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## The use of reflectance measurements in the determination of diffusion of reactive dyes into cellulosic fiber

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### ABSTRACT

Reactive dye fixation and color yield of a dyed cellulosic fiber significantly depend on the dye diffusion into the fiber polymer system. In case of pad dyeing processes, dye diffusion exerts a more significant influence on dye fixation and hence color yield. This paper proposes a new method for determining the extent of diffusion of reactive dyes into the fiber in pad dyeings using Kubelka-Munk equation. The  $K/S$  values are used as in an equation,  $\%D$  (extent of dye diffusion) =  $100 - [(K/S_{diffusion\ index}) / (K/S_{reference}) \times 100]$ . The paper introduces and explains how to determine the new  $K/S$  variables used in this equation. The new method is simple, non-destructive, relatively faster and applicable to industrial dyehouses, and was validated by a microscopic analysis of dyed fiber cross-section carried out in this work and to the dye manufacturer's recommendations for dyebath-ingredient concentrations.

**KEY WORDS** reflectance; Kubelka-Munk; dye diffusion; reactive dyes; pad dyeing

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### INTRODUCTION

Pad dyeing of cellulosic fabric with reactive dyes is a two stage process. First the dye is applied to the fabric by padding. The fabric is then subjected to a dye fixation step<sup>1</sup>. The dye commences to diffuse into the interior of the fiber when the fabric is padded. The diffusion continues till the subsequent

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3 fixation step. The extent of the diffusion depends on the electrolyte concentration (steam fixation) or  
4 the urea concentration (bake fixation) and swelling of the fiber during fixation step <sup>2</sup>.

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7 The selection of dyes and color matching are the most critical processes in cotton dyehouses. Color  
8 yield, the extent of dye fixation and colorfastness are the key results in these processes. However, the  
9 important goals are the shade reproducibility and right-first-time production <sup>3-5</sup>, thus producing the  
10 required quality with minimum costs including the costs involved in production time and rehandling.  
11 Dye diffusion exerts a significant influence on dye fixation, ultimate color yield and colorfastness of a  
12 dyed fiber <sup>6-9</sup>. Therefore, dye diffusion should be considered for dye selection and color matching as  
13 one of the primary parameters.  
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16  
17 To date, industrial dyehouses have not considered the measurement of dye diffusion as an everyday  
18 tool because the currently available methods are either time consuming, indirect, or involving the use  
19 of sophisticated laboratory procedures or complex mathematics <sup>10,11</sup>. The currently available methods  
20 for determining dye diffusion are: examination of fiber or yarn cross-sections <sup>10</sup>, multilayer of a clear  
21 polymer thin film diffusion methods <sup>12-14</sup>, microdensitometer method <sup>15</sup>, rate of dyeing method <sup>10</sup>, and  
22 laser scanning confocal microscopy (LSCM) method <sup>16-18</sup>. This paper presents an easy, direct and  
23 industrially applicable method for determining the dye diffusion. The diffusion measurement can be  
24 performed during the regular spectrophotometric measurements for dye selection and color matching  
25 processes.  
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30 Reflectance spectrophotometers are now a basic feature in the laboratories of most industrial  
31 dyehouses. There they are usually used for color matching and quality control. Fundamentally, such  
32 spectrophotometers provide the means to measure the reflectance of any surface across the visible  
33 spectrum. The surface reflectance value ( $R$ ) can be further used for determining other primary color  
34 measurement quantities such as tristimulus values ( $XYZ$ ),  $CIE L^*a^*b^*$  values, total color difference  
35 values and color strength value ( $K/S$ ) <sup>19</sup>.

36  
37  
38 The color strength of a dyed fabric is customarily expressed as its  $K/S$  value. ' $K$ ' and ' $S$ ' are  
39 absorption and scattering coefficients of the dyed fiber. The  $K/S$  value is calculated from ' $R$ ' using the  
40 Kubelka-Munk Equation 1 <sup>20-22</sup>.

$$41 \quad K/S = (1 - R)^2 / 2R \quad (1)$$

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3 'R' is preferably determined at the maximum absorption wavelength of the dyed fabric ( $\lambda_{max}$ )<sup>23</sup>. The  
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5  $K/S$  value has been shown to be a direct measure of the color yield<sup>24</sup>. The value can also be used to  
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7 determine other important dyeing factors such as dye uptake, migration index (MI) and dye fixation  
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9 <sup>4,23,25-27</sup>. A relation between  $K/S$  value and the cross-sectional distribution of dye molecules in the dyed  
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11 cellulosic fibers and yarns has been reported<sup>28</sup>. Based on this fact, studies were carried out for dye  
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13 diffusion in pad dyeings which resulted in a new method of measuring reactive dye diffusion into  
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15 cellulosic fiber using  $K/S$  values. The hypothesis for the new method is presented in following sub-  
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17 section. There are three basic methods for fixation of the dye after padding cellulosic fabric with  
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19 reactive dyes. These are steaming (saturated steam, 30–120 sec), baking (150–160°C, 30–90 sec) and  
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21 batching (ambient temperature, 6–36 hrs). The hypothesis was examined for these three fixation  
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23 methods. The specific dyeing processes selected for the study were pad-steam, pad-dry-bake and pad-  
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25 batch.

### 26 27 **Hypothesis**

28  
29 Immediately after padding a cellulosic fabric with a reactive dye, the dye is almost exclusively  
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31 concentrated on the surface of the fibers and yarns within the fabric. As dye diffusion progresses, the  
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33 number of dye molecules on the surface of the fiber becomes less and less. Consequently, the color  
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35 strength ( $K/S$ ) value, being a measure of surface reflectance, commences to decrease. In other words  
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37 there is an inverse relationship between the color strength and diffusion. As the dyeing process  
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39 continues, the diffusion of the dye will eventually approach an optimum level and the dye will be  
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41 distributed throughout the fiber. It was hypothesised that a measure of diffusion could be obtained  
42  
43 through using  $K/S$  values obtained at various stages of the dyeing process. These measurements would  
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45 need to be made before any washing-off because this removes unfixed dye from the surface and  
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47 interior of the fiber. The base value for the measure of diffusion should ideally represent the color  
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49 strength of the dyed fabric where effectively no dye diffusion has occurred. For this purpose, if the  
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51 fabric is padded with dye bath containing the dye only, the diffusion will be at minimum and there will  
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53 be the maximum number of dye molecules on the surface of the fiber. The color strength value  
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55 measured at this point was taken as the base or reference value. That value is represented by  
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57 ' $K/S_{reference}$ '. The color strength value of a dyed fabric where certain diffusion has occurred because of  
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3 either fixation step or using the auxiliaries (such an electrolyte, urea, alkali, antimigrant...etc) is  
4 represented by ' $K/S_{diffusion\ index}$ '. This value is obtained sometime later in the dyeing process after  
5 padding the fabric in a dyebath with or without auxiliaries before washing-off. Having known the  
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$$\%D = 100 - [(K/S_{diffusion\ index}) / (K/S_{reference}) \times 100] \quad (2)$$

Where ' $(K/S_{diffusion\ index}) / (K/S_{reference}) \times 100$ ' represents the percentage of the dye remaining on the surface of the fiber after a certain amount of dye diffusion has occurred. Ideally,  $K/S_{diffusion\ index}$  will be equal to  $K/S_{reference}$ , when there is no dye diffusion.

## EXPERIMENTAL

### Materials

A commercially bleached, un-mercerized and fluorescent brightener-free cotton cellulose woven fabric (282 g/m<sup>2</sup>) with an absorbency of 1 sec (AATCC 79 – 1995) and a CIE whiteness index of 78.6 was used. Commercial reactive dyes, Clariant's Drimarene Red K4BL (CI Reactive Red 147, difluorochloropyrimidine reactive group), Dystar's Remazol Navy RGB 150% (CI Reactive Blue 250, sulphatoethylsulphone reactive group) and Kisco's Synozol Blue SHF-BRN 150% (CI Reactive Blue 221, bifunctional reactive group: aminochlorotriazene + sulphatoethylsulphone) were used. A non-ionic emulsifying detergent (Felosan RGN-S, CHT) was used for washing-off of unfixed reactive dyes and Alginate NVS (CHT) was used as an anti-migrant for pad-dry-bake dyeing. All other chemicals were general laboratory grade.

### Methods

#### *Pad dyeings*

Fabric samples were dyed by padding (two dip-two nip, 70% liquor pick-up, Benz laboratory padder) with 20 g/l dye and dye manufacturers recommended padding chemicals (see Table I). For pad steam dyeing, the padded fabric samples were steamed (wet-temperature of 101 – 102°C, 100% moisture, Mathis laboratory steamer) for 60 sec. For pad-dry-bake dyeing, the samples were dried (70°C, suspended in a Brownbuilt automatic oven dryer) followed by baking (160°C for CI Reactive Red

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3 147, 150°C for CI Reactive Blue 250 and CI Reactive Blue 221, Mathis laboratory stenter) for 60 sec.  
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5 For pad-batch dyeing, the samples were carefully rolled, wrapped with plastic and left to batch  
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7 (ambient temperature,  $25 \pm 2^\circ\text{C}$ ) for 12 hrs. Dyeings were also carried out to study the effect of  
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9 variation in the magnitudes of sodium chloride concentration (0–60 g/l, pad steam dyeing), steaming  
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11 and baking time (0–120 sec), batching time (0–24 hrs) and dye concentration (0–20 g/l in pad-steam  
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13 dyeing, 0–30 g/l in pad-batch dyeing).

#### 14 ***Washing-off and DMF stripping***

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16 Dyed fabrics were rinsed with cold then hot tap water, soaped with 2 g/l Felosan RGN-S at the boil  
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18 for 15 min, and then rinsed with hot water until the bleeding stopped. The fabrics were finally rinsed  
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20 with cold water and dried. The dye fixation was measured after further boiling the fabrics with a 50%  
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22 aqueous solution of N,N-dimethyl formamide, DMF, (liquor-to-fiber ratio of 40:1, 15 min, reflux  
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24 condensation).

#### 25 **Measurements**

##### 26 ***Color strength (K/S) value***

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28 *K/S* value was obtained at the maximum absorption peak (Datacolor 600 spectrophotometer settings:  
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30 30 mm sample aperture, illuminant D65, UV included, specular component included, reflectance  
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32 mode and 1964 (10°) CIE Supplementary Standard Observer). Each fabric sample was conditioned  
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34 (30 min,  $\sim 25^\circ\text{C}$ ,  $\sim 65\%$  relative humidity) before measuring the *K/S* value.

##### 35 ***Color yield and dye fixation***

36  
37 The color strength after DMF treatment ( $K/S_{DMF}$ ) was obtained as the final color yield. The approach  
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39 for determining the extent of reactive dye fixation using *K/S* values, used by other researchers<sup>29-32</sup>,  
40  
41 was followed. The percentage of reactive dye fixed on the cotton fabric ( $\%F$ ) was measured as the per  
42  
43 cent ratio of the final color yield to the color strength of dyed sample before washing-off.

##### 44 ***Cross-section of dyed fiber***

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46 The sections of dyed yarn bundles were prepared on thin steel plate having 1mm holes. This was done  
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48 by pulling the bundle of yarns through the hole, using a strong nylon yarn, so that the yarns were  
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50 tightly packed. The cross-sections were then revealed by slicing them off on the metal surface. The  
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52 microscopic observations were made (500 times magnification, Leica DM 2500M Microscope).  
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## Procedures for the proposed method of measuring diffusion

### *Determining $K/S_{reference}$*

The fabric sample was padded with a known concentration of the dye (two dip-two nip, 70% liquor pick-up, Benz laboratory padder). The preparation of the dye solution and setting up the liquor pick-up were precisely done. The sample was then dried (15 min, 70°C, suspended in a Brownbuilt automatic oven dryer). Drying, in the absence of an electrolyte, urea or antimigrant, results in maximum number of dye molecules on the surface of the fabric because the dye solution migrates to the surface of the fabric as reported by Hunger<sup>1</sup>. The color strength ( $K/S_{reference}$ ) was then measured. It required only several hours to prepare the database reference color strength values. Those values can be used for dye diffusion measurements repeatedly for the same dye and fabric.

### *Determining $K/S_{diffusion\ index}$*

Having determined the ' $K/S_{reference}$ ', an undyed sample of the same fabric was padded with the dyebath containing the same concentration of the same dye together with known concentrations of the relevant auxiliaries in the same precised way. The color strength ( $K/S_{diffusion\ index}$ ) was then measured before and after the subsequent fixation steps (steaming, baking or batching).

## RESULTS AND DISCUSSION

### **Effect of electrolyte concentration on dye diffusion (pad-steam dyeing)**

In pad-steam dyeing, an electrolyte favours increased dye levelness in the fiber and the eventual color yield<sup>33-35</sup>. This is because the electrolyte promotes dye diffusion, even distribution of dye molecules throughout the fiber and ultimately dye fixation<sup>6</sup>. Therefore, pad-steam dyeing results were obtained to determine the effect of electrolyte concentration on dye diffusion.

### *Microscopic analysis*

Fiber cross-sections of the fabrics dyed with CI Reactive Red 147 showed that the core is more colored with increasing electrolyte concentration (Figure 1). This confirms that the electrolyte promotes dye diffusion in pad-steam reactive dyeing of cotton<sup>6,9,34,36-38</sup>.

### ***New method for the extent of dye diffusion (%D)***

Figure 2 shows the comparative effect of electrolyte concentration on color strength of dyed sample before washing-off ( $K/S_{diffusion\ index}$ ) and the proposed measure of dye diffusion (%D) using equation (1). The figure shows an inverse relation between  $K/S_{diffusion\ index}$  and %D. Figure 2 (b) shows that the dye diffusion increases with increasing sodium chloride concentration, at constant alkali concentration (15 g/l) and steaming time (60 sec). Maximum dye diffusion was obtained at 50 g/l sodium chloride. It is well known that the addition of an electrolyte in a dyebath increases the degree of aggregation of the dye molecules<sup>39</sup>. Therefore, an optimum electrolyte concentration generally exists. Electrolyte concentrations higher than the optimum are avoided to minimise excessive dye aggregation. The CI Reactive Blue 250 diffused less than the CI Reactive Red 147 and the CI Reactive Blue 221. The dye-fiber reaction and dye diffusion occur simultaneously in pad-steam dyeing. Therefore, CI Reactive Blue 250 may have higher relative rate of dye-fiber reaction to the rate of dye diffusion.

### **Effect of electrolyte concentration on dye fixation and color yield**

Figure 3 shows that the dye fixation and color yield increase with increasing sodium chloride concentration, at constant alkali concentration (15 g/l) and steaming time (60 sec). The maximum values were obtained at 50 g/l sodium chloride, after which a near-constant color yield was observed with CI Reactive Red 147. The optimum dye diffusion was also obtained at 50 g/l sodium chloride; therefore, it can be postulated that the dye fixation and color yield increase with the increase in dye diffusion. Further, the optimum dye fixation and color yield can be obtained at optimum dye diffusion. Thus, the optimum electrolyte concentration can be determined by obtaining optimum dye diffusion instead of obtaining dye fixation and color yield. This saves the experimental time as washing-off procedures are not required for determining dye diffusion.

### **Relation of dye diffusion to the dye fixation and color yield (pad-steam dyeing)**

The relationship between the extent of dye diffusion and dye fixation (and hence color yield) can be obtained through results presented in Figures 2 (b) and 3. The increase in dye diffusion is known to favour dye fixation and color yield in pad dyeings<sup>6</sup>. Figure 4 shows that the proposed measure of diffusion is directly related to dye fixation and color yield. This further confirms the validity of the new method for determining the extent of dye diffusion.



### Rate of dye diffusion during steaming, baking and batching

Figure 5 shows the extent of dye diffusion achieved at an industrially possible range of steaming times for CI Reactive Red 147, CI Reactive Blue 250 and CI Reactive Blue 221. For the CI Reactive Blue 250, the dye diffusion did not increase significantly with increasing steaming duration. Over 95% of its diffusion occurred within the first 30 seconds. The diffusion slightly increased up to a maximum at 60 seconds and slightly decreased at longer steaming time. This implies that the dye has higher dye diffusion initially when the extent of dye-fiber reaction is low. For the CI Reactive Red 147 and the CI Reactive Blue 221, a pronounced maximum value for %D was observed at 60 seconds of steaming time. This shows that the rate of diffusion of these dyes, relative to CI Reactive Blue 250, was expedited more by steaming. The diffusion maxima may be due to excessive dye hydrolysis with increasing steaming time and the hydrolysed dye is relatively more mobile within the fiber. However, the reasons for the maxima are not fully understood which may have industrial implications. The effect of dye molecular weight and the type and number of reactive groups may be worthy of further study.

For the pad-dry-bake method, Figure 6 shows that the patterns of behaviour of dye diffusion with baking time are similar to those obtained with steaming time. The absolute values of diffusion of the three dyes at any time were less with baking than with steaming. This implies the presence of water molecules in the cotton fiber at elevated temperatures has a marked effect on the rate and extent of diffusion. For the CI Reactive Blue 250, dye diffusion continued to increase marginally with increasing baking time. The maximum value for this dye was not determined because the time exceeds the industrial practice. Whereas the diffusion of CI Reactive Red 147 and CI Reactive Blue 221 increased up to 90 seconds of baking time and then decreased.

Figure 7 demonstrates the applicability of the proposed method for determining diffusion to the pad-batch dyeing method. There, the extent of diffusion of CI Reactive Red 147 and CI Reactive Blue 221 dyes increase then decrease with increasing batching time. The maximum value of diffusion was obtained at 12 hrs batching. For the CI Reactive Blue 250, diffusion continued to increase with increasing batching time. The maximum value for CI Reactive Blue 250 is of the order of 36 hrs of batching. As per the dye manufacturers' recommendations for optimum dyeing results, the batching

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3 time for CI Reactive Red 147 and CI Reactive Blue 221 ranges from 4 to 14 hrs and that for CI  
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5 Reactive Blue 250 is up to 36 hrs. The figure confirms that the dyeing results significantly depend on  
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7 the extent of dye diffusion and conforms to the dye manufacturers' recommendations.

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9 It is clear from Figures 5, 6 and 7 that the dye diffusion is promoted by steaming, baking and  
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11 batching. The CI Reactive Blue 250 diffuses more slowly than the other two dyes. Figure 8 shows that  
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13 the optimum dye diffusion obtained in pad-steam and pad-batch dyeings was similar and considerably  
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15 higher than that obtained in pad-dry-bake dyeing. This reflects that the penetration of dye molecules is  
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17 supported by the presence of water molecules which are not present in pad-dry-bake process.

### 18 19 **Effect of dye concentration on diffusion**

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21 The effect of dye concentration on diffusion was determined for the pad-steam and pad-batch  
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23 processes as these processes showed a significant extent of dye diffusion. In kinetic studies, dye  
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25 concentration dependence is an important factor because an increase in the number of dye molecules  
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27 results in increasing the competition for dye sites on the fiber. Most of such studies have been of  
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29 theoretical nature where diffusion coefficients are demonstrated for exhaust dyeings<sup>6,13,15,40,41</sup>. In  
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31 exhaust dyeing of cotton with anionic dyes, dye exhaustion gradually decreases with an increase in  
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33 the concentration of dyes applied. This is due to the gradual build-up of the dye at the fiber surface,  
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35 hence retarding the further adsorption of remaining dye from the dyebath<sup>42</sup>. The effect of dye  
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37 concentration on the extent of dye diffusion as determined by the proposed equation for pad dyeings  
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39 is shown in Figure 9. The results show that the dye diffusion decreases with increasing dye  
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41 concentration in both pad-steam and pad-batch dyeings. In order to obtain optimum dyeing results,  
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43 increased amounts of electrolyte may be used for increased dye concentrations, and that is also  
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45 recommended by dye manufacturers.  
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### 49 **CONCLUSION**

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51 The new, simple, non-destructive and relatively faster, method for determining the extent of dye  
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53 diffusion by using  $K/S$  values have been developed and validated for pad dyeings. The results  
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55 obtained by new method conform to a microscopic analysis of dyed fiber cross-section carried out in  
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57 this work and to the dye manufacturer's recommendations for dyebath-ingredient concentrations. This  
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3 can effectively be used in industrial dyehouses for precise determination of the dye efficiency and  
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5 thus improving dyeing results and assisting with cost reductions.  
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11  
12 technical advice and suggestions.  
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TABLE I Padding Chemicals and Concentrations

Dye	Chemicals and their concentrations		
	Pad-steam dyeing	Pad-dry-bake dyeing	Pad-batch dyeing
CI Reactive Red 147	Sodium chloride (30 g/l)	Urea (100 g/l)	<sup>38</sup> Be Sodium hydroxide (8 ml/l)
	Sodium bicarbonate (15 g/l)	Sodium bicarbonate (15 g/l)	Sodium carbonate (10 g/l)
CI Reactive Blue 250	Sodium chloride (30 g/l)	Alginate NVS (1 g/l)	
	Sodium carbonate (15 g/l)	Urea (50 g/l)	<sup>38</sup> Be Sodium hydroxide (14 ml/l)
		Sodium carbonate (15 g/l)	Sodium silicate (70 g/l)
CI Reactive Blue 221	Sodium chloride (30 g/l)	Alginate NVS (1 g/l)	
	Sodium carbonate (15 g/l)	Urea (50 g/l)	<sup>38</sup> Be Sodium hydroxide (12 ml/l)
		Sodium carbonate (15 g/l)	Sodium chloride (30 g/l)
		Alginate NVS (1 g/l)	

For Peer Review

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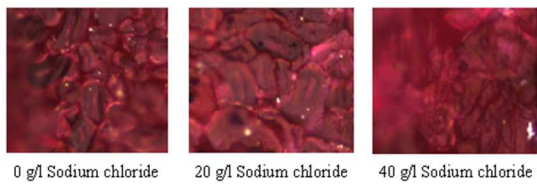


FIGURE 1 Fibre cross-sections of cotton fabrics dyed with 20 g/l CI Reactive Red 147 (before washing-off) at 60 sec steaming.

190x275mm (96 x 96 DPI)



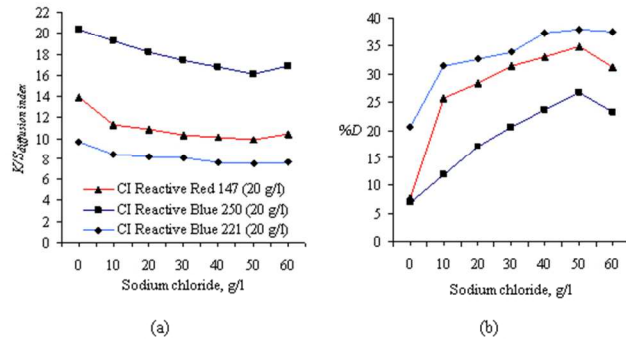


FIGURE 2 Effect of electrolyte concentration on dye diffusion

190x275mm (96 x 96 DPI)

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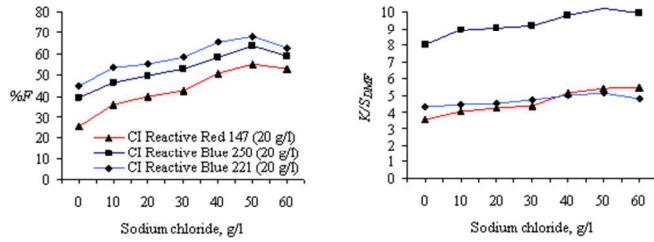


FIGURE 3 Effect of electrolyte concentration on dye fixation and colour yield

190x275mm (96 x 96 DPI)

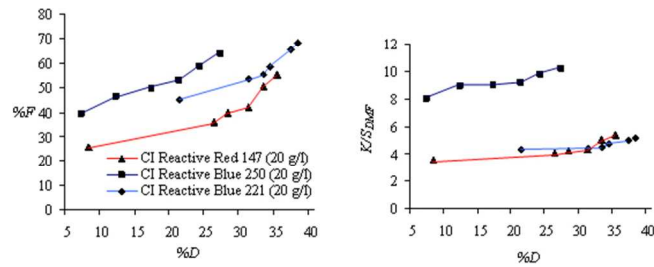


FIGURE 4 Effect of dye diffusion on dye fixation and colour yield

190x275mm (96 x 96 DPI)

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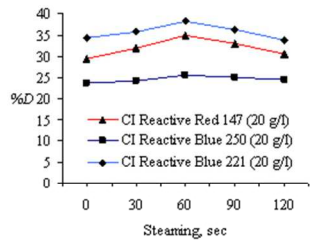


FIGURE 5 Rate and extent of dye diffusion during steaming

190x275mm (96 x 96 DPI)

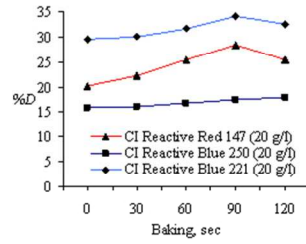


FIGURE 6 Rate and extent of dye diffusion during baking

190x275mm (96 x 96 DPI)

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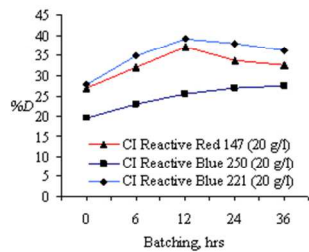


FIGURE 7 Rate and extent of dye diffusion during batching

190x275mm (96 x 96 DPI)

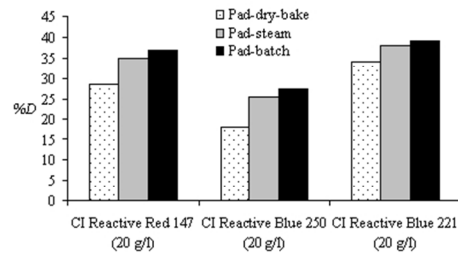


FIGURE 8 Optimum dye diffusion obtained with steaming, baking and batching

190x275mm (96 x 96 DPI)

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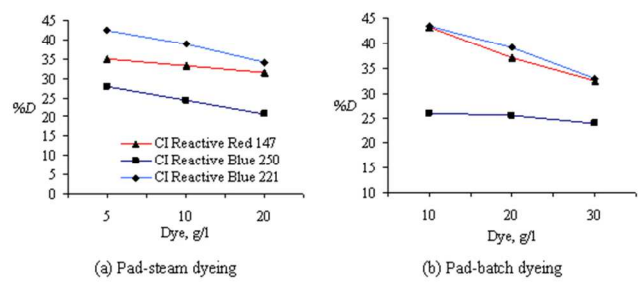


FIGURE 9 Effect of dye concentration on dye diffusion at constant electrolyte (50 g/l), alkali (15 g/l) and steaming time (60 sec)

190x275mm (96 x 96 DPI)