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Enhancement of Dyeability and Antibacterial Characteristics of Silk Fabrics Using Chitosan Nano-Particles

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THE present work was carried out to study the effect of prepared chitosan nanoparticles (CNPs) on the dye ability of silk fabric dyed with mono and bi functional reactive dyes. In order to obtain the optimum condition of using nano chitosan the silk fabric was treated with different concentration of nano-chitosan using three different methods (a)pre-treatment,(b)one bath process and (c)post-treatment. The colour strength (K/S) and colour parameters of dyed silk samples were measured for each method. The change in silk fabric surface morphology after using nano chitosan was examined with SEM. The K/S results of silk dyed samples treated with nano chitosan solution were higher comparable to the untreated samples.furthermore nanochitosan exhibit superior behaviour as antibacterial agent against both gram-positive and negative bacteria .

Keyword: Nano-chitosan, Silk, Antibacterial, Dyeing, Reactive dye.

Introduction

Silk fibres produced by silk worms have been discovered from 2700 BC. It consist of protein which consist of fibroin and sericin, a non-filamentous protein-and furthermore different polluting influences, for example, colours, wax, sugars, and inorganic salts. The sericin reinforces the silk fibre and makes it need gloss; in this way, it must be degummed before dying. [1-3]

Silk fibres have many advantage and used in different fields. [3-5]dyeing silk fibres have been widely studied using different dyes such as acid, metal complex and reactive dyes. Reactive dyes have extremely well known because of their brilliancy, give different hues, good wet fastness properties and advantageous utilization.[6-8]

In addition, a few properties of silk fibre, for example, wrinkle recovery, low maintenance properties, yellowing, water and oil staining resistance, colour capacity and fastness are powerless and must be studied to enhance them.

Therefore, development of silk surface using some physical and chemical processes have been studied for example, ɤ-Ray or plasma or UV initiated grafting, are being investigated as a physical methods for enhancing silk fibre properties.These methods are more important than chemical wet treatments since they are considered as clean, dry and eco-friendly methods.[9, 10]

One of the important chemical treatment for silk fibres is graft copolymerization which can enhance softness properties without losing whiteness or handle. Despite these points of interest, the chemical processes don't consent to ecological controls because of their producing of some toxic agents through processes due to using some monomers during the grafting process such as vinyl, methacrylate, acrylamide and fluoroacrylate monomers. [11-16]

Chitosan has been assessed for various textile application due to its chemical configuration and

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properties such as non-toxicity, hydrophilicity and antibacterial properties as man-made fibres, cotton,silk,wool and polypropylene dyeing, durable press, antimicrobial, antistatic, de-odourising finishing, sizing, printing and ink jet printing. [17]

Recently Chitosan nanoparticles have been used widely in textile application because it impart supriorer charactres than chitosan as increase in its bioactivity.[18-22]

The present work aims to study the efficiency of nano-chitosan to enhance dye uptake, colour measurement and colour fastness properties of dyed silk fabrics beside its antimicrobial characters using different types of reactive dyes.

Experimental

Materials

Silk fabrics (Natural silk (100%) was supplied by (Akhmem .Co) private sector.), mill-scoured and bleached were kindly supplied by Misr El-Beida Dyers Company, Kafr El-Dawar, Alexandria, Egypt

Chitosan of high M.wt. 600000 – 800000, with a viscosity of 800000 cps (Aldrich) was used. Six reactive dyes mono and bifunctional with different structure as given in **Table 1**. All other chemicals used in this work were of laboratory grad.

Preparation of nano chitosan

Preparation of nano chitosan was done according modified method from Jonoobi et al. [23] as followe: chitosan 2 wt.% was suspended in distilled water containning 1 % acetic acid using a mechanical blender at 500 rpm for 20 min. Then, the solution subjectud to ultrasonicator for 30 min at 35 power. After that it was used for further processes.

Treatment of silk fabric with nano chitosan

Silk fabric treatment using nano chitosan is carried out by three methods: (1) pre-treatment method, nano chitosan treatment was prior to silk dyeing. (2) In the second method the nano chitosan is incorporated in the dyeing bath of silk in one bath. (3) While in post treatment method the silk fabric is firstly dyed then finished with nano chitosan. Different amount of nano chitosan solution (0 - 20 %) were used through to study the optimum concentration given higher K/S.

Dyeing

Treated and untreated silk fabrics were dyed usingdifferent dyes by the conventional method. The dyeing bath was adjusted at pH 4,liquor ratio 1:40 and dye concentration 2%. The additive salt was ammonium acetate (2 g/l) and levelling agent (2 ml/l). Dyeing process temperature start at 45°C then raised to 100°C and samples were kept in the bath for 30 min. Dyed samples was then washed by warm water followed by cooled water and dried at room temperature.

Pre-treatment method

Samples of Silk fabric were treated with aqueous solutions containing different concentrations of nano chitosan (0- 20 ml) using material with liquor ration (1:40) and the temperature was set at 70°C for 30 min. The nano chitosan treated silk fabric was allowed to dry in the open air before dyeing with reactive dyes as described above.

One- bath method

In this method silk fabrics were treated with various nano chitosan solution concentrations to allow the interaction between silk fabric and the prepared solution then the dyeing process with reactive dyes was carried out under the previous conditions in the same bath.

Post- treatment method

The Silk fabrics dyed with reactive dyes were then treated with nano chitosan solutions using different ratios. After that the treated silk fabrics were thoroughly washed and dried as described before.

Characterisation

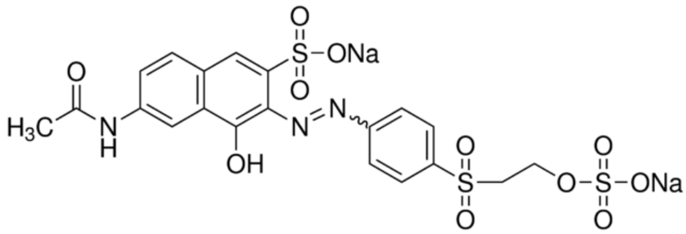
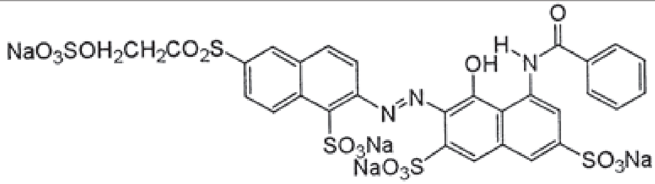
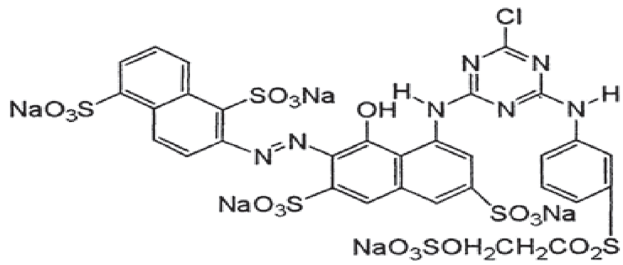
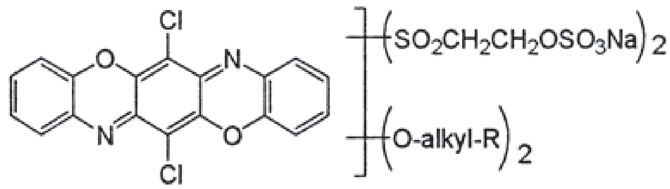
Colour measurements

The colour strength values of the treated dyed fabrics measured by reflection spectroscopy with a Hunter Lab UltraScanPRO USA, 2007 spectrophotometer according to a standard method at the wavelength of the maximum absorbance, expressed as K/S. [24-27]

Fastness properties [28-31])

The colour fastness to washing determined according to the AATCC Test method 61- 2007 [32] using Launder-Ometer. The colour fastness to rubbing (Dry and wet rubbing) determined according to the AATCC test method 8 - 2007. [33] The Colour Fastness to Perspiration determined according to the AATCC test method 15 - 2013. [34] Colour fastness to light determined according to AATCC test Method (16–2004). The evaluation established using the blue scale as reference of colour change. [35]

TABLE 1: Chemical structure of mono and bifunctional dyes

C . I . Reactive	Class	Structure
orange 16	Single azo mono functional	
Red 180	Single azo mono functional	
Yellow 145	Single azo bifunctional	
Red 195	Single azo bifunctional	
Blue 224	Triphenodioxazine bifunctional	

\Antibacterial Test Method

The disc diffusion method ([14, 36] used to evaluate the antimicrobial activity of treated and untreated silk fabrics. 10 mm discs of fabrics were placed onto the surface of inoculated Nutrient agar plates and incubated at 37°C for 48 h. The inhabitation zone was determined for each disc. Three parallel experiments were measured. The most of microorganisms used were ATCC registered strains except Bacillus cereus that was local isolate obtained from Agric. Microbiology Department, National Research Centre; Egypt. (*Streptococcus pyogenes* (19615), *Escherichia coli* (25922), *Aspergillus niger* (6275) and *Bacillus cereus)*.

Result and Discussion

Charachtrisan of prepared chitosan nanoparticles

The prepared chitosan particle size was confirmed by transmission electron microscope as **Figure 1a-b**presented homogeneous and uniform particle morphology in the range of 50 nm.

Effect of using different concentration of nano-chitosan on colour strength

The effect of nano chitosan solution concentration on both K/S and colour parameters (L, a, b, and ΔE) values of treated silk fabric dyed with red Reactive red 195 and Reactive orange 16 by using the three method (pre-treatment,

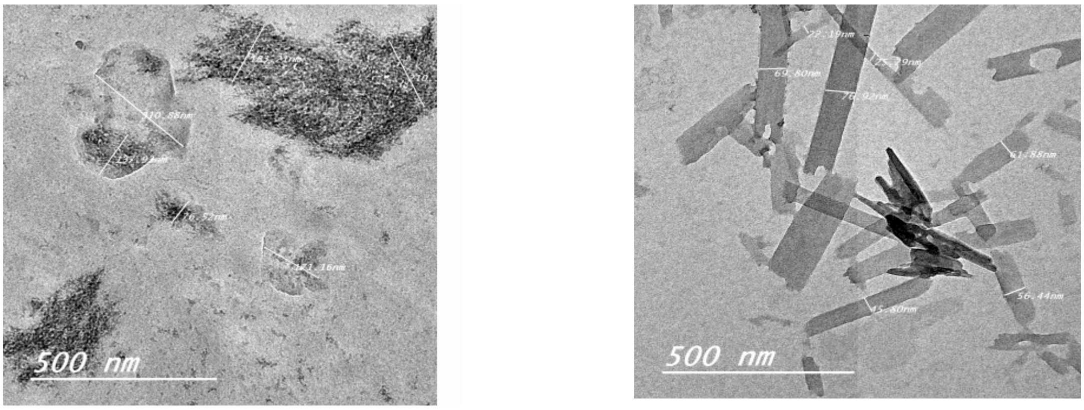


Fig. 1:TEM microscope image of chitosan particles (a) and chitosan nanoparticles (b)

one step bath and post treatment method) are shown in **Table 2**. From **Table 2**it can be seen that the K/S values of pre dyed samples treated with nano chitosan solution are higher than those of untreated dyed samples this is expected and has been proved in previous researches that the chitosan in normal size increase the rate of dye uptake and dye exhaustion in the case of acid, reactive dyes and natural dye as henna. [5, 37, 38]

Table 2 showed that the higher K/S value was obtained by adding 10 ml of nano chitosan solution this phenomena holds true regardless the treatment method, further increase in concentration after 20 ml leads to slightly decrease in the colour strength values.**Table 2** shows a colour strenght values for single – step (one- bath) process using different concentration of nano chitosan solution and two reactive dyes, and we can concolude that, the maximum K/S value was obtained by using (10 %) of nano chitosan concentration regardless to the type of reactive dye. Also, **Table 2** showed that by increasing concentration of nano chitosan more than (10 %) the K/S values has been decreased. In **Table 2** silk fabric samples were first dyed by mono-and bi functional reactive dyes, then dyed samples were treated with different concentration of nano chitosan in subsequent step.From **Table 2** it is obvious that the maximum K/S value was obtained for both used dye at (5 %) of nano chitosan concentration, and then K/S value decreased by increasing concentration of nano chitosan.

From **Table 2** it is observed that by increasing the conc. of nano chitosan up to (20 %) in case of pre- treatment method, up to (10%) in case of single – step process, and up to (5%) in case of post treatment process the K/S value decreased this may be due to the competitive nature between nano chitosan particles and dye molecules which leads to dye precipitation thus the silk fabric does not absorb dye evenly.

From the colour difference (ΔE) values which given in **Table 2** it can be seen that up to (20, 10 and 5 %) of nano chitosan concentration for pre-treatment process, one-bath process and post – treatment process respectively there is no significant colour difference (ΔE) between the samples by increasing of nano chitosan concentration.

It can be concluded from the previous data that silk fabrics can be dyed dyes used in this investigation, using various concentrations of nano chitosan solution via three methods as describe before under optimum conditions.The optimum concentration of nano chitosan used in pre and single-step treatment methods were 20%, and 10% respectively will be used for reactive dyes 180, 145, and 242, science in case of pre-treatment dyeing was carried out after treatment of sample by 20% of nano chitosan, while in single-step treatment methods (one- bath) treatment of sample by nano chitosan and dyeing take place in the same bath.

The influence of nano chitosan on colour strength (K/S) value were plotted in **Table 3**. It is obvious from these tables that, colour strength (K/S) value of treated samples higher than untreated samples, and this holds true for all dyes.

The optimum concentration of nano chitosan used in post-treatment methods were 5%

respectively will be used for Reactive red 180, Reactive yellow 145, and Reactive blue 224 dyes. **Table 3** shows the post-treatment methods, in this methods dyeing take place without interference between silk fabric and nano chitosan, hence the colour strength (K/S) value of treated samples less than untreated.

SEM of silk samples

In order to clarify the difference in the morphology structure of silk before and after treatment with nano chitosan the samples were subjected to SEM. **Figure 2a** state for the untreated silk fabric characterized by the smooth surface and lake of pores or cracks. **Figure 2b-d** state for treated silk samples with nano chitosan via different method conditions. Despite the treatment method, there is a formation of thin layer film on the fabric surface. The roughness of the silk surface cause increase dye ability and antibacterial properties of the silk yarns.

TABLE 2: Effect of different concentration of nono on colour strength values for silk fabric

Processes	nano chitosan%	ΔE		b		a		L		K/S	
		RD	OD	RD	OD	RD	OD	RD	OD	RD	OD
Untreated	0	0	0	66.45	75.42	22.01	5.52	6.75	14.30	0.03	0.04
Pre-treatment process	5	1.81	0.84	59.45	74.62	29.92	10.19	10.19	21.37	11.12	8.47
	10	2.27	1.04	56.97	73.54	32.86	12.29	10.91	24.41	15.01	12.27
	20	2.62	1.09	54.66	73.40	34.30	12.43	10.29	25.35	17.55	13.15
	30	2.60	0.95	55.99	73.97	32.16	11.99	10.05	23.45	14.28	11.04
	40	2.24	0.97	56.70	73.91	32.41	12.19	10.03	23.17	14.64	11.39
One- bath process	5	1.02	0.67	63.77	75.30	19.53	10.12	3.75	10.47	4.84	3.02
	10	3.99	2.92	49.25	76.99	36.90	21.76	9.27	40.67	22.91	2.12
	20	1.04	0.61	64.68	76.22	20.94	17.10	6.49	17.16	2.08	3.41
	30	0.68	0.58	69.16	76.73	16.20	6.98	5.03	16.97	6.62	3.75
	40	0.48	0.50	72.42	77.42	11.27	5.91	2.64	15.07	12.94	5.80
Post – treatment process	5	0.84	0.46	67.81	76.54	19.7	4.64	5.75	12.55	1.25	1.55
	10	0.80	0.45	67.89	76.80	18.59	4.42	5.16	12.36	1.42	1.24
	20	0.79	0.44	68.37	77.15	19.55	4.68	5.66	12.71	1.75	1.05
	30	0.78	0.44	68.80	77.10	20.12	4.63	6.04	12.62	1.95	1.05
	40	0.78	0.41	68.58	78.00	19.44	4.11	5.87	1.41	1.27	1.03

* RD: Red 195 OD: Orange 16

TABLE 3: Colour strength K/S, L, a, b, and ΔE value of silk fabrics using nano chitosan treatment and different reactive dyes via pre-treatment process.

Dyes used	Treatment process	K/S	L	A	B	ΔE
Reactive red 180	Untreated	5.81	48.58	49.02	-6.18	52.05
	Pre-treatment	9.99	44.85	54.81	-4.31	70.95
	One- bath process	9.35	46.06	55.42	-5.27	72.26
	Post – treatment	3.97	55.04	48.21	-8.72	73.69
Reactive Yellow 145	Untreated	2.39	76.29	11.61	47.88	87.22
	Pre-treatment	6.45	70.59	22.80	63.39	97.58
	One- bath process	6.13	71.83	22.04	64.87	98.74
	Post – treatment	2.45	76.93	11.78	49.00	91.52
Reactive blue 224	Untreated	1.25	63.36	-8.66	-19.59	51.59
	Pre-treatment	5.85	42.75	-2.29	-31.49	53.14
	One- bath process	6.62	40.93	0.63	-33.36	52.81
	Post – treatment	3.34	49.75	-6.87	-25.92	56.52

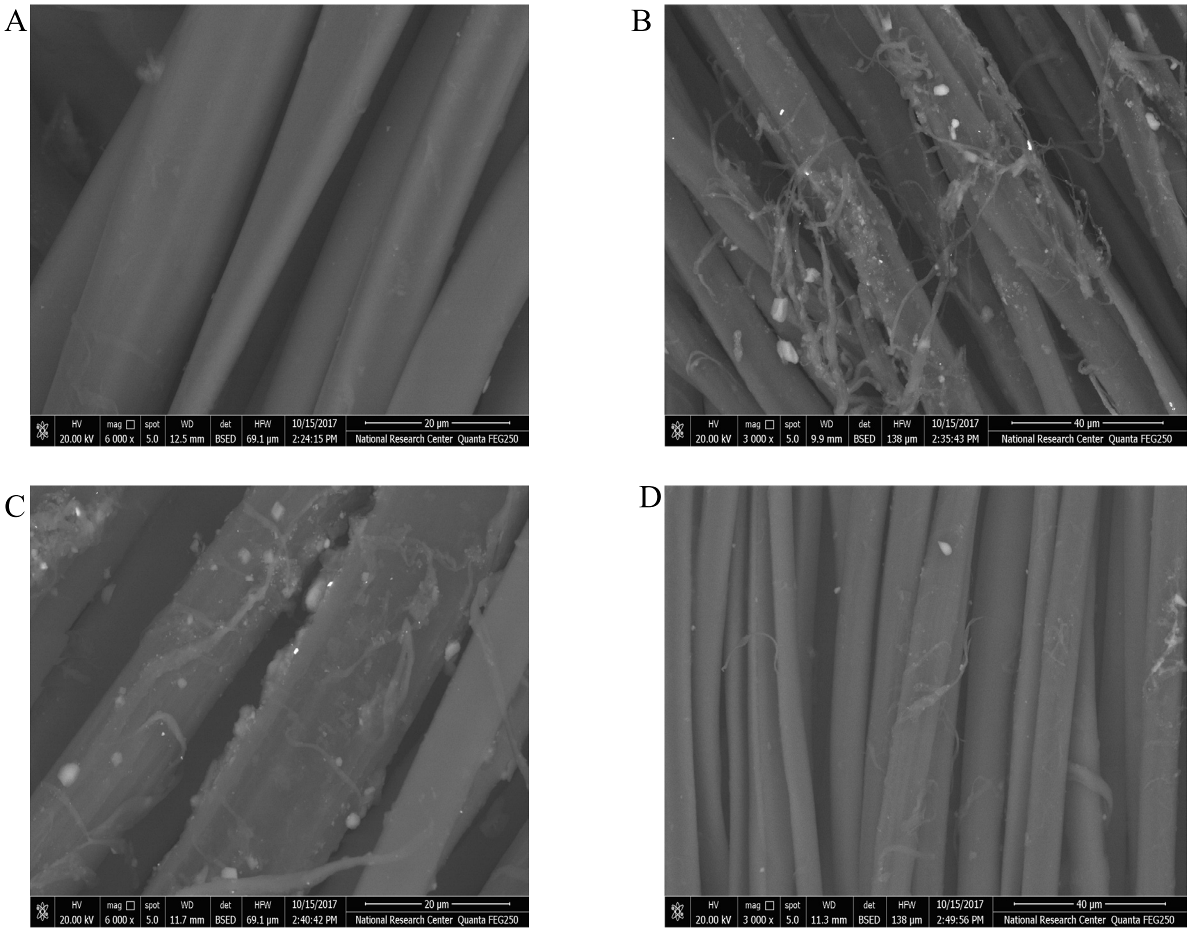


Fig. 2: SEM image of untreated and treated silk fabrics

a) untreated silk b) silk treated with nano chitosan by spontaneous method condition
c) silk pre-treated with nano chitosan d) silk treated with nano chitosan after dyeing

Agglomerates of nano-chitosan particlesis recognized at different positions of the fibre surface and also absent in certain places with distinctive edges, which may be due to the separation of the fibres that were in contact to each other during the nanochitosan coating formation during fabric handling.

Fastness properties

The dyed treated silk fabric with nano chitosan and mono or bi-functional reactive dyes were tested to washing, perspiration, rubbing, and light. The results are shown in **Table 4**, and from this table it is clear that there are improvements in fastness properties of reactive dyes used in this work.

Washing fastness

Treated silk fabric by nano chitosan dyed with mono and bi-functional reactive dyes have very good (4) fastness, while untreated silk fabric have humble (3-4) fastness comparable with treated silk fabrics.

Perspiration fastness

The fastness to both acid and alkaline perspiration were increased after treatment of silk fabric by nano chitosan. The fastness to acid perspiration was changed from (3-4) to very good (4) after treatment by nano chitosan. For alkaline perspiration a similar result was observed since the fastness increased from good (3) to very good (4) after treatment by nano chitosan.

Rubbing fastness

Dry rubbing test on the dyed silk fabrics were similar to washing fastness which gave very good after treatment by nano chitosan. The fastness to wet rubbing was improved after treatment by nano chitosan, science the fastness changed from good to very good.

Light fastness

From **Table 4**, it can be seen that Light fastness properties was also improved from good to very good by using of nano chitosan via pre-treatment method.

Antibacterial effect of nano-chitosan

The antibacterial finishing for silk textile goods to prevent or retard the growth of bacteria become required that silk are often considered to be more vulnerable to microbial attack owing to their hydrophilic structure and moisture transport characteristics. (30) [39] (39) Chitosan is an ideal candidate material as beside its antimicrobial activity is nontoxic, biodegradable and biocompatible natural polymer.

Several studies revealed that good antibacterial activity of chitosan was observed against Gram-negative bacteria than Gram-positive bacteria. [40-42] In this study the samples treated by nanochitosan exhibit superior behaviour as by decreasing the particle size of chitosan to nano size increase its permeability to cell membrane which causes death of the cell as shown in **Table 5**. Although all the tested samples has good antibacterial effect against both gram-positive and negative bacteria but the highest values of inhabitation zone were for the samples treated via post dyeing procedure. This expected as most of the protonated amine group (which is the responsible of the antibacterial activity) are consumed in the electrostatic bond with anion (Cl) of reactive dyes in the other two dyeing methods.

TABLE 4: Fastness properties of the untreated and treated silk fabrics by nano chitosanand reactive dyeing at optimum conditions

Dyes	Samples	Rubbing Fastness		Perspiration fastness						Light fastness
				Acidic			Alkaline			
		Alt	St.	Dry	Wet	Alt	Dry	Wet	Alt	
Reactive Red 195	Untreated	3-4	3-4	3-4	3	3-4	3-4	3	3	4-5
	Treated	4	4	4	4	4	4	3	4	7
Reactive orange 16	Untreated	3-4	3-4	3-4	3	3-4	3-4	3	3	3-4
	Treated	4	4	4	4	4	4	4	4	7
Reactive red 180	Untreated	3-4	3-4	3-4	3	3-4	3-4	3	3	5
	Treated	4	4	4	4	4	4	4	4	7
Reactive Yellow145	Untreated	3-4	3-4	3-4	3	3-4	3-4	3	3	4-5
	Treated	4	4	4	4	4	4	4	4	6
Reactive blue 224	Untreated	3-4		3-4	3	3-4	3-4		3	4
	Treated	4		4	4	4	4		4	7

TABLE 5: antibacterial activity of treated fabrics

Dyes	Fabric	Inhibition zone (mm)			
		Gram positive bacteria		Gram negative bacteria	
		A. niger	S. pyogenes	B. cereus	E.coli
Reactive Red 195	Blank	0	0	0	0
	Before dyeing	17	17	18	19
	Simultaneous dyeing	18	19	19	20
	post dyeing	19	20	20	21
Reactive orange 16	blank	0	0	0	0
	Before dyeing	16	17	17	17
	Simultaneous dyeing	17	18	18	19
	post dyeing	19	20	20	20
Reactive red 180	blank	0	0	0	0
	Before dyeing	15	15	15	16
	Simultaneous dyeing	16	17	17	18
	post dyeing	17	19	19	20
Reactive Yellow 145	blank	0	0	0	0
	Before dyeing	15	15	16	17
	Simultaneous dyeing	16	16	16.5	18
	post dyeing	17	17	18	19
Reactive blue 224	blank	0	0	0	0
	Before dyeing	15.5	15	16	17
	Simultaneous dyeing	17	16	16	18
	post dyeing	18	17	17	19

Conclusion

The effect of chitosan nanoparticles on the dye-ability of silk fabric is investigated using three different processes; pre-treatment, post and simultaneous. The silk samples are treated using different concentration of chitosan nano particles and dyed with mono and bifunctional reactive dyes. The higher colour strength values are obtained of pre-treated of silk samples with aqueous solutions ofchitosan nanoparticles as it improve the dye uptake and antibacterial activity of untreated silk.It was found that, increasing in chitosan concentration up to 3% (w/v), there was nosignificant in colour difference between the fabricsfor both reactive dyes.Increasing in the chitosan concentration didn't yield higher K/S and caused dyeprecipitation. Moreover,colour fastness properties to light and washing of treated silk with chitosan were improved.

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تحسين قابلية الصباغة لأقمشة الحرير بإستخدام النانو كيتوزان وإكسابها خصائص مميزة من حيث مقاومتها للبكتريا الموجبة والسالبة

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الهدف من البحث :-

إستبدال المعالجة التقليدية للاقمشة الحريرية بالكيتوزان في الحجم الجزئي الحالي لمحلول الكيتوزان المحفز في حجم النانو وذلك بإستخدام تركيزات مختلفة من محلول النانو كيتوزان علي مختلف المراحل من قبل واثناء وبعد عملية الصباغة .

التجارب المعمليه والقياسات :

١-تحضير الكيتوازن في حجم النانو

تم تحضير محلول النانو كيتوزان بإستخدام الصوديوم زاد بوي فوسفات كـ((Dispersing agent) وبتقنية الموجات فوق الصوتية وذلك لمدة ٣٠ دقيقة

٢- معالجة الحرير بمحلول النانو أخذت ثلاث تقنيات معالجة قبل وأثناء وبعد عملية الصباغة بالصبغات النشطة المذكورة .

٣- قد جرت عملية صباغة الحرير بالصبغات النشطة بالطريقة التقليدية .

تم التحقيق من تحضير الكيتوزان في حجم النانو من خلال إستخدام الميكروسكوب الإلكتروني وقد اوضحت العينات المقاسة الوصول الي أحجام متناهية الصغر .

- كما تم أخذ صور بالماسح الضوئي للعينات المعالجة بالتقنيات الثلاثة السابق ذكرها ولوحظ من خلال هذه الصور وجود ترسيبات علي هيئة فيلم رقيق من جزيئات النانو كيتوزان ذات حواف واضحة والذي بدوره أي إلي زيادة قابلية الأقمشة للصباغة .

- تم اجراء مجموعة من الإختبارات وذلك لقياس درجات الثبات للغسيل والعرق بنوعية والضوء) واظهرت العينات المعالجة خصائص ثبات من جيد جدا الي ممتاز .

- وبالنسبة للثبات ضد البكتريا أظهرت العينات المعالجة تفوق ملحوظ عن العينات الغير معالجة والتي لم تعطي أي خصائص مقاومة للبكتريا بشقيها الموجبة والسالبة .

- الاستفادة فى ميدان التطبيق العلمي والعملي:

يعرض البحث لفكرة جديدة وهى إستخدام النانو كيتوزان في إحداث تحسين كبير لقابلية أقمشة الحرير للصباغة بالصبغات النشطة وذلك بإستخدام أقل كمية من الصبغات وتحقيق علي درجات عمق لوني وثبات عالى.