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The inkjet printing process for Lyocell and cotton fibres. Part 1: The significance of pre-treatment chemicals and their relationship with colour strength, absorbed dye fixation and ink penetration

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ABSTRACT

A statistical study of the outcome of inkjet printing, using a reactive dye based ink, on three cellulosic fabrics, namley Lyocell (standard *Tencel* and *Tencel A100*) and cotton, pre-treated with urea, migration inhibitor, penetration agent and alkali is reported. Colour strength (as Integ value), absorbed dye fixation and ink penetration were analysed as main responses. A full factorial design method was applied to study the effect of the level of each ingredient in the pre-treatment formulation on the responses, together with the steaming time used for dye fixation. The interactions between variables were also evaluated. The statistically significant variables were determined for each response and contour plots constructed for the most significant interactions to assess where the optimum responses might be achieved.

1. Introduction

In recent years, digital inkjet printing applications on textiles have attracted increasing interest at both academic and industrial levels. The development of inkjet printing processes for textiles has benefited from the extensive research carried out on inkjet printing on paper, which has become a well-established technology. For textiles, however, there remain significant challenges [1,2], including issues associated with productivity, print quality, and process integration [3], which are being addressed by hardware development, colour management systems, ink formulation and fabric pre-treatment [4]. Fabric pre-treatment is essential for textile printing with reactive dyes to ensure efficient inkjet print performance, for example to achieve acceptable colour strength and fastness properties, and to control droplet penetration and spread for optimum image quality, because the auxiliary chemicals required, such as urea, alkali and migration inhibitor, cannot normally be incorporated into the inks [5]. Commercial inkjet reactive inks are usually based on dyes with low-to-moderate fixation properties (generally monofunctional reactive dyes), so it is important to maximize dye fixation for technical, economic and environmental reasons. Many printed textile applications involve controlled ink penetration to maximize colour strength, although not for applications such as flags and banner printing where double-sided viewing is involved. Ink penetration has been investigated for inkjet printing on paper [6,7], but there is limited information on this characteristic in relation to textile inkjet printing.

Lyocell was introduced in 1994 as a sustainable, biodegradable regenerated cellulosic fibre, manufactured by an environmentally-friendly process and commercialized under the trade name *Tencel* [8]. Standard *Tencel* offers certain advantages over other regenerated cellulosic fibres, such as high wet tenacity, enhanced water imbibition and improved wearer comfort [9]. *Tencel A100* is a variant that exhibits reduced fibrillation compared with standard *Tencel*, achieved by a crosslinking process. *Tencel A100* shows lower tenacity and modulus than standard *Tencel* and has a more open structure, providing higher water imbibition and enhanced dyeability [8,10]. It has also been reported that a higher colour strength is achievable in the reactive dyeing of *Tencel* compared with cotton using the same amount of dye [11]. While literature reports of the dyeing behaviour of Lyocell fibres have appeared frequently, there are no previous studies of inkjet printing on these fibres. In this paper, the inkjet printing performance on *Tencel* fibres (standard *Tencel* and *Tencel A100*) using a reactive dye based ink is reported, and some comparisons made with cotton. Using a full factorial design approach, the relationships between the selected variables, the concentrations of pre-treatment chemicals and steaming time, and the responses, colour strength, dye fixation and

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ink penetration, have been established to provide recommendations for optimum fabric pre- and post-treatment conditions.

2. Experimental

2.1. Materials

The Lyocell fabrics used were desized and scoured standard *Tencel* (3/1 twill weave, yarn count: warp 48/1, weft 46/1) and *Tencel A100* (plain weave, yarn count: warp 92/1, weft 93/1), supplied by Lenzing Fibres, Austria. Mercerized cotton fabric (plain weave, yarn count: warp 52/1, weft 48/1) was obtained from Downey-Caric Ltd, Macclesfield, UK,. The pre-treatment chemicals *Thermacol MP*, a polyacrylic acid-based migration inhibitor, *Alcoprint AIR*, a non-ionic de-aerating penetration agent and *Lyoprint RG*, an anionic reduction inhibitor for reactive inks, and the reactive inkjet ink *Novacron Red MI-500* (which contains a monochlorotriazinyl reactive dye) were supplied by Huntsman, Basle, Switzerland. Urea and sodium bicarbonate were supplied by Aldrich, UK. A non-ionic detergent, *Synperonic BD 100*, (Univar, UK), was used in the wash-off process.

2.2. Equipment and instrumentation

A Roaches roller padder was used to apply the pre-treatment solutions on to the fabrics which were then dried in a Star oven. Inkjet printing was carried out using an Epson Stylus Colour 3000 DOD inkjet printer. A Roaches steamer operating at atmospheric pressure was used for fixation. Absorbance values of the wash liquors were measured, at the λ_{max} for the ink, using a Perkin–Elmer Lambda 2 UV/Visible spectrophotometer. Reflectance measurements of the printed samples were obtained using a Datacolor SF600 spectrophotometer, using the small aperture (9 mm), for illuminant D65, with the specular and UV components included. For measurement, the fabric samples were folded four times and an average of three readings per sample taken.

2.3. Design of experiment (DOE)

To conduct a comprehensive study of the factors and responses involved, a balanced full factorial experimental design was applied. The five process variables selected for study were the concentrations of urea, alkali, migration inhibitor, and penetration agent in the pre-treatment formulation, in addition to steaming time. Each of these variables was set at two levels, as shown in Table 1. This experimental design gave 2^5 (32) experiments, which were carried out in the random order given in Table 2. The responses considered in the analysis were colour strength, absorbed dye fixation and ink penetration. All statistical analyses were performed using the statistical software package Minitab 15.1.

2.4. Pre-treatment and printing procedure

Pre-treatment was carried out using the formulations given in Table 2. Additionally, a reduction inhibitor *Lyoprint RG* was incorporated into the formulations, at a concentration of 15 g l^{-1} in each case. The liquors were applied to the fabrics cut to A4 size, by padding with 75–80% pick up. The fabrics were dried for 5 min at 120 °C and conditioned for 24 h at 20 °C and 65% relative humidity. Inkjet printing was carried out with a single pass at a resolution of 1440 dpi as a solid square print pattern to facilitate colour measurement. Printed samples were allowed to air dry for 5 min and then steamed at 102 °C for the appropriate times (Table 2). The wash-off to remove unreacted and hydrolyzed dye, using de-ionised water, followed an established literature procedure [12], and involved an initial cold wash (1 l) for 7 min, a second cold wash (1 l) for 5 min, a boil wash at 95–100 °C for 10 min in an aqueous solution of non-ionic detergent (*Synperonic BD 100*, 1 g l^{-1}), a warm (*ca* 40 °C) rinse and finally a cold rinse, The samples were then dried for assessment.

Table 1
Control factors in the design of experiment.

Code	Control factor	Experimental level 1	Experimental level 2
A	Urea	100 g l^{-1}	200 g l^{-1}
B	Alkali	20 g l^{-1}	40 g l^{-1}
C	Migration inhibitor	100 g l^{-1}	200 g l^{-1}
D	Penetration agent	3 g l^{-1}	10 g l^{-1}
E	Steaming time	5 min	10 min

2.5. Measurement of colour strength

Colour strength was assessed as the Integ value [13] which provides a measure of the visual depth and is given by the formula:

$$I = \sum E_\lambda \left(\frac{K}{S}\right)_\lambda (\overline{x_\lambda} + \overline{y_\lambda} + \overline{z_\lambda}) \quad (1)$$

where E_λ is the spectral energy distribution of the light source (Illuminant D65),

$(\frac{K}{S})_\lambda$ is the Kubelka-Munk function of reflectance.

$\overline{x_\lambda}$, $\overline{y_\lambda}$, $\overline{z_\lambda}$ are the CIE standard observer colour matching functions.

2.6. Determination of absorbed dye fixation

Determination of % absorbed dye fixation from the inkjet process used a methodology adapted from previously established procedures for textile dyeing with reactive dyes [14,15]. For

Table 2
Experimental design with five factors, varied at two levels.

Run	Urea (g l^{-1})	Alkali (g l^{-1})	Migration inhibitor (g $^{-1}$)	Penetration agent (g l^{-1})	Steaming time (min)
1	100	20	200	3	5
2	100	40	100	3	5
3	100	20	200	10	10
4	100	20	100	10	5
5	100	40	100	10	5
6	100	40	200	10	5
7	200	40	200	10	5
8	100	40	100	10	10
9	200	20	200	3	5
10	200	40	100	3	10
11	100	20	100	10	10
12	200	20	100	10	10
13	100	20	200	3	10
14	200	40	100	10	10
15	200	40	100	3	5
16	200	40	100	10	5
17	100	40	200	3	5
18	200	20	200	10	5
19	200	40	200	3	5
20	200	20	200	10	10
21	100	40	200	10	10
22	200	20	100	10	5
23	100	20	100	3	5
24	100	20	100	3	10
25	200	40	200	10	10
26	100	40	100	3	10
27	200	40	200	3	10
28	100	40	200	3	10
29	200	20	100	3	10
30	100	20	200	10	5
31	200	20	100	3	5
32	200	20	200	3	10

calibration purposes, the square pattern was printed with the reactive ink on 100% polyester fabric. This printed fabric was washed with de-ionised water (1 l). A comparison of the reflectance measurements of the original fabric with printed and washed fabric, confirmed that no dye remained on the fabric, so that the wash liquors collected represented 100% wash-off. This solution was diluted to concentrations of 80%, 60%, 40% and 20% and the validity of the Beer–Lambert law established by a linear correlation between absorbance and dye concentration. The linear calibration was used to establish the relative concentrations of dye washed-off from the printed fabrics in the combined first three wash liquors, from which the % absorbed dye fixation was determined. Confirmation that all unfixed dye was removed from the fabrics by the washing sequence was provided by soxhlet extraction of representative samples using 20% aqueous pyridine [16].

2.7. Calculation of ink penetration

The penetration of ink through the fabrics was expressed as the penetration factor (PF) determined by calculating the ratio of the Integ value obtained from the back of the print to the corresponding value from the face of the print, in each case after subtracting the Integ value given by the background (equation (2)).

$$\mathrm{PF} = \frac{I_b - I_g}{I_f - I_g} \tag{2}$$

where I_b: Integ value of the back of the print,
I_f: Integ value of the face of the print,
I_g: Integ value of the background

3. Results and discussion

The fabric pre-treatments were formulated using urea (moisture-retaining agent), sodium bicarbonate (alkali), a synthetic polyacrylic acid-based migration inhibitor, a non-ionic penetration agent and an anionic reduction inhibitor, on the basis of manufacturer's recommendations. The fabrics were printed using a commercial inkjet ink containing a red monochlorotriazinyl reactive dye. Statistical analysis, as detailed in Section 2.3, was carried out to identify those control factors, or variables, which were statistically significant and to study the effect on the responses of varying their levels in the pre-treatment. Response variation was calculated according to equation (3) [17].

$$\text{Variation} = \frac{\text{response}_{\max} - \text{response}_{\min}}{\text{response}_{\max}} \times 100 \tag{3}$$

The responses obtained from the analysis are given in Table 3. The mean colour strength of the printed fabrics (as Integ values) was found to decrease in the order – standard *Tencel* > cotton > *Tencel A100*, while the mean absorbed dye fixation values decreased in the order – *Tencel A100* > standard *Tencel* > cotton. The observation of higher dye fixation on Lyocell fibres compared with cotton is consistent with similar observations reported for reactive dyeing [18]. The crosslinked structure of Tencel A100, used to control fibrillation, enhances fixation compared with standard Tencel, a feature which is consistent with observations during reactive dyeing. However, lower colour strength is developed with inkjet printing on *Tencel A100,* contrasting with dyeing performance [8,10]. It is conceivable that its crosslinked structure limits the development of deep shades at the fibre surface by its effect on dye sorption properties, although further investigation would be required to investigate this feature mechanistically. There was significantly less colour strength variation on *Tencel A100* than on standard *Tencel* and cotton. The variation in dye fixation was similar for the three fibres. A study of the variation of the penetration factor (PF) with the pre-treatment applied and steaming time for each individual fabric is relevant in providing an understanding of substrate effects on the dye sorption properties. However, PF values cannot meaningfully be compared between the three fabrics, especially in terms of the mean values, since they were not identical in terms of yarn count and weave structure, factors which may have influenced ink penetration. The higher mean PF given by *Tencel A100* compared with standard *Tencel* and cotton is mainly due to the fact that the *Tencel A100* is a lighter fabric.

Table 3
Mean response and response variation for standard *Tencel*, *Tencel A*100 and cotton.

	Response					
	Colour strength		Absorbed dye fixation		PF	
	Mean	Response variation (%)	Mean	Response variation (%)	Mean	Response variation (%)
Standard *Tencel*	13.33	53.38	53.7	36.09	0.085	54.47
Tencel A100	6.66	16.64	60.34	37.60	0.44	57.14
Cotton	11.43	49.86	46.47	38.26	0.14	80.66

3.1. The statistical significance of process variables

Factorial analysis was conducted to identify the statistically significant factors that affect the three responses (colour strength, absorbed dye fixation, penetration factor), to assess the main effect of the variables and the 2-way and 3-way interactions between variables. The statistically significant variables were identified as illustrated in the Pareto charts given in Fig. 1. A Pareto chart is a useful graphical tool for illustrating the magnitude and significance of an effect. Each chart displays, in decreasing order, the absolute value of the standardized effect of the variables (x-axis) together with a reference line on the chart which corresponds to the level of significance, $\alpha = 0.05$. An effect that extends past this reference line is statistically significant. For simplicity, only the statistically significant 3-way interactions are included in the charts.

3.1.1. Effect of variables on colour strength

The dominant factors affecting colour strength developed on all three fabric types were urea concentration (A) and steaming time (E), although the latter was less significant on *Tencel A100*. On cotton, there were no other significant factors. On the *Tencel* fabrics some interactions were significant, notably that between alkali and migration inhibitor (BC) on standard *Tencel* and between migration inhibitor and penetration agent (CD) on *Tencel A100*. Both *Tencel* fabrics showed a significant 3-way interaction corresponding to urea, alkali and penetration agent (ABD).

3.1.2. Effect of variables on absorbed dye fixation

Steaming time (E) is a highly significant factor, influencing dye fixation on all three fabric types. The urea level (A) was highly significant on *Tencel A100* and cotton but was not a statistically significant factor on standard *Tencel*. The only significant interactions involved alkali level and steaming time (BE) on standard *Tencel*, migration inhibitor and steaming time (CE) on standard *Tencel* and cotton and urea and steaming time on Tencel A100. Although alkali is essential for fixing reactive dyes on cellulosic fibres, perhaps unexpectedly, no significant individual effect of alkali concentration (B) on fixation was detected on any of the fibres. However, this may well be because its effect on fixation depends on its significant interactions with other pre-treatment ingredients.

3.1.3. Effect of variables on penetration factor (PF)

The study of penetration factor was of particular relevance in the context of its anticipated influence on the colour strength developed on a specific fabric since increased ink penetration might be expected to reduce visual depth of shade [6]. Urea level (A) showed a dominating effect on *Tencel A100* (the only significant factor in this case) and on cotton, but no significant effect on standard *Tencel*. Migration inhibitor level (C) was significant on standard *Tencel* and cotton, but not on *Tencel A100*. The PF was influenced by the significant interaction between urea and migration inhibitor (AC) on both cotton and standard *Tencel*. Many more interactions between variables were observed on cotton than on the *Tencel* fabrics.

3.2. Contour plots of responses

Contour plots, selected in such a way as to demonstrate the main effects of individual variables, together with those representing the most significant variable combinations, were constructed. These plots (Figs. 2–4) show the variations in colour strength, absorbed dye fixation and penetration factor as a result of selecting different values of two variables while the values for the other variables are held constant (hold values 100, 20, 150 and 3 g l^{-1} for the levels of urea, alkali, migration inhibitor and penetration agent respectively, and 10 min steaming time). Analysis of these plots may be used to identify an optimized solution for a given response on a specific fabric. In the discussion that follows, the codes A – E represent the variables as given in Table 1 and, for example, 'plot BC' refers to the plot of alkali concentration vs. migration inhibitor concentration.

3.2.1. Standard Tencel

The trends exhibited by the selected key interactions between variables for standard *Tencel* are illustrated in Fig. 2. Inspection of colour strength plots DE, CE and BC demonstrates that higher colour strength may be achieved by lowering the concentrations of penetration agent and alkali, using a higher migration inhibitor concentration of 200 g l^{-1}, and a steaming time of 10 min. The plot between urea and penetration agent (AD) is consistent with the high statistical significance of urea and demonstrates that a higher urea concentration enhances colour strength. Plot BC, representing the significant interaction between alkali and migration inhibitor, reveals that a concentration of 20 g l^{-1} alkali is the optimum for producing deep shades.

In terms of absorbed dye fixation, plot AD indicates that urea has a positive effect, although not statistically significant. Urea is a humectant, forming a eutectic mixture with water. It swells the fibre during steaming and increases dye solubility, factors which might be expected to facilitate dye–fibre covalent bond formation and colour strength development. Indeed, this is the case with standard *Tencel*, although contrasting with the effects on the other two fabrics (see subsequent sections). Inspection of fixation plots BE, CE and DE all indicate that, to maximize fixation, a longer steaming time is beneficial, especially at higher alkali concentration. As illustrated in the Pareto chart (Fig. 1), the combination BE

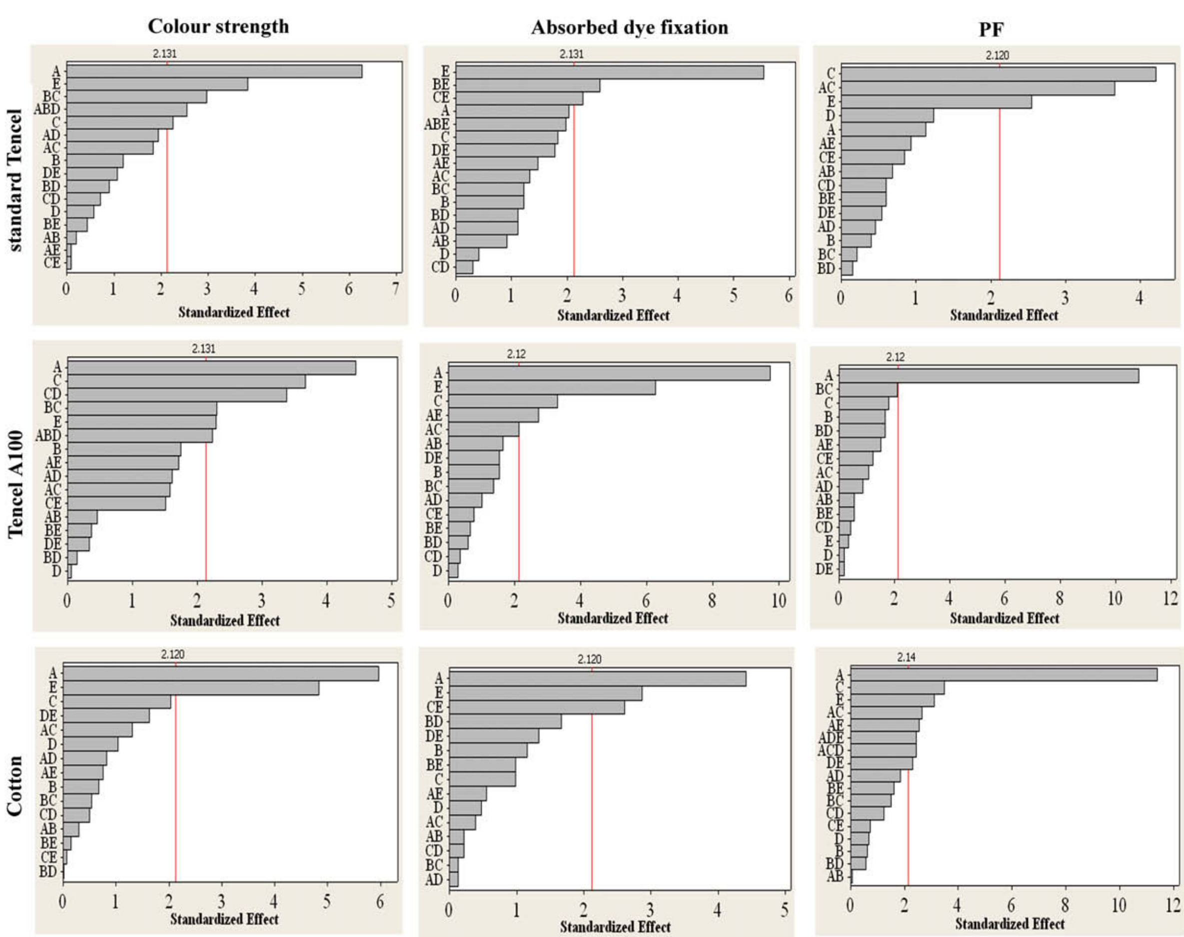

Fig. 1. Pareto charts illustrating the standardized effect for the variables and their significance on colour strength, absorbed dye fixation and penetration factor. *A: Urea, B: Alkali, C: Migration inhibitor, D: Penetration agent, E: Steaming time.*

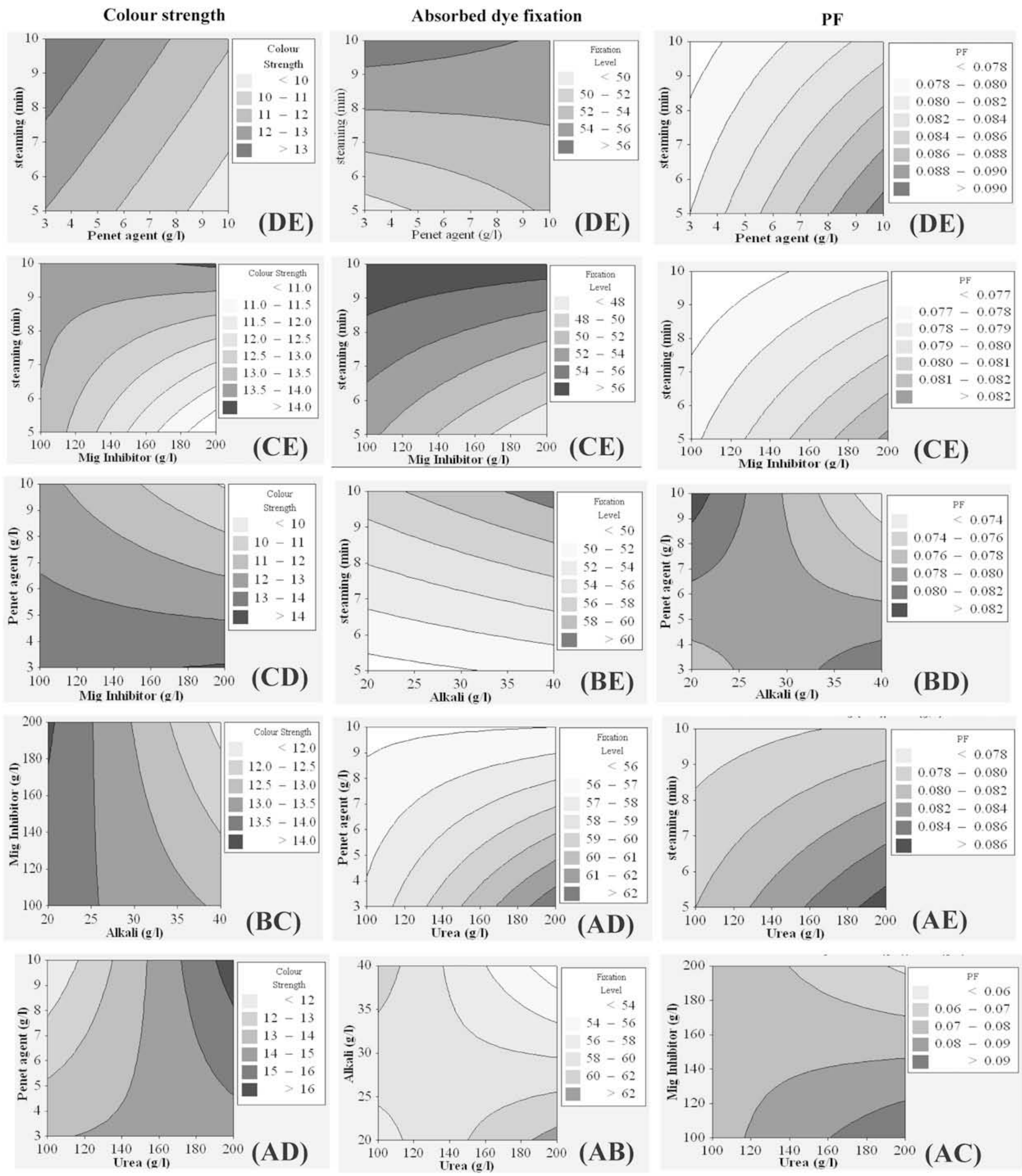

Fig. 2. Contour plots for the responses – colour strength, absorbed dye fixation and penetration factor (PF) measured on inkjet printed standard *Tencel*.

indicated a significant interaction in terms of fixation. While a low alkali level enhances colour strength, a high level is required for fixation, indicating the difficulty in simultaneously optimizing both responses in respect of this variable on standard *Tencel*. The interaction between alkali and urea (plot AB) reveals that a high level of urea is recommended if alkali is to be used at the lower level, and *vice versa*.

PF plots show that ink penetration increases with increasing penetration agent concentration and using a shorter steaming time (plot DE). Higher penetration also arises from increased urea concentration, especially when a shorter steaming time and less migration inhibitor are used (plots AE, CE and AC). An explanation which may be proposed is that with shorter steaming time, the dye does not adequately fix on the upper layer of the substrate and consequently the ink continues to be absorbed, facilitated by the low level of migration inhibitor. Consequently, lower colour strength results due to the excessive penetration.

3.2.2. *Tencel* A100

The selected contour plots for inkjet printing on to *Tencel* A100 are given in Fig. 3. The colour strength plots show that, to maximize colour strength, a high urea concentration and a longer steaming time is desirable (plot AE), and the concentration of penetration agent should be at the low value (plot CD). Steaming

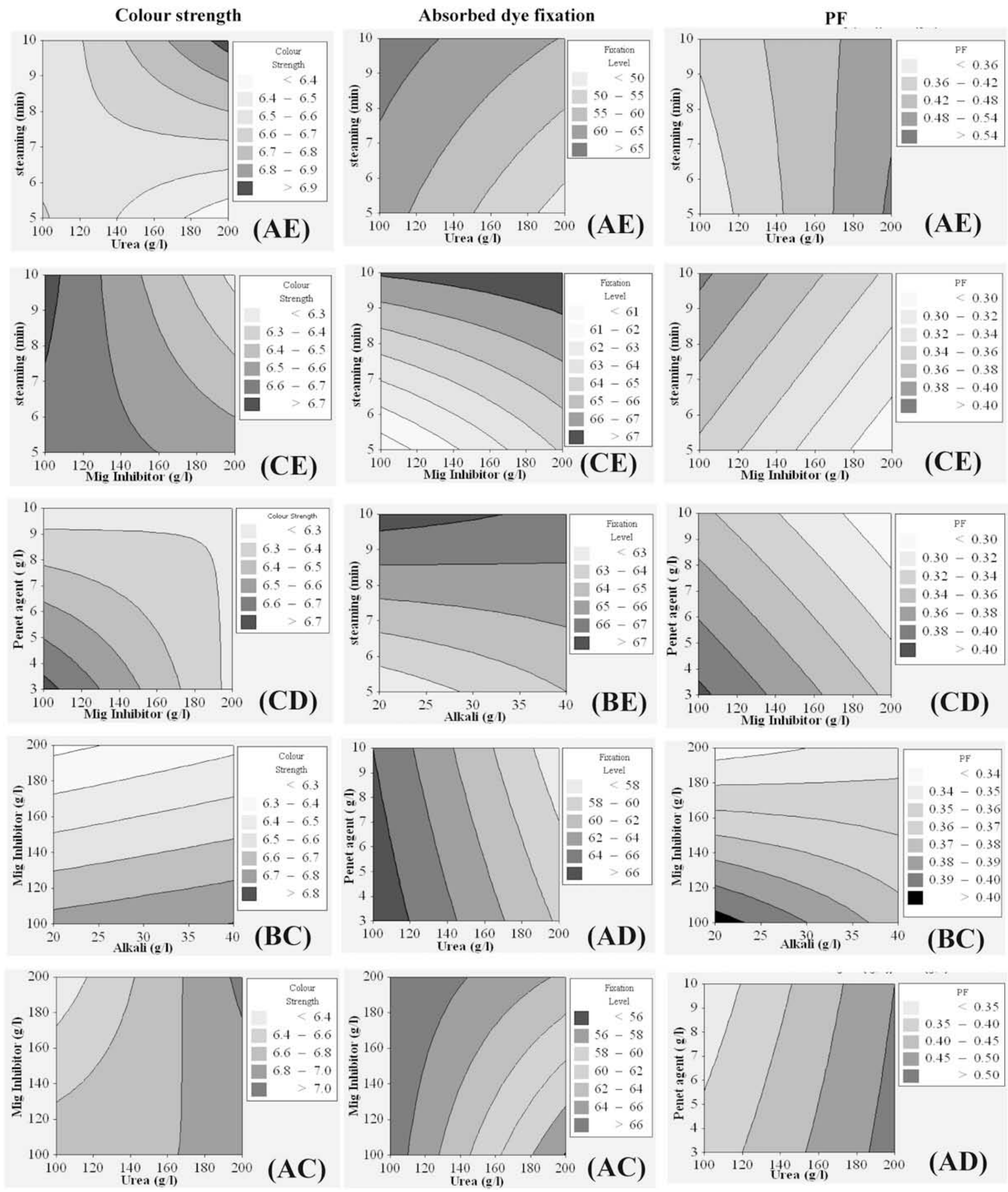

Fig. 3. Contour plots for the responses – colour strength, absorbed dye fixation and penetration factor (PF) measured on inkjet printed *Tencel A100*.

provides the high temperature and moisture required to swell the coating film, ease dye diffusion and ensure dye fixation. Previous studies have demonstrated the importance of steaming conditions on the colour strength developed on inkjet printed fabrics [19,20]. The recommendation to employ a penetration agent in the pre-treatment recipe for inkjet printing is probably inherited from its use in the continuous dyeing process to promote fabric wetting [21]. Some chemical manufacturers claim that it also acts as an antifoaming agent, and ensures a uniform substrate coating. In contrast to standard *Tencel,* as illustrated in plots CE and CD, a low level of migration inhibitor is recommended for enhanced colour strength by inkjet printing on *Tencel A100*. However, from plot AC, there is a region of high colour strength at a high level of migration inhibitor when the urea level is high. In this case, the statistically significant interactions are CD and BC and these plots are thus most relevant for determining an optimized response. The effect of alkali concentration on colour strength is of little significance on *Tencel A100*, contrasting with standard *Tencel* on which a decrease in colour strength was observed with the higher level of alkali.

The absorbed dye fixation contour plots for *Tencel A100* did not consistently show the same trends as those for colour strength. This is especially the case for urea, which reduces dye fixation at the higher level (plot AE) indicating a difficult optimization

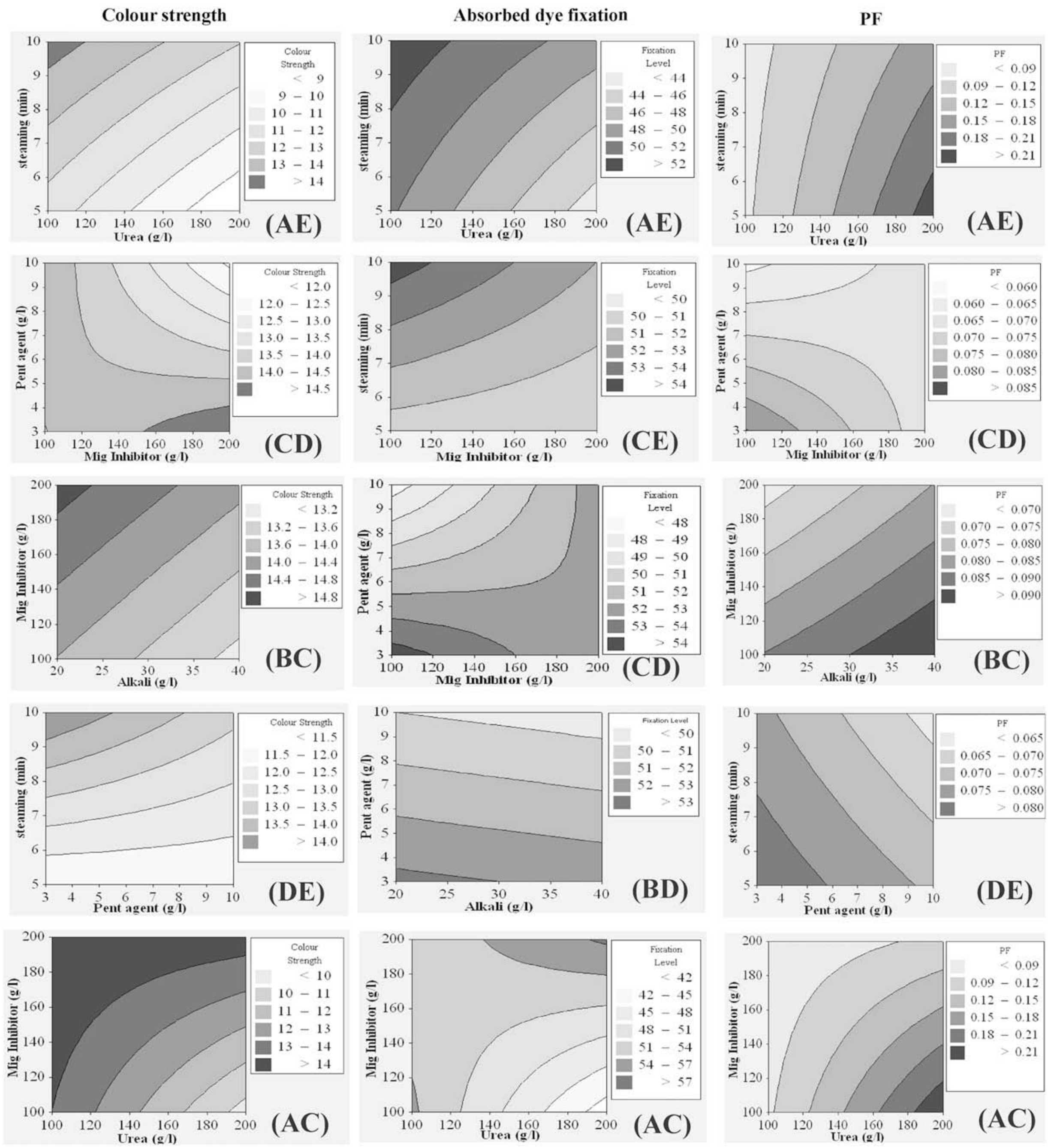

Fig. 4. Contour plots for the responses – colour strength, absorbed dye fixation and penetration factor (PF) measured on inkjet printed cotton.

compromise involving colour strength and fixation. With standard *Tencel*, there was also a difficult compromise required for optimization, although it involved alkali concentration. The contrast between Tencel A100 and standard *Tencel* in terms of the effect of urea is difficult to explain in detail, but is likely to be associated with the effect of crosslinking, both at the surface and in the bulk of the fibre, and the consequent effect of the urea on the swelling properties of the fibre. A possible explanation for a negative influence of urea on fixation is its effect on moisture retention during steaming which favours dye hydrolysis, thus reducing fixation. Negative effects of urea concentration on the fixation of Procion reactive dyes in screen printing of cotton have been reported [22]. The significant effect of migration inhibitor on fixation on *Tencel A100* (Fig. 1), which is positive as demonstrated by plot CE (Fig. 3), may be explained by considering its influence on the dye–fibre interactions during steaming. A longer steaming time increases the degree of swelling of the pre-treatment film, promoted by the migration inhibitor at a high level, is beneficial for the fixation process (plot CE), but influenced also by the other auxiliaries, especially low levels of urea and penetration agent (plots AC and AD).

Penetration factor (PF) is influenced predominantly by urea, and also by steaming time, both increasing ink penetration at high levels (plot AE). The migration inhibitor, which acts as filler for the capillary spaces in the fabric, unsurprisingly, appears to decrease PF (plot BC), especially with a longer steaming time (plots CE and CD).

In contrast to its role on standard *Tencel*, increased penetration agent concentration appears to reduce ink penetration on *Tencel A100* (plot CD).

3.2.3. Cotton

The selected contour plots for inkjet printing on to cotton are given in Fig. 4. Increased levels of migration inhibitor and steaming time enhance colour strength on cotton fabric (Fig. 4), while lower levels of urea and penetration agent are required (plots DE and AC). The influence of urea on colour strength observed on cotton thus contrasts with its effect on *Tencel* fabrics where it enhances colour strength. Thus, keeping urea at a low level (100 g l^{-1}) and migration inhibitor at high level (200 g l^{-1}) is beneficial for producing deeper shades on cotton. An optimized level of alkali (20 g l^{-1}) was also beneficial for enhanced colour strength on cotton as illustrated in plot BC. The effect of migration inhibitor concentration in enhancing colour strength is thus consistent with the effect on standard *Tencel*, but contrasts with its effect on *Tencel A100*. The migration inhibitor acts as thickener in the pre-treatment, inhibiting the wicking properties of the fabric by filling the fibre-to-fibre and yarn-to-yarn capillary spaces [23] and provides a barrier to swelling due to hydration. Thus, it inhibits penetration and spreading of the ink droplets during inkjet printing. The positive effect of migration inhibitor level on colour strength is explained by its effect on controlling ink penetration. However, while this explanation is plausible for the behaviour on *Tencel A100* and cotton (Figs. 3 and 4), standard *Tencel* showed the opposite behaviour, for reasons which are not clear.

The synthetic polyacrylic acid-based migration inhibitor used in this study has been shown to have superior performance in terms of colour strength and fixation compared with natural polymers such as sodium alginate and carboxymethyl starch [24,25] in screen printing of reactive dyes on to certain cellulosic fibres. It has also been suggested in a previous study that using a separate thickener in addition to a migration inhibitor in the pre-treatment is necessary for optimized colour strength and inkjet print quality [26]. The current study has demonstrated that use of this synthetic migration inhibitor is capable, in an optimum formulation, of producing high colour strength and fixation, arguably eliminating the need for an additional thickener. Increasing penetration agent level reduces colour strength (plot DE), similar to the behaviour on *Tencel* fabrics. Therefore, the penetration agent may well be an unnecessary ingredient in the pre-treatment. The use of this type of auxiliary in screen printing has commonly been associated with increased bleeding and haloing of the print, adding emphasis to this conclusion [27].

The dye fixation contour plots for cotton, in contrast with the *Tencel* fabrics, generally show similar trends to those for colour strength. As with standard *Tencel*, higher alkali concentration (40 g l^{-1}) resulted in a decrease in fixation (plot BD, Fig. 4), although its effect was not statistically significant. This behaviour, more apparent on cotton, may be explained on the basis of alkali increasing the concentration of cellulosate anions, which leads to repulsion of anionic dyes, thereby creating more opportunity for dye hydrolysis [21]. The effect of alkali on *Tencel A100* was different in that it had little effect on colour strength and fixation. Such differences may be attributed to the environment of the dye within the fibre which influences the extent of dye–fibre reaction compared with hydrolysis.

Although steaming time enhances colour strength and fixation on cotton (plots AE), it is recommended not to exceed 10 min due to an increased likelihood of dye hydrolysis [22]. As with *Tencel A100*, a high level of urea (200 g l^{-1}) decreased fixation, conceivably due to increased dye hydrolysis caused by excessive moisture retention during steaming. In the case of cotton, the reduced fixation at higher levels of urea correlates as expected with the observed reduction in colour strength. There has been some industrial interest in minimizing the use of, or replacing, urea as an auxiliary in textile processing for environmental reasons [28,29]. This present study is useful in demonstrating how the level of urea may be controlled to provide a balance which maximizes efficiency and cost-effectiveness (colour strength and dye fixation) while minimizing environmental effects.

Urea increases penetration into all three fabrics. Migration inhibitor and penetration agent levels decrease ink penetration on cotton and *Tencel A100*, but increase penetration on standard *Tencel*. Longer steaming time decreases PF on cotton and standard *Tencel*, but gives rise to PF on Tencel A100. In addition, as noted from Fig. 1, there are several significant interactions between variables for the PF on cotton. The relationship between ink penetration and colour strength is a particularly important feature in inkjet printing whose statistical significance for these three fibres will be reported in detail in Part 2 of this series of papers.

4. Conclusion

The study reported in this paper has provided a quantitative insight into the effect of pre-treatment chemicals on the colour strength, dye fixation and ink penetration on inkjet printed Lyocell (standard *Tencel*, *Tencel A100*) and cotton fibres, using a statistical analysis approach. As is the case with dyeing, standard *Tencel* offers advantages in inkjet printing over cotton in terms of the colour strength and dye fixation which may be achieved. In general, lower depth of shades were obtained by inkjet printing on *Tencel A100*, although it shows the highest dye fixation among the three fibre types, consistent with its dyeing behaviour. It is conceivable that the crosslinked structure of *Tencel A100* influences sorption of dyes and their reaction with the fibre, at the surface and within the bulk of the fabric, in such a way as to give paler shades than on standard *Tencel* and cotton.

Significant differences were seen with the main responses when the levels of the pre-treatment chemicals were varied. In each case, colour strength was enhanced using longer steaming time and a high level of migration inhibitor. Urea influenced dye uptake in different way for each fibre. Increasing urea concentration enhanced colour strength on both Lyocell fibres, but caused a decrease on cotton. Urea had a negative effect on dye fixation on cotton and Tencel A100, but a positive effect on standard Tencel. Alkali is a vital ingredient in printing with reactive dyes in producing deep shades with efficient fixation. However, this study illustrates that an excessive amount may have an adverse effect on fixation, especially for inkjet printing on cotton. Explanations for the observations, consistent with appropriate literature observations, have been proposed, although it is difficult to rationalise all of the results in detail because of the complexity of the systems which show several 2-way and 3-way interactions between variables.

Contour plots of the most significant interactions illustrate the conditions under which each response might be optimized on the three individual fabrics. The nature of the contour plots indicates the need for a compromise in optimizing colour strength and fixation simultaneously on the Lyocell fabrics, while cotton shows more consistent behaviour in terms of these two responses and thus easier simultaneous optimization. The statistical analysis provides an important tool for optimizing the pre-treatment formulation in terms of efficiency, cost-effectiveness and environmental issues. For example, the inclusion of a penetration agent in the formulation did not offer any apparent advantage since no significant interactions with the other ingredients were observed.

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