

UV/O₃ Preirradiated Cotton Fabric-Containing Chitosan for Effective Removal of Heavy Metals

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Abstract

The present work aims at studying the effect of Ultra-Violet/Ozone (UV/O₃) irradiation of cotton fabrics on their interaction with chitosan/citric acid based formulation and, the onset of this on the ability of treated fabrics to remove heavy metal ions from their aqueous solutions. To achieve the goal, the cotton fabrics were preirradiated for 90 minutes using UV/O₃ as radiation source. The irradiated and the unirradiated cotton fabrics were submitted to finishing formulation consisting essentially of chitosan as a finishing agent and citric acid as crosslinking agent in combination with sodium hypophosphite (SHP) as a catalyst. Finishing formulation containing different concentrations of chitosan (0 - 4) along with citric acid (8 g/l) and SHP (4 g/l) were used and the interaction of chitosan with the cotton fabric was assessed through nitrogen measurement. The preirradiated and chemically finished fabrics vis-à-vis the unirradiated fabrics were examined for metal ion removal. Results conclude that the preirradiated cotton fabrics exhibit higher nitrogen content than the unirradiated fabrics. The same holds true for heavy metal removal, exemplified by Zn²⁺ and Cd²⁺ ions from their aqueous solutions. That is, preirradiated fabrics containing chitosan display higher percent heavy metal removal than their unirradiated mates.

Keywords

Chitosan, Cotton, Irradiation, Preirradiation, Heavy Metal Removal

1. Introduction

In common practice aqueous waste streams of many industries, such as metal plating facilities, mining opera-

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tions, and tanneries are contaminating with heavy metals. The same is also the case with soils surrounding many military bases which beside being contaminated, they pose a risk of metals groundwater and surface water contamination of the metals related to these activities mention is made of cadmium (Cd), chromium (Cr), lead (Pb), and Zinc (Zn) [1]-[3]. Heavy metals are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders which represent a serious threat for human population as well as the fauna and flora of receiving water bodies as discharges in waste water [4] [5].

Treatment processes for metals contaminated waste streams include chemical precipitation, membrane filtration, ion exchange, carbon adsorption, and coprecipitation/adsorption [6]-[8]. Cost effective alternative technologies or sorbents for treatment of metals contaminated waste streams are needed. Natural materials that are available in large quantities, or certain waste products from industrial or agricultural operations, may have potential as inexpensive sorbents. Due to their low cost, after these materials have been expended, they can be disposed of without expensive regeneration [3] [9] [10].

In the past twenty years a special attention has been given to polysaccharides such as chitosan. Chitosan is a natural aminopolymer derived by deacetylation of the naturally occurring biopolymer chitin which is the second most abundant polysaccharide in the world after cellulose [11] [12]. Chitosan has been used in many industrial and environmental applications, such as a remediation agent to remove heavy metal ions from aqueous solutions [13]. Chitosan powders, flakes, or beads have been used as alternative sorbents and displayed excellent aqueous heavy metals removal ability [14]. Chitosan has also been used as a surface modification agent impregnated onto supporting surfaces as adsorption sites because its amines functional groups have strong bonding ability to various heavy metal ions [15] [16].

Current work is undertaken with a view to highlight the impact of irradiation of cotton fabric using UV/O₃ prior to treatment of the fabric with chitosan/citric acid formulation. Also highlighted is the ability of cotton fabric-containing chitosan to remove heavy metal ions such as Zn²⁺ and Cd²⁺ ions from their aqueous solutions.

2. Experimental

2.1. Materials

Woven cotton fabrics (120 g/m²) were kindly supplied by El Nasr Company for spinning, weaving and dyeing, Mehalla, Egypt, Chitosan (LMW) (Fluka), Hostapal C.V[®] (non-ionic detergent-Clariant), acetic acid, hydrochloric acid, sodium carbonate, caustic soda, zinc acetate, cadmium acetate, citric acid (CA) and sodium hypophosphite (SHP), were all of laboratory grade chemicals.

2.2. Methods

2.2.1. Scouring of the Fabric

The grey cotton fabric was scoured using Hostapal C.V (2.0 g/l) and sodium carbonate (5 g/l) at 90°C for 1 hour, then rinsed with hot and cold water and finally dried at 100°C.

2.2.2. UV/O₃ Irradiation

The samples under test in the form of stripes were firstly exposed to UV source in a medium of ozone for 90 minute. A high intensity, low pressure mercury lamp without outer envelope-LRF-02971, 200 watt, 220 volts, made in Poland, was placed in a cubic box with side length of 60 cm, and the samples were put around the source at a distance of 20 cm. Molecular oxygen was subjected to the 184.9 nm irradiation and when ozon was irradiated at 253.7 nm, the radiation was adsorbed by most hydrocarbons and also by ozon [17]. The products under this excitation underwent with atomic oxygen to form simpler, volatile molecules, which desorbed from the surfaces. Therefore, when both wavelengths were present atomic oxygen was continuously generated and ozone was continually formed and then destroyed [18] [19].

2.2.3. Chitosan Treatment

Different concentrations of chitosan (0% - 4%) were freshly prepared using 1% acetic acid for dissolution. To these chitosan solutions (8 g/l CA and 4 g/l SHP) were added. Fabric samples were then padded separately in each of these solutions to a wet pick-up of 100% followed by drying at 85°C for 5 min, and by thermo-fixation at 170°C for 2 min. The treated samples were washed several times with cold water, and then dried at ambient condition (temperature of 25°C ± 2°C and relative humidity 65% ± 5%) [20].

2.2.4. Preparation of Metal Ion Solutions

Zinc and cadmium acetate solutions were prepared at different concentrations (100, 200, 300, 500 and 1000 mg/l) in closed containers.

2.2.5. Heavy Metal Removal Process

In separate 125 ml Erlenmeyer flasks each containing a known weight of adsorbent material, 100 ml of metal ion solution of known concentration (100, 200, 300, 500 and 1000 mg/l) was added to the flask. A range of pH 2 - 9 was adjusted by using 0.1 M HCl or 0.1 M NaOH. The flask was closed and occasionally shaken in a shaking water bath at 30°C for certain periods of time. The content of the flask was then filtered by vacuum using Whatman-40 filter paper and the filtrate was analyzed for assessing the remaining cations using Variant Atomic absorption spectrometer-USA [21].

2.3. Testing and Analysis

2.3.1. Nitrogen Content Determination

Nitrogen content was monitored according to Kjeldahl's method [22]. The following equation was used to calculate the nitrogen content:

$$N\% = (0.014 \times N \times V) \times 100/W$$

where N and V are the normality and volume (ml) of HCl while, W is the weight of the sample in gram.

2.3.2. Scanning Electron Microscopy (SEM)

Scanning Electron microscopy (SEM) of the treated fabrics was studied using a scanning electron probe micro-analyzer (type JXA840A)-Japan. The specimens in the form of fabrics were mounted on the specimen stabs and coated with thin film of gold by the sputtering method. The micrographs were taken as 200 times magnification using 30 kV accelerating voltage.

2.3.3. Infra-Red Spectroscopic Analysis (IR)

Measurements were performed using Nexus 670-FTIR spectrometer Nicolet-USA at the range of 4000 - 400 cm^{-1} and a resolution of 4 cm^{-1} .

2.3.4. Atomic Absorption Spectrometry (AAS)

Measurements were carried out using variant spectrometer-USA, using Cd and Zn standard lamps.

3. Result and Discussion

3.1. Morphological Structure

Figure 1 illustrates the SEM micrographs of scoured cotton (**Figure 1(a)**), chitosan treated cotton (**Figure 1(b)**) and preirradiated cotton fabric using UV/O₃ radiation followed by treatment with chitosan (**Figure 1(c)**). As is evident, the micrographs obtained indicate that the cotton fabrics undergo morphological changes, the magnitude of which relies on the treatment given to the cotton fabric. For instance, fibers treated with chitosan without UV/O₃ irradiation exhibit relatively higher entanglement, the lowest entanglement of the cotton fibers is, indeed, observed after UV-irradiation; a point which could be associated with photoablation. This photoablation in UV-irradiated cotton fabric may induce changes in the material surface, such as foam formation and expansion of the whole material [23]. It is as well to report that, the fabric coated with chitosan acquire a veil-forming like that formed onto the cellulose fibers that were preirradiated by UV followed by chitosan treatment as is evident from **Figure 1(b)** and **Figure 1(c)**.

3.2. Chitosan Concentration versus Nitrogen Content

Nitrogen content of chitosan treated fabrics and chitosan treated preirradiated cotton fabrics were determined and plotted against the concentrations of chitosan used in fabric treatment as shown in **Figure 2**. It is clear that preirradiated fabrics contains lower amount of chitosan, expressed as nitrogen content, than the unirradiated fabrics when chitosan concentration used in treatment of both fabrics is 1.5%. The opposite holds true at chitosan concentration of 2%. Higher chitosan concentration (2.5%) decreases the nitrogen content and, therefore the

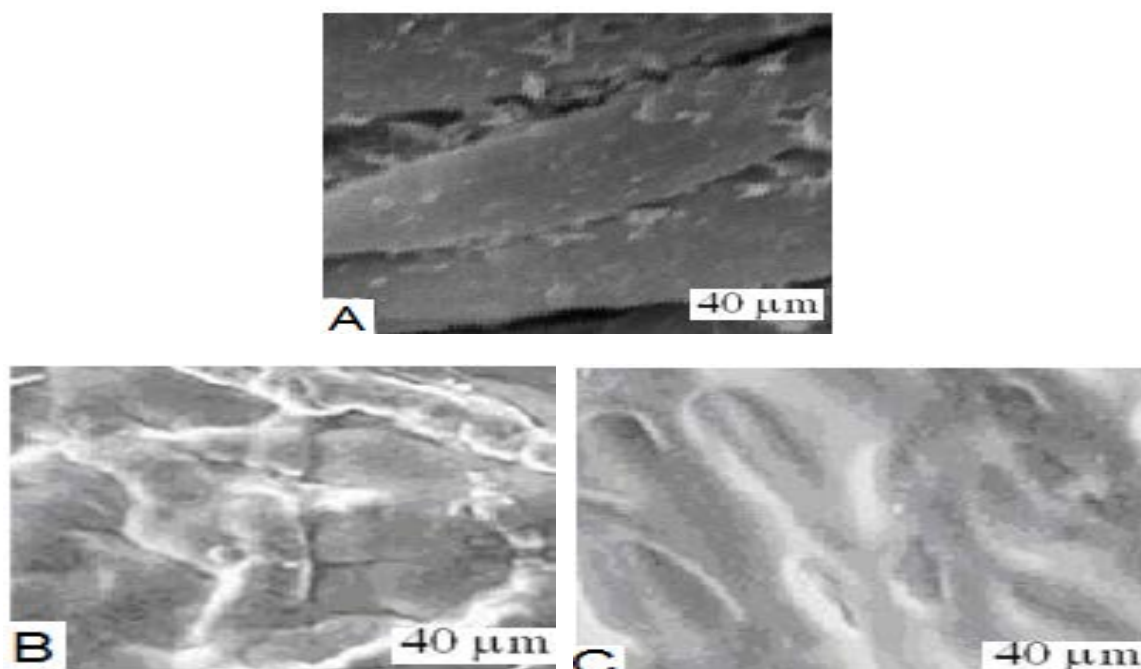


Figure 1. SEM micrograph for (a) scoured cotton, (b) 2% chitosan treated cotton without UV-irradiation and (c) 2% chitosan preirradiated by UV/O₃ followed by chitosan treatment.

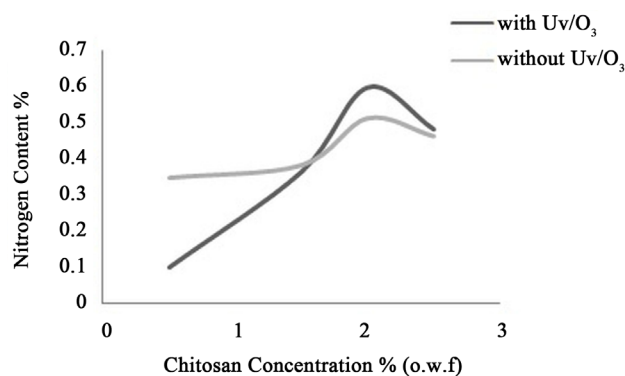


Figure 2. Effect of chitosan used in application of chitosan un-irradiated and irradiated cotton fabrics using UV/O₃ irradiation on nitrogen content of the fabrics. Treatment time (90 min), temperature 25°C.

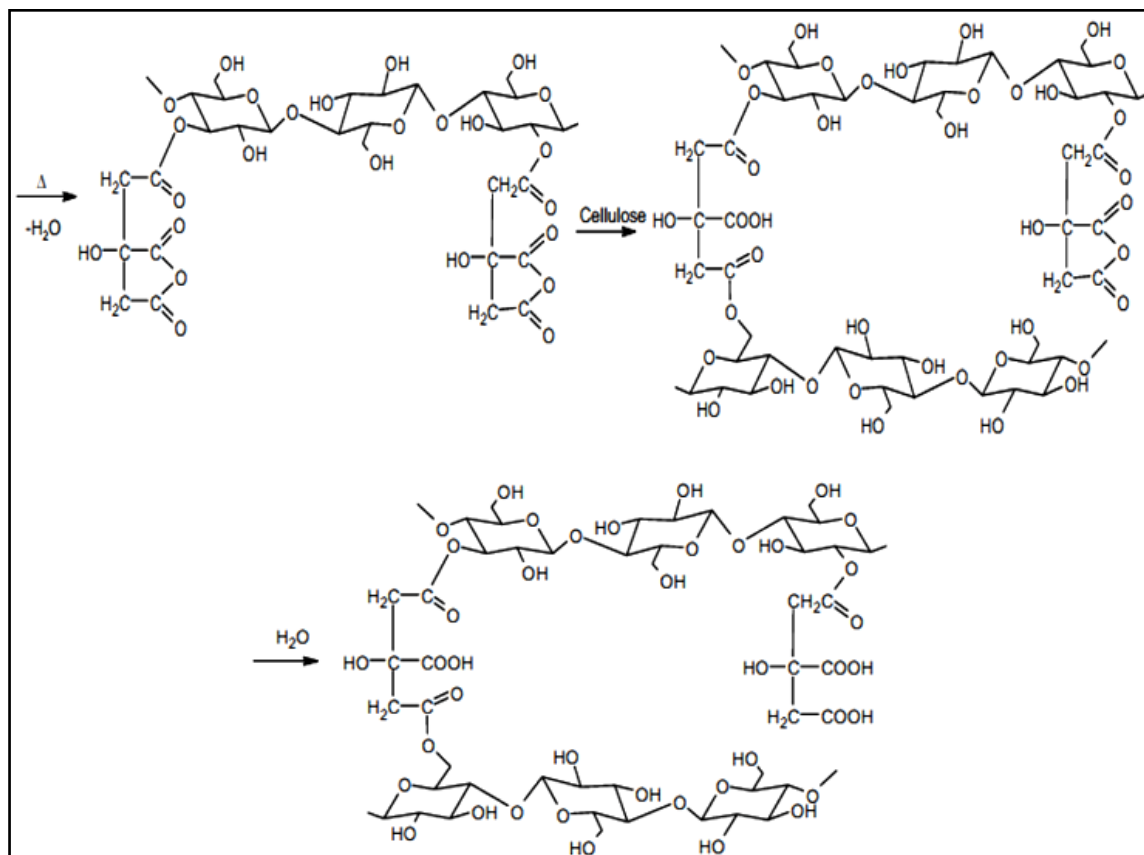
chitosan content on the fabric. This could be interpreted in terms of availability of chitosan molecules and interaction of the latter with the cotton cellulose molecules of the fabric. The interaction of the irradiated fabrics with chitosan seems to be affected substantially by morphological and microstructural changes and their impact on the nature and surface area of cotton cellulose, in addition to creation of reducing aldehydic and acidic carboxylic groups under the oxidative action of UV/O₃ radiation. Such changes would have a serious impact on the interaction of irradiated cotton fabric with chitosan vis-a-vis that of the irradiated cotton fabrics. The decline in chitosan content on the fabrics upon using chitosan concentration higher than 2% could be explained in terms of greater interaction of chitosan molecules at its higher concentration. The result is the formation of chitosan on the surface of the fabrics in amounts large enough to impede further significant deposition of chitosan.

3.3. Role of Citric Acid in Finishing UV/O₃ Irradiated Cotton with Chitosan

When the irradiated and un-irradiated cotton fabrics are treated with the chitosan finishing formulation contain-

ing citric acid, cotton cellulose enriched in free primary hydroxyl groups may bind some extra carboxyl groups through single ended reaction of citric acid with these hydroxyl groups. Hence, this treatment may further accentuate the cation uptake ability of cotton, *i.e.*, citric acid modification of cotton cellulose presents an effective means for increasing cation exchange capacity of cotton. Meanwhile the citric acid modified cotton exhibits much lower values of swelling and water retention capacity, including that cotton gains further stabilization by reacting with citric acid via crosslinking. It follows from this that single ended and crosslinking reactions of citric acid with cotton hydroxyl groups are responsible for introduction of carboxyl group for enhanced cation value and crosslinks for stabilizing of the modified cotton respectively.

Scheme 1 shows the chemical reactions of cellulose with citric acid, as is evident the mechanism of hypophosphite-catalyzed cross linking of cellulose with citric acid proceeds as two-step process. In the first step the formation of a cyclic anhydride from the acid takes place. In a subsequent step this intermediate reacts via its ring-opening with the alcohol groups of the cellulose forming the ester binding group [24]-[26] it is reported that pH of a solution containing polycarboxylic acids should be kept in the 1.5 - 4.5 range in order to successfully crosslink the fibers. It is certain however that, relatively high acidities are not advocated by virtue of their damaging effect on the fibers. Instead, more appropriate conditions are recommended using adequate catalysts in the pH range of 3.7 - 4.5 [24]. It was disclosed that addition of certain amount of sodium hypophosphite (6 g/l) in a solution containing chitosan and citric acid increases the stabilization of the cellulose which might be explained by the presence of the phosphite and the partial sodium salt of the polycarboxylic acid to accelerate the esterification reaction [24]. It is as well to report that the increase of phosphite at a fixed CA concentration (4%) results in a decrease of the chitosan content [27]. This may be attributed to the neutralization of free-carboxyl groups, thus restricting the esterification with the chitosan hydroxyl moieties. These results are in agreement with previous work reported by Coma, Sebti, Pardon, Pichavant, and Deschamps [28] in which the increase in the NaH_2PO_2 concentration resulted in decreasing crosslinking between hydroxylpropyl methylcellulose and citric acid.



Scheme 1. The chemical reactions of cotton cellulose with citric acid.

3.4. Effect of pH on Heavy Metal Removal

Figure 3 shows that the effect of pH of the heavy metal ions, namely Zn^{2+} and Cd^{2+} solution on removal of these ions using UV/ O_3 irradiated cotton fabric-containing chitosan. It is seen that heavy metal adsorption on the fabric and, therefore, metal removal increases by increasing the pH up to 9. This is observed with both cations. Nevertheless, removal of Zn^{2+} is higher than that of Cd^{2+} . Equilibrium within which chitosan reacts with metal ion may account for this. Given below is **Scheme 2** which signifies the mechanism of binding metal ion by chitosan.



The amine group of chitosan is protonated as follows:



According to Equation (2) the chitosan will be positively charged and metal ion also positively charged, so repulsive forces occur between chitosan and metal ions instead of attraction. However, increasing the alkalinity of the solution a reverse effect will take place leading to higher absorption of the metal cations [29] [30].

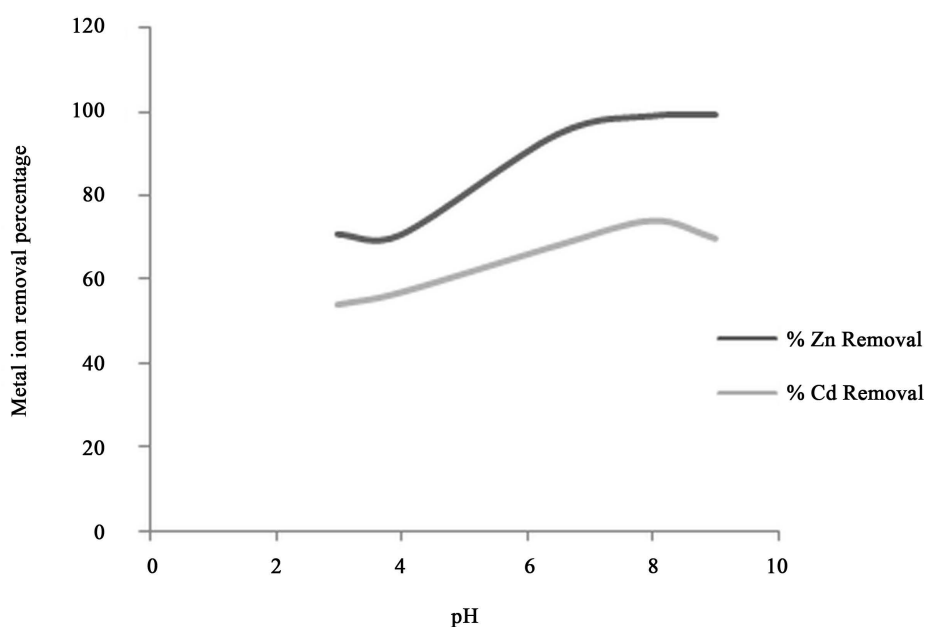
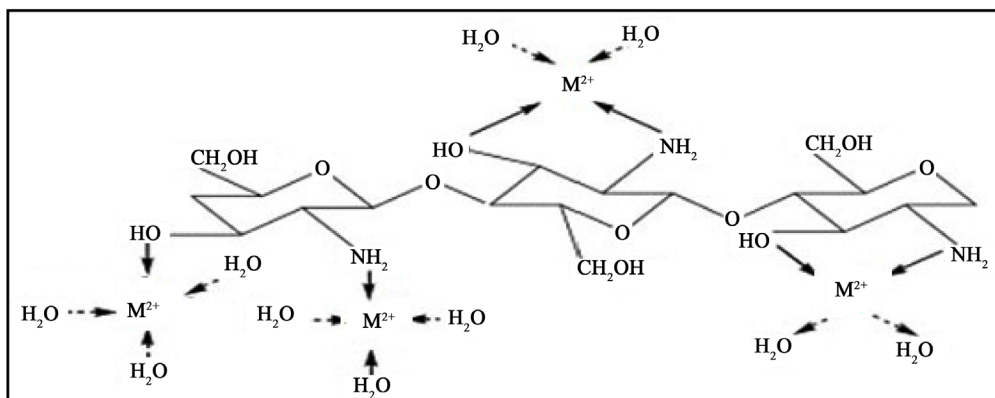


Figure 3. Effect of pH of the aqueous solution of heavy metal ions on their removal using chitosan-containing cotton fabrics. Chitosan concentration (2%), time (2 hrs), metal ion concentration (100 mg/l) and L:R (1:20).



Scheme 2. Mechanism of binding metal ion by chitosan.

3.5. Effect of Metal Ion Concentration

Figure 4 shows dependence of metal ion removal by chitosan loaded fabric on the metal ion concentration in solution. It is clear that metal ion adsorption (*i.e.* removal) increases by increasing metal ion concentration up to a certain concentration (200 mg/l) then decreases thereafter due to saturation of the absorbent with the metal ions [21]. Here too, removal of Zn ions is greater than removal of Cd ions at lower concentrations of these ions. The removal of the two types of ions becomes equal at metal ion concentration of 1000 mg/l.

3.6. Effect of Liquor Ratio

Figure 5 shows the effect of liquor ratio on the removal of metal ions from their solution. Obviously, the absorption for Zn and Cd ions increases by increasing the liquor ratio up to (1:20) then start to decrease. This could be associated with decrement in collision between reactants molecules by dilution *i.e.* by increasing the liquor ratio [31].

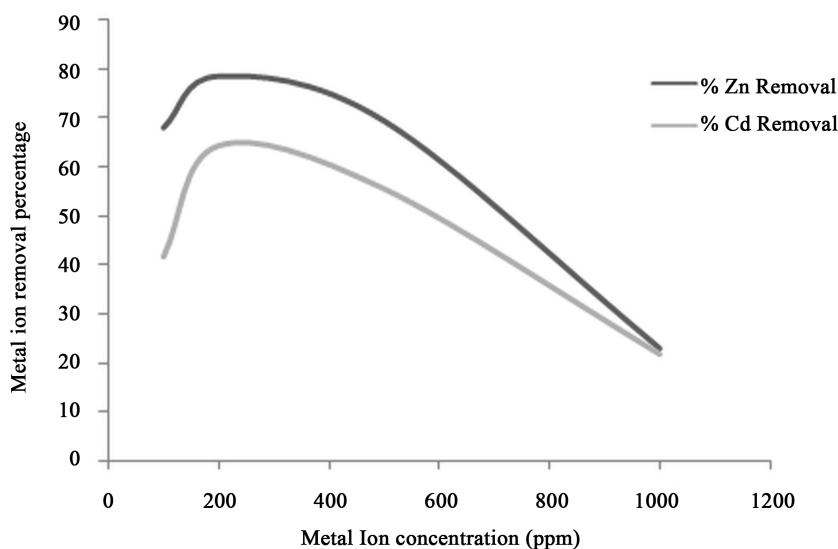


Figure 4. Effect of metal ion concentration on metal removal. Chitosan concentration (2%), time (2 hrs), pH (9), metal ion concentration (200 mg/l) and L:R (1:20).

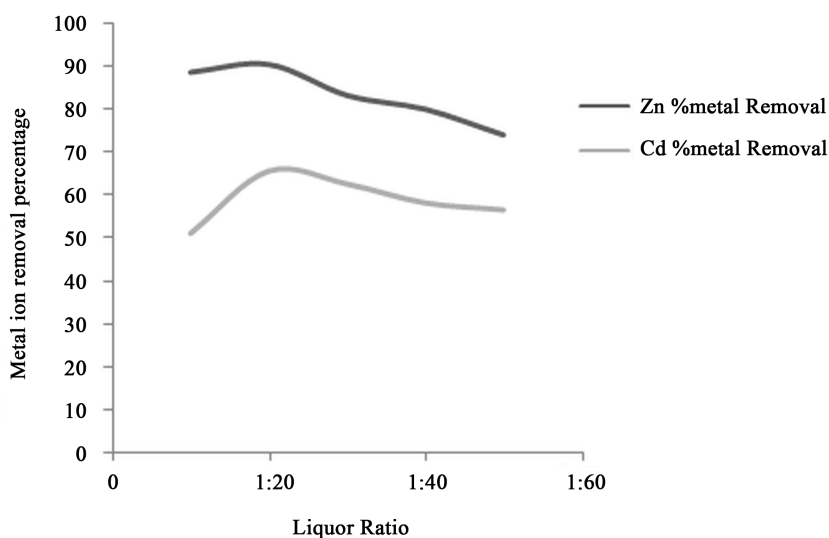


Figure 5. Effect of liquor ratio on metal removal. Chitosan concentration (2%), time (2 hrs), pH (9), metal ion concentration and (200 mg/l).

3.7. Effect of Duration of Adsorption

Figure 6 depicts the effect of duration of metal ions adsorption on their removal. It is seen that increasing the duration of adsorption is accompanied by increased metal ion removal. This is rather expected since increasing the duration of adsorption gives the opportunity for the adsorbent to capture more metal ions until a duration of 120 minutes. Thereafter removal of the metal ions decreases due to saturation [16].

3.8. FT-IR Spectral Analysis

Figure 7 shows the FT-IR spectra of unirradiated cotton and UV/O₃ irradiated cotton treated with chitosan at concentration of 2%. Comparing FT-IR spectra of the chitosan treated cotton with those of the untreated cotton fabrics would signify the presence of chitosan as revealed by its typical peak which appears only in the treated samples at 1560 cm⁻¹ and assigned to NH bending in amide group. This is in agreement with spectra reported by Sionkowska [23] related to UV-irradiated chitosan film. The other peaks due to chitosan at 3360 cm⁻¹ (OH group), 3290 cm⁻¹ (NH stretching) and 1648 cm⁻¹ (C=O amide bond) are overlapped to cellulose peaks.

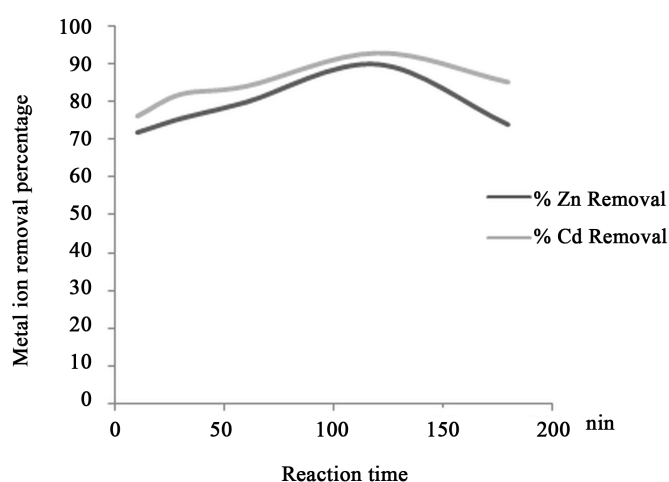


Figure 6. Effect of reaction time on metal removal. Chitosan concentration (2%), pH (9), metal ion concentration (200 mg/l) and L:R (1:10).

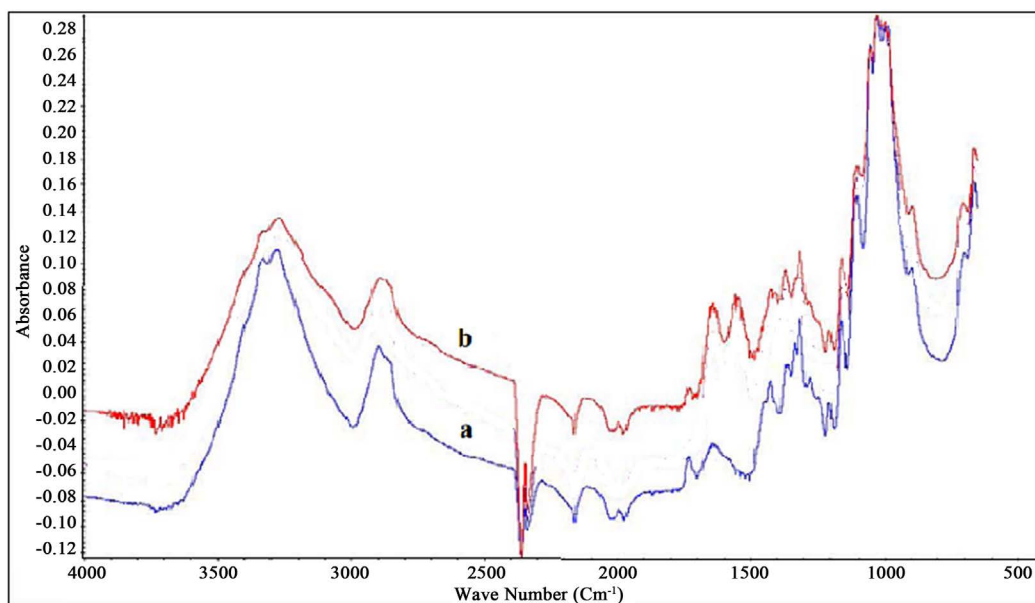


Figure 7. FT-IR spectra of a untreated cotton and b UV/O₃-chitosan 2% treatment.

4. Conclusion

Woven cotton fabrics were first irradiated using UV/O₃ for 90 minutes followed by treatment with chitosan at different concentrations (0% - 4%) dissolved in (1% v/v) of acetic acid, using citric acid (8 g/l) which was used as a crosslinking agent along with sodium hypophosphite (4%) as a catalyst for chitosan fixation on the fabrics. Fabrics so treated display a value of 2% nitrogen content and this was considered the greatest nitrogen content. The particular fabric was utilized to remove Zn²⁺ and Cd²⁺ metal ions from their aqueous solutions. The results indicated that the best conditions for this heavy metal removal were pH (9), L:R (1:20), [M] (200 ppm) for 120 minutes at ambient temperature.

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