In situ Production and Deposition of Nanosized Zinc Oxide on Cotton Fabric

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ABSTRACT: *In this research, the cotton fabric was modified with nanosized zinc oxide (ZnO) by a simple and novel approach. The nanosized zinc oxide was prepared and deposited onto cotton fabrics by in situ method using zinc acetate dihydrate ($Zn(OAc)_2.H_2O$) as precursors and sodium hydroxide, with and without starch as a capping agent. The size and morphology of nanosized zinc oxide on cotton fabric in the presence and absence of starch were investigated. The samples were characterized by X-Ray Diffraction (XRD), Fourier Transform InfraRed (FT-IR) spectroscopy, Scanning Electron Microscopy (SEM), Energy-Dispersive X-ray spectroscopy (EDS), Atomic Absorption Spectroscopy (AAS), and contact angle. The antibacterial activity of modified cotton was evaluated by measurement of the reduction of the gram-negative of Escherichia coli (E. coli) on the treatment of cotton. SEM images of treated fabrics were indicated that nanosized zinc oxide was well dispersed on the surfaces of samples. The results of the contact angle revealed the more hydrophobic character of treatment of cotton as compared with blank, which will have high potential applications in various fields.*

KEYWORDS: *Cotton fabric; In situ; Nanosized; Starch; Zinc oxide.*

INTRODUCTION

In recent years, the application of nanotechnology has attracted considerable interest from the high-performance textiles area [1]. Functional clothes are one of the goals of the textile industry for the improvement properties with potential advantages [2]. The antibacterial textiles are one of the most important applications of cotton, wool and silk fabrics [3]. The nanostructure materials are good ideas to attract the antibacterial applications of cotton fabrics [4]. Antibacterial agents can be classified into two types of organic and inorganic materials. The inorganic materials are expanded because of stability under harsh process conditions and safety to humans and animals [5].

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Nanomaterials are used onto fibers with a wide range of compositions and various structures. These materials are resulted in unique properties due to the high surface area [6]. The nanosized ZnO is one of the most common nanomaterials that is prepared with different methods such as hydrothermal [7], sol-gel [8], green synthesis [9], thermal decomposition [10], and laser ablation [11].

Cotton fabrics are widely used by people as a kind of natural textiles because of their excellent properties such as softness, breathability, biodegradation, safeness, and low-price, affinity to skin, regeneration performance, and hygroscopic property. However, cotton fabrics provide an excellent environment to retain moisture and grow microorganisms. To overcome such problems, numerous materials have been employed to impart antimicrobial activity to cotton fabrics [4]. Extensive research has been done for the treatment of fabric with nanomaterials. *In situ* production and deposition of the silver nanoparticle is a good candidate for the treatment of cotton [12] and knitted cellulose fabric [13] with antimicrobial property [14]. Among the antibacterial fabrics of interest are copper [15], copper oxide (CuO) [16-17], silver [18-19], titanium dioxide (TiO_2) [20-21], and zinc oxide [22-26]. Other uses of cotton fabric are also important, such as the decrease of wrinkle-able nature was reported by nanomaterial based on silicone [27]. The self-cleaning of cotton textile was reported by nanomaterial based on TiO_2 [28] and SiO_2/TiO_2 [29] as photoactive coatings. The super-hydrophobic textiles were reported via a wet chemical route using a modification of cotton fabric with ZnO nanorod array film [30]. The UV-blocking of textile was studied by ZnO with nanoparticles [31] and nanorods [32].

The objective of this study was to develop the *in situ* production and deposition of nanosized zinc oxide on cotton fabric with and without starch as a capable procedure for modification of the surface fabric. This simple chemical preparation was decreased the time of production and deposition of nanosized zinc oxide on cotton fabric and increased the advantage of this method. The novelty of this work was the investigation and evaluation of the starch effect on size and morphology, hydrophobic property, and Zn release of treatment of cotton fabric. The antibacterial activity of the modified cotton fabric sample was tested against gram-negative bacteria of *Escherichia coli*. The samples also were characterized by various techniques.

EXPERIMENTAL SECTION

Materials

Zinc acetate dihydrate ($Zn(OAC)_2.2H_2O$), sodium hydroxide (NaOH) and soluble starch were purchased from Merck (Darmstadt Germany). Cotton fabric was purchased from Sarab Baft Company.

Methods

Synthesis of nanosized zinc oxide

The nanosized zinc oxide was prepared by a wet chemical method using zinc acetate and sodium hydroxide as precursors based on the previous reports [33]. In this research, the nanosized zinc oxide was prepared without and with starch as a capping agent via hydrothermal synthesis. The zinc acetate dihydrate (0.2195g, 1 mmol) was dissolved in distilled water and the aqueous solution was prepared in two conditions including without and with starch (1% w/v). Then, 10 ml NaOH (1 M) was added drop by drop for 30 minutes. After that, the reaction was allowed to proceed for 2 h at 90 °C by an oil bath. Finally, the solution was centrifuged at 10,000 rpm for 10 min and the supernatant was discarded. The white product was washed three times using distilled water to remove the by-products and the excessive starch. The obtained samples were dried at 80°C for overnight.

In situ synthesis of nanosized zinc oxide on cotton fabrics

The cotton fabric (2.5×2.5cm) was immersed in NaOH (0.01 M) solution for 1 h at 90°C and washed using distilled water. *In situ* deposition of nanosized zinc oxide on the cotton fabrics was carried out a simple method in a short time and there is no need to re-prepare the fabric. Accordingly, the cotton fabric was immersed in the precursors of nanosized zinc oxide with and without starch for 2 h at 90°C. Then, the samples were washed for 3 times using distilled water and then dried at 37°C in an oven.

Characterization

The samples were characterized by X-ray diffraction, IR spectroscopy, scanning electron microscopy, EDS analysis, contact angle, and atomic absorption spectroscopy.

The X-ray diffraction patterns of the products were collected utilizing Cu Kα X-ray radiation with a voltage of 40 kV and a current of 30 mA by X'pert pro diffractometer (Equinox3000, France). Fourier transforms infrared spectra of control and treatment of cotton fabrics were recorded using a Nicolet 380 FT-IR spectrophotometer (Thermo electron corporation, USA). A scanning electron microscope (EM-3200 model, KYKY, China) was employed to observe the morphology and size. Atomic absorption spectroscopy measurements were performed (Perkinelmer model). The contact angle was evaluated (OCA 15 plus model, Dataphysics Swiss). The antibacterial activities were evaluated by disk diffusion method against *E. coli*, strain ATCC 8739, a gram-negative bacterium. The samples were placed on germ-containing agar plates, inoculated with *E. coli* then incubated in an agar media. Bacteriostatic activity of the sample was evaluated after 24 h of incubation at 37°C and investigated the percent reduction of bacteria and inhibition zone.

RESULTS AND DISCUSSION

X-ray diffraction

The X-ray diffraction patterns of samples were measured from 10 to 110 (2θ) (Fig. 1). XRD results were indicated the successful formation of nanosized ZnO in present or absent of starch. There are four strong peaks at 14.7, 16.3, 22.5, and 34.6° attributed to (100), (002), (101), and (102) crystal planes of ZnO respectively. Based on the results, the crystalline structure was similar to the previous report (JCPDS card No. 36-1451) [9]. For the treatment of cotton fabric, the XRD results were approved in the previous report [34].

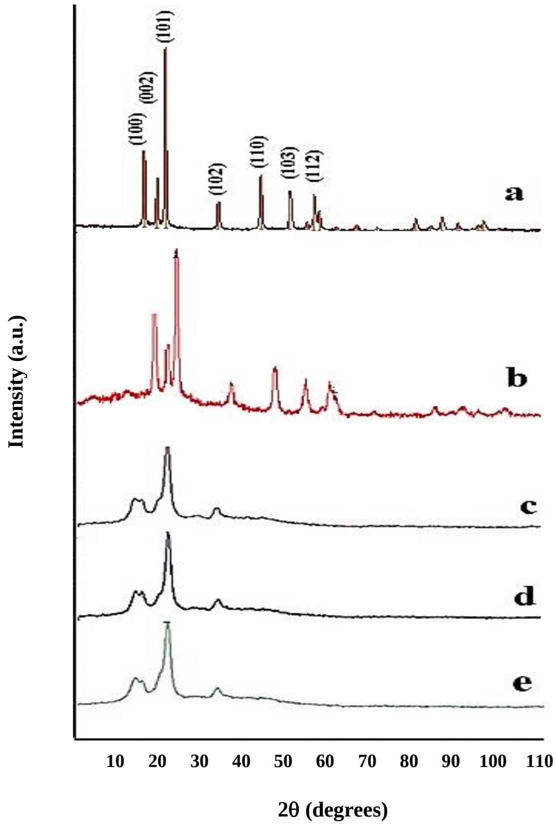

Fig. 1: XRD pattern of (a) nanosized ZnO in absence of starch, (b) nanosized ZnO in presence of starch (c) control cotton fabric, (d) treatment of cotton fabric in the present of starch, and (e) treatment of cotton fabric in absent of starch.

Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy are shown in Fig. 2 for nanosized zinc oxide, control cotton fabrics, and treatment of cotton fabrics. The absorption band at 422.79 cm^{-1} was attributed to the stretching vibration of metal-oxygen (Zn-O). The result was corresponding with the previous report [30]. Two broad bands were illustrated between 3000-3700 cm^{-1} and 2800-3000 cm^{-1} that the first one is related to O–H stretching vibrations for functional groups and the second one can be related to C=O residues due to CO_2 atmospheric and C-H stretching in the cellulose of cotton fabric. Peaks at the 1428 and 1369 cm^{-1} were assigned to C-H bending, and the result was corresponding with the previous report [35]. According to previous reports of cotton fabric, the minor shifts of peaks may be due to hydrogen bond as the interaction between ZnO nanostructures and cotton fabrics in comparison with control fabric [36-37]. The FT-IR results indicated the qualitative formation of ZnO deposition on the surface of treatment of cotton fabric with or without soluble starch.

Scanning electron microscopy

Scanning electron microscopy has investigated the size and morphology of ZnO nanostructures and nanosized zinc oxide on the surface of treatment of cotton fabrics with or without starch (Fig. 3). The result images were shown ZnO nanostructures spherical and rod shape with and without starch, respectively. The size of ZnO nanostructures was the average diameter of 52 nm and length of micrometer for nanorods (Fig. 3.a), and

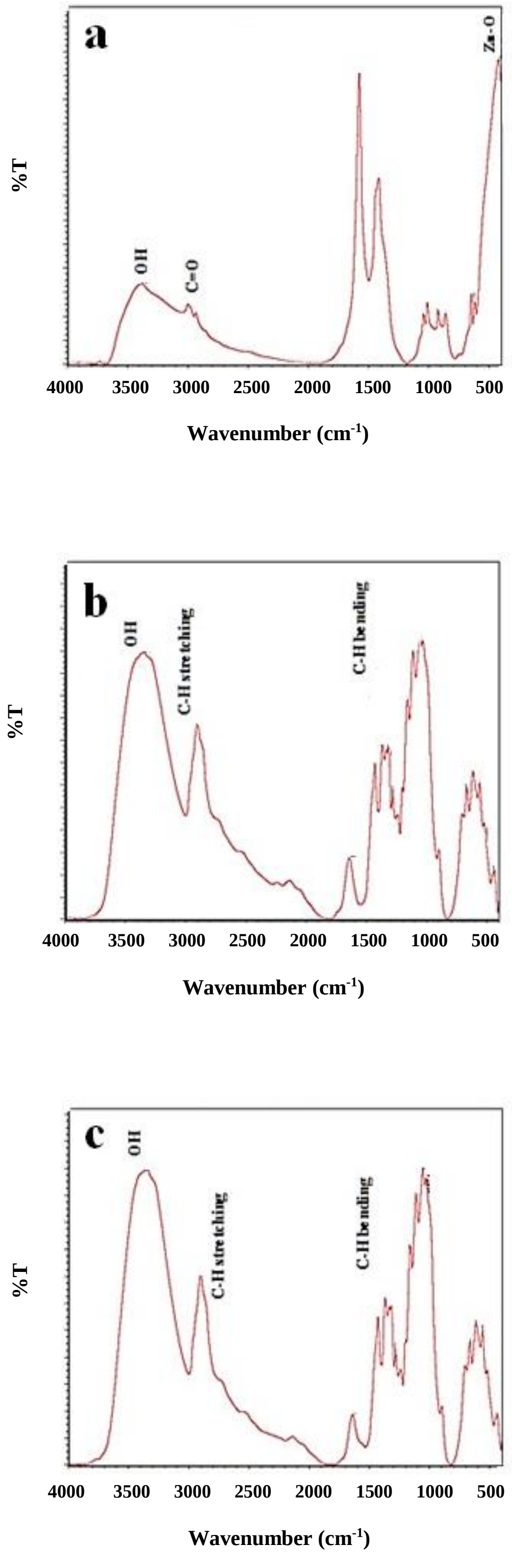

Fig. 2: *FTIR spectra of (a) nanosized ZnO, (b) control cotton fabric, and (c) treatment of cotton fabric.*

the average size of 88 nm (Fig. 3.b) for nanoparticles. Starch acted as a capping agent and affected on size and morphology of nanosized zinc oxide by increasing nucleation and controlling the growth of nanostructure. Scanning electron microscopy was shown the successful deposition of ZnO nanostructure on the cotton fiber with and without starch. Zinc oxide with a spherical shape can be observed on cotton fiber with a rough tube surface. The average size of ZnO nanoparticles without and with starch on the surface of fabrics was 67 nm (Fig. 3.c) and 87 (Fig. 3.d), respectively. These results indicated the successful formation of ZnO nanoparticles on the surface of treatment of cotton fabric with or without soluble starch. The ZnO nanoparticles were observed with spherical morphology that corresponded to the previous report for the treatment of cotton fabric with chitosan/ZnO nanoparticles [38].

Energy-dispersive X-ray spectroscopy

Energy-dispersive X-ray spectroscopy was used to evaluate the chemical composition of treatment of cotton fabrics with ZnO nanoparticles (Fig. 4). This analysis was clearly showed strong peaks of elements such as carbon (C), oxygen (O), and zinc (Zn) after deposition of nanosized zinc oxide, indicating that ZnO nanostructures were deposited on the surface of cotton fabrics. The EDS results indicated the presence of zinc and oxygen in treatment of cotton fabric with or without soluble starch, and approved the previous report [34].

Contact angel

The wettability of the cotton fabrics was investigated using the digital photograph image of spherical water droplets. Generally, if the water contact angle is smaller than 90°, the solid surface is considered hydrophilic and if the water contact angle is larger than 90°, the solid surface is considered hydrophobic [39]. The control cotton fibers were hydrophilic with the water contact angle of 69.3° and 69.1° because of the abundant surface hydroxyl groups and observed water absorption easily (Fig. 5.a). The treatment of cotton fabrics with zinc oxide were turned hydrophilic in absent starch with the water contact angle of 76.4° and 73.6° because of the presence of nanosized zinc oxide (Fig. 5.b). The decrease in water contact angle was assigned to the decrease in hydrophobicity. The treatment of cotton fabrics with zinc oxide were shown hydrophobic in presence of starch with the water contact angle of 90.9°

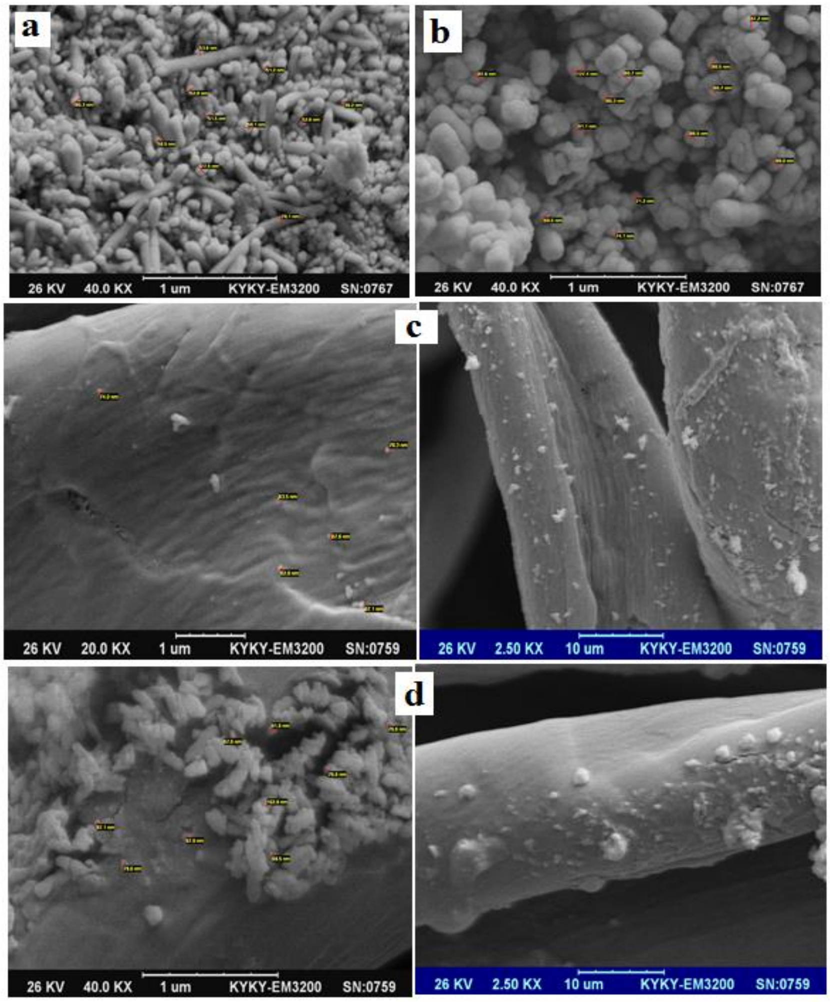

Fig. 3: SEM image (a) nanosized ZnO (b) nanosized ZnO in the presence of starch (c) nanosized ZnO on cotton fabric (d) nanosized ZnO on cotton fabric in the presence of starch.

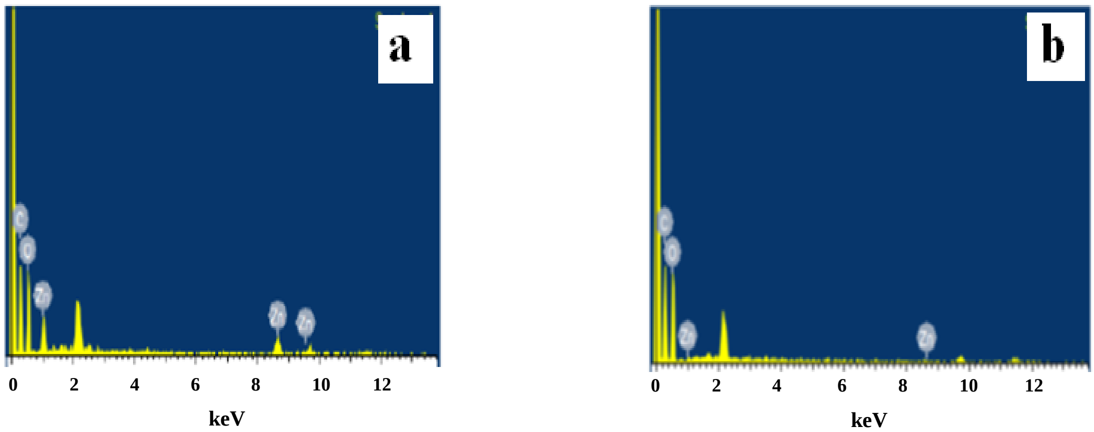

Fig. 4: EDS of (a) nanosized ZnO on cotton fabric (b) nanosized ZnO on cotton fabric in presence of starch.

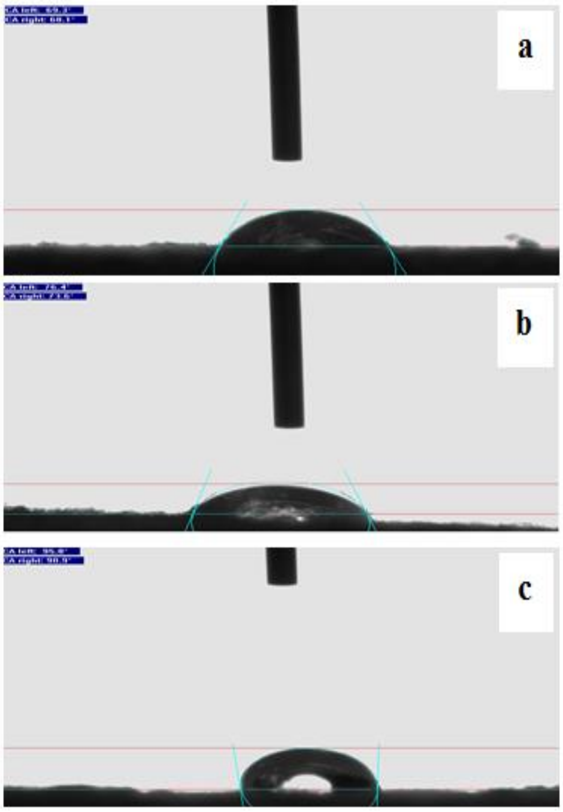

Fig. 5: The water contact angle of (a) cotton fabric, (b) nanosized ZnO on cotton fabric, and (c) nanosized ZnO on cotton fabric in presence of starch.

and 95.5° that is clearly revealing the surface was changed (Fig. 5.c). These results indicated the hydrophobic and hydrophilic properties in the treatment of cotton fabric with or without soluble starch, respectively. The contact angel results were approved the wettability properties of treatment of cotton fabric with ZnO nanoparticles [40].

Atomic absorption spectroscopy

The released zinc was evaluated using atomic absorption spectroscopy at 213.9 nm during washing. The treatment of the cotton fabric sample (1 g) was washed 3 times using a washing bath at room temperature and 40 °C. The measurement of Zn content is shown in Fig. 6 for the treatment of cotton fabrics with and without starch. Based on the results, the higher amount of released Zn was shown for the treatment of cotton fabric without starch and washing at more temperature. There was the hydrogen bond as the interaction between nanosized zinc oxide and cotton fabrics, and the nanostructures didn't have the covalent bond with the fabric surface. Also, the amount of released zinc was decreased through three rinse cycles of treatment of cotton fabrics. Therefore, starch improved the washing durability of treatment of cotton fabrics with nanosized ZnO at two tested temperatures. The results of released Zn were presented for the first time at 40 °C, and corresponded to the previous report for various shapes of zinc oxide nanoparticles at room temperature [33].

Antibacterial test

The antibacterial activity of treatment of cotton fabrics with zinc oxide was evaluated using agar diffusion and the absorption method against *E. coli* (strain ATCC 8739) at 37 °C for up to 24 h (ISO 20645:2004). The inhibition zone was 1 mm for the treatment of cotton fabrics with and without starch, indicating antibacterial activity in comparison with control cotton fabrics. The antibacterial results were approved the previous reports by *in situ* synthesis and sol-gel method [41], and using the pad-dry-cure method [42] in modified textiles with zinc oxide nanoparticles.

CONCLUSIONS

In this study, cotton fabrics were modified with zinc oxide nanostructure using *in situ* immersion method. The presence of starch affected the size, morphology, hydrophobicity, and release of ZnO nanostructure deposited on the fabric surface. Starch controlled morphology and size by capping of growth planes and observed more nanoparticles with a slightly larger size on cotton fabrics. Also, starch caused an increase in the amount of nanosized zinc oxide and a decrease of hydrophilic and release in treated fabrics. The change of hydrophilic property is possible for cotton fabric with surface modification to hydrophobic properties. The presence of starch cased to decrease of zinc release from cotton fabric. Based on the result, treatment of cotton fabrics showed antibacterial agent against *E. coli* compared to untreated fabrics and can have huge potential for different applications in future goals. The important innovation is *in situ* production and deposition of nanosized zinc oxide on cotton fabric with and without starch for expansion of other uses to be concentrated.

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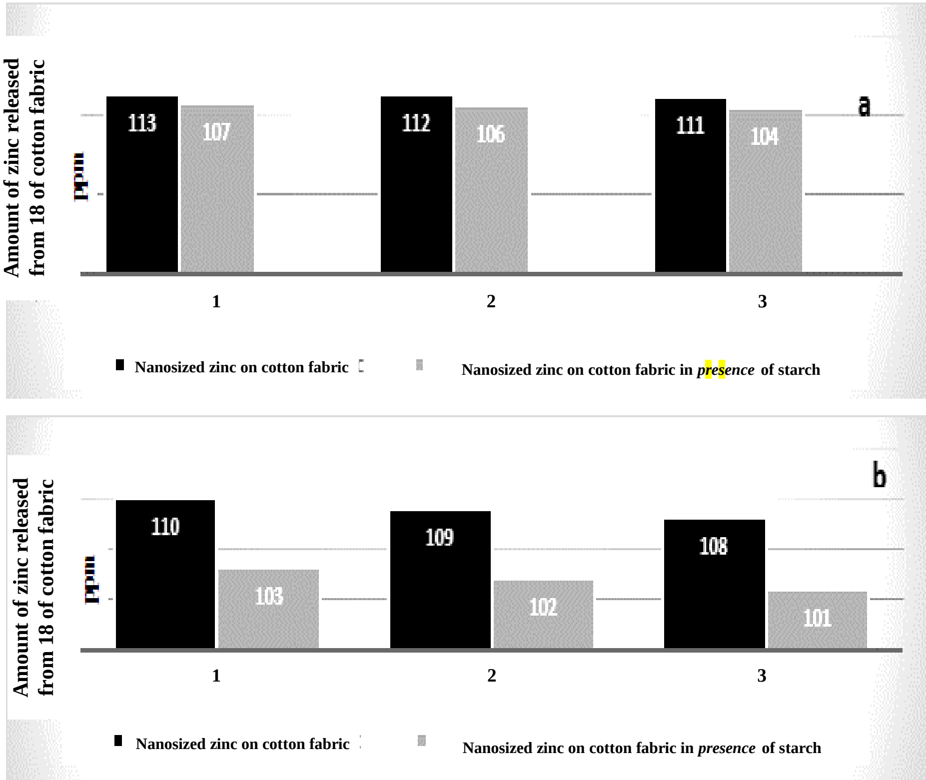

Fig. 6: The released zinc from the treatment of cotton fabrics during washing (a) at 40 °C and (b) at room temperature.

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