Influence of Chitosan Posttreatment Parameters on the Fixation of Pigment-Based Inks on Ink-Jet-Printed Cotton Fabrics

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ABSTRACT: Cotton fabrics were digitally printed with pigment-based black ink with an HP Desk-Jet 880C printer. These ink-jet-printed fabrics were posttreated with chitosan samples for the fixation of the pigment-based ink on the cotton. The influence of various parameters, including the molecular weight (MW), application method (pad–dry–cure vs pad–batch), concentration, and pH, on the degree of fixation (DF) of the pigment-based inks was examined. The chitosan-posttreated cotton samples were evaluated for their color strength, DF, color difference, and whiteness index values and their colorfastness properties. Chitosan samples with MWs of 150,000 and greater than 375,000 showed 100% (complete) fixation of the pigment-based inks on the cotton fabrics. DF drastically decreased in the chitosan with an MW of less than 5000. Both the pad–dry–cure and pad–batch methods were found to be suitable for chitosan application onto ink-jet-printed fabrics. Chitosan with an MW of 150,000 showed 100% fixation at concentrations ranging from 0.3 to 1%. A further decrease in the concentration significantly decreased the fixation. High fixation values were achieved at acidic pH, whereas a neutral to alkaline pH resulted in poor fixation. The colorfastness properties for each parameter studied are also discussed. The posttreatment of the digitally printed cotton with chitosan was found to be very effective in fixing the pigment-based inks. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 2495–2501, 2011

Key words: biopolymers; coatings; dyes/pigments; molecular weight distribution/molar; polysaccharides

INTRODUCTION

Ink-jet printing is a technology wherein droplets of ink are produced and deposited on various substrates, such as paper or textile materials, in response to an electronic signal. The ink-jet printing of textiles has emerged as a technology with tremendous opportunities and significant challenges.[1] It has full potential to meet market demands, such as for quick response and mass customization. It offers unlimited design possibilities with respect to repeat size and color range. It eliminates the setup costs associated with screen preparation and can potentially enable cost-effective, short-run production.[2] It allows visual effects, such as tonal gradients and infinite pattern repeat size, that cannot be practically achieved by a screen-printing technique.[3] It is a simple and environmentally clean technology. Other benefits include flexibility, reproducibility, creativity, and competitiveness.

Both dye-based and pigment-based inks can be used for the ink-jet printing of textiles. Despite their high color quality, dye-based inks suffer from some serious disadvantages. They are substrate-dependent, requiring different sets of inks for different textile fibers. This substrate dependence requires frequent changes in the color ways and/or fabrics; this leads to costly machine downtime. The ink-jet printing of blends is not yet possible with dye-based inks. Dye-based inks require costly pretreatments and posttreatments, such as steaming and washing, for their fixation. This may also add to the effluent and may require further expensive effluent treatment.[1] Because of the specific issues of the solubility and stability of dyes in the inks, the calibration of colors is difficult.

Pigment-based inks are substrate-independent and can be applied to most textile substrates, including blends. Other significant advantages include their ease of application, simple fixation via curing, better productivity, lower production costs, elimination of costly washing and steaming processes, and low effluent.[4] Our previous research[5] showed that chitosan, a well-known natural polysaccharide, could be successfully used as a posttreatment reagent for the fixation of pigment-based inks on ink-jet-printed cotton. The mechanism behind the fixation of pigment-based inks on cotton is believed to be possible

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TABLE I
Chemical Composition of the Pigment-Based Ink

Component	wt %
Carbon black pigment	<5
Isopropyl alcohol	<2.5
2-Pyrrolidone	<15
Water	>70

because of the strong film-forming ability of chitosan due to intramolecular and intermolecular hydrogen bonding.[6] The possibility of fixing existing pigment-based inks (intended for paper printing) onto cotton fabric was realized through this approach. In this study, the effect of various chitosan posttreatment parameters on the fixation of pigment-based inks was investigated.

EXPERIMENTAL

Materials

Desized, scoured, and bleached plain-weave cotton fabric (190 g/m^2) was used. Chitosan samples with three different molecular weights (MWs) were purchased from Sigma-Aldrich (Sydney, Australia). The ink cartridge (51645A) containing aqueous pigment-based black ink was purchased from Hewlett Packard (Melbourne, Australia) (Table I). All of the chemicals used in this study were laboratory grade.

Methods

Ink-jet printing of the cotton fabric

The cotton fabric samples (17 × 25 cm^2) were secured with sticky tape onto A4 paper as a backing material. A solid rectangle (15 × 20 cm^2) with a maximum print quality of 600 × 600 dots per inch was printed with a Hewlett Packard 880C Desk-Jet printer. The print head was based on thermal drop-on-demand technology. The samples were printed with aqueous pigment-based black ink with the chemical composition shown in Table I.

Chitosan posttreatment

Solutions of chitosan samples (I, II, and III) were prepared by the dissolution of a known amount of chitosan sample in a 1% (v/v) acetic acid solution. The chitosan solutions were magnetically stirred for 4 h to ensure complete dissolution. The physicochemical properties of the chitosan samples are shown in Table II. The ink-jet-printed cotton fabrics were posttreated with chitosan samples by the pad–dry–cure and pad–batch methods. The printed fabrics were padded to give 80% wet pickup. In the pad–dry–cure method, after padding, the fabrics were dried at 50°C for 5 min. The posttreatment parameters studied were (1) the MW of chitosan (<5000, 150,000, and >375,000), (b) the curing temperature (110, 130, and 150°C), (3) the batch time (3, 12, and 24 h), (4) the concentration of chitosan (0.03–1%), and (5) the pH of the chitosan solution ranging (4–9).

Analysis and measurements

Color measurement of the ink-jet-printed fabrics

The color characteristics of the printed samples were evaluated with a HunterLab colorQUEST II (Reston, Virginia) reflectance spectrophotometer with the following settings: illuminant D65 and 10° standard observer, specular component included, port size = 30 mm, and reflectance mode. Premier Colorscan software (Mumbai, India) was used for color measurement. The fabric was folded twice to ensure opacity and measured on three different portions of the fabric surface. The average value was recorded.

Color strength (K/S)

K/S of the ink-jet-printed samples was calculated with the Kubelka–Munk equation:

$$K/S = \frac{(1-R)^2}{2R}$$

where R is the reflectance of the colored sample; K is the absorption coefficient, which depends on the concentration of the colorant; and S is the scattering coefficient caused by the colored sample.

All the K/S values in this study were determined at the maximum absorption wavelength, at which the reflectance value was the lowest.

Degree of fixation (DF; %)

DF was calculated with the following equation:

$$DF(\%) = \frac{K/S \text{ after washing}}{K/S \text{ before washing}} \times 100$$

Washing was carried out according to ISO 105-C06: 1994 (E). Condition1 Multiple (C1M) test conditions were used. The results of one C1M(multiple) test was considered equivalent to approximately five

TABLE II
Physicochemical Properties of Chitosan Samples

Chitosan sample	DD (%)	MW	Viscosity (cP)[a]
I	>90	<5000	6
II	95	150,000	24
III	98	>375,000	>200

[a] At 25°C and a 1% concentration.

TABLE III
Effect of the Chitosan MW on DF

Application method[a]	MW of the chitosan samples	*K/S*		DF (%)	ΔE^*
		Before washing	After washing		
Pad–dry–cure[b]	<5000	5.4	1.5	27.8	19.9
	150,000	5.3	5.4	101.9	0.2
	>375,000	5.4	5.5	101.8	0.1
Pad–batch[c]	<5000	5.5	0.7	12.7	32.8
	150,000	5.3	5.3	100.0	0.1
	>375,000	5.4	5.4	100.0	0.1

[a] All the ink-jet-printed fabrics were padded at a 1% concentration of chitosan samples.
[b] The fabrics padded with chitosan samples were cured at 150°C for 5 min.
[c] The fabrics padded with chitosan samples were batched for 24 h.

domestic or commercial launderings at temperatures not exceeding 60°C.

Shade difference

To compare the shade difference of the samples before and after washing, the color difference (ΔE^*) values were measured with the HunterLab colorQUEST II reflectance spectrophotometer.

Fabric whiteness

The Commission Internationale de l'Eclairage (CIE) whiteness index of the untreated and chitosan-treated fabric samples were measured with a Datacolor 600 spectrophotometer. Datacolor Tools version 1.1.1 software (Lawrenceville, New Jersey) was used to calculate the whiteness index.

Colorfastness of the ink-jet-printed fabrics

The colorfastness of the ink-jet-printed fabrics to crocking and laundering were assessed according to ISO 105-X12:1993 and ISO 105-C06:1994(E), respectively. Multifiber fabric (DW), supplied by the Society of Dyers and Colorists (Bradford, England), was used as an adjacent fabric to assess the colorfastness to laundering.

RESULTS AND DISCUSSION

Effect of the chitosan MW on the fixation of the pigment-based inks

Both the pad–dry–cure and pad–batch methods were used to study the effect of the chitosan MW on the fixation of the pigment-based inks on the ink-jet-printed cotton fabrics. As shown in Table III, DF significantly decreased for the chitosan sample with an MW of less than 5000. Higher MW chitosans have been reported to have good film-forming properties as a result of intramolecular and intermolecular hydrogen bonding.[6] In the case of the chitosan sample with an MW of less than 5000, there would not have been enough intramolecular and intermolecular hydrogen bonding to form a good film. Hence, the fixation decreased significantly. The complete fixation of pigments was achieved with *K*/S values slightly above 100% for the ink-jet-printed fabrics treated by chitosan samples with MWs of 150,000 and greater than 375,000. The chitosan-treated samples showed increased *K*/S values after washing and, therefore, high fixation values exceeding 100%. This may have occurred because, during padding, the pigments deposited on the fabric surface may have been pressed into the interior of the fabric structure because of the pressure of the padding rollers. This may have led to a decreased initial *K*/S values before washing. Because of mechanical action during washing, the pigments may have migrated back to the fabric surface. Also, the strong film formed by chitosan on the fabric surface may have firmly held the pigments back, caused the redistribution of pigments, and thereby, prevented the loss of pigments into the wash liquor.[5]

Both the pad–dry–cure and pad–batch methods followed almost the same trend. The *degree ofdeacetylation*(DD; %) is defined as the average number of d-glucosamine units per 100 monomers and is expressed as a percentage. It determines the content of free amino groups ([bond]NH_2) in chitosan. Although the DD values for all of the chitosan samples were above 90%, the results suggest that the MW of chitosan significantly affected its film-forming ability, which in turn, affected the fixation of the pigments. A chitosan MW of less than 5000 was not sufficient to provide adequate crosslinking for durable film formation.

Table IV shows the effect of the chitosan MW on the colorfastness properties of the ink-jet-printed cotton fabrics. The dry and wet rubbing fastness values were acceptable for the ink-jet-printed fabrics treated by chitosan with MWs of 150,000 and greater than 375,000, whereas the fabrics treated by chitosan with an MW of less than 5000 showed poor rubbing fastness. Excellent washing fastness was observed in all of the samples, except for the chitosan with an MW of less than 5000. The results reinforce the proposition that at a low MW (<5000), there was insufficient crosslinking to bind the pigment within the chitosan film. Hence, the MW of chitosan had a significant effect on the binding ability of chitosan. However, the method of application of the chitosan, namely, pad–dry–cure or pad–batch, had no significant effect on the binding ability of the chitosan. Because of these results, the chitosan sample with an

TABLE IV
Effect of the Chitosan MW on the Colorfastness of the Ink-Jet-Printed Cottons

Application method	Chitosan MW	Rubbing fastness[a]			Washing fastness					
					Staining[a]					
		Dry	Wet	Color change[b]	Cellulose acetate	Cotton	Nylon 6,6	Polyester	Acrylic	Wool
Pad–dry–cure	<5000	3	1–2	1–2	4–5	4–5	4–5	4–5	4–5	4–5
	150,000	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	>375,000	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
Pad–batch	<5000	2–3	1–2	1–2	4–5	4–5	4–5	4–5	4–5	4–5
	150,000	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	>375,000	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5

[a] ISO Grey Scale for staining assessment (illuminant D_{65}).
[b] ISO Grey Scale for color change assessment (illuminant D_{65}).

MW of 150,000 was chosen for further evaluation and study.

Effects of the application methods

Whiteness index

The results of the CIE whiteness index measurement of the fabrics treated with chitosan (MW = 150,000) by the pad–dry–cure and pad–batch methods are shown in Table V. The whiteness of the fabrics decreased with increasing curing temperature. This agreed well with the previous study.[7] At 110°C, the change in whiteness was minor, whereas at 130°C, the loss of whiteness was marked but commercially acceptable. With the pad–batch method, an increase in the batch time decreased the whiteness of the fabrics. As shown by the results of the pad–batch method, chitosan alone had an effect on the whiteness, as no heat was involved in this method. However, the whiteness of the fabrics treated with chitosan by both application methods was within commercial acceptability.

DF and ΔE*

Table VI shows the effect of the curing temperature and batch time on DF. At 110°C, the fixation value was highest compared to those at the other temperatures (130 and 150°C). This was due to the high initial *K*/S value of the fabric sample before washing. During ink-jet printing, we observed that not all of the ink was retained on the fabric. Some of the ink wicked through the fabric onto the backing paper. We believe that this was because the amount of ink jetted on the fabric by the print head varied slightly from sample to sample. Also, the different *K*/S values among the samples and within a sample suggested the uneven absorption of ink onto the fabric surface. The increases in the *K*/S values after washing and the fixation values were believed to due to the reason explained in the previous study.[5] The Δ*E** values were found to be in agreement with the values of DF. Most importantly, the results show that chitosan could fix the pigment-based inks at a curing temperature of 110°C.

As shown in Table VI, there was no marked effect of the batch duration on DF. Furthermore, the *K*/S and DF values were effectively the same as those obtained by the heat curing of the prints. In the pad–batch method, the samples were simply rinsed and then allowed to dry in air under ambient laboratory conditions. These results suggest that chitosan was self-crosslinking, which means that it could form a crosslinked network upon exposure to ambient conditions without any application of energy or

TABLE V
Effect of the Curing Temperature and Batching Time on the CIE Whiteness Index

Posttreatment parameter		CIE whiteness index[a]
Curing temperature (°C)	110	74.46
	130	71.93
	150	70.37
Batching duration (h)	3	72.17
	12	71.23
	24	70.79

[a] CIE whiteness index of the untreated fabric (control) = 75.09.

TABLE VI
Effect of the Curing Temperature and Batching Time on DF and ΔE*

Posttreatment parameter		*K/S*			
		Before washing	After washing	DF (%)	Δ*E**
Curing temperature (°C)	110	6.4	7.4	115.6	1.9
	130	5.4	5.6	103.7	1.6
	150	5.4	6.0	111.1	1.7
Batching duration (h)	3	5.7	6.0	105.3	1.6
	12	6.0	6.2	103.3	1.6
	24	6.3	7.3	115.9	1.9

TABLE VII
Effect of the Curing Temperature and Batching Time on the Colorfastness

Posttreatment parameter		Rubbing fastness[a]			Washing fastness					
					Staining[a]					
		Dry	Wet	Color change[b]	Cellulose acetate	Cotton	Nylon 6,6	Polyester	Acrylic	Wool
Curing temperature (°C)	110	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	130	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	150	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
Batch duration (h)	3	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	12	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
	24	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5

[a] ISO Grey Scale for staining assessment (illuminant D65).
[b] bISO Grey Scale for color change assessment (illuminant D65).

curing and without the need for a bifunctional crosslinking agent. This may have an important commercial implication, in that the cotton fabrics ink-jet-printed with pigment-based inks could simply be batched after chitosan treatment and then air-dried to provide a durable print.

The high fixation value for the batch duration of 24 h was again due to the initial high K/S value of that sample. Interestingly, DF slightly decreased as the batch duration increased from 3 to 12 h, despite the high K/S value of the sample batched for 12 h. This might have been due to the variability of ink distribution and absorption on the fabric surface. The ΔE^* values showed a similar trend to that seen in the case of the pad–cure method.

Colorfastness to laundering and rubbing

As shown in Table VII, the colorfastness values to laundering and rubbing of the ink-jet-printed cotton fabrics posttreated with chitosan by the pad–dry–cure and pad–batch methods were found to be acceptable. The wet rubbing fastness for all of the samples was close to commercial acceptance (industry will accept a wet rubbing value of 3 for pigment-printed fabrics).

It was clear from the results obtained that both application methods produced similar results in terms of whiteness, DF, ΔE^*, and colorfastness to laundering and rubbing. Both methods were found to be suitable for the application of chitosan as a surface finish for the fixation of pigment-based inks.

Effect of the chitosan concentration

The effect of the chitosan concentration on the fixation of pigment-based ink on ink-jet-printed cotton fabrics is shown in Table VIII. DF of the pigment-based inks was effectively 100% for chitosan concentrations ranging from 0.3 to 1%. The fixation decreased from around 96 to 80% as the chitosan concentration was further decreased from 0.1% to 0.03%. Hence, DF of the pigment-based inks was markedly affected at the concentrations studied (i.e., 0.1, 0.05, and 0.03%).

As shown in Table IX, the dry rubbing fastness decreased as the concentration decreased but was acceptable for concentrations down to 0.1% and further decreased for 0.05 and 0.03% concentrations. The wet rubbing fastness was nearly acceptable for concentrations down to 0.1%, whereas for concentrations of 0.05 and 0.03%, it was found to be poor. The washing fastness was excellent for all concentrations down to 0.1%. For concentrations of 0.05 and 0.03%, the color change was satisfactory, and the staining was acceptable to good. These results demonstrate the suitability of chitosan for binding the ink-jet-printed pigments on cotton at a concentration as low as 0.3%.

Effect of pH

As shown in Table X, DF was significantly affected by pH. Initially, the fixation increased slightly as the pH increased from 4.0 to 6.0. At lower pH values,

TABLE VIII
Effect of the Chitosan Concentration on DF

Chitosan concentration (%)	K/S		DF (%)	ΔE^*
	Before washing	After washing		
1.0	5.3	5.5	103.8	1.2
0.75	5.1	5.1	100.0	0.6
0.5	5.4	5.7	105.5	1.3
0.3	5.1	5.2	102.0	0.3
0.1	5.2	5.0	96.2	0.9
0.05	5.0	4.2	84.0	2.7
0.03	5.0	4.0	80.0	3.1

TABLE IX
Effect of the Chitosan Concentration on the Colorfastness

Chitosan concentration (%)	Rubbing fastness[a]			Washing fastness					
				Staining[a]					
	Dry	Wet	Color change[b]	Cellulose acetate	Cotton	Nylon 6.6	Polyester	Acrylic	Wool
1.0	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
0.75	4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
0.5	3–4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
0.3	3–4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
0.1	3–4	2–3	4–5	4–5	4–5	4–5	4–5	4–5	4–5
0.05	3	2	3–4	3	3	4–5	3–4	4	4
0.03	3	2	3–4	3	3	4–5	3–4	4	4

[a] ISO Grey Scale for staining assessment (illuminant D65).
[b] ISO Grey Scale for color change assessment (illuminant D65).

the free amino groups of chitosan were highly protonated. Because of this, the pigments might have been held more strongly, and their migration to the surface might have been limited. As the pH was further increased to 6.0, the equilibrium of protonation may have shifted more toward unprotonated amines. As a result, the pigments may have more easily migrated to the fiber surface along with the loosely held chitosan molecules during the washing cycle. This contributed to the higher K/S values after the washing cycle and, hence, the increase in DF. Also, this could have simply been due to the variability of the ink distribution and absorption onto the fabric surface after washing. After 6.0, as the pH was further increased, the fixation decreased significantly. It is well known that chitosan dissolves in dilute mineral and organic acids by the protonation of free amino groups at pH values below about 6.5.[8] Above pH 6.5, chitosan exhibits poor solubility because the amino groups are less likely to be protonated.[9,10] The poor binding capability beyond pH 6.0 suggested that the film-forming ability of chitosan and, hence, DF depended on the cationic nature of chitosan.

TABLE X
Effect of the pH on DF

pH of the chitosan solution	K/S		DF (%)	ΔE^*
	Before washing	After washing		
4.0	5.0	5.1	102.0	0.4
5.0	5.1	5.3	103.9	0.5
6.0	5.0	5.3	106.0	0.6
7.0	5.1	4.5	88.2	4.1
8.0	4.9	3.3	67.4	9.5
9.0	4.8	2.7	56.3	11.7

As shown in Table XI, there was no significant effect of pH on the colorfastness up to pH 6.0. From pH 7.0 to 9.0, there were decreases in the dry and wet rubbing fastnesses. This was again due to the poor binding capability of chitosan beyond pH 6.0. Washing fastness in terms of staining remained unaffected at all pH values.

TABLE XI
Effect of the pH on the Colorfastness of the Cotton Fabric

pH	Rubbing fastness[a]			Washing fastness					
				Staining[a]					
	Dry	Wet	Color change[b]	Cellulose acetate	Cotton	Nylon 6.6	Polyester	Acrylic	Wool
4.0	3–4	2–3	4–5	4–5	4	4–5	4–5	4–5	4–5
5.0	3–4	2–3	4–5	4–5	4	4–5	4–5	4–5	4–5
6.0	3–4	2–3	4–5	4–5	4	4–5	4–5	4–5	4–5
7.0	2–3	1–2	3–4	4–5	4	4–5	4–5	4–5	4–5
8.0	2	1–2	2	4–5	4	4–5	4–5	4–5	4–5
9.0	1–2	1	1–2	4–5	4	4–5	4–5	4–5	4–5

[a] ISO Grey Scale for staining assessment (illuminant D65).
[b] ISO Grey Scale for color change assessment (illuminant D65).

CONCLUSIONS

In this study, we evaluated the effect of various chitosan posttreatment parameters on the fixation of pigment-based inks on ink-jet-printed cotton fabric. Chitosan with an MW of less than 5000 was found to be unsuitable for achieving the desired fixation. Both the pad–dry–cure and pad–batch methods were equally effective for chitosan application on the ink-jet-printed fabrics. A curing temperature of 110°C and a batch duration of 3 h were sufficient for optimum fixation of the pigment-based inks on the cotton. Complete fixation of the pigment-based inks on cotton was achieved with chitosan (MW = 150,000) at concentrations of 0.3% and higher. The fixation of the pigment-based inks was markedly affected at concentrations of 0.1, 0.05, and 0.03%. Complete fixation was observed at acidic pH values from 4.0 to 6.0. Neutral and alkaline pH values caused a significant decrease in the fixation values. Colorfastness to laundering was acceptable to excellent for all of the parameters studied, except with a chitosan MW of less than 5000 and alkaline pH values. The rubbing fastness values were acceptable in all cases, except with a chitosan MW of less than 5000, chitosan concentrations of 0.05 and 0.03%, and pH values of 7.0, 8.0, and 9.0.

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