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Surface Modification of Wool Fabrics Using Chitosan Nanoparticles Before Dyeing with Synthesized Direct Dye and Antimicrobial Activity Evaluation

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Abstract

Pretreatment of wool fabrics with chitosan and chitosan nanoparticles (1%, 2%, 3%) with synthesized direct dye is used to enhance the exhaustion of dye, fastness properties, and antimicrobial activity of dyed fabrics. Also, we use the ultrasonic technique for wool dyeing to reduce the temperature and time of dyeing. The results assessed for dyeing and antimicrobial activity indicate high-quality dyeing properties, However, direct dyes showed higher exhaustion and fixation values, fastness properties, and the colorimetric CIE L*a*b* C*h° data of the dyed wool fabrics. Also, these direct dyes have higher antibacterial activity toward Gram-positive and Gram-negative bacteria therefore they can be used in biomedical applications.

Keywords: chitosan; Antibacterial activity; direct dyes; fastness; Dyeing; wool; bacteria

1. Introduction

Chitosan is a polysaccharide-based cationic biopolymer. It has been studied for its effect on the dyeability of wool fabrics in recent years. Julia proposes a method for improving the dyeability of wool fabric with reactive dyes that involve pre-treating the fabric with an oxidizing agent and then applying chitosan to the fabric. Other researchers reported that the dyeability of wool fabrics pre-treated with chitosan/nonionic surfactant mixture improved, as did colour strength with reactive dye [1-10]. Chitosan, a cationic (1-4)-2-amino-2-deoxy- -d-glucan partially acetylated to a typical extent of 0.20, is manufactured industrially from marine chitin, a linear (1-4)-2-acetamido-2-deoxy-d-glucan. Both have attracted attention due to their superior characteristic properties, including biocompatibility and lack of toxicity. Because of their favourable properties such as affinity, fabric compatibility, and water solubility, chitosan and chitosan derivatives are polymers of interest in cotton coloration [11-16].

In the textile industry, chitosan nanoparticles usage is a relatively new material. Nanoparticles possess unique properties, such as a large ratio of surface to volume, surface-active multi-centers, and high surface reactivity [17-22]. The advantages of chitosan and nanomaterials have emerged as chitosan nanoparticles with excellent physicochemical properties. It is bioactive and frequently used in many industrial areas including textiles [23-29]. It has been previously reported that chitin nanofibers can be grafted onto cotton fabrics, with or without surface modification, to achieve special properties such as improved moisture management [30-32]. Alkaline deacetylation was used to create nano chitosan from crab shell chitin nanofibers. The nano chitosan was dyed with acid blue 25 (2 anthraquinonesulfonic acid) and used as a colourant for cotton fabrics [33, 34].

In addition, the polyester fabric was alkali-treated and nano-chitosan-coated. Following that, all samples were dyed with reactive dyes. The water absorption and bending resistance of pristine polyester, alkaline polyester, and chitosan-treated polyester were determined. FTIR/ATR was used to investigate

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surface chemical bonding. [35]. The effect of chitosan nanoparticles on the dye-ability of silk fabric is studied using three different methods: pre-treatment, post-treatment, and simultaneous. The silk samples are dyed with mono and bifunctional reactive dyes after being treated with different concentrations of chitosan nanoparticles [36-39].

Nanotechnology offers a novel approach to improving the antimicrobial activity of textile fabrics. Wool fabrics were dyed with a natural dye extracted from red prickly pear using microwave heating. Chitosan-Propolis nanocomposite was applied as the treatment on wool fabrics before dyeing by using microwave and ultrasonication methods [40-43]. Wool fabrics were pretreated with chitosan and chitosan nanoparticles before being dyed with a saffron red and yellow mixture as a natural dye using a microwave heating method [44-47]. To impart antimicrobial properties, previously synthesized reactive dyes were applied to wool fabrics along with varying degrees of molecular weight chitosan [48-50]. The effect of chitosan application on the dyeing properties of wool fabrics was investigated by measuring the colour strength (K/S) values of the treated substrates at different chitosan and dye concentrations. The treated fabrics have higher antimicrobial activity than the untreated fabrics, and the chitosan treatment improves the antimicrobial properties of the dyes [51-56].

In our study, wool fabrics were dyed with synthesized direct dyes after being treated with chitosan at various concentrations to improve dyeability and impart antimicrobial activity.

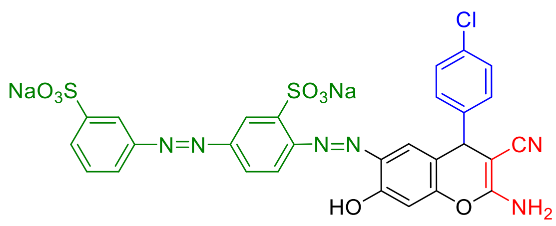
2. Material and methods

2.1. Materials

Chitosan (high molecular weight) (Aldrich). Chitosan nanoparticles were purchased from (Aldrich). All chemicals used in this study were of laboratory grade.

2. 2. Synthesis of dye

We synthesized direct 2-amino-4-(4-chlorophenyl)-7-hydroxy-4H-chromene-3-carbonitrile was synthesized according to the literature procedure (Makarem , 2008) as briefly in a one-pot reaction, a solution of 4-chlorobenzaldehyde (1.41g, 0.01 mol), malononitrile (0.73g, 0.01 mol), and resorcinol (1.10g, 0.01 mol) were reacted together in EtOH (20 ml). The reaction mixture was reflexed for 4 hours to complete the reaction by monitoring TLC. 4-Aminoazobenzene-3,4'-disulfonic acid diazotized in hydrochloric acid with sodium nitrite, followed by coupling reaction with compound **1** in 1:1 molar ratio at room temperature and until the pH is fixed at 5. Finally, the diazo direct dyes (**D) was** formed named 2-(2-amino-4-(4-chlorophenyl)-3-cyano-7-hydroxy-4H-chromen-6-yl)diazenyl)-5-((3-sulfophenyl)diazenyl)benzenesulfonic acid (D) [57].



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2.3. Characterization and analysis

UV-Vis spectra have been used to confirm the formation of madder dye nanoparticles. The spectra were collected over a range of 250-800 nm. The dyed fabrics were tested according to AATCC and ISO standards [58, 59]. The colour strength values (K/S) were determined using CIE Lab: D-65 10 standard. The ISO-CO6 D1M was used to determine the colour fastness to laundering. The colour fastness to laundering, perspiration and rub were measured using AATCC-15, AATCC-8, and AATCC-16 standards respectively. The test specimen and the two adjacent fabrics (cotton and wool) were compared using a grayscale. The rating scale of wash fastness for color change was from 1 (very poor), 2 (poor), 3 (fair), 4 (good) to 5 (excellent). The rating scale of lightfastness was from 1 (very poor), 2 (poor), 3 (fair), 4 (moderate), 5 (good), 6 (very good), 7 (excellent), to 8 (outstanding).

2.4. Evaluation of Antibacterial activity

2.4.1. Materials

Two bacterial strains were *E. coli* ATCC 11229 (Gram-negative) and *S. aureus* ATCC 6538 (Gram-positive). These bacterial strains were selected as test cells because they are the most frequent bacteria in wound infection. Fresh inoculants for antibacterial assessment were prepared on nutrient broth at 37°C for 24 hours.

2.4.2. Test methods

All antibacterial activity tests were made in triplicate to ensure reproducibility. The antibacterial activity of the dyed fabrics against *S. aureus* and *E. coli* was evaluated by using the colony counting method [16, 43] where; a liquid culture was prepared by mixing 0.5 g peptone and 0.3 g beef extract in 100 ml water. 1 cm diameter blended film samples were cut and put into 10 ml of liquid culture, to which 10 µl of microbe culture was inoculated. All samples were incubated for 24 h at 37°C. From each incubated sample, 100 µl of the solution was taken, diluted, and distributed onto an agar plate. All plates were incubated for 24 hours and the colonies formed were counted [41].

The percentage bacterial reduction was determined as follows:

$$Reduction\ in\ CFU(colony\ forming\ units)\% = \frac{C-A}{C}\ x100$$

Where A is CFU/ml after contact (end test) and C is CFU/ml at zero contact time.

3. Results and discussion :

3.1. Effect of treatment with chitosan and nano chitosan for wool fabric dyed with direct dye by Conventional method

The results in figure 1 indicated that the samples treated with nano chitosan and dyed with the synthesized direct dye exhibit higher absorption than that treated with chitosan [57, 60]. The results also indicated that the treatment with nano chitosan 2% gave a higher absorption wavelength than 1% and 3% concentration as shown in Fig. 1.

3.2. Effect of pH on untreated wool and treated with chitosan and nano chitosan dyed with direct dye by the ultrasonic method:

The results in Fig. 2 indicated that the samples treated with nano chitosan and dyed with the synthesized direct dye exhibit higher absorption than that treated with chitosan at the same pH [51]. the results also indicated that the treatment with nano chitosan 3% gave a higher absorption wavelength than 2 and1% % concentration at pH 5 and higher than the values at pH 4 as shown in figure 2.

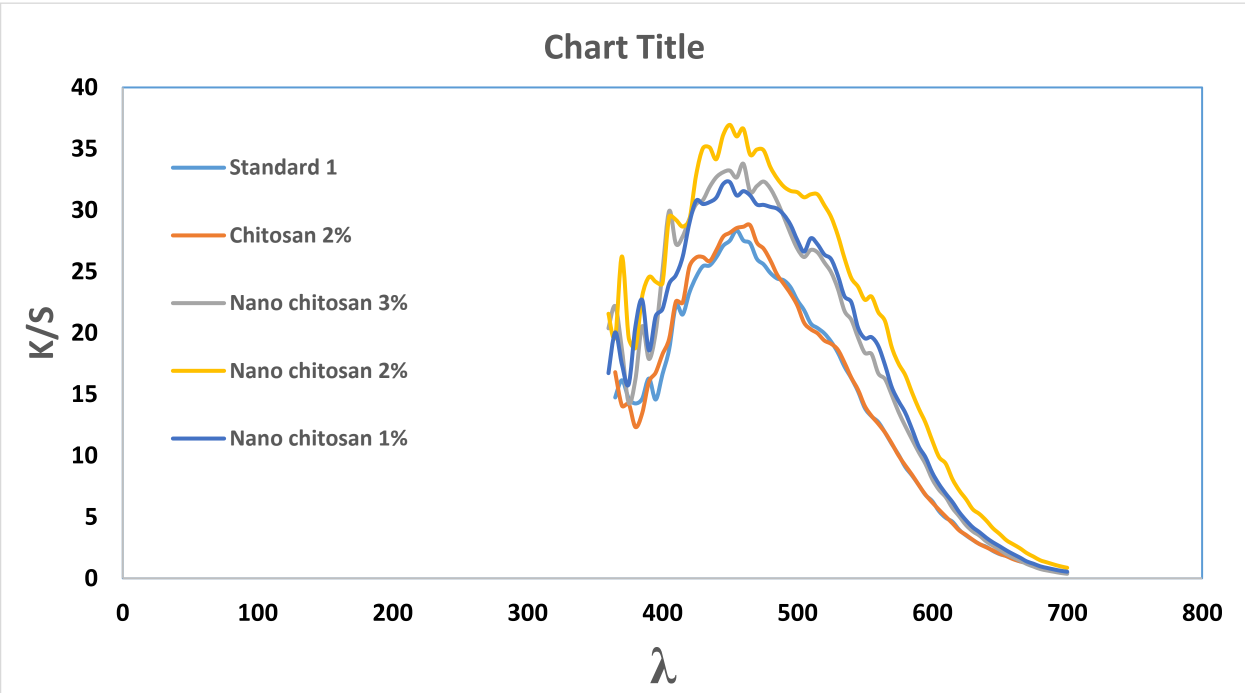

Fig. 1. Effect of wavelength on K/S for wool dyed with direct dye by Conventional method

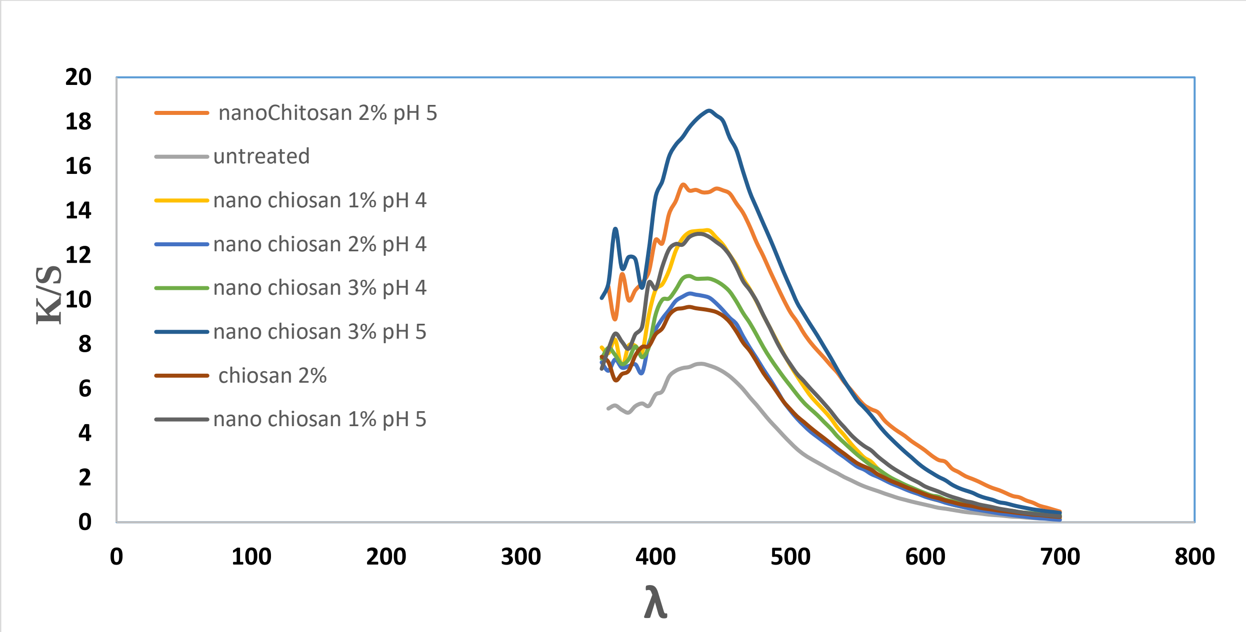

Fig. 2: Figure 1: Effect of wavelength on K/S for wool dyed with direct dye by ultrasonic method

3.3. Colorimetric data of the dyed wool fabrics using direct dye at pH4 by Conventional method

The colorimetric CIE L*a *b*C*h data were evaluated for dyed wool fibers with the conventional method as shown in T**able 1**. The color strength changes remarkably as different concentrations of chitosan and nano chitosan were used while the Lab values show that samples treated with 2% nano chitosan are darker in shades. The results obtained revealed that the pretreatment with chitosan and nano chitosan enhanced the leveling properties [61].

3.4. Colorimetric data of the dyed wool fabrics using direct dye by Ultrasonic method:

The colorimetric CIE L*a *b*C*h data were evaluated for dyed wool fibers with the ultrasonic method **at pH4,5** were shown in T**able 2**. The color strength changes remarkably as different concentrations of chitosan and nano chitosan were used while the Lab values show that samples treated with 3% nano chitosan are darker in shades compared to the untreated. The results obtained revealed that the pretreatment with chitosan and chitosan nanoparticles enhanced the leveling properties [62].

Table 1. Colorimetric data of the dyed wool fabrics using direct dye at pH4 by Conventional method

Fabrics	K/S	L*	a*	b*	dE*
Sample 1; (Chitosan 2%)	28.77	26.15	21.72	20.10	0.48
Sample 2; (chitosan nanoparticles 3%)	33.78	22.24	21.00	16.20	5.28
Sample 3; (chitosan nanoparticles 2%	36.63	19.18	17.29	12.24	11.15
Sample 4; (chitosan nanoparticles 1%	31.55	21.52	20.28	14.40	7.18

3.4. Fastness Properties of Printed Fabrics

Table 3 shows the fastness properties of dyed wool (W) fabric at pH 4 using direct dye D at the conventional method. These data show that all dyed samples have very good to excellent of fastness properties towards the light, rub, perspiration and washing.

Table 4 shows the effect of pH change on the fastness properties of the dyed fabrics. At pH 4 they show very good fastness properties whereas at pH 5 they show excellent fastness properties.

Table 2. Colorimetric data of the dyed wool fabrics(W) using direct dye by Ultrasonic method

Fabrics	K/S	L*	a*	b*	dE*
Sample 5; (Chitosan 2%)	14.94	35.65	14.73	23.86	12.59
Sample 6;untreated	7.10	53.61	18.00	36.32	32.35
Sample 7; chitosan nanoparticles 1% pH 4	13.03	45.04	23.97	36.39	25.33
Sample 8; chitosan nanoparticles 2% pH 4	10.28	47.34	19.37	34.49	25.99
Sample 9; chitosan nanoparticles 3% pH 4	11.07	44.85	19.92	32.66	22.86
Sample 10; chitosan nanoparticles 3% pH 5	17.75	36.60	20.38	29.08	14.16
Sample 11; chitosan nanoparticles 2% pH 5	9.68	46.33	18.11	32.02	23.96
Sample 12; chitosan nanoparticles 1% pH 5	12.82	42.17	20.32	31.36	19.90

Table 3. Fastness properties of dyed wool (W) fabric at pH 4 using direct dye D at conventional method

Dye	Treated Fabric W	Fastness to Wash			Fastness to Perspiration						Fastness to Rubbing		Light
					Acidic			Alkaline					
		SC	SW	Alt	SC	SW	Alt	SC	SW	Alt	Dry	Wet	
D	**Sample 1**	4	4	4	4	4	4	4	4	4	4	4	4
	Sample 2	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4
	Sample 3	4	4	4	4	4	4	4	4	4	4	4	4
	Sample 4	4	4	4	4	4	4	4	4	4	4	4	4

SC, staining on cotton**; SW,** staining on wool**; Alt,** color change of the dyed sample**.**

Table 4. Fastness properties of dyed wool (W) fabric at pH 4 and pH 5 using direct dye D at ultrasonic method

Dye	Treated Fabric W	Fastness to Wash			Fastness to Perspiration						Fastness to Rubbing		Light
					Acidic			Alkaline					
		SC	SW	Alt	SC	SW	Alt	SC	SW	Alt	Dry	Wet	
D	**Sample 5**	4	4	4	4	4	4	4	4	4	4	4	4
	Sample 6	4	4	4	4	4	4	4	4	4	4	4	4
	Sample 7	4	4	4	4	4	4	4	4	4	4	4	4
	Sample 8	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	Sample 9	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	Sample 10	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	Sample 11	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5
	Sample 12	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	5

SC, staining on cotton**; SW,** staining on wool**; Alt,** color change of the dyed sample

3.5. Antibacterial Activity

Table 6 illustrates that chitosan with 2 wt.% concentration shows antibacterial towards both Gram-positive and Gram-negative bacterial due to conversion of amino groups into ammonium ions that can attach to the bacterial cell causing bacterial death [15, 43]. In addition, wool fabrics treated with chitosan show higher antibacterial activity towards Gram-positive bacteria than Gram-negative bacteria due to bacterial cell wall structure [4, 15].

Also, wool fabrics treated with chitosan nanoparticles shows antibacterial activity at pH values 4 and 5 due to the large number of amino groups that converted to ammonium cations that can attach to bacterial cell wall causing bacterial death. In addition, antibacterial increased as both chitosan nanoparticles concentration and pH values increased [63].

Also, wool fabrics show higher antibacterial activity towards both Gram-positive and Gram-negative bacteria because of nanostructure and dimensionless nanoparticles that can penetrate bacterial cell wool for both Gram-negative and Gram-positive types [19, 53, 64].

4. Conclusion

The dyeability of treated fabrics increased with increasing chitosan concentration and imparted a high antibacterial action with high growth reduction, according to the findings of this study. Additionally, traditional methods lowered salt and alkalinity in treated materials, while ultrasonic approaches reduced not only salt and alkalinity, but also temperature and dying time. The K/S of treated fabrics is higher than that of untreated fabrics. Overall, Chitosan-poly (propylene imine) has the potential to be a new, efficient, and environmentally friendly finishing agent for multifunctional wool fabric treatment.

Table 6. Antibacterial activity expressed in reduction percent (%) of wool fabrics treated with chitosan (CS) and chitosan nanoparticles (CSNPs) via ultrasonic method Vs. conventional method

Sample No.	Reduction percent	
	S. aureus	*E. coli*
	%	
CS (2%) treated wool fabrics	77.54	43.27
CSNPs wool fabrics		
Conventional method		
1%	82.28	52.01
2%	87.51	61.82
3%	91.38	62.66
Ultrasonic treated method		
pH 4		
1%	85.13	75.79
2%	95.13	83.71
3%	96.34	93.67
pH 5		
1%	91.33	80.84
2%	95.74	87.72
3%	97.69	95.59

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