

Self-cleaning and Antibacterial Properties of ZnO Nanoparticles on Cotton Fabric Treated with Maleic Acid

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Bi-functional finishes of cotton such as wrinkle resistance with antibacterial and self-cleaning are important in textile industry. In this case, reducing cost and duration of processing is essential. For this purpose, cotton fabric was treated with different concentrations of maleic acid (MA), sodium hypophosphite (NaH_2PO_2) and ZnO nanoparticles in two methods of separately and simultaneously. Crease recovery angle and weight gain tests were examined. For measuring carboxylic acid groups of maleic acid treated cotton fabrics, methylene blue dye was used, and then, reflective spectrophotometer was used to determine methylene blue dye absorption. Self-cleaning and antibacterial properties of ZnO nanoparticles treated cotton fabric were investigated. For measuring zinc amount, atomic absorption analysis was applied. As the Maleic acid concentration increased, weight gain, crease recovery angle and color yield increased. Color difference or self-cleaning effect of treated fabrics showed that by increasing maleic acid concentration, color difference increased in separate method. In simultaneous treatment, by increasing MA concentration and cross-linked sites, there was a slight increase in ΔE and self-cleaning effect. Results indicated that ZnO nanoparticles amount in separate method of treatment increased as compared with simultaneous method. Antibacterial performance decreased for simultaneous method of ZnO nanoparticles treatment.

Keywords: self-cleaning, antibacterial, cotton, wrinkle resistant, maleic acid, separate method.

1. INTRODUCTION

Polycarboxylic acids such as BTCA and Citric acid (CA) are non-formaldehyde durable press and crease resistant finishes for cotton fabrics. Maleic acid, succinic acid and tartaric acids are bifunctional carboxylic acids. Maleic acid (MA) in the presence of NaH_2PO_2 is used for wrinkle free or crease resistant of cotton fabric. By treating cotton and MA, esterification of MA happens which causes single ester linkage with cellulose. By increasing the temperature of curing, the cotton fabric treated with MA and sodium hypophosphite shows higher crease resistance, that forms cross-link of cellulose [1–3]. Yang showed that bi-functional acids and polycarboxylic acids had similar flame retardant properties. CA or BTCA treated cotton fabric indicated increase of cross-linking, stiffness, dimensional stability and crease resistance. [4–7]. Maleic acid (MA) at the present of NaH_2PO_2 reacts with cellulose to form cellulose-ester linkage, crease resistance and fire performance without change in fabric properties [2, 8, 9].

Zinc oxide is widely used in sun-screens, UV absorbers, anti-reflection coatings, photo-catalysis and catalyst [10–13]. ZnO particles are used in polymers, fabrics and cosmetic materials for UV blocking properties. This property of zinc oxide is produced when it has a nano-size. The band-gap around 3.37 eV belong to ZnO and ZnO can absorb light around 376 nm. Range of ultra violet light occurs at 376 nm and zinc oxide can absorb this light [14–16]. Because of this photo-catalytic property, zinc oxide acts as antimicrobial and self-cleaning agents under UV light [17–19]. The metal oxides like CaO, MgO and

ZnO have antibacterial activity due to the formation of reactive oxygen species on the surface of metal oxides [17, 20]. Abdelhady synthesized chitosan/ZnO nanoparticles and loaded it on cotton fabric to achieve antibacterial performance and UV blocking. Improvement in UV protection and antibacterial properties were obtained by increase of ZnO/chitosan concentrations [21]. Zinc oxide-soluble starch nanocomposites (nano-ZnO) synthesized using water as a solvent and soluble starch as a stabilizer and coated on paper. By the treatment, antibacterial, surface strength and smoothness of paper were enhanced [11]. Self-cleaning and antibacterial properties of ZnO treated textiles were investigated in previous works [22–26].

In this research, cotton fabric was treated with ZnO nanoparticles and Maleic acid (MA) for self-cleaning effect and crease resistance. For this purpose the cotton fabric was coated with maleic acid, and then was treated using ZnO nanoparticles (separate method). In the second method, cotton fabric was treated with maleic acid and zinc oxide nanoparticles simultaneously.

2. EXPERIMENTAL

2.1. Materials

The used fabric in this study was a plain woven cotton fabric with weight of 143.3 g/m^2 . The chemical used was ZnO nanoparticles colloidal solution with average size between 10 to 35 (nm) from Sigma Aldrich (Germany), Maleic acid and sodium hypophosphite from Merck (Germany). Methylene blue dye and nitric acid from (Applichem., Germany) were applied in this research.

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2.2. Methods

Cotton fabrics were treated in two methods of separate and simultaneous. In a separate method, the cotton fabric was coated using maleic acid at concentrations of % 2 to 6 (% owf) with NaH_2PO_2 (% 1 to 3) at 60 °C for 30 minutes. Then they were dried at temperature of 100 °C, cured at temperature of 180 °C for 2 minutes. Then treated fabrics were loaded with zinc oxide nanoparticles at concentration of % 0.1 (% owf) and temperature of 60 °C for 30 minutes, and cured at temperature of 180 °C for 2 minutes. In the simultaneous method, the ZnO nanoparticles and maleic acid were loaded at the same time with the same amount as much as separate method were applied at temperature of 60 °C for 30 minutes, then cured at temperature of 180 °C for 2 minutes.

For determining the carboxylic acid groups, fabrics were immersed in % 0.1 solution of methylene blue. After 30 minutes the samples were dried at ambient temperature. K/S value of methylene blue stained fabrics were measured by reflectance spectrophotometer (X-rite 7000A) according to AATCC standard EP6 at reflectance of 482 nm [27].

The weight of fabrics was obtained before (W_1) and after (W_2) ZnO nanoparticles and MA treatment of cotton fabric according to AATCC D3776 method [28]. Weight change (ΔW) was measured according to the Eq. 1:

$$\Delta W (\%) = \frac{W_2 - W_1}{W_1} \times 100. \quad (1)$$

Crease recovery angles of treated fabric were determined using AATCC standard test method 66. The sum of the crease recovery angles of the warp and weft (fill) were measured.

AATCC-100 standard method was used for antibacterial performance [29]. The test was carried out with two bacteria: *Escherichia coli*, and *staphylococcus aureus*. Colonies were counted after 24 hours and bacteria reduction was measured from the Eq. 2:

$$\text{Bacteria reduction (\%)} = (C - A/C) \times 100, \quad (2)$$

where C is the counted colonies of cotton fabric and A is the counted colonies of ZnO nanoparticles coated cotton fabric.

Atomic absorption spectroscopy was used for determining zinc ion amount. The cotton fabrics treated with ZnO nanoparticles were placed in furnace (Naberthern, Germany) at temperature of 600 °C for 3 hour, and then a solution prepared for analysis. The zinc ions content were reported in terms of grams per 100 grams of fabric.

Self-cleaning properties of ZnO nanoparticles treated fabrics were determined by staining methylene blue dye (% 0.01) on treated fabrics. After drying the samples, the stained fabrics were exposed to sun light for 30 and 60 hours, and color difference of fabrics were measured before and after exposing to light using X-rite 7000A reflectance spectrophotometer. The color difference was measured according to the Eq. 3 and according to AATCC standard EP7 [30]. Where L^* is lightness, and a^* and b^* are indicators of red-green and yellow-blue colors, respectively.

$$\Delta E = \sqrt{\Delta a^{*2} + \Delta b^{*2} + \Delta L^{*2}}. \quad (3)$$

FTIR/ATR Bruker Tensor 27 was used for infrared spectra of cotton, MA treated cotton and ZnO nanoparticles treated cotton. Spectra were collected at a resolution of 4 cm^{-1} and 32 scans.

3. RESULTS AND DISCUSSION

Fig. 1 shows SEM micrographs of ZnO nanoparticles treated cotton fabric with maleic acid in separate methods. The particles with the size of nano meter were related to ZnO nanoparticles.

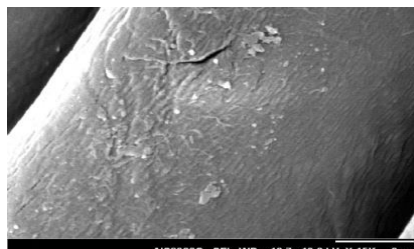


Fig. 1. SEM micrographs of treated fabrics with ZnO nanoparticles

Table 1 shows weight gain percentage, crease recovery angle and color yield of Maleic acid treated cotton fabric. By increasing the maleic acid concentration to 6 %, the weight gain and crease recovery angles of fabrics increased (Table 1).

Table 1. Weight gain, crease recovery angle and color yield of maleic acid treatment of cotton fabrics

Maleic acid, %	Weight gain, %	Crease Recovery angle, degree	Color yield, k/s
–	–	260.2	12.54
2	2.207	290.8	15.27
4	3.125	316	15.36
6	3.523	320.2	16.53

Un-treated cotton had crease recovery angle of 260.2, by treatment of 6 % maleic acid it reached to 320.2. As the maleic acid concentration increased, cross-linking of cellulose caused more wrinkle resistant fabric. Methylene blue dye is a cationic dye which has a tendency to anionic surface, so that is able to absorb on the carboxylic groups of cotton. By maleic acid treatment of cotton fabric, carboxylic acid groups increase on cotton. In Table 1, color yield of stained cotton fabric, increased from 12.54 to 16.53. The increase of color yield indicates the higher absorption of methylene blue dye, therefore higher carboxylic acid formation. Maleic acid can reach to higher wrinkle resistance of cotton fabric, and cause a single ester bond to cellulose, and disable to form the second cyclic anhydride intermediate for cross-linking of cellulose. Sodium hypophosphite and two MA esterify with cellulose and cross-linking of two celluloses happens [31]. The increase of fiber wrinkle resistance has dependency on MA and NaH_2PO_2 concentration. H-P bond of NaH_2PO_2 adds to C=O of MA to bond cotton by esterification and generating crosslink between two celluloses (Fig. 2).

After ZnO nanoparticles treatment, weight gain and crease recovery angle and color yield of treated fabrics were measured (Table 2).

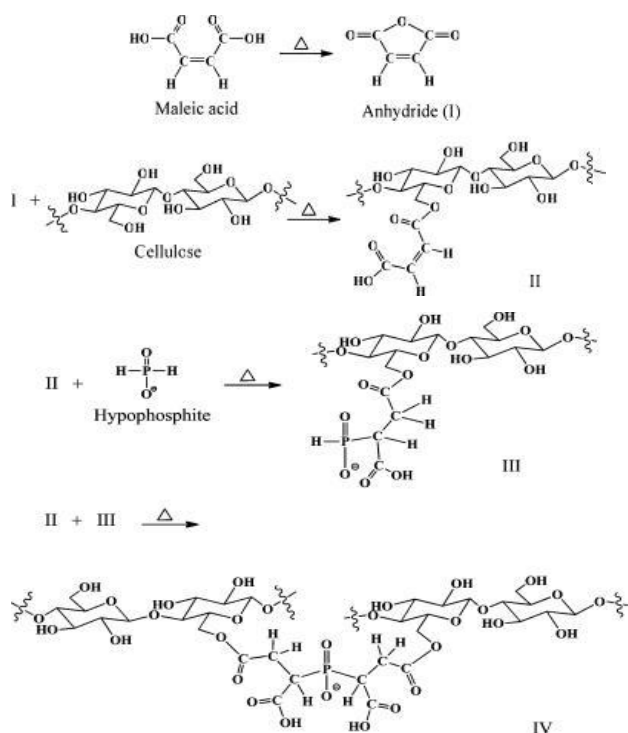


Fig. 2. Crosslinking of cellulose and maleic acid [2]

Table 2. Weight gain, crease recovery angle and color yield of ZnO nanoparticles treatment (Separate method:A, Simultaneous :B, MA: Maleic acid

	A	B	A	B	A	B
MA, %	Weight gain, %		Crease recovery angle, degree		Color yield	
–			218	218	–	–
2	0.155	0.46	223.4	283.6	16.44	16.81
4	0.291	0.643	252.6	314.6	17.38	18.34
6	0.37	0.773	267.5	316.6	20.23	20.38

By increasing maleic acid concentration from 2 % to 6 %, Color yield of ZnO nanoparticles in two steps of treatment (Separate ZnO) increased (Table 2). In simultaneously treatment, color yield reached to 20.38. There was not any significant difference between two methods in color difference. It means that there was not any difference between two methods in absorption of methylene blue dye, therefore the same carboxylic acid groups' sites. Crease recovery angle of fabrics were increased by increasing concentration of maleic acid. In simultaneous treatment of ZnO nanoparticles with maleic acid, Crease recovery angle was higher than separate method (two steps method) of treatment. In separate method and second step of treatment, ZnO nanoparticles were treated at temperature of 180 °C, this fixing temperature might be a reason for breaking the cross-linked cellulose-maleic acid bonding. The other possible reaction might be related to catalytic effect of metal oxide such as ZnO which act as a catalyst in reaction between maleic acid and cellulose that affected on cross-linking [32]. Weight gain of treated cotton with maleic acid and ZnO nanoparticles increased in simultaneous method (one step) as compared with separate method.

Table 3 shows color difference of ZnO nanoparticles treated cotton fabric after exposing to sunlight for self-cleaning effect. By increasing maleic acid concentration,

color difference increased in separate method. In first step of the treatment of separate method, cross-linking of cellulose and MA happened, and then, by adding ZnO nanoparticles, ZnO nanoparticles penetrate into the fiber structure, more cross-linked sites, and more ZnO nanoparticles penetration.

Table 3. Color difference (ΔE) of ZnO nanoparticles treated cotton

	Separate ZnO	Simultaneous	Separate ZnO	Simultaneous
MA, %	30 h	30 h	60 h	60 h
2	3.878	3.342	4.765	4.301
4	4.519	3.637	5.504	4.384
6	8.319	3.864	9.924	4.386

The increase in ZnO nanoparticles penetration caused increase in ΔE (color difference), therefore self-cleaning effect increased (Table 3). ZnO nanoparticles are capable of photo-degrading and self-cleaning of organic stain and dirt because of its photo catalytic performance. The self-cleaning happens by direct photo oxidation of the organic compounds in the presence of H^+ which leads to the decomposition of materials and removal from textiles. Exciting of ZnO leads to the formation of e^- and h^+ . In the presence of water and oxygen, two photochemical reactions occur at the surface of ZnO: oxidation of ZnO that photo induced positive holes are involved and the other reaction is reduction which photo induced negative electrons are involved and the HO_2° and O_2° were produced. Therefore, combination of the HO_2° leads to the formation of H_2O_2 (Fig. 3) [33, 34]. In simultaneous treatment, by increasing MA concentration and cross-linked sites, there was a slight increase in color difference. It showed lower amount of ZnO nanoparticles absorption.

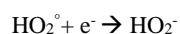
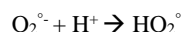
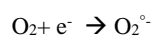
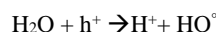
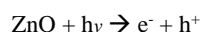


Fig. 3. Scheme of antibacterial and sel-cleaning mechanism of ZnO [30]

Fig. 4 shows zinc ion content on ZnO nanoparticles treated cotton fabrics which obtained from atomic absorption analysis.

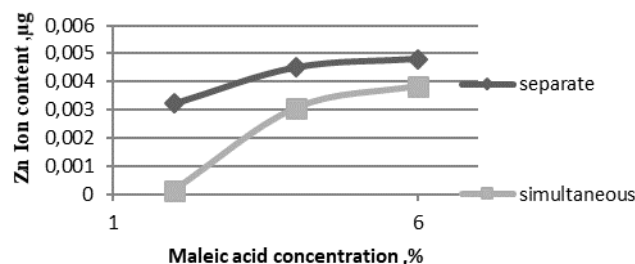


Fig. 4. Zn ion content per 100 grams of cotton fabric (in terms of µgrams)

By increasing maleic acid concentration, the zinc ion content increased. In comparison between separate and simultaneous method, separate method showed higher Zn ion content. It means that in separate method of ZnO nanoparticles loading on wrinkle resistant cotton, higher amount of ZnO nanoparticles were absorbed. In simultaneous reaction of ZnO and Maleic acid with cotton fiber, the possible dominate reaction is related to maleic acid bonding with cellulose, then absorption of ZnO nanoparticles happens on cross-linked cellulose. ZnO nanoparticles and Maleic acid can be absorbed by hydroxyl groups of cellulose.

Fig. 5 shows bacteria reduction percentage of ZnO nanoparticles treatment of maleic acid treated cotton fabrics. The higher amount of antibacterial performance was related to separate method of treatment. In separate method of treatment, maleic acid treatment caused cross-linked sites, then ZnO nanoparticles were added to treated cotton. The antibacterial effect of ZnO nanoparticles is because of very reactive species of superoxide, hydrogen peroxide and hydroxyl that acts on the surface of ZnO after exposing to UV and light. The possible reaction according to researches is the formation of H_2O_2 on the surface of the ZnO which inhibits growth of bacteria. The formation of H_2O_2 is according to Fig. 3 [34]. These results were agreement with self-cleaning results (Table 3) which separate method indicated higher color difference. The atomic absorption analysis also indicated the same results (Fig. 4) and the separate method showed higher Zn ion content, therefore higher ZnO nanoparticles.

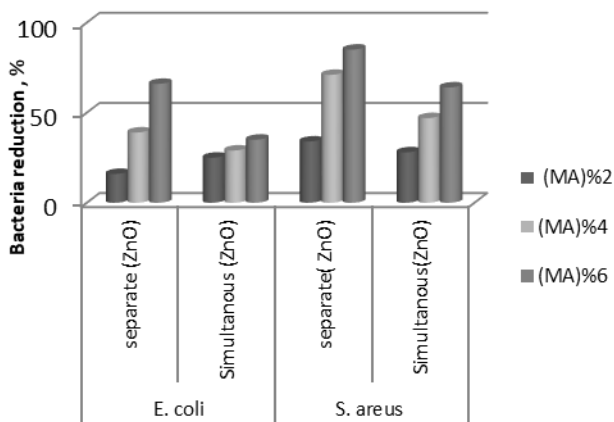


Fig. 5. Bacteria reduction percentage of ZnO nanoparticles treatment on maleic acid treated cotton

Fig. 6 a shows the FTIR/ATR spectra of un-treated cotton and treated cotton with maleic acid. It can be seen that the band at 1644 cm^{-1} is due to the bending mode of the hydrogen-bonded hydroxyl group of cellulose [31], and for maleic acid treated cotton, band at 1740 cm^{-1} is due to the carbonyl bands carboxyl and ester [2], which was formed between MA and cotton at curing. Fig. 4 b indicates FTIR/ATR spectra of cotton, treated cotton with maleic acid (cotton+MA), maleic acid treated cotton with ZnO in one step (simultaneous) and two steps (separate) methods. The band at 3371 cm^{-1} is related to hydroxyl groups of cellulose [35]. The intensity of peak at 3371 cm^{-1} for separate method increased as compared with

simultaneous method. The higher intensity might be related to the higher banding between ZnO and cellulose or higher absorption of ZnO nanoparticles. The peak at 530 nm is due to the ZnO nanoparticles [31, 35].

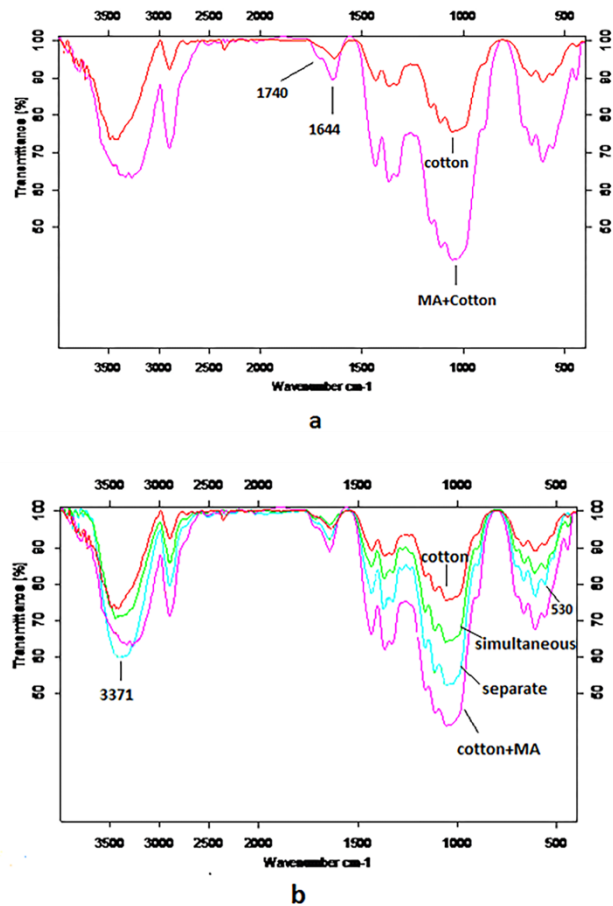


Fig. 6. FTIR/ATR spectra of cotton, Maleic acid treated cotton (Cotton+ MA): a – spectra of cotton, Cotton +MA, ZnO loading in simultaneous method; b – in separate method

4. CONCLUSIONS

The ZnO nanoparticles treatment of cotton fabric with maleic acid increased wrinkle resistance, weight gain and methylene blue dye absorption. Color difference or self-cleaning effect of treated fabrics showed that by increasing maleic acid concentration, color difference increased in separate method. In simultaneous treatment, by increasing MA concentration and cross-linked sites, there was a slight increase in ΔE and self-cleaning effect. Results indicated that ZnO nanoparticles amount in separate method was higher than simultaneous method. Antibacterial performance increased for separate method of ZnO nanoparticles treatment. Although, simultaneous methods of treatment can be useful in textile industry for reducing costs and duration of processing, the separate method is useful for self-cleaning and anti-bacterial performance of wrinkle resistant cotton fabric.

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