Effects of chitosan on the metal absorption and UV protection properties of woven cotton fabric

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ABSTRACT – REZUMAT

Effects of chitosan on the metal absorption and UV protection properties of woven cotton fabric

In this research, cotton fabrics were treated with carboxylated chitosan, loaded by the pad-dry-cure method for investigating the absorption of heavy metals and the Ultraviolet (UV) protection properties of fabric samples. For this purpose, chitosan-treated cotton fabrics were soaked in $CuSO_4$ solution to investigate the rate of heavy metal absorption. The surface morphology of cotton fabrics was investigated using Scanning electron microscopy (SEM) analysis. Inductively coupled Plasma Spectroscopy (ICP) analysis was employed to examine the amount of heavy metals' absorption upon chitosan-treated cotton fabrics. Besides, reflection and transmission spectrophotometry analyses were used to examine the optical properties of cotton fabrics. The results show a noticeable increase in copper absorption by increasing the amount of chitosan. The maximum absorption of Cu belongs to the %6 chitosan-treated sample by 7357.6 PPM, equal to %7.35. The more concentration of chitosan in the fibre causes a higher rate of UV protection. Therefore, the chitosan-treated cotton fabrics examined in this work can be used for various water filtration purposes, notably to eliminate toxic metals. Also, It can be used as a wearable textile for protecting against harmful UV rays.

Keywords: *chitosan, metal absorption, UV protection, cotton, fabric*

Efectele chitosanului asupra absorbției metalelor și proprietăților de protecție UV ale țesăturii din bumbac

În această cercetare, țesăturile din bumbac au fost tratate cu chitosan carboxilat, aplicat prin metoda de fulardare-uscare-condensare pentru analiza absorbției metalelor grele și a proprietăților de protecție la ultraviolete (UV) ale mostrelor de țesături. În acest scop, țesăturile din bumbac tratate cu chitosan au fost înmuiate în soluție de $CuSO_4$ pentru a investiga viteza de absorbție a metalelor grele. Morfologia suprafeței țesăturilor din bumbac a fost investigată folosind analiza microscopiei electronice cu scanare (SEM). Analiza prin spectroscopie cu plasmă cuplată inductiv (ICP) a fost utilizată pentru a examina cantitatea de metalelor grele absorbite de către țesăturile din bumbac tratate cu chitosan. În plus, analizele de spectrofotometrie de reflexie și transmisie au fost utilizate pentru a examina proprietățile optice ale țesăturilor din bumbac. Rezultatele arată o creștere vizibilă a absorbției cuprului prin creșterea cantității de chitosan. Absorbția maximă de Cu aparține probei tratate cu chitosan 6% cu 7357,6 PPM, egal cu 7,35 %. Cu cât concentraţia de chitosan pe fibră este mai mare, cu atât rata de protecție UV este mai mare. Prin urmare, țesăturile din bumbac tratate cu chitosan examinate în această lucrare pot fi utilizate în diferite scopuri de filtrare a apei, în special pentru eliminarea metalelor toxice. De asemenea, pot fi utilizate ca textile purtabile pentru protejarea împotriva razelor UV dăunătoare.

Cuvinte-cheie: *chitosan, absorbție de metal, protecție UV, bumbac, țesătură*

INTRODUCTION

Throughout the vast and quick evolution of new science regarding the textile industry in the latter years, numerous global scientists have actively conducted investigations about practical textiles.

Practical textiles have been well-known as the most significant element of the textile industry. Nevertheless, a need for applicable high-tech textiles regarding UV protection, waste refinement, fireproofing, etc., could not be reached just by an ordinary single-step preparation [1, 2]. The growing requisition regard to the increasing demand for multipurpose fabrics needs powerful multidimensional scientific knowledge and process [3–5].

One of the most prevalent forms of textiles around the world is cellulose-made cotton fibres [6]. Sweat-absorbing, comfortableness, softness, and flexibility of cotton fibres made them one of the most common types of textiles worldwide. Besides, cotton textiles create a perfect habitat for microorganisms to be raised the cause of their ability to moisture preserving [7–10]. And one of the best natural biopolymers with numerous specific characteristics such as non-toxicity, antibacterial and cationic nature features is chitosan [11, 12].

Seeking an environmentally friendly method that replaces poisonous textile chemicals has been a permanent goal for the textile industry. In this regard, chitosan could be used as a superb alternative to environmentally friendly textile chemicals. A recent paper has already reviewed the multiple applications of chitosan for dyeing and finishing textiles [13–15]. It also has been proven as a wonderful alternative for dyes and metal ions absorption [16].

Another perspective refers to the progressive awareness of consumers about an urgent need for sunlight protection which is connected with the prevalence of skin damage and its correlation with growing exposure to UV light. Intense and chronic drawbacks of ultraviolet radiation (UVR), such as speeding up skin deterioration and ageing, are apparent to everyone. Overexposure to UV radiation is the main cause of diverse eye, skin, and DNA problems. The main clusters of the UV radiation band include the UV-A band (320–400 nm), UV-B band (290–320 nm), and also UV-C band (200–290 nm). The ozone layer and existing oxygen in the atmosphere are responsible for the total absorption of the UV-C band, which has the highest energy among other regions. The rest of the total UV radiation which radiates to the ground consists of 94% UV-A and 6% UV-B. Despite UV-A which causes minor skin reactions and somehow immunological disorders, UV-B has been proven to be the main cause of skin cancer [17, 18]. Moreover, in recent years, studies have been conducted on the absorption of heavy metals using chitosan-treated cotton fabrics. For example, Ferrero F et al. prepared a chitosan-coated cotton gauze by UV-curing and tested it as an adsorbent to remove copper (II) and chromium (VI) ions from water solutions. The adsorption capacities increased with increasing metal ion concentration for both metal ions, while the temperature did not significantly affect the metal ions' adsorption process [19]. Rongjun Qu et al. investigated the adsorption of Au (III) from an aqueous solution using cotton fibre/chitosan composite adsorbents. The results show that chitosan-treated fibres can selectively adsorb Au (III) from binary ion systems in the presence of the coexistent ions Pb (II), Cu (II), Ni (II), Cd (II), Zn (II), Co (II), and Mn (II) [20].

Nauman, A et al. coupled chitosan and copper nanoparticles on the fabric surface. The fabricated chitosan/copper cotton cloth (Cu/Chi-CC) can be used as an easily removable substrate for the absorption of Congo red (CR) dye [21].

In this study, deposited chitosan upon cotton fabrics was employed for investigating its UV protection and heavy metals absorption properties. Pad dry cure method was employed for depositing chitosan on cotton fabrics. Chitosan-treated cotton fabrics were overwhelmed on $CuSO_4$ solution to investigate the absorption rate of heavy metals. Reflective and transmission spectroscopy, SEM and ICP analyses were performed to evaluate Cu absorption by chitosan-treated cotton fibres.

EXPERIMENTAL

Materials

In this study, chitosan modified polysaccharides with medium density and cotton fabrics was used to investigate the chitosan impact on the metals' absorbance by cotton fabrics. Also, the UV protection properties of treated cotton samples were studied. Employed chitosan was made by Fluka Co with a deacetylation degree of 70%, and the utilized cotton fabric was weaved by 18.8 Tex warp and weft yarns composed of 56.88 threads in each square inch. All the other agents such as acetic acid and copper sulphate ($CuSO_4$) were purchased with commercial grade.

Method

At first, specimens containing 1, 3 and 6 percentage of chitosan were made. This procedure was done by dissolving chitosan powder in deionized water containing 3% Acetic acid. Then chitosan was deposited on the cotton samples using the pad-dry-cure method. Cotton fabric was cut off into four equal pieces and was overwhelmed in solutions containing chitosan. Cotton specimens were padded with different concentrations of chitosan (e.g., 1, 3 and 6%) at 80% wet pick-up and dried at 90°C for 5 minutes. A stenter machine was used for curing samples at 120°C for 2 minutes. Afterwards, 1 wt% of $CusO_4$ solution was prepared, and specimens were immersed in the produced solution separately. For investigating the reflection factor of all treated samples and comparing them with untreated ones, reflection Spectroscopy (D.R.S., Xrite Sp64 Spectcophotometer, Varion Co., Italy) analysis was used over a range of 400–700 nm. Also, transmission spectroscopy analysis was used to investigate the protection capability of samples against UV radiation (NIR, Analytic Jena Spectrophotometer 250) over a range of 280–400 nm. Besides, for inspecting the surface morphology of all treated and untreated samples, Scanning electron microscopy (SEM-Model EM-3200, KYky China) was employed. Inductively coupled Plasma Spectroscopy (ICP-OES simultaneous Model VISTA-PRO-Varian Co.- Australia) was used to measure the rate of metal absorption. The amounts of absorbed copper on the surface of untreated and chitosan-treated cotton with different concentrations of chitosan were compared using ICP analysis. For studying the fastness properties, samples were washed. The samples were washed with nanionic detergent for 20 times and dried in an oven at 60°C for 5 min. The maintenance of Cu on washed samples was analysed and determined using the ICP method.

Moreover, this research investigated the mechanical properties of untreated, chitosan-treated cotton and chitosan-treated cotton/$CuSO_4$ loaded samples. The load and elongation at break value (tensile strength) were measured by a Dinamometer TIRA test 2300 (made in Germany). The size of samples used for measurement was 120 × 300 mm^2 and the standard of the measurement was CSN EN ISO 13934-1. The maximum cell of the system was 5000 N. The distance between the jaws and the extension rate was 200 mm and 100 $mm \cdot min^{-1}$, respectively.

RESULTS AND DISCUSSION

This study investigated the rate of metals absorption and UV protection properties of chitosan-treated cotton. As was explained in the experimental part, cotton samples were deposited with different concentrations of chitosan (e.g., 1, 3, and 6%). Chitosan-treated samples were loaded with 1% wt of copper sulphate solution. Reflection spectrophotometry analysis was employed to investigate the samples' optical properties. The reflection properties of both untreated and chitosan-treated cotton after immersion in copper sulphate solution are shown in figure 1. The reflection factor for 6% chitosan treated cotton after immersion in Cu solution is less than the others. Comparing the reflection percentage of samples, it was concluded that by increasing the amount of used chitosan, the reflection factor decreased (figure 1). The above statements are evidenced by checking the results of the 6% sample with the highest rate of chitosan and consequently is darker, which may be a result of copper & chitosan's presence on the fabric surface. The results show that, by increasing the percentage of chitosan on cotton samples, more amounts of copper sulphate can be adsorbed which corresponds with recent research [22].

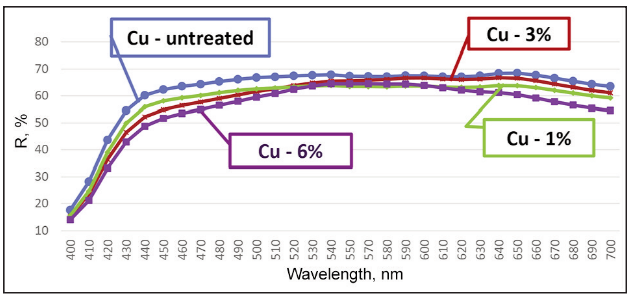

Fig. 1. Reflection factor for all chitosan-treated cotton fabrics with different percentages after $CuSO_4$ loading

Investigating the surface morphology of samples was done by SEM analysis (figures 2 and 3). Chitosan particles are apparent on the samples' surface as expected. The more amount of chitosan results in the higher particles' surface agglomeration and roughness. Consequently, the 6% chitosan-treated sample shows the most agglomeration and roughness. Another SEM analysis was done after overwhelming chitosan samples in the $CuSO_4$ solution (figures 4 and 5). As expected, the 6% chitosan-treated specimen absorbed more amounts of $CuSO_4$. These results confirm the achieved results by Perumal et al., which demonstrated that chitosan/gelatin hydrogel particles absorb a higher amount of Hg ion [23] that, coincides with the results of Shahraki et al. and Naeimi et al., which showed more rate of heavy metals ions absorption by utilizing chitosan [24, 25].

Structures that were observed on the samples' surface might be CuO nanoparticles. That is, chitosan-treated fabrics absorbed $CuSO_4$ and transformed it into CuO nanoparticles (chemical reaction 1).

$$CuSO_{4\,(aq)} + 2H_2O \rightarrow CuO + 2H_2SO_4 \qquad (1)$$

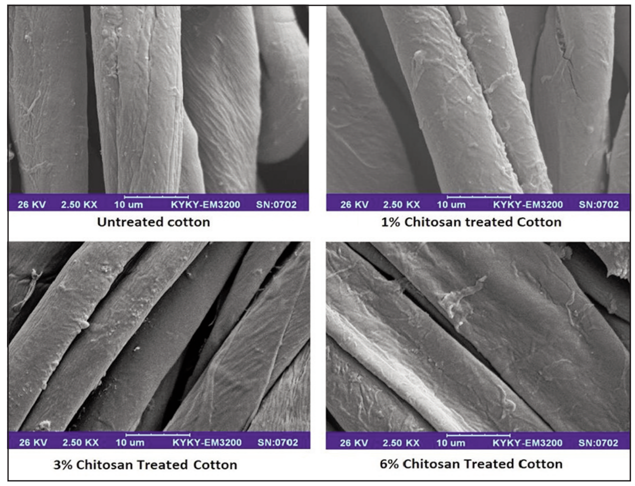

Fig. 2. SEM images of chitosan-treated cotton fibres, magnification of 2.5 kx

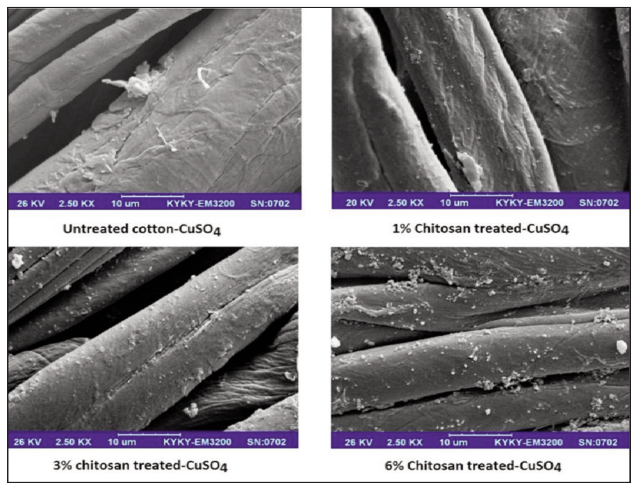

Fig. 3. SEM images of chitosan-treated cotton fibres, magnification of 10 kx

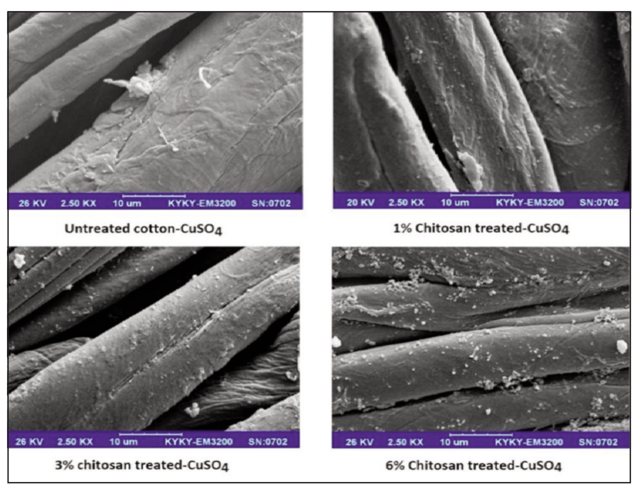

Fig. 4. SEM images of Chitosan treated + $CuSO_4$ cotton fibres, zoom in 2.5 kx

ICP analysis was employed for a precise investigation of the Cu amount on the fabrics' surface. The results show that chitosan-treated fabrics absorbed more Cu compared to raw fabrics. This rate of absorption had risen by increasing the amount of chitosan. The results show that the 6% chitosan-treated

Table 1

ICP ANALYSIS DATA OF CHITOSAN-TREATED/$CuSO_4$ LOADED COTTON FABRICS				
Sample	**EL.**	**Wavelength**	**Cal. conc. (ppm)**	**Cal. conc. (%)**
Untreated/Cu-loaded cotton	Cu	324.754	6060.3	6.06
1% Chitosan treated/Cu-loaded cotton	Cu	324.754	6060.3	6.3
3% Chitosan treated/Cu-loaded cotton	Cu	324.754	6698.3	6.6
6% Chitosan treated/Cu-loaded	Cu	324.754	7357.6	7.3
Untreated/Cu-loaded cotton after 20 times of washing	Cu	324.754	4100.2	4.1
1% Chitosan treated/Cu-loaded cotton after 20 times washing	Cu	324.754	5070.5	5.07
3% Chitosan treated/Cu-loaded cotton after 20 times washing	Cu	324.754	6078.5	6.07
6% Chitosan treated/Cu-loaded cotton after 20 times washing	Cu	324.754	7124.7	7.12

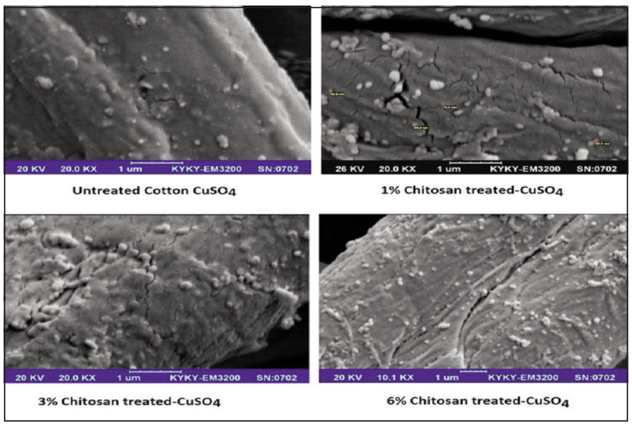

Fig. 5. SEM images of Chitosan treated+ $CuSO_4$ cotton fibres, higher magnification

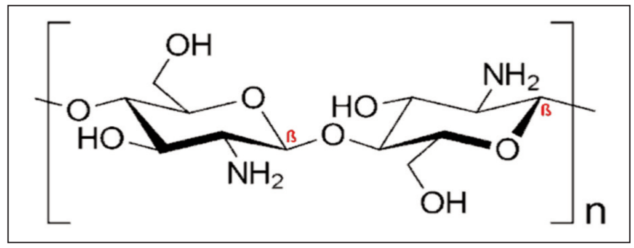

Fig. 6. Structure of chitosan

sample absorbed 7,357.6 ppm, equivalent to 7.35% Cu (table 1). These results correspond to the outcomes of the SEM analysis. According to the structure of chitosan (figure 6), more particles appear on the surface by treating cotton samples with chitosan. Also, as seen in figures 3 and 4, the unevenness on the surface of cotton fibres increases with chitosan treatment. By increasing the concentration of chitosan, this roughness will be increased. So more amounts of copper can be absorbed by chitosan-treated cotton. Copper makes a bond with the surface of cotton, both chemically and physically. Some copper particles are imprisoned in rough surfaces, and some bond chemically with polar groups. The results related to fastness properties against washing, show that, after 20 times of washing, more amounts of copper remain on the chitosan-treated cotton as compared with untreated cotton. By increasing the concentration of chitosan, the fastness properties will be improved.

In this study, transmission spectroscopy was employed to examine protection against UV radiation (figure 7). As seen by increasing the amount of Chitosan, more Cu was absorbed by samples, and consequently, fewer UV rays passed through the fabrics. Finally, we can conclude that the chitosan-treated samples are more resistant to UV radiation. Besides, Rehan et al. and Shahidi et al. concluded in their research that, chitosan-treated cotton fabrics illustrated a higher protection level against UV light compared to untreated cotton fabrics [2, 24]. The investigations of Vilela. C et al. show remarkable UV absorption characteristics in both UV-A and UV-B regions by using an active component of chitosan which is similar to the obtained results in this research [26].

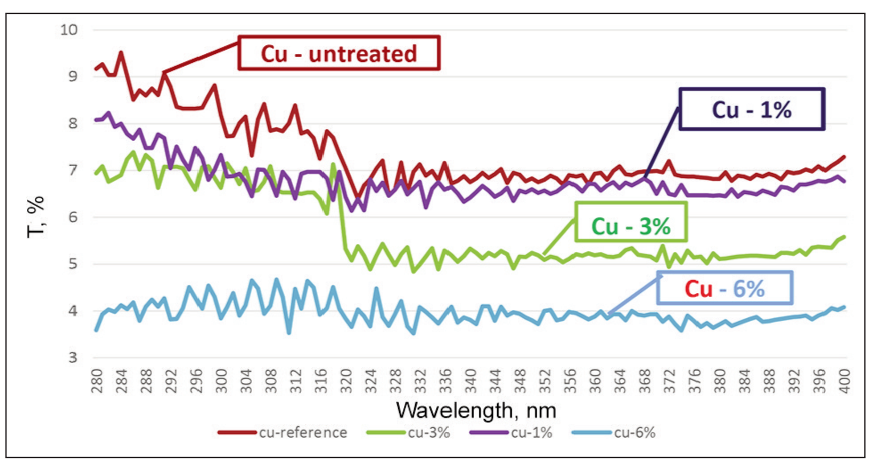

Fig. 7. Transmission factor for untreated and all chitosan-treated cotton fabrics after loading with $CuSO_4$

Table 2

MECHANICAL PROPERTIES OF UNTREATED AND TREATED SAMPLES IN WARP DIRECTION				
Sample	**Max elongation (mm)**	**Load F max (N)**	**Young's modulus *E* (MPa)**	**Time (s)**
Untreated cotton	23.27	2120.6	241.81	15.54
6% Chitosan treated	23.95	2123.7	241.41	15.55
6% Chitosan treated/Cu loaded	18.88	1839.95	235.1	12.66

Table 3

MECHANICAL PROPERTIES OF UNTREATED AND TREATED SAMPLES IN WEFT DIRECTION				
Sample	**Max elongation (mm)**	**Load F max (N)**	**Young's modulus *E* (MPa)**	**Time (s)**
Untreated cotton	14.69	1197.07	232.49	8.92
6% Chitosan treated	14.61	1196.35	231.46	8.93
6% Chitosan treated/Cu loaded	10.55	989.12	213.44	6.19

To investigate the effect of the chitosan treatment on the strength of the cotton fabrics, the maximum load and elongation at break (mechanical properties) were investigated. Both weft and warp directions were measured. The results are shown in tables 2 and 3. As it is seen, the mechanical properties of cotton samples after chitosan treatment will not change. However, coincident treatment with both chitosan and $CuSO_4$ solution causes reducing the mechanical properties. This reduction for 6% chitosan treated/ loaded with $CuSO_4$ is more significant. However generally, this tensile strength reduction is not considerable.

CONCLUSIONS

In this research, the effect of chitosan on the metal absorption of cotton fabrics was carried out. Also, the effect of chitosan-treated/Cu-loaded cotton with different concentrations of chitosan on the UV protection properties of cotton was investigated.

The samples were prepared using different concentrations of chitosan and a certain percentage of $CuSO_4$. Reflective-Spectrometry analysis results presented that the 6% chitosan-treated sample has the most reduction in reflection. SEM analysis confirmed the presence of chitosan and copper on the cotton fabrics. By increasing the percentage of chitosan, more amounts of copper are adsorbed by cotton samples. The results of the ICP analysis confirm the outcomes of the SEM test. To examine the resistance of samples against UV radiation for wearable applications, NIR spectroscopy analysis was done. The results showed a significant decrease in UV passing by increasing the amount of chitosan and copper. It can be worth mentioning that this biopolymer has plenty of desirable properties such as low price, renewability, high absorption, and recoverability, which make it a good filter for absorbing contaminants such as heavy metal ions and wastewater from the textile industry. Also for use as cloth, it can be a good choice for protection of the body against UV and bacteria [12].

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