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ORIGINAL ARTICLES

Chitosan Induced Bactericidal Properties And Improved Printability to cotton fabrics

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ABSTRACT

Chitosan at concentration up to 4%in 1% acetic acid was chemically attached to cotton fabric using citric acid (8g/l) as a crosslinking agent in presence of sodium hypophosphite (4g/l) as a catalyst. The application was practiced as per the pad-dry-cure method. Fabric padded in the chitosan solution to 100% wet pick up, dried at 85°C for 5 minuets then cured at 170°C for 2 minuets. Thus, fixed chitosan would be expected to perform dual function: it imparts antibacterial activity to the fabric meanwhile it enhances the response of the fabric towards reactive printing. Indeed such dual function was evidenced by the antibacterial activity of the chitosan-containing fabric towards Gram-positive (S.aureus) and Gram-negative (E. coli) bacteria, along with higher color strength and overall fastness properties. Surface characteristics of the fabric before and after treatment with chitosan were also examined using SEM.

Key words: Chitosan, cotton, antibacterial, color strength.

Introduction

Chitin is a polysaccharide found in the outer skeleton of arthropods; the later encompasses insects, crabs, shrimps, and lobsters (Elnagar *et al.,* 2012 and Huang e*t al.,*2008). It is the second most plentiful, naturally occurring polymer after cellulose (Li *et al.,*2002). Chitosan is derived from Chitin; it is prepared by partial deacetylation of chitin (55-60%) (Kuo-Chein *et al.,*2008). Chitosan has a high molecular weight, usually tens of thousands or even millions of kDa. Chitosan can be hydrolyzed into low molecular weight oligosaccharides. The nitrogen content of chitosan amounts to(6.89%) .Chitosan comprises copolymers of glucosamine and N-acetyl-glucosamine (Li, Dunn, Grandmaison& Goosen, 1997). It has attracted considerable interests by virtue of their biological activities such as antimicrobial (Lim & Hudson, 2003). Chitosan is inexpensive, non-toxic, biodegradable and possessing reactive amino groups. It has been useful in many areas of applications, such as wastewater treatment, food and recently in drug industry and as a hydrating agent in cosmetics. (Chung, Lee, & Kim, 1998; Davidson & Xue, 1994; Kim, Choi, & Yoon, 1998; Rippon, 1984 and Tsuragai, Yoshikawa, Tajima, & Ishii, 1991). The molecular structure of chitosan is also similar to that of cellulose. Chitosan is usually applied as a cotton finish to produce fabrics with improved properties as well as to confer some properties such as bactericidal. As a matter of fact, this has become an area of increased research interest (Zhao *et al.,*2003 and Knittel *et al.,*2006).

In current work, cotton fabric is rendered bactericidal via treatment of the cotton fabric with chitosan. This chitosan-treated fabric displays also better reactive printing properties than the untreated fabric. Furthermore, treatment of cotton fabric with chitosan alters the surface characteristics of the fabric as revealed by scanning electron microscopy

2. Experimental:

2.1 Materials:

Cotton: Mill desized, scoured and bleached cotton fabric (130 g/m^2) produced by Misr/ Helwan for Spinning and Weaving Company, Egypt. Chitosan (LMW) (Fluka), citric acid(CA),sodium hypophosphite(SHP), Espycon 1030 (non-ionic detergent), (C.I reactive Red 84) Ciba Co., sodium alginate, urea, acetic acid, sodium carbonate and caustic soda were of chemical grade.

2.2 Methods:

2.2.1 Chitosan treatment:

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Different concentrations of chitosan were prepared (0-4%) using 1% acetic acid for dissolution. Then the samples were padded in previously prepared chitosan solutions each containing (8 g/l CA and 4 g/l SHP). Squeezing the treated samples to a wet pick-up of 100% and dried at 85^{o}C for 5 min, then cured at 170^{o}C for 2 min. The treated samples were washed several times with cold water, then dried at ambient condition (temperature of 25±2^{o}C and relative humidity 65%±5).

2.2.2 Printing with Reactive dye:

2.2.2.1 Preparation of printing paste:

Application of screen printing technique with Reactive Red 84 dye and the following printing paste.

Thickening agent (Sod. alginate suspension)	500 g
(C.I Reactive Red 84)	30 g
Urea	100 g
Sodium carbonates	30 g
Water	340 g
	1000 g

Fixing the printed samples using thermofixation at 160˚C for 5 minutes , then washing the printed samples with cold water ,then soaping with 2 g/l Espycon 1030 at 45˚C for 15 minutes , rinsing with cold water and finally drying at 100°C prior to assessing for color strength (K/S) and over all fastness properties..

2.3. Testing and Analysis

Scanning electron microscopy:

Examination of the surface morphology of the chitosan-cellulose blend films using a JEOL–840X scanning electron microscope, from Japan, magnification range 35–10,000, resolution 200 Å, acceleration voltage 19 kV. The films were deposited onto a copper holder with conductive carbon paint and coated with gold under vacuum before observation

The nitrogen content:

It is measured according to Kjldahle's method. The following equation was used to calculate the nitrogen content on sample weight (Wt).

$$N\% = \frac{0.014 \times N \times V \times 100}{Wt}$$

Where; V= Volume of HCl; N= Normality of HCl and Wt= Sample's weight.

Antibacterial Activity:

To evaluate the antibacterial activity for gram-positive (S.aureus) and gram-negative (E. coli) bacteria we have used AATCC 6538 and AATCC6538p methods. Six samples named S1-S6 represent samples with different chitosan concentration (0 up to 4%), mixing 0.5 gm of each sterilized samples with 100 ml of 10^6 CFL/ml bacteria solution respectively. Putting the mixture in a conical flask and cultured in a shaker incubator (Shang-Mei, CWF) at 37°C for 24hrs.with 120 rpm. S1 was used as the control sample, where S2-S6 samples were considered as the test samples. We diluted the bacterial culture solutions by a factor of 10^4 using the ten-time dilution method, then, 1 ml of each diluted bacterial solutions mixed with 15 ml nutrient agar and placed into Petri dishes. After mixing and coagulation, the samples were cultured at 37°C for 24hrs; the colony count on the plate was then calculated using the following formula:

Percentage of viable cell = K/K_0:

Where K_0 is the control colony count and K is S2–S5 colony count.

Determination of fastness properties:

The treated samples were washed as per the conditions specified in the test AATCC test method [1].The color fastness to rubbing, to perspiration and to light were determined according to the AATCC test methods.

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Determination of color strength:

The color strength of the treated and untreated samples was evaluated by Hunter Lab Ultra scan PRO at λmax.

Results And Discussions

3.1 Morphology of chitosan/cellulose blends:

Fig.1 shows the SEM images that magnify 100 times the treated samples. Where S1 sample does not contain chitosan and shows cavernous surface while the S2 (1% Chitosan) sample shows a smoother surface. The S5 sample containing (2% chitosan) exhibits the smoothest surface. On the contrary, when the chitosan increased into 4% as in S6 , a subsequent coarse surface appeared again. In case of films produced by the high-pressure method, the water molecules in the solvent will vaporize immediately after the pressure is relieved and a cavernous structure waked form on the S1 surface. As chitosan content in blend films increases, their surface tends to become smoother. Since the molecular weight of chitosan is up to 960 kDa, but that of cellulose is only 178 kDa, the change of viscosity of viscous films caused by chitosan cannot be overlooked. When the viscosity of cotton films increases, it will suppress the water molecules from vaporizing, causing S3 and S5 to lose the cavernous structure and their surface to become smoother. However, the surface of S6, which contains more chitosan, becomes coarse again probably because chitosan agglomerates and causes phase separation.

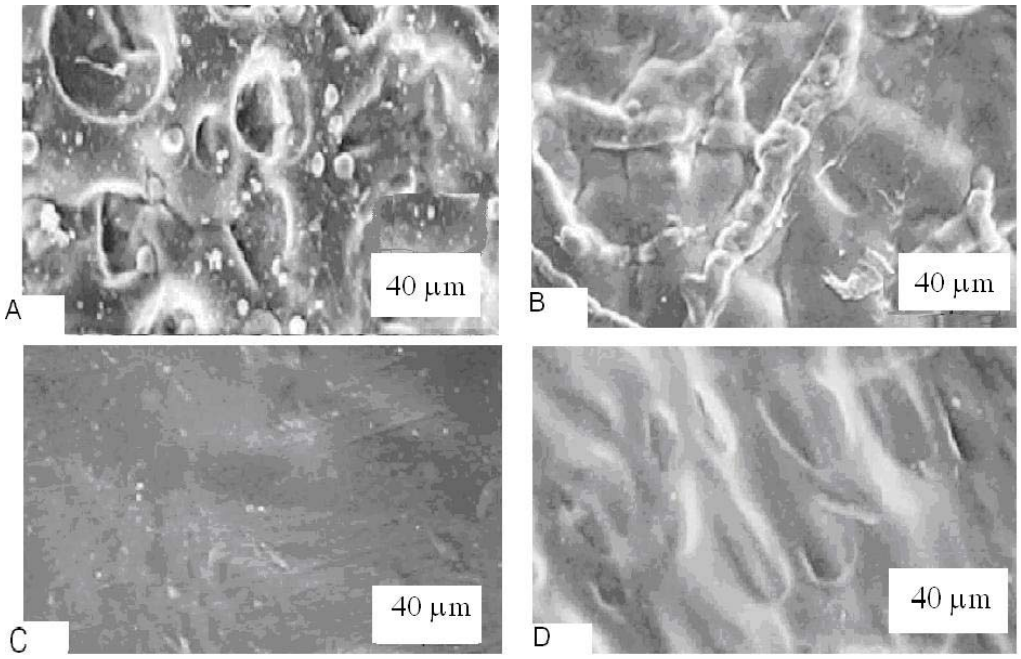

Fig. 1: SEM images for cotton samples treated with different chitosan concentrations a)S1 (0 % chitosan), b) S3 (1% chitosan), c) S5 (2% chitosan) and d) S6 (4% chitosan) (o.w.f).(magnification 100 time).

3.2 Nitrogen Content:

The theoretically N content of chitosan is 6.89%. This nitrogen content is attributed to the presence of amide and amino groups in chitosan molecules. Table 1 shows the N content of cotton samples treated with different concentrations of chitosan.

Table 1 shows the chemical properties of treated cotton fabrics where, increasing the chitosan concentration up to 2% is concomitant with increasing the nitrogen content. This returns to the increase in the viscosity of the padding solution, decreasing the crosslinking efficiency and different penetration into the cellulose matrix [K.F. El-tahlawy et al.2005-M.M.G. Fouda et al.,2009] Also, chitosan agglomeration explains the phase separation**.**

Table 1: Relation between chitosan concentration and nitrogen content

Chitosan concentration % (o.w.f)	N %
0	0
0.5	0.542
1.0	0.914
1.5	1.080
2.0	1.630
4.0	1.080

CA (8 g/l), SPH (10 g/l), pick up 100%.

3.3 Antibacterial activity:

Table 2 shows the antibacterial activity of the treated cotton fabric at different chitosan concentrations (0.5-4.0%). All samples showed inhibition zones that reveal the antibacterial activity and this is attributed to the amino groups found in chitosan, which acquire an effect on the inner cell membranes and organelles such as mitochondria and thus killing bacteria [Nasr *et al.,* 2009]. The best effect found at 2%.

Table 2: Effect of chitosan concentration on Antibacterial activity

Chitosan Concentration (%)	Antibacterial behavior	
	G(+)	G(-)
0	-	-
0.5	+	+
1.0	++	++
1.5	++	++
2.0	+++	+++
4.0	++	++

G (-) gram negative (Escherichia Coli); G (+) gram positive (Staphylococcus aurous) Sign (+) means that the sample shows inhibition zone and it has antibacterial activity.

3.4 Fastness properties and color strength:

The fastness properties of sample treated with 2% chitosan as well s those of the untreated samples as shown in table 3. As is evident, the chitosan-treated fabric displays an enhancement in fastness properties especially light, rubbing, washing and alkali perspiration fastness, in addition to higher color strength (K/S increased from 9.1 to 11.8) as compared with the untreated fabric.

Table 3: Fastness properties of treated cotton fabric with and without chitosan

Fastness properties			Untreated samples	Treated samples
Rubbing	wet		3-4	4-5
	dry		4	4
Washing	Alt.		3-4	4
	St-c		3-4	4-5
	St-w		3-4	4-5
Perspiration	alkali	Alt.	4	4-5
		St.	4	4-5
		St.	4	4-5
	acid	Alt.	4-5	4-5
		St.	4-5	4-5
		St.	4-5	4-5
Light fastness			6	7
Color strength			9.1	11.8

Chitosan concentration 2%.

Conclusion:

Bactericidal properties along with improved reactive printability could be imparted to cotton fabric by treatment of the latter with chitosan. Such dual action of chitosan-containing fabric is a manifestation of the biological activity of chitosan, which is concomitant with provision of amino and hydroxyl groups in the molecular structure of cotton cellulose of the fabric. These amino and hydroxyl groups afford additional reactive sites on the cotton backbone thereby enhancing its reaction with the reactive dye during printing. As a result of such dual function the chitosan-treated fabric exhibits antibacterial activity. Meanwhile this same fabric displays higher color strength and overall fastness properties than the untreated fabric. It is as well to emphasize that treatment of cotton fabric with chitosan changes the surface characteristics of fabric as shown by SEM images.

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