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Faisal, S, Ali, M, Siddique, SH et al. (1 more author) (2021) Inkjet printing of silk: factors influencing ink penetration and ink spreading. *Pigment & Resin Technology*. ISSN 0369-9420

<https://doi.org/10.1108/prt-12-2019-0120>

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Inkjet Printing of Silk: Factors Influencing Ink

Penetration and Ink Spreading

Abstract

Purpose - Pretreatment of fabric with a number of chemicals and auxiliaries is a prerequisite for inkjet printing. Owing to the rapidly increasing use of inkjet printing for textile fabrics, the study of the effects of process variables on various characteristics of the resulting print has drawn considerable interest recently. The primary purpose of the work reported here was to study the effects of different variables associated with the inkjet printing process on the quality of the resulting print. Specifically, the effects of chemicals and auxiliaries used in the pretreatment of fabric prior to printing and factors such as steaming time were studied.

Design/methodology/approach - In the present study, which forms a part of a larger study by the authors, the influence of the nature of thickener, the amounts of thickener, urea and alkali, pH of the pretreatment liquor and the duration of steaming on ink penetration into the printed fabrics and the ink spreading across the fabrics were studied. The nature of ink penetration and ink spreading are known to have pronounced effects on the quality, and in turn, the overall appearance of the resulting print. A set of experiments based on a blocked 2^{5-1} fractional factorial design (FFD) with four centre points were conducted in order to evaluate the role of the aforementioned five variables. Ink penetration was quantified on the basis of the principles of Kebulka-Munk theory while ink spreading was analysed by image analysis.

Findings - Detailed statistical analyses of the experimental data obtained show that different thickeners perform differently and can have a marked influence on ink penetration and ink spreading. In the case of polyacrylic acid (PAA)-based thickener, changing the levels of the factors has a marked effect on ink penetration and in-turn on ink spreading. In the case of polyacrylamide (PAM)-based thickener, on the other hand, the effect of changing the levels of various factors on the ink penetration and ink spreading is considerably less pronounced. In addition, PAM treated samples exhibited better performance in terms of ink penetration and spreading.

Originality/value - This study provides useful information for textile printers and highlights the importance of selecting the right type of thickener to make the printing process and the quality of the resulting print more predictable and controllable.

Keywords: Ink Penetration; Ink Spreading; Silk; Pretreatment; Inkjet printing; Reactive ink

Introduction

A number of physico-chemical phenomena take place as soon as a liquid ink drop comes into contact with a receiver substrate. These phenomena include drying by imbibition, diffusion, evaporation and eventually long-term stability (Van Roost and Desie, 2006). The rate at which these phenomena occur has a direct and significant impact on the overall appearance of a print (Yang and Kruse, 1999). In addition, the interaction of incident light in terms of reflection, transmittance, scattering and absorption is affected by the concentration of the colorant in the deposited ink film, and the thickness and the chemical composition of the ink film (Yang, 2003). In the context of the present study, ink film thickness was considered a more important factor compared to the other factors affecting print appearance as stated in the aforementioned text. Clearly, the spreading of ink across the substrate and the penetration of ink into the substrate affect the ink film thickness. When ink spreads, the ink film becomes thinner and the dot size becomes larger. One of the main factors that affect the transmittance of colorants is the thickness of the ink film. Other factors that affect the colour density of printed dot include surface reflectance (light that is scattered on the dot's surface before being absorbed or transmitted through), scattering of light within the ink, and amount of light that is absorbed by the substrate. However, these factors are generally considered to be minor compared to the thickness of the ink film. Hence, ink spread causes a dot to decrease in colour density whilst to increase in size. Thus, the extent of penetration into the substrate, the extent of lateral spreading and the time duration in which these phenomena take place to a given degree are the relevant factors to be taken into account when the quality of inkjet printing is evaluated in terms of print sharpness and visual appeal (Arney and Alber, 1998 ; Kim, 2006).

In the case of inkjet printing of textiles, it is well known that appropriate pretreatment of a fabric, subsequent to bleaching/mercerisation and prior to the deposition of ink, is necessary (Cie, 2015 ; Kaimouz et al., 2010b). The aim of the pretreatment process is to furnish, on the fabric surface, various chemicals and auxiliaries that are crucial to achieving the desired print quality which cannot be added into the ink formulation owing to the problems that are centred around clogging of nozzles and dispensability of the ink (Ding *et al.*, 2019). Such a dedicated pretreatment operation is the marked difference in the process route of inkjet printing of textiles from that of

the screen printing (Kiatkamjornwong *et al.*, 2005). Attempts have been made to devise novel approaches aimed at eliminating the need for elaborate pretreatments prior to inkjet/digital printing (Ma *et al.*, 2017 ; Ushiku *et al.*, 2010). However, from the point of view of practical viability, a dedicated pretreatment operation, as outlined in the preceding text, is more widely practiced for inkjet printing using pigmented inks (Leelajariyakul *et al.*, 2008 ; Sapchookul *et al.*, 2003) and inkjet printing using reactive dyes (Fang *et al.*, 2007 ; Hosseini *et al.*, 2013 ; Iriyama *et al.*, 2002 ; Xie *et al.*, 2007). A typical pretreatment recipe for preparing a substrate for inkjet printing using reactive dyes essentially contains thickener, alkali and urea. Regardless of the colorant type, the interplay of chemicals and auxiliaries that are used in the pretreatment liquor directly affect the interaction of the ink droplet with the substrate and, in turn, the amount of ink that penetrates through the substrate, the final geometry of the print dot on the substrate, and the diffused area etc. (Moutinho *et al.*, 2007). In order to characterise the interaction of ink droplet with the substrate, different techniques, including spectroscopic techniques (Yang *et al.*, 2006), microscopic techniques (Tse *et al.*, 1998), modulation transfer function (Janasak *et al.*, 2007) and image analysis (Fan *et al.*, 2003), can be employed. Image analysis is a relatively simple yet effective technique for analysis of ink penetration and spreading on sheet materials such as papers and textiles (Daplyn and Lin, 2003 ; Yang *et al.*, 2005).

The present study is part of a broader investigation conducted by the authors on the major factors that influence various aspects of inkjet printing of silk fabric. In the first phase, the authors carried out statistical analysis of the influencing factors on colour strength and %fixation (Faisal *et al.*, 2019). In the second phase of the same investigation, which is reported in this manuscript, the authors have conducted a statistical analysis of the same influencing factors, *i.e.* the amount of thickener, the amount of urea, the amount of alkali, pH of pretreatment liquor, type of thickener and the duration of steaming, on ink penetration and ink spreading. Owing to the advantages as outlined in the preceding text, image analysis was employed as the main analytical tool for characterisation of ink spreading while ink penetration was characterised using the well-established Kebulka-Munk equations.

Materials and Methods

Materials

Ready-to-print 100% silk fabric (plain weave of weighting 35 g/m²) was purchased from local market. The digital printing auxiliaries for wool fabric pretreatment included polyacrylic acid thickener (Huntsman, Pakistan), polyacrylamide thickener (Diamontex, Italy), urea, sodium bicarbonate, acetic acid and Revatol S (Archroma, Pakistan), which were supplied by SU & Company. Ladipur RSK (anionic detergent), supplied by Archroma Pakistan, was used in washing-off process. The fabrics were printed using a commercial reactive ink, Antelos R-KY XD (monochlorotriazine, Solunaris GmbH).

Method

Pretreatment of the Substrate

The required quantities of urea, alkali, reduction inhibitor (15 g/L) and thickener were added in 500 mL water with constant stirring to ensure proper dissolution. Remaining amount of water was then added to make up 1 L of padding liquor, of which the pH was adjusted to the required value using acetic acid. Padding of fabric samples was carried out on a Mathis laboratory padder (horizontal configuration) at a pressure of 1.7 bar and a speed of 1.5 r.p.m. The pick-up was maintained between 75-80%. After padding, the fabric was dried at 100 °C for 5 minutes using Mathis LTE dryer and then conditioned for 24 hours prior to printing.

Inkjet Printing and After-treatments

Inkjet printing was carried out on JP5 evo (MS Printing Solutions, Italy) digital printing machine. The resolution was set at 600×600 dpi (two pass) and a solid geometric pattern was printed. For fixation, the printed fabrics were steamed at 102 °C (saturated steam) for the duration as required for each individual experimental run. After fixation, the fabrics were washed for 10 minutes in 200 mL cold water, subsequently rinsed in 200 mL of water containing 2 g/L of Ladipur RSK at 95 °C for 10 minutes and then cold washed again for 10 minutes in 200 mL water (Faisal *et al.*, 2019).

Analytical Techniques

Analysis of Ink Penetration

For printing on textile substrates, Kebulka-Munk theory is more effective in assessing ink penetration. This is because that it considers both absorption and scattering of the incident light. A common practice that uses the relation given in Equation 1 to assess ink penetration within a textile substrate was adopted in this study.

$$\%Penetration (\%P) = \frac{100(K/S)_B}{0.5[(K/S)_F + (K/S)_B]} \quad (1)$$

Analysis of Ink Spreading

Ink spreading refers to migration of ink outside the desired area in a design. In this work, ink spreading was analysed separately for warp direction and for filling direction. For this purpose, the printed samples were scanned using an HP 8350 scanner at 300 dpi. Image J was used to measure the dimensions of the printed pattern. As shown in Figure 1, the width and length of multiple rectangular segments on the scanned printed design were recorded for each sample, followed by the calculation of the average width and length of the printed design. Lastly, Equation 2 and Equation 3 were used to quantify ink spreading in warp and filling directions, respectively.

$$S_w = Ink\ Spreading_{(w)} = L_p - L_0 \quad (2)$$

$$S_f = Ink\ Spreading_{(f)} = W_p - W_0 \quad (3)$$

In Equation 2, L_p is the length of the printed pattern in warp direction, L_0 is the length of the designed pattern in warp direction; whereas in Equation 3, W_p is the length of the printed pattern in filling direction and W_0 is the length of the designed pattern in filling direction.

Insert Figure 1 here

Experimental Design

The present study forms a part of a larger set of experiments conducted by the authors. Thus, the experimental design and the process factors are the same as reported by the authors in one of

their previous works (Faisal *et al.*, 2019). For the study reported here, a set of experiments based on a blocked 2^{5-1} fractional factorial design (FFD) with four centre points were conducted to evaluate the role of five variables namely, amount of thickener (100–200 g/L, Factor A), amount of urea (80–150 g/L, Factor B), amount of alkali (10–25 g/L, Factor C), pH of pretreatment liquor (6–9, Factor D) and steaming time (10–15 min, Factor E) on ink penetration (%P) and ink spreading in filling (S_f) and warp (S_w) directions. Three coded levels, -1, 0, and +1, were considered for each variable (see Table I), and accordingly 20 experiments were carried out. The block was used to check for any statistical significant effect of the type of thickener on the properties of interest. Linearity in design space was checked and confirmed by additional runs at centre points. All statistical analyses were performed using the statistical software package Minitab 18 (Minitab Inc.).

Insert Table I here

Results and Discussion

Statistical Analysis

The experimental runs and the data pertaining to ink penetration and ink spreading in filling and warp directions thus obtained are provided in Table II. The data was fitted into a second order polynomial model by forward selection regression analysis. Regression models were statistically evaluated by analysis of variance (ANOVA) and is presented in Table III and visualised by using Pareto charts shown in Figure 2. The adequacy of the models was determined by evaluating the F -test value, lack of fit, and the coefficient of determination (R^2) obtained from the analysis of variance (ANOVA).

Insert Table II here

It can be seen from Table III that, for ink penetration, the ANOVA of the regression model demonstrated that the model was highly significant, as was apparent from the F -test with a very low p -value ($p < 0.000$) with the coefficient of determination (R^2) at 0.8915. The lack of fit F -value of 6.07 and the p -value of 0.149 reveals that the pure error was not significantly relative. The block effect among the type of thickeners was not significant at 95% confidence level. A p -value of curvature greater than 0.05 indicates a satisfactory linearity in the region studied. The p -values ($p < 0.05$) of each model term presented in Table III indicate that the ink penetration of inkjet-

printed silk fabrics was significantly affected by the main effects of the amount of urea, the pH pretreatment liquor and steaming time and an interaction terms of the amount of thickener and the amount of urea (A*B), the amount of alkali and pH of the pretreatment liquor (C*D) and the amount of alkali and steaming time (C*E).

For ink spreading in filling direction, the *F*-test value of 127.14 with a very low *p*-value ($p < 0.000$) and the coefficient of determination (R^2) of 0.9893 demonstrate that the model was highly significant. The lack of fit *F*-value of 2.50 and *p*-value of 0.317 implies that the lack of fit is not significantly relative to the pure error. The block effect was found to be not significant at 95% confidence level amongst type of thickener. A *p*-value of curvature greater than 0.05 indicates a satisfactory linearity in the region studied. It can be observed from Table III that all of the five factors viz. amount of thickener (A), amount of urea (B), amount of alkali (C), pH of pretreatment liquor (D) and steaming time (E) significantly affected the ink spreading in filling direction of inkjet-printed silk. The interaction between amount of thickener and amount of urea (A*B), amount of alkali and steaming time (C*E), pH of pretreatment liquor and steaming time (D*E) had a significant effect on the ink spreading in filling direction of inkjet-printed silk.

For ink spreading in warp direction, the *p*-value (0.001) and the corresponding *F*-test value of 9.29 for the model shows that the model is highly significant. The Lack of Fit *F*-value of 0.82 implies that lack of fit is not significant relative to the pure error. The block effect among the type of thickeners was not significant at 95% confidence level. A *p*-value of curvature greater than 0.05 indicates a satisfactory linearity in the region studied. It can be observed from Table III that, only the main effect of steaming time affected the ink spreading in warp direction. However, interactive terms of amount of thickener with amount of urea (A*B), amount of alkali (A*C), pH of pretreatment liquor (A*D) and steaming time (A*E) affected the ink spreading in warp direction.

Insert Table III here

Pareto Charts

Pareto charts shown in Figure 2 depict the relative significance of effects of factors on ink penetration and ink spreading in warp and filling direction of inkjet-printed silk fabrics. In Pareto charts, the bar lengths are proportional to the absolute value of the estimated effect. In addition, all bars that are located to the right of the vertical red dashed line represent significant factors,

which mean that the corresponding factors and/or their interactions influence the response above a statistically significant level of 95% confidence. Thus, it is evident from the charts shown in Figure 2(a-c) that the interaction of amount of thickener and amount of urea (A*B) has the most pronounced influence on all three responses, *i.e.* ink penetration, ink spreading in filling and warp directions. Moreover, in the case of ink spreading in warp direction (S_w), the effect of interaction of amount of thickener and amount of urea (A*B) is only preceded by steaming time (E).

Insert Figure 2 here

Influence of Factors and Their Levels on Ink Penetration

From the mean plots presented in Figure 3, it can be seen clearly that in the case of fabrics pretreated using PAA, the ink penetration is minimum at the centre point (Level 0) of all the factors, while for higher levels and lower levels, the ink penetration increases. Another significant pattern in the results is that ink penetration is comparable for all the factors at their respective centre points (Level 0). Furthermore, ink penetration for the samples pretreated using PAM is lower in all cases. In addition, the effect of varying amounts of PAM is considerably insignificant on ink penetration.

Influence of Factors and Their Levels on Ink Spreading in Filling Direction (S_f)

As far as ink spreading in filling direction is concerned, changing the levels for the considered factors affect it in more or less the same manner. Both the thickeners seem to be comparable in terms of their effect on ink spreading in filling direction at 150 g/L (Level 0). However, at higher and lower levels, *i.e.*, 100 g/L and 200 g/L, respectively, the ink spreading in filling direction is more in the case of fabric pretreated with PAM. In contrast, in the case of fabric pretreated using PAA, the final dimensions of the print in the filling direction deviate considerably from the actual dimensions in the design at higher and lower levels of the considered factors.

Influence of factors and their levels on Ink Spreading in Warp Direction (S_w)

For the samples pretreated using PAA, a more or less similar trend is observed for ink spreading in the warp direction, *i.e.* the difference between the dimensions of the printed pattern and the actual dimensions in the design is minimum at factor level 0 (150 g/L of PAA). For factor levels -1 and +1 (100 g/L and 200 g/L), the dimensions of the printed area are rather smaller than the actual dimensions in the design. It is also evident from the mean plots, shown in Figure 3, that

except for steaming time, the effect of the different levels of the factors concerned on ink spreading in warp direction when printing is done on PAM treated substrate is rather negligible.

Insert Figure 3 here

Relationship between Ink Penetration and Ink Spreading

The scatter plots provided in Figure 4 provide insight into the relationship between ink penetration and ink spreading. From Figure 4, it can be seen that in the case of PAA, for all the factors, an increase in ink penetration was accompanied by a more pronounced reduction in the dimension of the printed area. In the case of PAA, the levels of a factor that resulted in increased ink penetration also resulted in a more pronounced difference (decrease) in the dimensions of the printed design. This is in-line with the fact that with increased penetration, the volume of ink that is available for potential spreading across the plane of the fabric is reduced (Kaimouz *et al.*, 2010a).

In the case of PAM, a similar relationship between ink penetration and ink spreading also existed. As discussed in the preceding text, various levels of the factors have a rather less pronounced effect on %Penetration which is found to be approximately 70% for the factors and their different levels. The results obtained for S_f and S_w and the influence of various factors on these parameters are in-line with the results of %P and indicate clearly that S_f and S_w are not affected significantly by different factors and their levels.

Insert Figure 4 here

The boxplots shown in Figure 5 provide a clear picture of ink penetration and corresponding ink spreading that is observed for the two thickeners that are considered in this study. It can be seen, from Figure 5 that, for PAM treated fabrics, ink penetration at various factor levels vary in a narrower range compared to the variation observed for PAA. Furthermore, ink penetration on PAM treated fabric is lower than that observed on PAA treated fabric and correspondingly the ink spreading is such that the final dimensions of the printed pattern are close to the actual dimensions in the design. The differences in the ink penetration and ink spreading values within the range of thickeners used, could be due to differences in their physical and chemical nature and number and location of functional groups (e.g. hydroxyl, carboxylic, and amide groups)(Ibrahim *et al.*, 2008 ; Ibrahim *et al.*, 2003 ; Ibrahim *et al.*, 1994). Both PAM and PAA are hydrophilic polymers. However, functionality, copolymerisation and molecular mass

directly affect their hydrophilic nature. Since molecular mass and comonomer content are one of the key determinants of the usefulness of a polymer for specific applications, it is proposed that as a future embodiment of the present study, characterisation of these features could provide further insight into the observed differences in ink penetration and ink spreading.

Insert Figure 5 here

Conclusions

In the present study, the effect of the nature/type of thickener; the amounts of thickener, urea and alkali; pH of pretreatment liquor and steaming time on two properties namely ink penetration and ink spreading was studied. The experimental runs were planned on the basis of a blocked 2^{5-1} fractional factorial design (FFD) with four centre points. The models for ink penetration and ink spreading in warp and weft direction were found to be significant at 95% confidence level. The results show that the interaction of the amounts of thickener and urea (A*B) has the most pronounced influence on all three properties, *i.e.* ink penetration, ink spreading in filling direction and ink spreading in warp directions. In addition, the results of this study clearly indicate that the two thickeners used separately in the pretreatment liquor had a significant impact on the extent of penetration of ink.

In the case of PAA, changing the levels of the factors studied has a marked effect on ink penetration and in-turn on ink spreading. In the case of PAM on the other hand, the effect of changing the levels of various factors on the properties of concern is considerably less pronounced. All of the findings presented in this paper provide useful information for textile printers and highlight the importance of selecting the right type of thickener in order to make the process and the resulting print quality more predictable and controllable.

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Table I Factors and levels used in the 2^{5-1} fractional factorial design

Symbol	Factor	Levels		
		Low (-1)	Center (0)	High (+1)
A	Thickener (g/L)	100	150	200
B	Urea (g/L)	80	115	150
C	Alkali (g/L)	10	17	25
D	pH of pretreatment liquor	6	7.5	9
E	Steaming Time (min)	10	12.5	15

Table II Coded design matrix and the results of 2^{5-1} fractional factorial design

Blocks	Factors					%P	S_w (mm)	S_f (mm)
	A	B	C	D	E			
1	1	-1	-1	-1	-1	83.46	-0.064	-0.951
1	-1	1	-1	1	1	81.75	-0.384	-1.447
1	1	-1	-1	1	1	79.67	0.089	-0.185
1	-1	1	1	1	-1	98.21	-0.840	-0.130
1	1	-1	1	-1	1	62.18	0.163	-0.110
1	0	0	0	0	0	73.58	-0.040	-0.001
1	0	0	0	0	0	72.78	-0.087	-1.228
1	-1	1	1	-1	1	78.39	-0.359	-0.846
1	1	-1	1	1	-1	88.34	-0.385	-4.209
1	-1	1	-1	-1	-1	83.53	-0.420	-2.970
2	1	1	-1	1	-1	67.69	-0.147	-1.424
2	-1	-1	1	-1	-1	69.01	0.106	-0.693
2	1	1	1	-1	-1	72.01	-0.164	-1.524
2	-1	-1	-1	1	-1	66.78	0.130	0.130
2	0	0	0	0	0	73.89	-0.142	-0.736
2	-1	-1	-1	-1	1	70.56	0.395	0.297
2	1	1	1	1	1	76.16	0.159	0.415
2	1	1	-1	-1	1	74.84	0.107	1.502
2	-1	-1	1	1	1	72.55	0.352	1.398
2	0	0	0	0	0	71.23	-0.113	0.467

Block 1 (PAA): runs 1-10, block 2 (PAM): runs 11-20, centre points: runs 6, 7, 15 and 20

Figure 1 (a) Scanned printed area, (b) filling-wise dimensioning and (c) warp-wise dimensioning

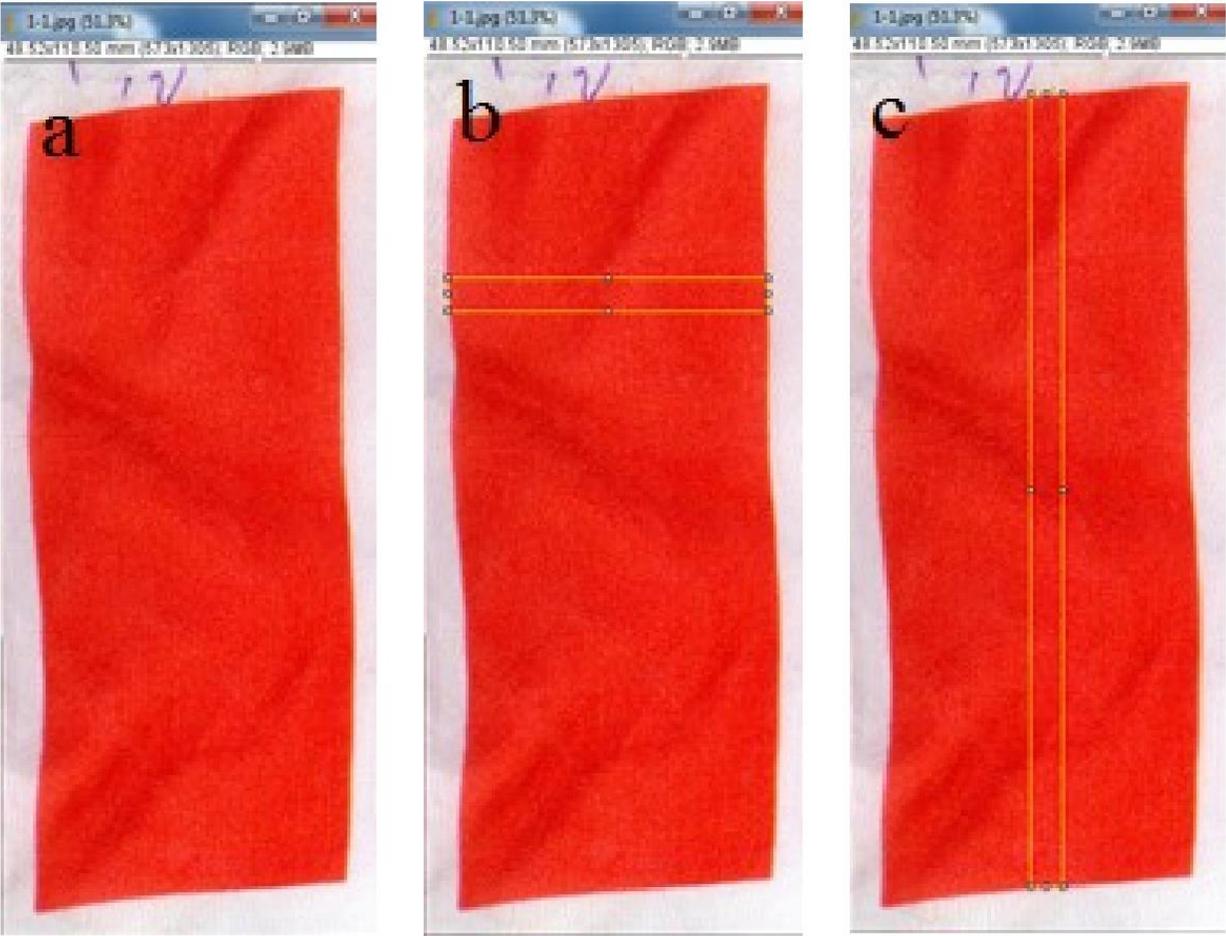
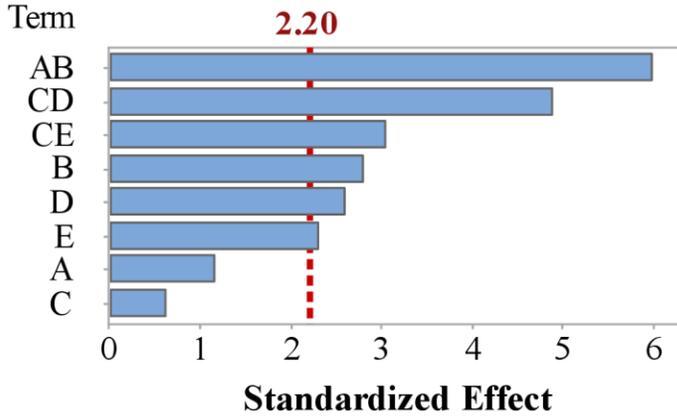
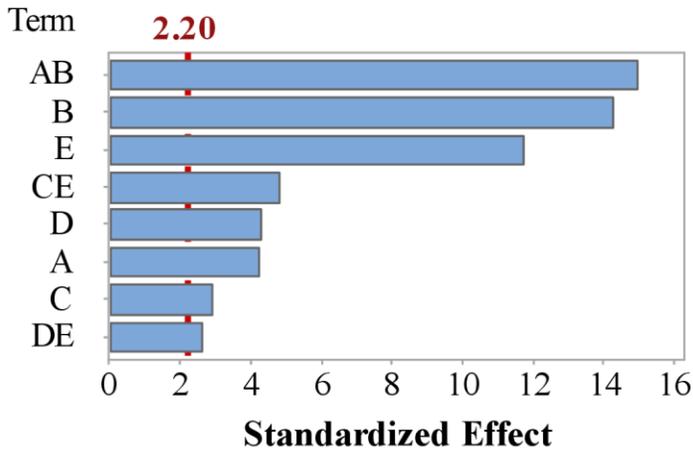


Figure 2 Pareto charts for standardised effects at $p = 0.05$ for (a) Ink Penetration (%P), (b) Ink Spreading in filling direction (S_f) and (c) Ink Spreading in warp direction (S_w)

(a)



(b)



(c)

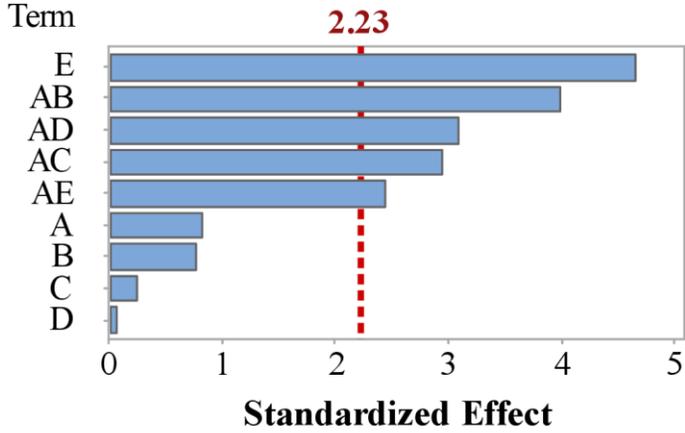


Figure 3 Mean plots illustrating the effects of factors and their levels on Ink Penetration (%P), Ink Spreading in warp direction (S_w) and Ink spreading in filling direction (S_f)

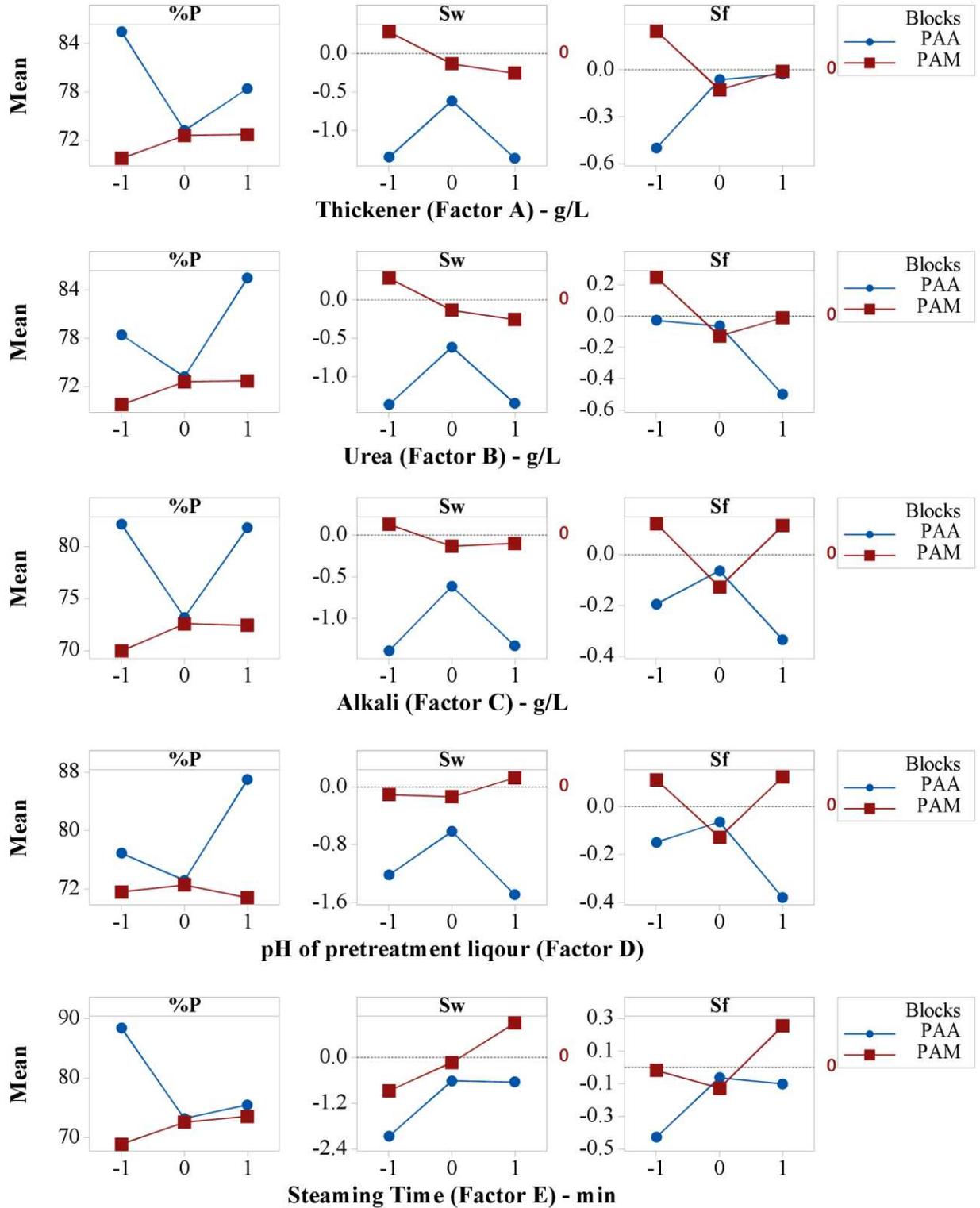


Figure 4 Scatter plots for ink penetration *vs.* ink spreading in warp direction and ink penetration *vs.* ink spreading in filling direction

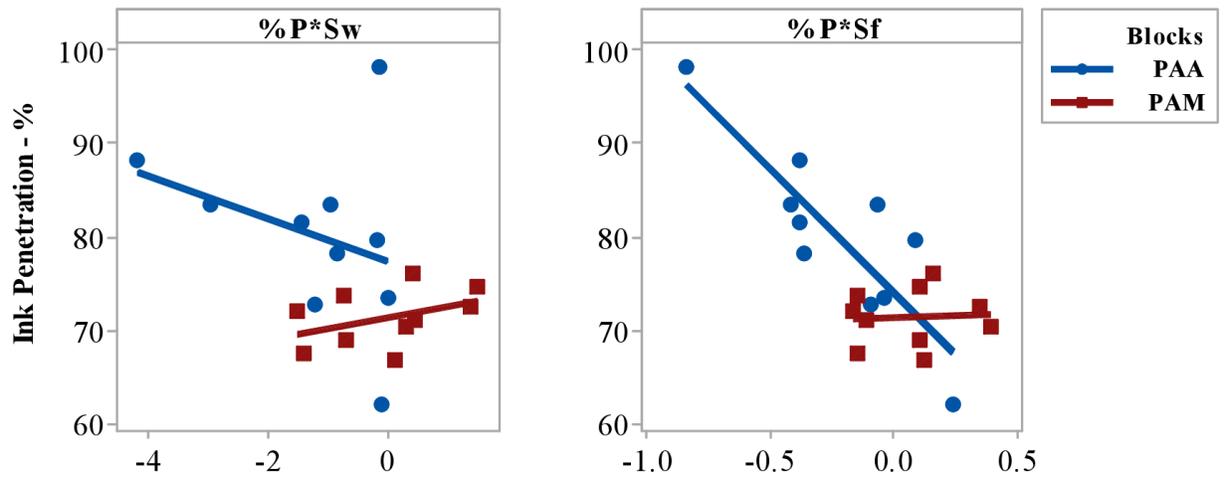


Figure 5 Boxplot showing the effect of thickeners on ink penetration and corresponding effect on ink spreading

