

Enhancement of dyeing cotton fabrics using nanocomposite

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ACTUALLY, the present current work addresses an innovative approach for benign enhancement of dyeing of cotton fabrics assisted with nanocomposite based on chitosan (Cs), silver nanoparticles (Ag NPs) and clay. The Cs/Ag NPs and Cs/Ag NPs/clay nanocomposites were prepared using UV irradiation and applied on the cotton fabrics by pad-dry-curing method. The blank and treated cotton fabrics were dyed with C.I. Acid Orange 74. The Dye exhaustion, colorimetric analysis as well as fastness properties of the treated fabrics was evaluated. The obtained data proves that the nanocomposite of Cs/Ag NPs/clay is a promising mixture for durable dyeing for cotton fabrics.

Keywords: Cotton Fabrics; Dyeing; Nanocomposite; Acid Dye; Chitosan; Silver NPs; Clay.

Introduction

High performance cellulosic based fabrics with dual or multi-functional surfaces, is usually used to increase the textiles versatility [1-13]. Coloration of technical textiles is performed either by dyeing to form uniform color or by printing to introduce a design or a pattern to a fabric. Dye molecule structure contain of two key parts: the chromophores, responsible for creating the color, and the auxochromes, which can not only increment the chromophore but also render the molecule soluble in water and give improved affinity toward the fibers [14]. Acid dye is a material used in textile dyeing. The naming of the acid dye develops from the use of an acid in the solution. Acid dye is usually a sodium salt of sulfonic acid, or at least, it is an ionic carboxylic acid which turns into an anion in a solution. It will dye the fibers in the cationic part [15]. It is usually substituted with ammonium ion groups in fibers such as wool, silk and nylon [16-19]. This fiber will absorb the acid. The acid will protonate the amino group of fibers, so they become cationic. Dyeing process involves exchange of the anion associated with an ammonium ion in the fiber with a dye anion in the bath [20-22]. The cotton fabrics are hydrophilic fabrics whose structures are cellulose polymers with varying degree of polymerization (DP). The primary -OH group of cellulose is a

functional group that acts to bond with the dyestuff [23, 24]. The use of acid dye causes less affinity to cotton fabric because of no cationic part in cotton surfaces. The Chemical modification of cotton fabrics for improving utilization of acid dyes has attracted much attention. The process is primarily to attach the cationic compounds on cotton fabrics by chemical binding or physical adsorption for enhancing the substantively between anionic dye and cotton [25-27]. Such treated cotton would be dyeable with acid dyes under neutral or mildly acidic conditions in the absence of electrolyte in the dyebath [28, 29]. In this research, the utilization of nanocomposite in the textile fields is examined by preparation and deposition of the Cs, Cs/Ag NPs and Cs/Ag NPs/clay nanocomposites. The nanocomposites deposited on the cotton surface will be dyed by the acid dyes.

Materials and methods

Materials

Scoured and bleached 100% cotton fabric (140 g/m²) was supplied by El-Mahalla El-Kobra Company. The fabrics were scoured in aqueous solution with a liquor ratio 1:50 containing 5 g/l nonionic detergent solution (Hostapal, Clariant) and 2 g/l Na₂CO₃ at 95°C for 4 hr. to remove impurities, then rinsed thoroughly in cold tap water, and dried at room temperature. C.I. Acid Orange 74 as model acid dye.

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Methods

Preparation of Cs/Ag NPs/clay nanocomposite

Clay solution (1%) was prepared by dissolving 1 g in 100 mL deionized water under magnetic stirring for 1 h then sonicated for another 1 h. The CS/Ag⁺ composite was prepared by dissolving 2 g of Cs in 100 ml of 1.0 Wt % acetic acid under constant magnetic stirring for overnight. After complete dissolution, 100 mg/l of silver nitrate (AgNO₃) was added slowly to Cs solution under contentious stirring. Then, the clay suspension was added to the CS/Ag⁺ composite vigorously stirred for 4 h at room temperature. The CS/Ag⁺/clay was irradiated using the UV range (320-400 nm) with a UV lamp at $\lambda = 350$ nm, photon energy 2.26-3.94 eV emitting 668 $\mu\text{W cm}^{-2}$ at a distance of 5 cm for 2 h. The color of prepared sample was gradually changed from colorless to light gray, then to gray and finally to dark gray [30].

Finishing cotton fabric with the as prepared nanocomposites

Bleached cotton fabrics were washed with 1 g/L nonionic detergent at 90 °C for 15 min to remove impurities. The pretreated cotton fabrics were immersed for 1 h in Cs (2%), and the same concentrations of the as prepared Cs/Ag NPs and Cs/Ag NPs/clay nanocomposites that are mentioned before. The treated samples were squeezed to 100% wet pick-up using pad-dry-cure method at constant pressure. Samples were dried at 90 °C for 5 min, followed by curing at 140 °C for 1 min to fix the as prepared nanocomposite into and on the surface of cotton fabrics.

Dyeing process of untreated and treated cotton fabric with Cs/Ag NPs/clay

Dyeing process of blank and treated cotton fabric was carried out at 100 °C, L.R 1:40. Dyeing baths contains 2 % (w.o.f) dye at pH 5. The fabric was immersed and the temperature was raised to 100 °C and the dyeing continued for 60 min. At the end of dyeing, the dyed samples were rinsed with tap water and allowed to dry in open air. The dyed samples were rinsed with water and washed in a bath L.R. using 3 g/l of nonionic detergent Hostabal Clarinet at 60 °C for 30 min. Subsequently, they were rinsed and dried at ambient temperature [31].

Characterization

Dye exhaustion

The extent of dye exhaustion (%) achieved for an appropriate dye concentration on each of the two types of dyes was determined by the absorbance spectroscopic analysis of the dyebath before and after dyeing on Shimadzu UV-2401PC

UV/vis spectrophotometer at maximum wave length for each dye. The percentage of dye-bath exhaustion (% E) was calculated using Eq. (1):

$$\% E = A_1 - A_2 / A_1 \times 100 \quad (1)$$

where A_1 , A_2 are the concentrations of the dye-bath before and after dyeing, respectively.

Colorimetric analysis

The colorimetric analysis of the colored fabrics was recorded using a spectrophotometer with pulsed xenon lamps as a light source (UltraScan (Pro, Hunter Lab, USA) 10° observer with D65 illuminant, d/2 viewing geometry and measurement area of 2 mm. All measurements were acquired at λ_{425} nm wavelength. The corresponding color strength value (K/S) was assessed by applying the Kubelka-Munk equation (Eq. 2) [1,2] sunlight can also be a source of such deleterious effects as sunburn, and suntanning, as well as premalignant and malignant lesions. These may all occur in individuals with normal responses to sunlight. In addition, there exist a variety of 'abnormal' photosensitivity responses to sunlight that may result from either endogenous imbalances (e.g. the porphyrias):

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

where R is the decimal fraction of the reflection of the colored fabric, K is the absorption coefficient and S is the scattering coefficient. The total color difference (ΔE) was represented in terms of CIE LAB color space data. The CIE LAB color space data (L^* , a^* , b^*) of colored samples was measured by the same spectrophotometer used for measuring color strength at the same setup, and then the color difference was calculated using (Eq. 3),

$$\Delta E = L^2 + [(a^2 + b^2)]^{1/2} \quad (3)$$

where, ΔE is the total difference between the sample and the stander, L is the lightness from black (0) to white (100), a^* is the red (+) / green (-) ratio and b^* is the yellow (+) / blue (-) ratio. The color fastness of the treated fabrics was tested according to ISO standard methods. The specific tests were ISO 105-X12 (1987) for color fastness to rubbing; ISO 105-C02 (1989) for color fastness to washing and ISO 105 E04 (1989) for color fastness to perspiration [9].

Results and discussion

Preparation and characterization of CS/Ag NPs/clay composite and treated cotton fabrics

The formation of Ag NPs was visually observable by the color change in the treatment

solution from colorless to a color. The UV-vis spectroscopy is a simple and sensitive technique for the characterization of Ag NPs. The typical UV-vis absorption spectra of the resulting solution of Ag NPs synthesized under UV radiation are shown in Figure 1.

It is clearly observable in figure 1 that, there is no absorption in visible region for the composite of Cs/Ag⁺/clay. However, the absorption band around 400 nm is appearing in the absorption spectra composite sample of Cs/Ag NPs/clay prepared using 2 h irradiation time. Hybrid nanocomposites of Cs/Ag NPs/clay were applied to cotton fabric using pad-dry-cure method. The treated cotton fabrics with the as prepared nanocomposites of Cs/Ag NPs/clay when compared with the blank cotton fabric only are visibly evident as the color is changed from colorless to yellow darkness. The morphological of the untreated and treated cotton fabric surface with Cs, Cs/Ag NPs and Cs/Ag NPs/clay is observed by using FE-SEM and the resulted images are showed in Figure 2.

It is clearly seen that the untreated sample has a relatively smooth surface as shown in Figure 2 (A, a) at low and high magnification indicating that there are no nanoparticles on the surface. After treating with Cs solution, film of Cs is covered on the fabric surface, beside the formed Cs film, the surface becomes rough due to the effect of Cs polymer as displayed in Figure 2 (B,b). Figure 2C shows the SEM micrographs of the treated cotton fabrics with Cs/Ag NPs. It is observed that the Ag NPs are deposited on the surface of cotton fabrics. In addition, the obtained SEM graph at high magnification (8000X) indicating that the particles of Ag NPs are distributed on the fabrics uniformly. It is noticed that the Cs/Ag NPs nanocomposites is spherical with small diameter. It can be seen that the treated cotton fabric sample with colloidal dispersion of hybrid composite Cs/Ag NPs/Clay appears to be covered with aggregated nanocomposite and the rough surface is obtained. The low and high magnification (Figure 2 D and d) show a formation of aggregated clay minerals on the surface of cotton.

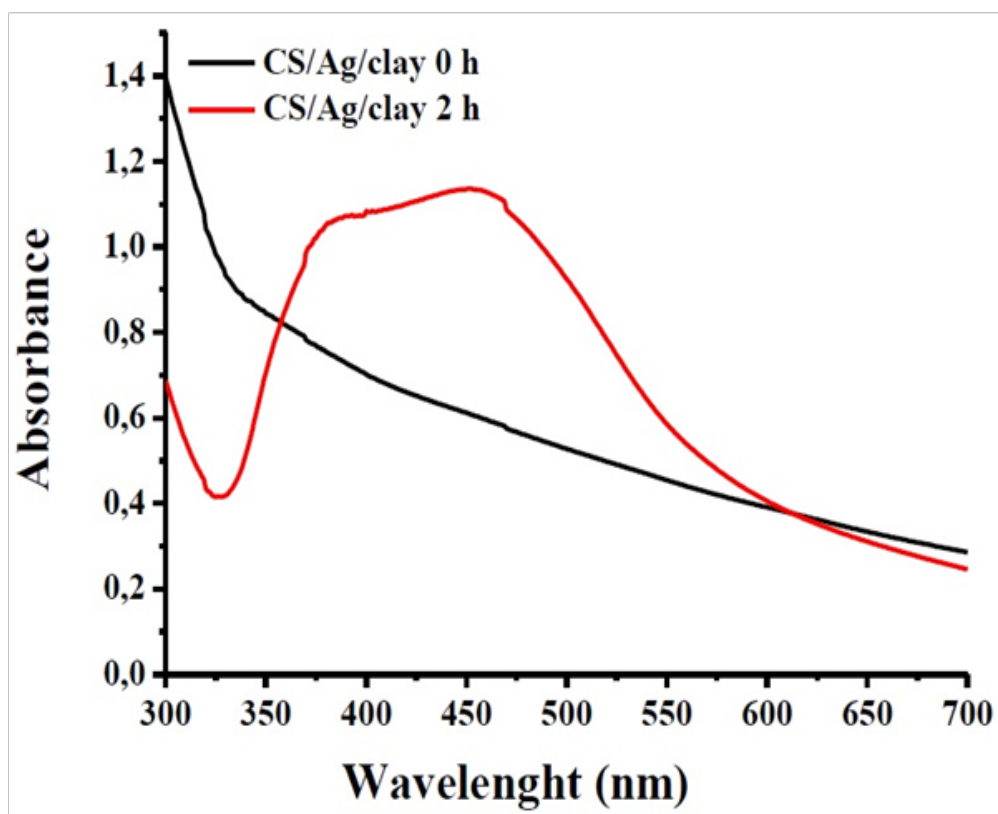


Fig. 1. Surface Plasmon resonance peak of Cs/Ag NPs/clay nanocomposite.

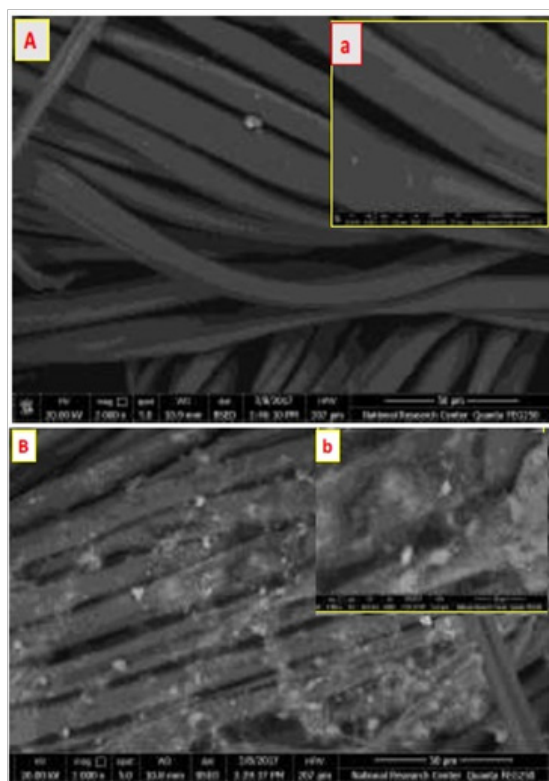


Fig. 2. Low and high magnification SEM images of (A,a) untreated cotton fabrics, (B,b) cotton fabrics treated with Cs/AgNPs/clay.

The nanocomposites of the Cs/Ag NPs with and without clay were coated on the cotton fabric using pad- dry –cure method. The influence of these hybrid nanocomposites on the fabric the mechanical properties and compared with untreated cotton fabrics are examined and the value of Tensile strength (N/cm²) and elongation (%) are recorded in Table 1.

It is seen that the tensile strength of untreated cotton fabric is 555 N/cm² and increase to 567 N/cm² for the treated fabric with Cs and Cs/Ag NPs. The value of tensile strength is sharply increase (667 N/cm²) with the addition of clay to the nanocomposite of Cs/Ag NPs. Opposite trend is observed with the elongation at break. The latter decreases by the addition of clay to Cs/Ag NPs nanocomposite. It is concluded that the high improvement in tensile strength may be achieved by adding clay to the hybrid nanocomposite of Cs/Ag NPs.

Dyeing of raw and treated cotton fabrics with acid dye

The coloration process of textile fabrics is an important aesthetic property for textiles end use and this process has evolved to be a crucial process. The Acid dyes are the salts of carboxylic acids or sulfonate can be dissolve in aqueous *J. Text. Color. Polym. Sci.* **16**, No. 1 (2019)

solutions leaving the anionic character to the chromophore unit, so these dyes show higher affinity towards cationic charged fibers like wool, silk and nylon. According to this behavior, the acid dyes are not used for cellulosic fibers/fabrics coloration. However, the fact that, the modification of cellulosic fibers/fabrics can be effectively on coloration of cotton fabrics with acid dye. In this study, the cotton fabric as cellulosic fabric was modified with different agents to show the influence of these modified on the coloration of cotton with acid dye.

To clarify the influence of different modified agent on the color of treated cotton fabrics, the dye exhaustion (%) as well as the color strength (K/S) was evaluated (Table 2. The color strength (K/S) value is commonly used to refer the amount of the dye content of the colored fabrics where, higher K/S value shows greater color intensity and hence better amount of dye incorporated into the fabrics.

It was observed from Table 2 that; the dye exhaustion (%) and the color strength (K/S) of treated cotton fabrics are higher than untreated cotton fabric. This result indicates that, the modification of cotton fabrics with different agent can improved significantly the dyeability

TABLE 1. Mechanical Properties of cotton fabrics.

Sample	Tensile strength (N/cm ²)	Elongation (%)
Blank cotton fabric	590±20	16.12±3
CS/ cotton fabric	563±17	16.11±1
CS/Ag NPs/ cotton fabric	564±13	15.93±1
CS/Ag NPs/clay/ cotton fabric	656±11	17.32±1

TABLE 2. The dye exhaustion (%) and color strength (K/S) of the dyed cotton fabric with C.I. Acid orange 74).

Sample	Exhaustion %	K/S
Untreated cotton fabric	12	1.2
Chitosan/ cotton fabric	62	13.58
Chitosan /Ag NPs/ cotton fabric	45	10.12
Chitosan /Ag NPs/clay/ cotton fabric	38	7.67

of cotton fabrics with acid dye. The cotton fabric treated with chitosan show the highest dye exhaustion (%) and the color strength (K/S). This is the enhancement may be attributed to that, the most of the amino groups of chitosan will be existed in protonated form -NH^{+3} in pH (4-5), so the chitosan treatment can be created of a positive cationic -NH^{+3} sites on cotton macromolecules resulting in higher accessibility to attract anionic negatively charged acid dye via ionic attractive forces. Thus, the number of positive charges on the cotton fabric surface will be a decisive factor in determining the extent of ionic attraction between acid dye and fabric and subsequently the obtained dye exhaustion (%) and the color strength (K/S). The presence of Ag NPs or clay with chitosan may be decrease the positive cationic charge occupied surface and decrease the attractive forces between acid dye and cotton fabric surfaces decreasing the dye exhaustion (%) and the color strength (K/S) for cotton sample.[32-36]

The CIE (L^* , a^* , b^*) system was used to investigate and evaluate the color coordinates where, L^* refers to lightness/darkness values from 100 to 0 representing white to black, a^* is the values run from negative (green) to positive (red) and, b^* values run from negative (blue) to positive (yellow). The colorimetric data of blank and treated fabrics were provided in Table 3.

Data of this table illustrated that, the original color of raw cotton fabrics is white with high L^* value (82.86), low a^* value (1.91) and negative b^* values (-7.82). On the other side, the dyed cotton samples have significantly change of (L^* , a^* , b^*) values compared to the blank ones. Where, (L^*) is significantly decreased while a^* and b^* values are increased dramatically. The deeper color leads to higher total color difference values. A more decrease in L^* values could be due to more colorant penetrations into cotton samples indicating more dyeability. The results showed that both the color components a^* and b^* almost increased by cotton surface modifications indicating more dye-ability of all modified cotton fabrics. The b^* values became more positive indicating more yellow part in color and the more positive a^* value indicating more red in color and the combination of b^* and a^* changes thus evaluating the increase in orange color of treated cotton fabrics. The more scientific change in b^* and a^* values was obtained in the cotton fabric treated with chitosan. It can be easily concluded that the cotton fabric treated with chitosan show the highest deep color intensity indicating more dye-ability. This behavior was also in agreement with dye exhaustion (%) and the color strength (K/S) data discussed earlier. In addition, the different color fastness properties of colored fabrics were measured and the results are reported in Tables 4.

TABLE 3. Colorimetric data of the dyed cotton fabric with C.I. Acid orange 74).

Sample	L*	a*	b*
Raw cotton fabrics	82.86	1.91	-7.82
Untreated cotton fabric	76.66	7.38	12.94
CS/ cotton fabric	49.44	42.87	46.39
CS/Ag NPs/ cotton fabric	54.09	41.01	47.61
CS/Ag NPs/clay/ cotton fabric	61.39	36.47	47.79

TABLE 4. Fastness properties of the dyed cotton fabrics with the acid dye.

Samples	Rubbing		Washing			Perspiration						Light
	Dry	Wet	Alt.	St.*	St.**	Acidic		Alkaline				
						Alt.	St.*	St.**	Alt.	St.*	St.**	
Blank cotton fabric	2	2-3	2-3	3	2-3	2	2-3	2-3	3	2-3	3	2
CS/ cotton fabric	3-4	3	4	4	4	4	4	4	4	4	4	6
CS/Ag NPs/ cotton fabric	4	3	4	4-5	4	4	4	4	4	4	4	5-6
CS/Ag NPs/clay/ cotton fabric	4	4	4-5	4-5	4	3-4	4	3-4	4	4	3-4	5

Alt.: alteration St.*: staining on cotton St.** staining on wool

In this table it is clear that, modified cotton fabrics were exhibited very good results for rubbing, washing, perspiration, and for light properties rather than un-modified cotton fabrics. these good fastness properties for the applied acid dye can be discussed in terms of the chemical structure of the dye used and that of modified cotton fabrics which may cause the dye to be tightly adhered to the modified cotton fabrics to the degree that caused this observed improve in fastness properties.

Conclusion

Research adopted in this study is undertaken with a view to enhancement of dyeing of the cotton fabrics with acid dyes using nanocomposites. Blank cotton fabrics and modified with Cs, Cs/ Ag NPs and Cs/Ag NPs/clay was dyed with C.I. Acid Orange 74 that otherwise have little substantively towards cellulosic fabrics. The results showed that, the good exhaustion %, K/S, colorimetric data and good fasteners properties were obtained

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for modified cotton fabrics in this order CS/ cotton fabric > CS/Ag NPs/ cotton fabric > CS/Ag NPs/ clay/ cotton fabric.

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تحسين صباغة الأقمشة القطنية باستخدام مخاليط نانوية

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¹ قسم التحضيرات والتجهيزات السليلوزية ، شعبة بحوث الصناعات النسيجية ، ² قسم الصباغة والطباعة والمواد الوسيطة ، شعبة بحوث الصناعات النسيجية ، المركز القومي للبحوث ، الدقي ، الجيزة ، مصر

يعالج العمل الحالي طريقة مبتكرة لتحسين صبغ الأقمشة القطنية بمساعدة المكمورات النانوية المستندة إلى الكيتوزان (Cs) والجسيمات النانوية الفضية (Ag NPs) والطين. تم تحضير Cs / Ag و Cs / Ag NPs Nanosomposites باستخدام الأشعة فوق البنفسجية للأشعة فوق البنفسجية وتطبيقها على الأقمشة القطنية من خلال طريقة المعالجة بالوسادة الجافة. كانت الأقمشة القطنية الفارغة والمعالجة مصبوغة بصبغة حامضية برتقالية اللون ٧٤. تم تقييم استنزاف صبغ ، تحليل اللوني وكذلك خصائص ثبات الأقمشة المعالجة. البيانات التي تم الحصول عليها تثبت أن مركب النانو من Cs / Ag NPs / الطين هو خليط واعد لصبغة دائمة للأقمشة القطنية