

Using of chitosan as an alternative biodegradable thickener in reactive Ink Jet Printing

Shrerif. H. Abd El-Salam

Professor of Textile Printing, Dyeing and Finishing Department, Faculty of Applied Arts, Helwan University.

Jacklin. I. Abd El-thalouth

Professor of Textile Printing, Dyeing and Finishing Department, Faculty of Applied Arts, Helwan University.

Nermeen. M. Mohammed

Textile Printing Dyeing and Finishing Department, Faculty of Applied Arts, Helwan University.

Abstract:

In this paper, cellulosic fabrics (cotton-viscose) was treated with chitosan as a biodegradable thickener as a replacement for convention thickener (Alginate and Carboxymethyle cellulose). To achieve this goal, many trials have made in order to adjust the best conditions ever to apply chitosan as a thickener. this trials lie on five main fields, the first one concerning the neutralization process its self, in order to adjust it in one or two baths, second was concerning by the best way and time for neutralization, then the third one is to choose the concentration for chitosan to use, then the fourth object is to compare the optimum concentration of using chitosan by the conventional methods, i.e. Alginate and C.M.C. and at last is to test the effect of mixing chitosan with alginate with different ratios. The color yield and color fastness were measured far all of printed sample and the result showed that chitosan could be uses as a pretreatment agent for ink jet reactive prints weather alone or in mixture with alginate.

Keywords:

*Cotton,
Viscose,
Alginate,
Carboxymethyle Cellulose,
Chitosan,
Ink-Jet Printing,
Reactive Dyes*

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1. Introduction:

Recently, there has been great interest in ink-jet textile printing, and how to improve this technique. Ink-jet printing has more benefits than conventional printing methods such as speed, flexibility, creativity, cleanliness, eco-friendliness, and competitiveness.⁽¹⁻³⁾

Nowadays, a huge ratio of ink-jet printing for cotton fabrics in markets have mainly produced by reactive dyes, but the application method is very different from the conventional printing process. Because the chemicals of the conventional printing, such as alkalis, urea, and sodium alginate (as a thickener) cannot be directly added to the ink formulations. it is ought to the purity and specific conductivity requirements of ink-jet printing because of the narrow nozzles of the print head. Therefore, the other chemicals applied to the fabrics in a treatment process before the ink-jet printing.⁽⁴⁻⁶⁾

Currently, sodium alginate is an important thickener commonly used for preparing the pretreatment print paste for ink-jet printing because of its ready solubility and excellent

stability even after high temperature fixation treatments. Similar to sodium alginate, chitosan is also widely used as a novel biomaterial. As natural polymers, sodium alginate and chitosan are biocompatible, biodegradable, and nontoxic.⁽⁷⁾

2. Experimental:

2.1 substrates:

2.1.1 Cotton fabrics: 100% desized, scoured, bleached, unmercerized poplin cotton fabric (140 g/m²) produced by Misr-Helwan for Spinning and Weaving Company-Egypt was used throughout the present work.

2.1.2 Viscose fabrics: 100% scoured viscose fabric (130 g/m²) produced by Misr-El Mahalla for Spinning and Weaving Company -Egypt was also used.

2.2 Chemicals:

2.2.1 Thickeners: the thickeners used comprise :

- **Sodium alginate:**

High viscosity sodium alginate in a solid form from brown algae supplied by (Fluka Chemicals Company) was used, and its chemical composition is shown in Fig. (1).

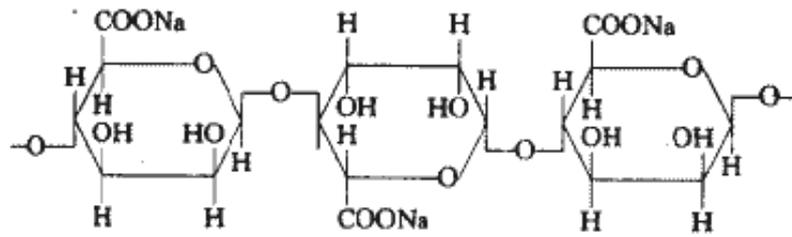


Figure (1) chemical structure of Sodium alginate

- **Sodium Carboxy methyl cellulose:**
High viscosity Sodium Carboxy methylcellulose was used in a solid form manufactured by (CISME

Italy s.n.c company), and its chemical composition is shown in Fig. (2).

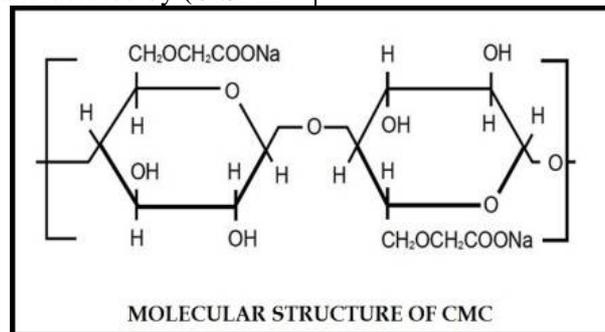


Figure (2) chemical structure of Sodium Carboxy methyl cellulose

- **Chitosan :**
Chitosan ($R-NH_2$) medium molecular weight (100.000) in a solid form was purchased from

(ROTH, Germany) was used, and its chemical composition is shown in Fig. (3).

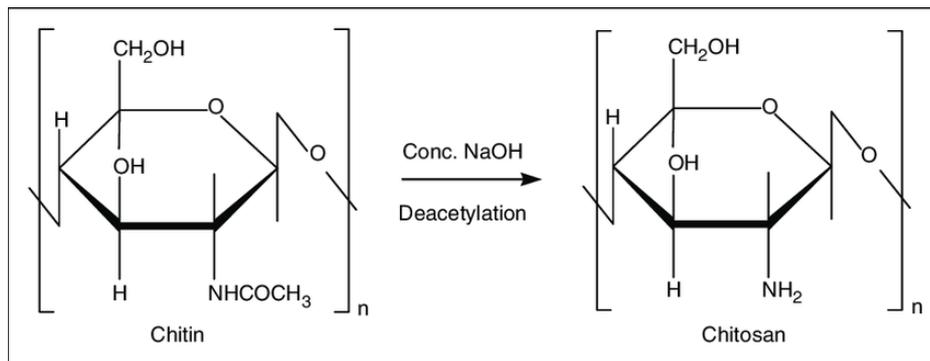


Figure (3) chemical structure of Chitosan

2.2.2 Other Chemicals: sodium bicarbonate, urea, acetic acid and non-ionic detergent were purchased from (Egptol PLM).

2.2.3 REACTIVE DYES :

Reactive inks (Cyan, Magenta, Yellow and Black) was supplied by (Piporin Co., Indonesia).

2.3 methods:

2.3.1 Preparation of chitosan pretreatment printing paste:

A stock chitosan solution with a medium viscosity was prepared through the complete dissolution of (10-20-30-40-50 g) of chitosan into 1000 mL of (10%) acetic acid with constant stirring. The stock solution was then filtered to remove any suspended impurities. The amount of chitosan used in the pretreatment print paste was measured

directly from the stock chitosan solution. 10g of urea and 8g of sodium bicarbonate were also added to the pretreatment print paste to study the effect of chitosan amount on each color.

2.3.2 Preparation of C.M.C. pretreatment printing paste:

Another pretreatment print paste was prepared that contained 150 g of Carboxy methyl cellulose (this amount was obtained from a stock Carboxy methyl cellulose solution through the dissolution of 10 g of Carboxy methyl cellulose in 990 mL of deionized water), 10 g of urea, and 8 g of sodium bicarbonat.

2.3.3 Preparation of Sodium alginate pretreatment printing paste:

a control pretreatment print paste was prepared that contained 150 g of sodium alginate (this

amount was obtained from a stock sodium alginate solution through the dissolution of 50 g of sodium alginate in 950 mL of deionized water), 10 g of urea, and 8g of sodium bicarbonate.

2.3.2 Fabric pretreatment :

2.3.2.1 One-Bath

The pretreatment print paste was padded to the fabric with a padding machine to give a final wet pickup of 80%. The chitosan treatment fabrics were cured at 170°C for 1.5 min. The fabrics were then washed with deionized water for 5 min after curing. The acid-bath-treated fabrics were then dried completely in an oven at 80°C., whereas the control fabric and C.M.C were only dried in the oven at 80°C.

To study the effect of alkali adding, it was made treated sample without adding sodium bicarbonate.

2.3.2.2 Two-Bath without storing:

To make a lower effect of neutralization during the mixing of the chitosan solution and sodium bicarbonate in the pretreatment print paste preparation process, a two-bath chitosan treatment was proposed that included:

(1) **The acid bath:** the fabrics were first soaked for 2–3 min in the stock chitosan solution and then padded uniformly on the fabric with a padding machine until a wet pickup of 80% was achieved. Second, the padded fabrics were cured at 170°C for 1.5 min. The fabrics were then washed with deionized water for 5 min after curing. The acid-bath-treated fabrics were then dried completely in an oven at 80°C.

(2) **The alkali bath:** in the alkali bath, a pretreatment paste containing 5 g of urea and 4 g of sodium bicarbonate in 500 ml of deionized water was prepared, and this pretreatment paste was padded onto the chitosan-pretreated fabrics by means of a padding machine until a wet pickup of 80% was achieved. Then the treated fabrics dried in an oven at 80°C before inkjet printing.

2.3.2.3 Two-Bath with storing:

To minimize the effect of neutralization during the pretreatment process, a two-bath chitosan treatment was proposed that included (1) an acid bath and (2) an alkali bath.

In the acid bath, the fabrics were treated same as in the two bath without storing process then were further conditioned at 20°C with a relative humidity of 65% for 24 h before the treatment with an alkali bath.

After that, the fabrics were padded same as the alkali in two bath without storing process then a wet pickup of 80% was achieved.

The two-bath chitosan treatment fabrics were then dried completely in an oven at 80°C and

subsequently conditioned at 20°C with a relative humidity of 65% for 24 h before inkjet printing.

2.3.2.4 The (chitosan –alginate) mixture:

It was used the (chitosan-alginate) mixture with: One bath process: the fabrics were padded in a one paste contained a mix of chitosan-alginate with ratio 50:50; a wet pickup of 80% was achieved. Then dried completely in an oven at 80°C and subsequently conditioned at 20°C with a relative humidity of 65% for 24 h before inkjet printing.

• Two bath process:

(a) With the ratio of 50:50, the fabrics were treated first with chitosan with two baths with storing process. Second with alginate; a wet pickup of 80% was achieved. Then dried completely in an oven at 80°C and subsequently conditioned at 20°C with a relative humidity of 65% for 24 h before inkjet printing.

(b) With the ratio of 50:50, the fabrics were treated first with alginate, second with chitosan with two baths with storing process. Then dried completely in an oven at 80°C and subsequently conditioned at 20°C with a relative humidity of 65% for 24 h before inkjet printing.

• It was also made samples treated with chitosan before alginate with the ratio of (25:75) and (75:25).

2.3.3 Ink-jet printing:

An ink-jet printer (DGI FD-1908, Digital Graphics Incorporation Co., Korea) was used machine at (BG group Co. Egypt). Four commercially available reactive ink-jet inks (cyan, magenta, yellow, and black), supplied by (Digital Graphics Incorporation Co., Korea), were used directly without further purification.

A square pattern of 120 mm 120 mm was printed for each color at 600 dpi 1200 dpi for easy comparison.

2.3.4 Fixation:

After ink-jet printing, the printed fabrics were air-dried and then steamed at 105°C for 10 min for color fixation.

2.3.5 washing:

The steamed fabrics were washed with cold running water then in 15ml/L nonionic detergent (10 g/L) at 100°C for 10 min. to remove all the unreacted reactive inks and chemicals and finally were washed with cold running water then air dried.

2.4 Analysis and Measurements:

2.4.1 Color yield measurement:

The printed fabrics were conditioned at 20°C with a relative humidity of 65% for 24 h before the color yield measurements with Spectrophotometer (Model:CM-3700A , SN: 10001434-JAPAN). The measurements were taken under a speculum

excluded with a large aperture. The fabric was folded two times to ensure opacity and then measured twice; that is, the measurement was conducted in both the warp and weft directions. The measured results were finally averaged.

The color yield, expressed as a K/S value (where K is the absorption coefficient depending on the concentration of the colorant and S is the scattering coefficient caused by the colored substrate), ranging from a wavelength of 400 to 700 nm with 20-nm intervals within the visible spectrum was calculated. The K/S values were summed according to eq. (1). The higher the K/S (sum) value was, the greater the dye uptake was, resulting in a better color yield:

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

where R is the reflectance of the colored sample.

2.4.2 Color fastness of the fabrics:

The colorfastness of the ink-jet-printed fabrics to:

- Washing was assessed by ROTOWASH (MODEL : M228BB – SN: 628D0028 – U.K.)
- Crocking was assessed by CROCKOMETER (MODEL : KMS – SN: 090129 – JAPAN)
- **Prespiration** was assessed by PRESPIROMETER (SN: 090182 – U.K.).

3. Result and discussions:

The color yield of chitosan treated samples:

Previous study reported that chitosan could be applied to different textile printing for improving the dye uptake and color fastness. For this purpose and as said before the main aim of this work is to replace alginate thickening by chitosan one, in order to gain the benefit of bio finishing printing i.e. printing with reactive dyes and improving the

color yield in one step process.

To achieve the goal many trials were made in order to adjust the best conditions ever to apply chitosan as a thickener, while the main problem face us was adjusting the PH.

As it is well known that chitosan dissolved in water only in acidic medium and hence in order to treat the cotton by chitosan, it was a must to be in acidic medium. Where in such conditions it's very hard or impossible to print with reactive dye. so it is a must that the fabrics undergo neutralization process so our trials lie on five main fields, the first one concerning the neutralization process its self, in order to adjust it in one or two baths, second was concerning by the best way and time for neutralization, then the third one is to choose the concentration for chitosan to use, then the fourth object is to compare the optimum concentration of using chitosan by the conventional methods, i.e. Alginate and C.M.C. and at last is to test the effect of mixing chitosan with alginate with different ratios.

The samples were first treated-printed, steamed, washed and dried finally were adjusted to K/S measurements and the results were illustrated in the following table.

1.1 Effect of neutralization process:

In this part trials were concerning the neutralization process its self, as said before. For this purpose four samples were prepared one the alkali was incorporated in the same bath with that of chitosan, while the other was after the treatment with chitosan (two for cotton and two for viscose) the result were illustrated in table 1 and 2.

(Table 1) Effect of neutralization process on k/s values for cotton printed fabrics

	cyan	magenta	yellow	black
Treating without sodium bicarbonate	1.57	1.46	1.68	2.51
Treating with sodium bicarbonate	2.79	2.55	3.67	2.21

(Table 2) Effect of neutralization process on k/s values for viscose printed fabrics

	cyan	magenta	Yellow	black
Treating without sodium bicarbonate	1.87	0.97	1.25	2.73
Treating with sodium bicarbonate	2.39	2.85	2.97	2.37

Tables (I-II) show the different values of K/S of two samples: chitosan treated fabrics with sodium bicarbonate adding and chitosan treated fabric without sodium bicarbonate adding in the pretreatment print paste.

Tables clearly show that the color yield of the printed fabrics was increases when sodium

bicarbonate has used in pretreatment processes for the color development of all shades.

This result was expected, that chitosan to react with cotton fabrics it must be acidic medium in order to obtain the reaction fig. (4) ⁽⁸⁾ with chitosan with the free hydroxyl group of cotton fabric.

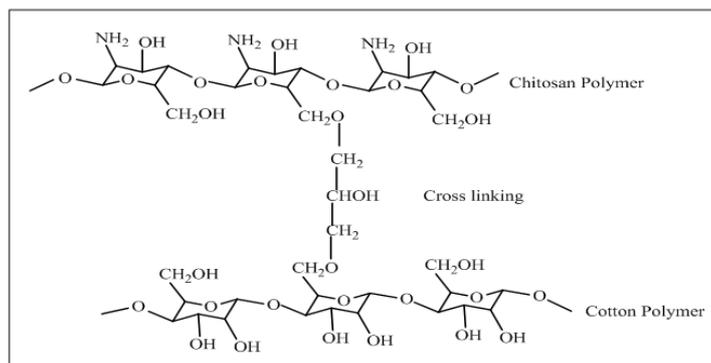


Figure (4) chemical reaction of chitosan with cotton.

After reaction occurred, chitosan at this moment compacted with cellulosic fabric it is no need for the acidic medium so we have to adjust fabrics for ionization reaction attraction with reactive dye so we have to treat fabrics with alkali at this point. The fabrics with NH_3^+ group (as a result of chitosan reaction) can easily uptake the reactive particles results high K/S values.

Using alkali in the printing process has the same role as in dyeing processes so it will enhance the color yield of all of shades. The adding of alkali is important to produce ionization in accessible cellulose hydroxyl groups, which can then react with the reactive dyes in the fixation stage.

Sodium bicarbonate is cheap and can give sufficient pretreatment print paste stability with most reactive dyes available in the market so it has

been the preferred alkali. During steaming process, carbon dioxide is lost from sodium bicarbonate and the ionization of cellulose is increased so it enhances the interaction between the dye and fiber in the fixation stage. ⁽⁹⁻¹¹⁾

1.2 Effect of time of neutralization:

As cleared from previous section that it is a must to treat samples before printing with alkali.

For this purpose two samples were prepared. One with two bath (i.e. treating with chitosan, curing at 80°C and the neutralization).

The other was left for 24 hours storing after curing before neutralization process and the samples were illustrated in table 3 for cotton and 4 for viscose, in addition to the result of the sample with one bath as a sack of comparison.

(Table3) Effect of time of neutralization on k/s values for cotton fabric

	Cyan	magenta	yellow	black
One bath	2.79	2.55	3.67	2.21
Two bath without storing	4.08	4.18	5.55	5.05
Two bath with storing	5.86	5.85	4.91	6.27

(Table 4) Effect of time of neutralization on k/s values for viscose fabric

	Cyan	magenta	yellow	black
One bath	2.39	2.85	2.97	2.37
Two bath without storing	7.94	11.6	13.42	10.15
Two bath with storing 24h.	9.45	8.15	13.5	12.15

Tables (3-4) show the effect of chitosan treatment method on the color yield of ink-jet printed cotton and viscose fabrics. It was observed that the two-bath with storing method treated fabrics had better color yield when compared with the chitosan-treated one-bath and the two-bath without storing method treated fabrics.

This color yield increase was expected that leaving samples for storing for 24 h. after neutralization permits a better condition for reaction to occur with chitosan particles and hence increase in up take groups for reactive dyes to react ended in highly K/S values.

This neutralization minimizing the amount of sodium bicarbonate in the pretreatment paste and the amount of the neutralization product as water was increased. and during the fixing process, the

low of sodium bicarbonate amount will minimize the fixation of reactive dyes with the fiber while the increased amount of water cause a higher risk of reactive dye hydrolysis which leading to the color yield reducing. So the best color yield values of the two-bath with storing method could be due to the neutralization effect occurred only inside the pretreatment print paste. ⁽¹²⁾

1.3 Effect of concentration of chitosan on color yield of the prints:

The section concerning with adapting the best conc. for chitosan in order to pretreat fabrics with. For this purpose, samples treated with two bathes after storing for 24 h. as said before were prepared but with different conc. of chitosan. viz., 10, 20, 30, 40 and 50 gm/l and the samples were printed, fixed and washed and adjusted to K/S

testing and the results were set in table 5 and 6.

(Table 5) Effect of concentration of chitosan on k/s values for cotton printed fabrics

	Cyan	magenta	yellow	black
10 gm./l	4.02	4.89	4.66	4.56
20 gm./l	4.64	4.32	5.09	5.9
30 gm./l	4.14	4.72	4.49	4.62
40 gm./l	5.17	7.24	4.87	5.59
50 gm./l	5.5	5.19	6.32	6.34

(Table.6) Effect of concentration of chitosan on k/s values for viscose printed fabrics

	cyan	magenta	Yellow	black
10 gm./l	8.13	9.32	12.46	11.72
20 gm./l	8.19	9.44	13.03	12.25
30 gm./l	9.05	0.45	14.19	12.17
40 gm./l	10.03	11.4	15.41	13.71
50 gm./l	9.74	10.74	15	14.12

As shown in tables (5-6) the color yield of the printed fabric was increased with the chitosan amount which used in the pretreatment printing paste so the best color yield achieved with 50 gm./L. of chitosan, but the samples was have a harsh handle with this amount so we could considered that the best color yield was achieved when 40 gm. of chitosan used in the pretreatment print paste.

This means that chitosan have important role in increasing the color yield of inkjet printing and this action could be expounded on the basis that the amino groups present in the chitosan take up a proton from the acidic dye bath to make the $-NH_3^+$ groups. These amino groups being electropositive in nature make the transfer of the negatively

charge dye anion easy from the bath onto the fabric, thereby improving the color fixation. ⁽¹³⁻¹⁵⁾

1.4 Comparing chitosan with conventional thickeners:

After adjusting and detect the best and optimum consternation and process to apply chitosan as a thickener. So we have to compare the best result achieved on using chitosan as a thickener with the convention methods i.e. thickener (alginate and CMC)

Sample were preprend with different thickener viz. chitosan, CMC, Alginate and adjusted to reactive inkjet printing and their K/S results were illustrated in table VII for cotton samples and VIII for viscose one.

(Table 7) Effect of different thickeners on k/s values for cotton printed fabric

	Cyan	magenta	yellow	black
Chitosan (4%)	5.17	7.24	4.87	5.59
C.M.C (5%)	3.47	1.85	3.34	3.47
Alginate (5%)	4.93	6.5	4.76	9.24

(Table 8) Effect of different thickeners on k/s values for viscose printed fabric

	cyan	magenta	yellow	black
Chitosan	10.03	11.4	15.41	13.71
C.M.C	7.54	9.24	13.32	16.26
Alginate	9.59	10.44	15.11	14.46

As shown in tables (7-8) the best color yield was achieved in case of using chitosan as a thickener and that was explained as when chitosan was applied on the cotton fabric, it is believed that the amino group present in chitosan take up a proton from the slightly acidic bath and so form NH_3^+ which Direct proportion with the amount of chitosan in pretreatment paste so the amount of the negative anions dye which transfer from the bath onto the fabric will increased and thereby improve exhaustion. ^(14, 15)

So we finally achieved the goal, that treating cotton or viscose with chitosan after reactive ink

jet printing can be a perfect alternative for alginate as a thickener as well as its cationic effect which increase the affinity of cellulosic fabrics towards the anionic dyes and hence increase color yield.

As cleared from the result that at all conditions viscose samples acquire high k/s values when compared to that of cotton at all cases with different conc. And this was expected data, that viscose simples are highly modified cellulosic fabrics with high percent of amorphous regions when compared to that of cotton and hence highly free hydroxyl groups capable of reaction when adjusted in appropriate condition.

1.5 Using chitosan in a mixture with sod.

Alginate:

Another trial was made, which is examine the compatibly of mixing chitosan with the conventional reactive thickener which is alginate.

In this part three samples were prepared first

(Table 9) Effect of using chitosan in a mixture with sod.alginate treatment on k/s values for cotton fabric

	Cyan	magenta	Yellow	black
Chitosan before Alginate (50:50)	4.93	3.81	5.09	4.34
Alginate before Chitosan (50:50)	4.5	4.17	4.71	3.98
One bath(50:50)	1.76	1.19	1.8	1.51

(Table 10) Effect of using chitosan in a mixture with sod.alginate treatment on k/s values for viscose fabric

	cyan	magenta	Yellow	black
Chitosan before Alginate (50:50)	6.54	7.41	11.14	11.17
Alginate before Chitosan (50:50)	6.5	5.68	10.7	9.25
One bath (50:50)	1.7	0.77	6.02	1.85

As shown in tables (9-10) the best color yield achieved with applying Chitosan before Sodium Alginate.

In case of one-bath pretreatment, the reduction on color yield due to the neutralization effect that caused by mixing the chitosan solution with sodium bicarbonate in the same printing paste. As known, the neutralization effect reduces the amount of sodium bicarbonate so reduce the fixation of reactive dyes inside the fiber and generates water that increase the risk of reactive dyes hydrolysis, thereby reducing the color yield.⁽⁹⁾

In case of applying Sodium Alginate before Chitosan neutralization was occurred also but with lower k/s values. This result was expected, chitosan is well known as a finishing compound for cellulosic fabrics so treating cellulosic fabrics

samples treated with chitosan then by alginate (50:50) the second sample was treated by alginate before chitosan and the last sample was mixing the two thickeners in one bath and the values of k/s for prints were illustrated in tables 9 and 10.

with chitosan first increase the reaction with cellulose and hence high present of NH_3^+ groups ends in highly values for k/s values, while when the alginate treated first this makes a sever condition for the finishing agent (chitosan) to react in such condition and hence cause lower k/s values.

In all cases it's obvious that using or applying both chitosan and alginate acquires achievement in ink jet reactive printing.

1.6 Effect of using different ratios of (chitosan - sod. Alginate):

Three samples were prepared with different ratios of chitosan and alginate.

In all samples, viz. (75:25), (25: 75) and (50:50) chitosan-alginate respectively, Cellulosic fabrics were adjusted to chitosan treatment first then with alginate.

(Table 11) Effect of using (chitosan-alginate) treatment different ratios on k/s values for cotton fabric

	cyan	magenta	Yellow	black
(75:25)	5.17	5.26	5.73	4.47
(25:75)	5.27	5.28	5.36	4.43
(50:50)	4.93	3.81	5.09	4.34

(Table 12) Effect of using (chitosan-alginate) treatment different ratios on k/s values for viscose fabric

	cyan	magenta	Yellow	black
(75:25)	7.23	9.2	12.37	11.2
(25:75)	7.67	10.17	14.17	12.46
(50:50)	6.54	7.41	11.14	11.17

Tables (11-12) show that the best color yield was achieved with using (chitosan: alginate) mixture

with ratio (25:75) because of the lowest neutralization effect with the smallest amount of acid solution.

That result was expected, that as the concentration of alginate increased the k/s value increase too.

So we can easily apply chitosan before alginate as a finishing agent to increase k/s values as well as a partially thickener to reduce the amount of alginate used.

2. Color fastness of the ink-jet-printed fabrics:

Table (13) shows that the inkjet printed fabrics that treated with chitosan two-bath with storing

method had very good color fastness properties to laundering, crocking and perspiration like the alginate sample and that might be explained with the introduction of primary amino groups into the cellulosic fiber structure. After these groups were deposited into crevices between the fibers, they most likely imparted a cationic surface that attracted the oppositely charged reactive dye anions. As a result, the ink-jet printability of the two-bath chitosan treatment fabrics was very good. ⁽¹⁵⁾

(Table 13) Color fastness of the ink-jet-printed fabrics

Test Sample		Chitos-an one bath	Chitos-an two bath without storing	Chitos-an with storing	Alginate	C.M.C.	Mixture (50:50)
CF. to Crocking	Dry	4	4-5	4-5	4-5	4-5	4-5
	Wet	1-2	2	3-4	3	2-3	3
CF. to Laundering	Change of color	4-5	4	4-5	4-5	4-5	4-5
	Wool	4	4-5	4-5	4-5	4-5	4-5
	Acrylic polyester	4-5	4-5	4-5	4-5	4-5	4-5
	Nylon	4-5	4-5	4-5	4-5	4-5	4-5
	Cotton	4-5	4-5	4-5	4-5	4-5	4-5
	Acetate	5	4-5	5	5	5	4-5
CF. to Perspiration	Change of color	4	4-5	4-5	4	4-5	4-5
	Wool	4-5	4-5	4-5	4-5	4-5	4-5
	Acrylic polyester	4-5	4-5	4-5	4-5	5	4-5
	Nylon	4-5	4-5	4-5	4-5	5	4-5
	Cotton	4	3-4	4-5	4	4-5	4
	Acetate	4-5	4-5	4-5	4-5	5	4-5

4. Conclusion:

From all previous results of the color yield and color fastness we can illustrates that chitosan can be easily used as a pretreatment agent for ink jet reactive prints for cellulosic fabrics as an excellent alternative for alginate weather alone or in mixture with alginate and it was confirmed that the best method of using chitosan is the two bath method with storing for 24h.

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