NATURAL DYES

Edited by **E. Perrin Akçakoca Kumbasar**

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Preface

As it is known dyes are colored materials that have an affinity to the substrate. Textile materials without colorants cannot be imagined and according to archaeological evidence dyeing has been widely used for over 5000 years. In the past, dyes were obtained from plants, animals or minerals. After the discovery of the first synthetic dye by W. H. Perkin in 1856, many thousands of synthetic dyes have been found and by the 1870s commercial dyeing with natural dyes was in the decline. For textile finishing processes first stage is pre-treatment. In general, dyeing and printing, chemical and mechanical finishing processes follow pre-treatment. With the development of chemical industry all finishing processes of textile materials are developing continuously and, ecological and sustainable production methods are very important nowadays. So some researchers show the ecological values of natural dye.

The first section of the book "Natural Dyes" considers eco-friendly pre-treatments. The application of natural dyes and approach to ecological dyeing with natural dyes is discussed in the following section. As it is known, the efficiency of finishing treatments can change with chemical properties. Accordingly, in the last Chapter, the importance of surface modification of wool fibers is evaluated.

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Contents

Part 1

Eco-Friendly Pretreatment

1

Eco-Friendly Pretreatment of Cellulosic Fabrics with Chitosan and Its Influence on Dyeing Efficiency

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1. Introduction

Wet processing of textiles uses large quantities of water, and electrical and thermal energy. Most of these processes involve the use of chemicals as assisting, accelerating or retarding their rates and are carried out at elevated temperatures to transfer mass from processing liquid medium across the surface of textile material in a reasonable time. So, we can use some natural materials and some physical tools to reduce the chemicals, water, energy and pollution. This tools and natural material can use also to improvement the fabrics surface via introduce new active groups on its. Improvement of the fabrics surface can increase the efficiency of bleaching, dyeing and finishing processes.

Chitin, a major component of the shell of crab and shrimp is one of the most abundant natural polysaccharides with a large unexplored commercial potential. Chitosan is partially or completely N-deacetylated chitin and mainly consists of B-(1,4)- linked 2- amino-2-deoxy-B-D-glucopyranose. In recent years, a number of investigations have been carried out to exploit the potential applicability of chitosan(1). Chitosan have many applications in the medical and textile fields.

Cellulose which has been known to have good physical properties has been widely used as construction material, paper and clothes. Cellulosic fabrics can be oxidized by several oxidizing agents such as hydrogen peroxide (H_2O_2), sodium persulphate ($Na_2S_2O_8$) and potassium periodate (KIO_3). Oxidation of cellulosic fabric using sodium metaperiodate ($NaIO_4$) has been extensively investigated in the literature, since it leads to selective cleavage at the C2 and C3 vicinal hydroxyl groups to yield a product with 2,3-dialdehyde units along the polymer chain(2). The latter is an important functional polymer for further derivatisation to specialized products.

As a naturally deriving substances, chitosan and cellulose share several common beneficial properties such as being nontoxic and biodegradable. Structurally, chitosan is slightly different from cellulose as the monosaccharide in chitosan chain is 2-amino-2-dehydroxy-D-glucose instead of D-glucose. The presence of amino group is responsible for the complete solubility of chitosan in a diluted aqueous acidic solution as a polycationic polymer whereas cellulose is totally insoluble. This polycationic nature makes chitosan very appealing as a surface treating agent for cellulose fabrics to improve its dyeability to reactive and acid dyes. Chitosan used for surface modification of cellulose fabrics. Direct padding or exhausting chitosan solution onto cotton fiber was reported to show significant improvement in dyeability of the fiber with some reactive dyes (3). Oxidations of cotton fiber or fabric prior to the treatment of chitosan have been reported. Chitosan was applied after oxidation of cotton fabric by H_2O_2 show improved dyeability with reactive dyes (4). Potassium periodate is known to selectivity convert 1,2-dihydroxyl groups to a pair of aldehyde groups without significant side reaction and is widely used in structural analysis of carbohydrates(2). This oxidizing agent was used successfully for surface oxidation of cotton fiber prior to the treatment with chitosan to produce chitosan coated cotton fiber.

We undertake this work with a view to establish appropriate conditions for synthesis of cotton fabric containing chitosan. We plan to incorporate chitosan in the molecular structure of cotton surface through strong interaction between chitosan molecules and cotton molecules. To achieve the goal, creation of functional groups such as aldehyde groups is effected by $NaIO_4$ oxidation and thus obtained oxidized cotton is treated with chitosan. This is ratter a two-step process for producing cotton fabrics containing chitosan. A novel one-step process is also devised for preparation of the same modified fabrics; the fabric is treated in an aqueous solution containing the oxidant and chitosan. All modified fabrics are monitored for fixed amount of chitosan expressed as nitrogen content, carbonyl content, dyeability, strength properties and IR spectra.

2. Experimental

2.1 Materials

A bleached cotton fabric was kindly supplied by Misr Company for Spinning and Weaving, Mehallah El-Kubra, Egypt. All chemicals used in current investigation were of analytical grade. Dyes used include acid dye which was of laboratory grade and, reactive dye, namely, Procion Turquoise® MXG which was of technical grade.

2.2 Methods

2.2.1 Oxidation of cotton fabrics with sodium periodate

Unless otherwise stated, bleached cotton fabric was immersed in an aqueous solution containing different concentrations of sodium periodate (30-80 mg/100ml). The solution was stirred for 1 hr at 60 °C using a material to liquor ratio (M/L) 1:50 .The oxidized sample was washed several times with water to remove the oxidant .This oxidized sample was used for the next reaction without drying.

2.2.2 Treatment with chitosan

A chitosan solution was prepared by stirring a dispersion of chitosan (0.5-2%) in 1% (v/v) aqueous acetic acid solution. The aforementioned oxidized cotton fabric was immersed in the chitosan solution with constant shaking for different periods of time (30, 60, and 120 min.).The treatment process was performed at different temperatures (40, 60 and 80 °C). A material to liquor ratio of 1:50 was used. The treated sample was washed several times with 1% (v/v) aqueous acetic acid solution followed by water and dried at 60 °C.

2.2.3 Oxidation of cotton fabrics and chitosan with sodium periodate in one bath

An aqueous solution containing 2% chitosan, 50 mg/100ml sodium periodate was prepared. Fabric was impregnated in this aqueous solution at liquor ratio 1:50 for 1 hr at 60 °C. The cotton fabric was then washed several times with 1% (v/v) aqueous acetic acid solution followed by water and dried at 60 °C.

2.2.4 Dyeing

Dyeing with reactive dye

Cotton fabrics treated with solution periodate as described above were dyed using aqueous bath containing 1% of the reactive dye along with 5% sodium chloride and liquor ratio 1:50 at room temperature. The temperature of the dyeing bath was raised to 60 °C for 45 minutes. The fabrics were then rinsed with water and treated with an aqueous solution containing 1% wetting agent at 60 °C for 30 minutes at a liquor ratio 1:50. The dyed fabrics were rinsed with hot water followed by cold water and finally dried at ambient conditions

Dyeing with acid dye

After being treated with solution periodate, the cotton fabrics samples were dyed in an aqueous bath containing 1% of the acid dye together with 2% sodium sulphate using a liquor ratio 1:50 at pH, 5-6 and temperature 40 °C. The latter was then raised to 100 °C for 60 minutes. At this end, the fabrics were squeezed, washed with water and treated with an aqueous solution containing 2% wetting agent at 60 °C for 30 minutes. The dyed fabrics so obtained were rinsed with hot water followed by cold water and finally dried at ambient conditions.

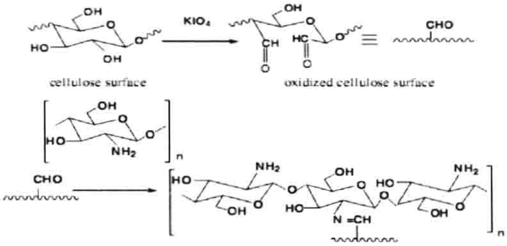
2.2.5 Testing and analysis

- The Nitrogen content was determined according to Kjeldahl method(5)
- The carbonyl content, expressed as meq./100g fabric, was determined according to a reported method (6).
- Tensile strength and elongation at break were measured using the strip method according to ASTM (7)
- The colour strength of dyed fabrics, expressed as K/S, was determined as described elsewhere (8).
- FT-IR spectroscopy

The FT-IR spectra of cotton fabrics treated with NaIO4 and chitosan in two subsequent steps as well as concurrently were recorded on a Nexus 670 FT-IR spectrophotometer, Nicolet, USA, in the spectra range 4000-400 cm^{-1} using the KBr disc technique.

3. Results and discussion

Complexation of chitosan with cotton cellulose to produce cotton –based new textiles is of paramount concern. To render these new cotton textiles more durable and stable for subsequent treatments such as dyeing and finishing, it is a must to induce strong interactions between chitosan and cotton. One of the approaches to achieve this is to create aldehyde groups in the molecular structure of cotton cellulose. These aldehyde groups undergo coupling with the amino groups of chitosan to form iminic bonds (1) whereby chitosan is fixed to cotton surface through a series of reactions as shown under:



Our work involves fixation of chitosan to bleached cotton fabric as per two methods. The fist method is based on oxidation of the fabric with $NaIO_4$ in acidic medium to yield fabric containing 2,3-vicinal diol of the glucose unit of cotton cellulose or what is called dialdehyde cellulose. The so obtained oxidized fabric is then subjected to chitosan treatment where interaction occurs thereby causing fixation of chitosan on the fabric surface. In the second method, the fabric is treated in a bath containing $NaIO_4$ and chitosan where simultaneous oxidation and chitosan fixation take place.

When dealing with the second method, mention should be made of the effect of $NaIO_4$ on the chitosan. According to previous reports(9), the chitosan molecule is susceptible to oxidation by sodium periodate at five points: the terminal aldehyde group at C1, the secondary hydroxyl on C4 at the non reducing end of the chain, the secondary hydroxyl on C3, the primary hydroxyl at C6 and the amino group at C2 position. In addition molecular chain scission of chitosan occurs through the attack of the oxidant on the 1-4 glucosidic linkages. When chitosan is oxidized the action takes place chiefly at C3 and C6 hydroxyls, the amino group at C2 and at the 1-4 glucosidic linkage by virtue of the greater abundance of these sites as compared with the two types of terminal groups.

With the above in mind, factors affecting the major technical properties of cotton-based new textile brought about by the first and second methods were thoroughly investigated. Factors studied include concentrations of chitosan and $NaIO_4$ as well as time and temperature of the treatments. On the other hand, the obtained cotton products were monitored for nitrogen content, carbonyl content, tensile strength, elongation at break and dyeability. IR spectra for cotton fabrics-containing chitosan which were processed as per the two methods are also presented.

3.1 Sodium periodate concentration

Table 1 shows the effect of sodium periodate concentration on the amount of fixed chitosan (expressed as nitrogen content), carboxyl content, tensile strength and elongation at break of the oxidized fabric. Obviously the amount of fixed chitosan on the cotton fabric increases by increasing the oxidant concentration up to 50 mg/100ml. Further increase in the oxidant concentration to 80 mg/100ml causes no significant increase in nitrogen content. This phenomena could be explained by considering the difference in the reaction site of the oxidation and Schiff's base formation in the cellulosic fabric. During the oxidation, the small periodate ion might be able to enter the cellulosic fabric interior and the glucose unit both inside and on the surface of the cellulosic fabric may be oxidized. On the other hand, chitosan is a huge molecule that cannot enter the fabric, and the modification with chitosan occurs on the surface of the fabric. Results of table 1 reveal that the carbonyl content of the fabric in question, i.e. periodate-oxidized fabric containing chitosan, increases by increasing the oxidant concentration. This is rather a manifestation of oxidation of hydroxyl groups of the cotton fabric to aldehydic groups under the progressive action of the oxidant at high concentrations.

Tensile strength decreases substantially after the fabric was subjected to periodate treatment at a concentration of 30 mg/100ml followed by chitosan treatment. Increasing the periodate concentration up to 80 mg/100ml causes no further significant decrease in the tensile strength of periodate oxidized fabric-containing chitosan. While the loss in tensile strength of the fabric upon using sodium periodate at 30 mg/100ml concentration could be interpreted in terms of degradation of cotton cellulose, the no significant change in tensile strength upon using higher concentrations calls for extra strength imported to the fabric by higher amounts of fixed chitosan as evidenced by the higher contents. The chitosan molecules seems to form a film fixed on the cotton cellulose (in the fabric form) through strong interactions thereby compensating for the higher losses in tensile strength expected at higher sodium periodate concentrations. It is also possible that the cotton cellulose undergoes modification during the initial oxidation and such modification makes the cellulose less susceptible for farther oxidation particular via chain scission. On the other hand, elongation at break marginally reduced after oxidation and chitosan treatment regardless of the oxidant concentration used within the range studied.

[$NaIO_4$] mg/100ml	Nitrogen content (%)	Carbonyl content Meq/100g S	Tensile strength (Newton)	Elongation at Break (%)
0.0	0.112	15.122	38.25	11.34
30	0.181	20.098	30.17	11.02
50	0.243	25.105	29.53	10.71
80	0.252	31.167	29.42	10.45

Conditions used for oxidation of cotton fabric: time,1hr; temperature,60 °C, M/L, 1:50
Condition used for chitosan treatment: [chitosan], 1%; time, 1.5 hr; temp, 60°C; M/L, 1:50.

Table 1. Effect of sodium periodate concentration on some chemical and mechanical properties of chitosan-containing cotton fabric.

The above findings indicate that modified cotton fabrics which enjoy the presence of chitosan as evidenced by their nitrogen content and acidic properties as evidenced by the carbonyl content while retaining much of their strength properties can be achieved using $NaIO_4$ concentration 30-5- mg/100ml followed by treatment with chitosan at a concentration of 1%.

3.2 Chitosan concentration

Table 2 shows the dependence of the modification effect- expressed as nitrogen content, carboxyl content and strength properties including tensile strength and elongation at break on the chitosan concentration. As is evident the nitrogen content and strength properties of modified fabric increase as the chitosan concentration increases. At higher concentrations, chitosan molecules would be greatly available in the proximity of the macrostructure of cotton and fabric surfaces both containing aldehyde groups thereby leading to Schiff's base formation between aldehyde groups and chitosan amino groups.

[chitosan] %	Nitrogen content (%)	Carbonyl content Meq/100g S	Tensile strength (Newton)	Elongation at Break (%)
0.0	0.000	36.231	28.22	10.31
0.5	0.191	32.143	28.52	11.02
1.0	0.243	25.105	29.53	11.71
1.5	0.292	22.125	31.21	12.44
2.0	0.324	20.176	31.51	12.53

Conditions used for cotton fabric oxidation: [$NaIO_4$], 50 mg/100ml; time, 1hr; temp, 60 °C, M/L, 1:50
Condition used in chitosan treatment: time, 1.5 hr; temp, 60 °C; M/L, 1:50

Table 2. Dependence of the magnitude of modification of cotton fabric on chitosan concentration.

It is further observed that the values of the carbonyl content of the modified fabric decrease substantially by increasing chitosan concentration. This indicates that the carboxyl groups of the modified cotton fabric under investigation are involved in chemical reactions with chitosan. Once this is the case, the carbonyl groups are masked and their values decreases by increasing the chitosan concentration where opportunities of interactions are better.

3.3 Temperature of chitosan treatment

Table 3 discloses the effect of chitosan treatment temperature of oxidized cotton on some chemical and mechanical properties of the modified fabric. The treatment was carried out at different temperatures for 90 minutes using chitosan concentration of 2%. The results signify that the nitrogen content increases by raising the chitosan treatment temperature from 40°C to 80 °C .This could be ascribed to increased amount of incorporated chitosan into the oxidized fabric as a result the favorable effect of temperature on swallability of cotton and mobility of chitosan molecules; both enhance the magnitude of interactions between cotton and chitosan. On the other hand, raising the chitosan treatment temperature adversely affects the tensile strength of the modified fabric. Most probably greater penetration of the highly mobile chitosan at higher temperatures in the fibriller structure of swollen cotton causes rigidity and, in term, decrement in tensile strength. The results of elongation at break are in confirmation with this.

Temp. °C	Nitrogen content (%)	Carbonyl content Meq/100g S	Tensile strength (Newton)	Elongation at Break (%)
40	0.196	24.116	34.21	13.13
60	0.324	20.176	31.51	12.53
80	0.513	18.523	29.53	11.71

Conditions used for oxidation of cotton fabric: [$NaIO_4$], 50 mg/100ml; time, 1hr; temp, 60 °C, M/L, 1:50
Condition used in chitosan treatment: [chitosan], 2%; time, 1.5 hr; M/L, 1:50

Table 3. Effect of chitosan treatment temperature of the oxidized cotton on major technical properties of the modified fabric

Table 3 depicts that the carbonyl content decreases by raising the chitosan treatment temperature from 40°C to 80 °C. This state of affairs implies that higher temperature acts in favour of the interactions of chitosan with cotton cellulose and that these interactions involve, inter alia, the carbonyl groups of cotton.

3.4 Time of chitosan treatment

Table 4 shows the effect of time of chitosan treatment on major technical properties of the obtained modified cotton fabrics. It is seen that the nitrogen content increases by prolonging the time of chitosan treatment within the range studied. Provision of better contact and intimate association of chitosan with cotton cellulose at longer duration period would account for this. On the other hand, results of carboxyl content, feature that the carbonyl content decreases by increasing the time of chitosan treatment. This is rather similar to the effect of chitosan treatment temperature discussed above and could be explained on the same basis.

Time (hr)	Nitrogen content (%)	Carbonyl content Meq/100g S	Tensile strength (Newton)	Elongation at break(%)
0.5	0.237	23.335	31.32	13.14
1.0	0.272	19.812	30.52	12.53
1.5	0.513	18.523	29.53	11.71

Conditions used for oxidation of cotton fabric: [$NaIO_4$], 50 mg/100ml; time, 1hr; temp, 60 °C, M/L, 1:50
Condition used in chitosan treatment: [chitosan], 2%; temp, 80 °C ; M/L 1:50

Table 4. Effect of time of chitosan treatment of oxidized fabric on some chemical and mechanical properties of the obtained modified fabric

Table 4 shows that the tensile strength decreases from ca 31 to ca 29 Newton when the duration of chitosan treatment increases from o.5 to 1.5 hr. This little effect of time of chitosan treatment is also seen with respect to elongation at break. At any event, however, these decrements are considered to be a direct consequence of rigidity conferred on cotton caused by chitosan penetration. As already stated, rigidity is proportionally related to the amount of chitosan on the fabric and the extent of penetration of former in the latter.

3.5 One step method for modification

In the foregoing sections, innovative modified cotton textiles could be achieved by a two-step method namely, periodate oxidation of cotton fabrics in one step followed by treatment of these fabrics in a second step by chitosan. In order to avoid detrimental effects of oxidation and in order to save time, chemicals and energy the two steps were combined in a single stage process where the cotton fabrics were treated in an aqueous solution containing the periodate oxidant and the chitosan under conditions emanated from the studies of the factors discussed above.

The nitrogen content, the carbonyl content and strength properties of cotton fabrics before and after being processed as per the two-step process and the one-step process are set out in table 5. For convenience the untreated cotton fabric and the two-step processed modified cotton fabric and the one-step processed cotton fabrics will be referred to as substrate I, substrate II and substrate III, respectively.

Substrate	Nitrogen content (%)	Carbonyl content Meq/100g S	Tensile strength (Newton)	Elongation at Break (%)
I	0.000	17.341	38.32	11.3
II	0.513	18.523	29.53	11.71
III	0.652	16.535	45.12	13.52

Where Substrate I: bleached cotton fabric; substrate II: two-step processed modified cotton; substrate III: one -step processed modified cotton. Two-step process involves oxidation by $NaIO_4$ and treatment with chitosan in two consecutive steps. One-step process involves treatment of the cotton fabric with an aqueous solution containing oxidant and chitosan.
Conditions used for oxidation of cotton fabric: [$NaIO_4$], 50 mg/100ml ;time,1hr; temp,60 °C, M/L, 1:50
Condition used for chitosan treatment: [chitosan], 2%; temp, 80 °C ; M/L 1:50
Condition used in one -step process: [$NaIO_4$], 50 mg/100ml; [chitosan], 2%; temp, 80 °C ; M/L 1:50

Table 5. Comparison among substrates I, II, III.

A comparison among the substrates I , II and III with respect to nitrogen content, tensile strength and elongation at break and carbonyl content as shown in table 5 would reveal that substrate II exhibits the carbonyl content and the lowest tensile strength as compared with substrates I and III; indicating the determinal effect of oxidation prior to chitosan treatment. On the contrary, substrate III acquires the highest nitrogen content and the highest tensile strength and elongation at break while retaining a carboxyl content which is equal to that of substrate I. This, indeed, signifies the advantages of the one-step process in avoiding prior oxidation entailed in the two-step process.

Presence of chitosan during oxidation of the cotton fabric with $NaIO_4$ seems to protect the cotton against oxidation. At the same time $NaIO_4$ oxidizes chitosan to produce chitosan products with better solubility and more uniform structure and, in turn, better film-forming properties. Such properties will be reflected on strength of the film and the extra strength brought about thereof when the cotton fabric is coated with this film. Achieving substrate III with its potential properties is regarded to be the most salient output of the current work.

Dyeability

Substrate I, substrate II and substrate III were dyed independently with reactive and acid dyes. The results obtained are shown in table 6. It is observed that regardless of the dye used. Substrate III exhibits colour strength which is higher than those of substrate II. This again signifies the superiority of substrate III which, indeed, together with its other properties (table 5) advocate the one-step process for preparation of innovative modified cotton fabrics-containing chitosan.

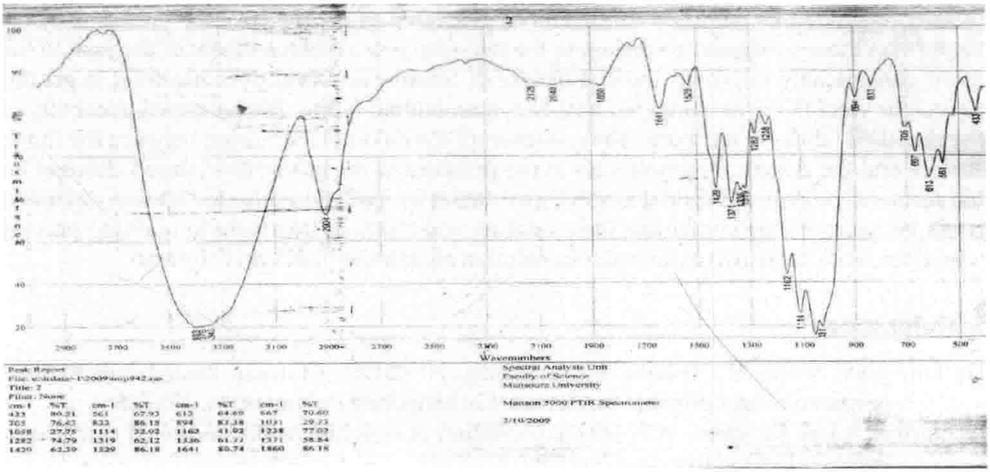
Dye	Colour strength (K/S)		
	Substrate 1	Substrate II	Substrate III
Reactive dye	16.4	22.78	23.05
Acid dye	2.8	4.47	4.49

Substrate 1: Bleached cotton fabric
Substrate II: processed as per the two-step process
Substrate III: processed as per the one-step process

Table 6. Dyeing of the different substrates under investigation with reactive and acid dyes

IR analysis

The IR spectrum of substrate II, as shown in figure 1, discloses the presence of a broad band at 3300-3500 cm^{-1} attributable to the NH_2 and OH groups , a weak absorption band appeared at nearly 1730 cm^{-1} due to the stretching vibration of the C=O double bond of the aldehydic group and a strong absorption band at 1641 cm^{-1} which is assigned to the C=N group was formed between the aldehydic group and chitosan. Also the IR spectrum of substrate III, as shown in figure 2 reveal the presence of a broad band at 3300-3500 cm^{-1} attributable to the NH_2 and OH groups , a strong absorption band at 2644 cm^{-1} which is assigned to the C=N group and a weak absorption band at nearly 1730 cm^{-1} foe C=O group.. These IR spectrums confirm the presence of chitosan in both substrates. That is, chitosan interacted with preoxidized cotton fabric as well as with cotton fabric subjected to concurrent oxidation and chitosan treatments.



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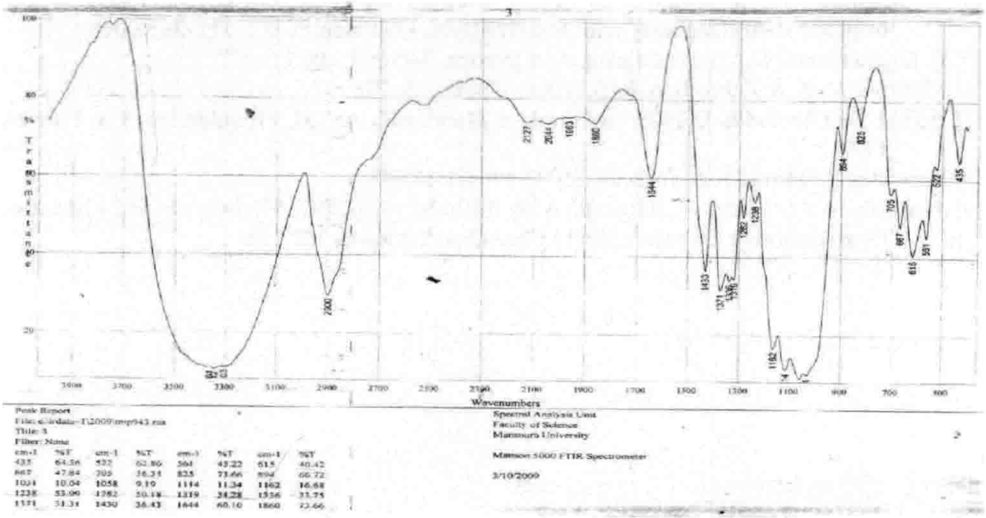
cm-1	%T	cm-1	%T	cm-1	%T	cm-1	%T
433	80.21	561	67.93	613	64.69	667	70.60
705	76.43	833	86.13	894	83.38	1031	29.73
1058	27.75	1114	32.02	1162	41.92	1238	77.02
1282	74.79	1319	62.12	1336	61.77	1371	58.84
1420	63.39	1529	86.18	1641	80.74	1860	86.18

Spectral Analysis Unit
Faculty of Science
Mansoura University

Mattson 5000 FTIR Spectrometer

3/10/2009

Fig. 1. IR spectrum of substrate II



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cm-1	%T	cm-1	%T	cm-1	%T	cm-1	%T
435	64.56	522	62.86	561	43.22	615	40.42
667	47.84	705	56.31	825	73.66	894	66.72
1031	10.04	1058	9.10	1114	11.34	1162	16.68
1238	53.90	1282	50.18	1319	34.28	1336	33.75
1371	31.31	1430	36.43	1644	60.10	1860	73.66

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Fig. 2. IR spectrum of substrate III

4. Conclusion

Chemical modification of cotton cellulose in the fabric form was effected through periodate oxidation treatment and chitosan treatment in the consecutive steps under different conditions. A single-step process was also devised for preparation of the same modified cotton. The idea in both cases was to create functional groups in the molecular structure of cotton such as aldehyde and carboxyl groups to expedite strong interactions with chitosan. Modified cotton fabrics processed as per the two processes were monitored, nitrogen content, carbonyl content, tensile strength and elongation at break in addition IR spectra.