup>	1.60	19.87	54.36	54.42	14.20	57.31	14.63
2.0	nil	—	—	47.81	58.87	21.97	56.24	20.46
	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	0.62	4.43	48.78	59.96	22.27	62.83	20.37
	$\text{Na}_2\text{CO}_3$ 1 g litre <sup>-1</sup>	2.09	14.93	47.12	59.00	21.91	62.74	20.30
	$\text{Na}_2\text{CO}_3$ 2 g litre <sup>-1</sup>	2.29	16.36	47.77	58.97	21.41	61.17	19.96
	$\text{Na}_2\text{CO}_3$ 5 g litre <sup>-1</sup>	3.09	22.08	49.97	57.86	19.85	63.98	18.94
4.0	nil	—	—	40.57	59.19	29.69	66.22	26.64
	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	0.14	0.61	41.15	60.69	30.80	68.06	26.91
	$\text{Na}_2\text{CO}_3$ 1 g litre <sup>-1</sup>	2.65	11.5	42.30	59.31	28.31	65.72	25.52
	$\text{Na}_2\text{CO}_3$ 2 g litre <sup>-1</sup>	3.07	13.4	41.82	59.41	28.05	65.70	25.27
	$\text{Na}_2\text{CO}_3$ 5 g litre <sup>-1</sup>	3.70	16.1	41.41	58.62	27.37	64.69	25.03
6.0	nil	—	—	37.78	57.22	30.61	64.89	28.15
	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	0.68	2.71	38.79	58.93	31.14	66.65	27.85
	$\text{Na}_2\text{CO}_3$ 1 g litre <sup>-1</sup>	1.40	5.58	39.67	58.08	29.15	64.98	26.65
	$\text{Na}_2\text{CO}_3$ 2 g litre <sup>-1</sup>	2.18	8.69	39.24	58.65	29.98	65.87	27.08
	$\text{Na}_2\text{CO}_3$ 5 g litre <sup>-1</sup>	3.90	15.5	38.79	58.14	29.47	65.19	26.89



alkali used, although in the cases of 0.1% and 0.5% omf dyeings there was little difference in proficiency between the three concentrations of sodium carbonate employed. The colorimetric parameters displayed in Tables 4 and 5 show that wash-off changed the shade of the dyeings, these changes being bluer in the case of CI Reactive Red 5 and redder in the case of CI Reactive Yellow 22; also, the extent of these shade changes increased with increasing extent of dye removal.

Tables 6 and 7 show the effect of wash-off on the fastness of the dyeings of CI Reactive Red 5 and CI Reactive Yellow 22, respectively, to the ISO CO6/

**TABLE 5**  
Colorimetric Data for CI Reactive Yellow 22

Dye (% omf)	Wash-off	$\Delta K/S$	% $\Delta K/S$	$L^*$	$a^*$	$b^*$	$C$	$h^\circ$
0.1	nil	—	—	89.58	-3.80	29.58	29.82	97.32
	Lanapex R 1 g litre <sup>-1</sup>	0.04	7.27	90.16	-3.82	29.73	29.97	97.33
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	0.09	16.36	89.51	-2.64	27.39	27.53	95.61
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	0.15	27.27	90.43	-2.84	25.52	25.68	96.35
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	0.15	27.27	90.82	-3.31	25.89	26.10	97.29
0.5	nil	—	—	87.58	-3.65	49.27	49.40	94.24
	Lanapex R 1 g litre <sup>-1</sup>	0.11	6.54	88.26	-3.41	49.78	49.90	93.92
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	0.33	19.64	86.96	-1.07	44.68	44.69	91.37
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	0.42	26.7	88.20	-2.55	46.07	46.15	93.17
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	0.42	26.7	88.58	-2.92	46.37	46.46	93.61
1.0	nil	—	—	86.63	-2.33	62.24	62.29	92.14
	Lanapex R 1 g litre <sup>-1</sup>	0.10	3.13	87.01	-2.20	63.42	63.45	91.99
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	0.60	18.8	86.30	-1.12	59.24	59.25	91.08
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	0.77	24.1	86.20	-0.58	56.87	56.88	90.58
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	0.93	29.1	86.93	-1.73	57.77	57.79	91.71
2.0	nil	—	—	84.95	0.57	74.89	74.89	89.57
	Lanapex R 1 g litre <sup>-1</sup>	0.04	0.70	85.33	0.75	76.96	76.97	89.44
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	0.91	16.1	84.59	1.70	71.22	71.24	88.63
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	1.36	24.11	84.17	1.80	66.40	66.42	88.45
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	1.84	32.62	79.08	6.41	57.14	57.49	83.60
4.0	nil	—	—	83.01	5.24	86.94	87.10	86.55
	Lanapex R 1 g litre <sup>-1</sup>	0.43	4.24	83.64	4.97	88.54	88.67	86.79
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	1.53	15.10	82.68	5.55	83.47	83.66	86.20
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	1.99	19.64	82.23	5.61	80.01	80.21	85.99
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	2.23	22.0	82.72	5.48	81.41	81.60	86.15
6.0	nil	—	—	81.61	8.27	91.87	92.24	84.86
	Lanapex R 1 g litre <sup>-1</sup>	0.07	0.57	82.49	8.02	93.33	93.67	85.09
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	1.79	15.30	81.63	8.36	88.26	88.66	84.59
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	1.85	14.80	81.04	8.18	86.23	86.61	84.58
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	2.09	17.28	81.31	8.22	86.25	86.64	84.56

C2 wash test. Furthermore, dyeings which had been washed-off using the surfactant generally exhibited slightly lower wash fastness than dyeings which had been washed-off using alkali. In the case of the sodium carbonate wash-off, wash fastness was generally unaffected by an increase in concentration of alkali from 1 to 5 g litre<sup>-1</sup>. This latter finding differs from that obtained for dyeings of CI Reactive Blue 163, for which it was observed that wash fastness increased marginally with increasing concentration of sodium carbonate.

For each of the three dyes and at each of the five concentrations employed, none of the dyeings exhibited excellent wash fastness (i.e. ratings of 5 for shade change and staining of adjacent materials), which, in turn, indicates that none of the four wash-off methods used was fully effective in removing

TABLE 6  
Wash Fastness (ISO C06/C2) of CI Reactive Red 5

Dye (% omf)	Wash-off	Change in shade	Staining	
			Lyocell	Cotton
0.1	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	4	4	3/4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4
0.5	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	5	4/5	4/5
1.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	4	4	3/4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4
2.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	3/4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4
4.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	3/4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4	4
6.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	3/4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4

**TABLE 7**  
Wash Fastness (ISO C06/C2) of CI Reactive Yellow 22

Dye (% omf)	Wash-off	Change in shade	Staining	
			Lyocell	Cotton
0.1	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4/5
0.5	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4
1.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4
2.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4
4.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4
6.0	<i>Lanapex R</i> 1 g litre <sup>-1</sup>	4	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 1 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 2 g litre <sup>-1</sup>	4/5	4/5	4
	Na <sub>2</sub> CO <sub>3</sub> 5 g litre <sup>-1</sup>	4/5	4/5	4

unfixed reactive dye from the dyeings. This finding concurs with those previously recounted for commercial monochloro-, dichloro- and bis-monochlorotriazinyl reactive dyes on cotton fabric.<sup>1,2</sup>

## CONCLUSIONS

The results showed that of the four wash-off methods used, that employing *Lanapex R* was less effective than the three Na<sub>2</sub>CO<sub>3</sub>-based methods in removing unfixed dye from the dyeings. It was also evident that, overall, the effectiveness of the sodium carbonate wash-off procedures in removing unfixed dye increased with increasing concentration of alkali used. Clearly, sodium carbonate was very efficient at removing unfixed dye from the lyocell

substrate; this is in agreement with the findings made earlier for such dichlorotriazinyl dyes<sup>1</sup> and also monochloro- and bis-monochlorotriazinyl dyes on cotton.<sup>2</sup> An argument has previously been forwarded<sup>1,2</sup> to account for the proficiency of the  $\text{Na}_2\text{CO}_3$ -based wash-off method and it is considered that the same argument applies in the case of the lyocell fibre used in the present work.

The colorimetric parameters of the rinsed and washed-off dyeings demonstrated that the four wash-off treatments caused a slight change of shade, the extent of which increased with increasing extent of dye removal.

In the context of the effect of wash-off on the fastness of the dyeings to the ISO CO6/C2 wash test, it was apparent that dyeings which had been rinsed only in tap water displayed lower fastness than samples which had been washed-off using *Lanapex R* or each of the three concentrations of sodium carbonate. Dyeings which had been washed-off using the surfactant generally exhibited slightly lower wash fastness than dyeings which had been washed-off using alkali. In the case of the sodium carbonate wash-off, the results showed that in the cases of the yellow and red dyes wash fastness was not dependent on the concentration of alkali used, whereas for the blue dye concentrations of 2 and 5 g litre<sup>-1</sup> yielded higher wash fastness results than 1 g litre<sup>-1</sup> alkali.

It thus seems reasonable to conclude that the use of surfactant may be unnecessary for the wash-off of dichlorotriazinyl reactive dyes from lyocell fabric and, instead, that a wash-off comprising 2 g litre<sup>-1</sup> sodium carbonate at 98°C for 30 min is sufficient to achieve adequate levels of both wash-off and wash fastness.

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