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Multifunctional Cellulosic Fabrics via Nanocoating

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Abstract

A simple method for coating various cellulosic containing fabrics has been developed. Coating layer made of metal nanoparticles and binder were uniformly distributed using direct coating process (flat screen) and time-saving microwave fixation method. A textile coating of viscous paste was prepared using binding agent based on acrylate copolymer, thickening agent based on ammonium polyacrylate and individual inclusion of Ag, ZnO, ZrO_2 and TiO_2, nanoparticles to provide durable antibacterial properties as well as UV-protection. The multifunctional effect of the coatings, such as antibacterial efficacy, UV-blocking ability, metal and nitrogen content were investigated. Surface alteration of the coated fabrics were identified as well by SEM, EDX analysis. The experimental results suggested that these coated fabrics showed a remarkable improvement in their antimicrobial activity against both Gram-positive and Gram-negative bacteria, irrespective of the used nanoparticle. The finished cellulosic fabrics demonstrated superior UV protection compared to untreated fabrics.

Keywords: Nanoparticles; coating; functional properties; cellulosic fabrics.

1. Introduction

Multifunctional cellulosic substances have been receiving a lot of attention due to their natural characteristics, interest for sustainability and improved for the comfort and safety of consumers.[1]

To impart or enhance the multifunctional characteristics and properties of a textile material different types of materials and several techniques can be used including plasma processing, enzymatic treatments, electrospinning, nanotechnology and polymer coating. Coated fabrics play an essential role in technical textiles with added value properties and considered as one of the most important technological methods in the textile manufacturing sector.[2-4]

Presently, the textile industry uses nanotechnology extensively, and there are a large number of nanomaterials which used in textiles presented in the markets. The use of nanocoating technologies are widely considered to be a new insight into textile finishing approaches. [5-8]. The introduction of nanocoating technologies in the textile area could provide specific benefits in manufacturing advanced textiles in the functional and apparel sectors. Several investigations are being performed to produce multifunctional textile fabrics coated with various nanoparticles. Researchers have used nanoparticles such as silver (Ag) [9-11]titanium dioxide (TiO_2)[12]and zinc oxide (ZnO)[13-16], to impart and enhance a variety of functionalitiesto fulfill various needs of current markets. Nanocoatings based modified fabrics produce multiple functionalities including antibacterial activity [17, 18],ultraviolet protection [19-22], self-cleaning[23-25] ,flame retardant and water repellency[26, 27]etc.

Cellulosic fabric incorporation with nanoparticles is growing rapidly, since it exhibits potential for unique multifunctional properties, including photocatalytic self-cleaning, UV protection, antimicrobial activity, flame retardancy, hydrophobicity, electrical and thermal conductivity.

In this study, thin coating layer made of Ag, ZnO, ZrO_2 and TiO_2, metal nanoparticles were uniformly deposited onto cotton, viscose and linen/ cotton cellulosic fabrics to provide durable antibacterial characteristics as well as UV-protection. The possibility to enhance the quality of produced cellulosic substrates and obtain coated layer of metal nanoparticles strongly attached to the fabric were investigated.

2. Experimental

2.1. Materials

Plain wave 100% mill-scoured and bleached cotton (120 g/m^2), viscose (110 g/m^2) and linen/cotton blend (65/35,180 g/m^2) was used throughout the study.

printofix® thickener 160 EG liquid (synthetic thickening agent based on ammonium polyacrylate, clariant);printofix® Binder MTB-01 liquid (acrylate-based copolymer, anionicclariant), Durex® Silicone -1020(based on modified polysiloxanemicroemulsion, Texchem, Egypt), GBresin® CPN (based on hy-

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droxymethylated 4,5-dihydroxyethylene urea, GB Chem, BASF, Egypt).

Zirconium oxide nanoparticles, (10 wt% in water, particle size <100nm), Zinc oxide nanoparticles, (50 wt% in water,particle size <35 nm), and Silver dispersion nanoparticles, (0.02 mg/l particle size 40 nm)were purchased from Sigma, Aldrich).

The additional chemicals utilized in this investigation included Ti-tetraisopropoxide (analytical grade, Sigma), while ammonium persulphate [$(NH_4)_2S_2O_8$], nitric acid and 2-propanol were graded as laboratory reagents.

2.2 Methods

2.2.1. TiO_2 sol–gel preparation

TiO_2 -nanoparticles wereprepared, as already stated using titanium tetraisopropoxide precursor with 2-propanol and nitric acid, particle size <10 nm [23,24]

2.2.2. Coating of cellulose substrates with nanoparticles

The cellulosic samples were coated directly by flat screen with a thin layerof coating formulation containing of thickening agent (20 g/kg),printofix® Binder MTB-01 as a binding agent100 g/kg),GB Chem ,BASF as crosslinking agent (20 g/kg), Durex® Silicone -1020as a softener(10 g/kg),ammonium persulphate as a catalyst (2 g/kg), acetic acid (1 g/kg), along with one of the nominatednanoparticles, namely Ag, ZnO, ZrO_2 and TiO_2, (20 g/kg), and up to 1000 g/kg water). Coated fabrics were dried and fixed in a microwave oven with an output power of 386 W/5 minutes. In order to remove unfixed active components, coated cellulosic substrates were rinsed with cold water, after that, they were completely washed using 2g/l non-ionic detergent at 90–95°C for 15 minutes.

2.3 Tests and analysis

2.1.1. Nitrogen content

The nitrogen content (N %) was measured using Kjeldhal process.

2.1.2. Metal content

The metal content of the produced coated cellulosic substrates was assessed by a flame atomic absorption spectrophotometer (GBC- Avanta, Australia).

2.1.3. Ultraviolet (UV) Protection

UV-protection factor (UPF) was evaluated based on the Australian/New Zealand Standard Method 135-2000. In accordance with, the Australian classification method, textiles can be classified as good, very good, and excellent protection if their UPF values fall between15 - 24, 25 - 39, and above 40, respectively [30].

2.1.4. Antibacterial activity test

Assessment of antibacterial activity against G−ve bacteria (E. coli) and G+ve(S. aureus)was evaluated according to AATCC Test Method 100-1999.

E. coli, a gram-negative bacterium and *S. aureus*, a pathogenic gram-positive bacterium were used to test the antimicrobial effectiveness of NPs treated cellulosic fabrics per jar. The dilution medium was nutrient broth and the neutralizer was 1N sodium hydroxide. To evaluate the reduction rate of coated fabric, reduction in the number of colonies between the coated and uncoated fabrics after incubation were determined. The results are expressed as percent reduction of bacteria(R) by eq. (1).

$$R\ \% = \frac{A - B}{A} X\ 100 \quad (1)$$

Where A and B are the numbers of bacteria recovered from the uncoated and NPs coated-fabric, respectively, after inoculation and incubation in a jar over the desired contact period.

2.1.5. Scanning electron microscopy(ESEM) and energy dispersive X-ray (EDX)

Scanning electron microscope (SEM) images of the coated and uncoated fabric samples were obtained using electron probe micro-analyzer (JEOL, JXL 840A) with energy disperse X-ray (EDX) spectroscopy for the elemental analysis.

All determinations were done in triplicate and the average was taken as final results.

3. Results and Discussion

The aim of this study is to investigate the effects of adding different nanomaterials to the coating formulation on the performance of cellulosic substrates. The achieved results are discussed below.

3.1 Metal content and N% of NPs coated fabrics

For a given set of coating conditions, the data in figure 1 summarize that the presence of 20g/ Kg of any of the nominated nano-materials in the coating formulation along with binder and other ingredients results in noticeable increase in the metal %. The increase of the metal content most properly is attributed to the positive role of the binding agent to pick up and retain the metal nanoparticles in the forming coating film. In addition, the differences in the metal NPs percentages is a direct sequence of their differences in the level of loading and attachment onto the binder/substrate matrix during the microwave fixing, as well as the chemical structure, size distribution, and concentration of the nanoparticles.

Also, the data in figure 1 demonstrate that adding crosslinker and softener to the coating layer when each of the selected nanomaterials is individually added to the coating mixture of the nominated substrates results in a notable increase in the N% as a direct sequence of the ether-crosslinking of cellulose chains and the formation of an elastic network due to the amino func-

tional groups in the silicone softener. The little increase in the N % highlight the beneficial of the added nanoparticles in accelerating and improving the degree of crosslinker and silicone softener attachment and fixation onto and within the fabric structure, and this is also controlled by the substrate type, fabric structure, which demonstrate the differences among the cellulosic substrates in fabric thickness and weight, surface area, cellulose content, non-cellulosic component, for example lignin in linen, amorphous/crystalline ratio.[31-34]

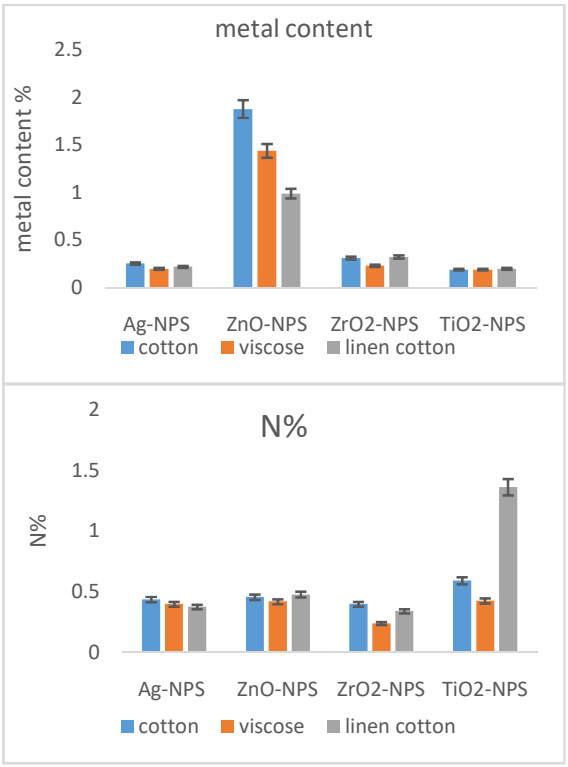

Figure 1: Coating components: nano-material (20g/kg); Printofix® Thickener 160 EG (20g/kg); Printofix® Binder MTB-01(100g/kg); resin (20g/kg); softener (10g/kg) ;$(NH_4)_2S_2O_8$ (2g/kg). Fixation:at 386W/5min.

2.4 UV-Protection property of coated fabrics

The effect of coating layer on improvement the UV protection factor (UPF) was evaluated and results are presented in figure 2. The UPF ratings for the cotton, viscose and cotton/linen blank fabrics were (10,5,15) respectively. However, the UPF values were increased when same blanks were treated with the coating layer containing NPs and binder. This achieved increase in UPF reveal that: (i) the given UV-protection characteristics, represented as UPF values, is controlled by the substrate type i.e. coated > uncoated, besides the loaded nanoparticles type and goes in the descending order: ZnO-Ps > TiO_2 -NPs > Ag- NPs > ZrO_2 -NPs > none > uncoated, (ii) the achieved increase in UPF ratings have been made by involving a binder in the coating layer, as a fixing agent for nanoparticles deposition. [14]

Moreover, the notable enhancement in the UPF values of the cellulosic substrates coated with nanoparticles may be examined in relation to their capacity to block and provide protection against detrimental UV radiation, particularly UV-B (l: 280-315 nm). The enhancement in UV-protection efficiency is affected by the cellulosic substrate type, i.e. difference in fabric construction, pore structure, surface area, the morphology of the fabric, degree of polymerization, amorphous to crystalline regions, cellulose content and the degree of coating, as well as the capacity of the nanoparticles to be loaded / fixed onto the surface of fabric.[20, 35, 36]

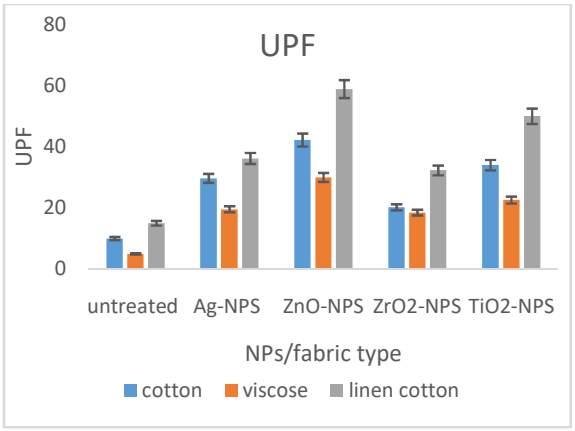

Figure 2: Coating components: nano-material (20g/kg); Printofix® Thickener 160 EG (20g/kg); Printofix® Binder MTB-01(100g/kg); resin (20g/kg); softener (10g/kg) ;$(NH_4)_2S_2O_8$ (2g/kg). Fixation:at 386W/5min.

2.5 Antimicrobial activity of NPs-loaded fabrics

Textile products, particularly those made from natural materials can provide microorganisms an ideal medium to grow, due to their large surface area as well as ability for moisture retention.

Thus, it has been discovered that chemical modification is a highly effective field of study to impart strong antibacterial activity to natural materials [37]. Therefore, the goal of the current study is to increase cellulosic fabrics antibacterial activity by coating formulation containing specific bioactive nanomaterials.

The antibacterial properties of uncoated and coated fabrics were tested qualitatively against *(Staphy- lococcus aureus)* as an example of G+ve bacteria and *(Escherichia coli)* as an example of G−ve bacteria, and results were summarized in figure 3. The data in figure 3 shown the antibacterial properties of coated cotton, viscose and linen/cotton blended fabric samples in the presence of the nominated nanoparticles (20g/kg coating formulation) and reveals that: (i) all the coated samples performed splendid antibacterial activities owing to the presence of the nominated nano-materials in

the coating formulation. (ii) regardless of which kind of nanomaterials utilized, inclusion of a binder has a positive role on antibacterial performance characteristics. (iii) also, from the data, it could be concluded that, the degree of enhancement in antibacterial activity of the developed coatings follows a declining order:ZnO- NPs >TiO_2- NPs > Ag-NPs >ZrO_2- NPs >none, and the difference in the antibacterial efficiency is influenced by the size, shape, morphologyand mode of attack on microorganisms of the nominated nanoparticlesfixation. In addition, the previouslymentioned antimicrobial activity of coated cellulosic fabrics are determined by the type and nature of substrate, that is, chemical and physical structure, cellulose content, amorphous-to-crystalline ratio and fabric construction.[38-40]Also, from the data, it could be concluded that, differences in the antibacterial activity of cellulosic fabrics coated with a layer containing of nanoparticles and a binder are clarifying the important role of binder in loading and fixation of nominated nanoparticles into fabric matrix during the microwave. [41]

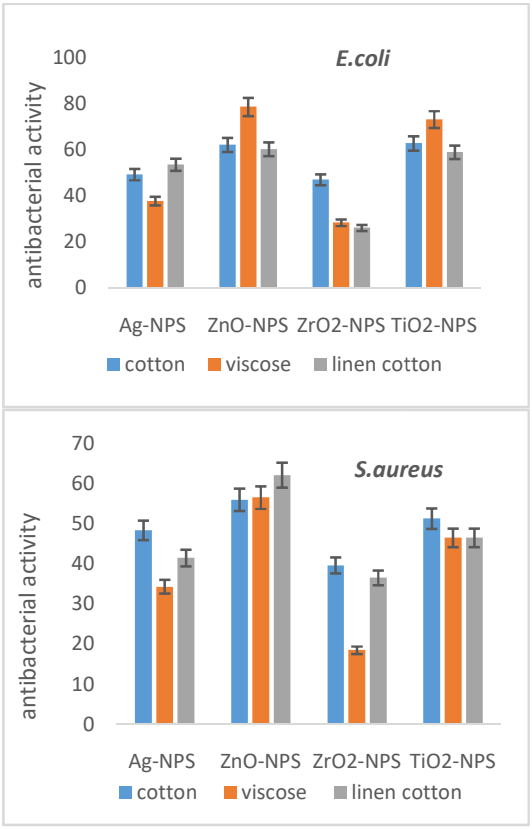

Figure 3: Coating components: nano-material (20g/kg); Printofix® Thickener 160 EG (20g/kg); Printofix® Binder MTB-01(100 g/kg); resin (20g/kg); softener (10g/kg) ;$(NH_4)_2S_2O_8$ (2g/kg). Fixation: at 386W/5min.

2.6 SEM with EDX of NPs coated fabrics

Coated cotton fabrics are chosen in this work for scanning electron microscope (SEM). The morphological changes of cotton fabrics result from deposition of various NPs were determined by scanning electron microscope (SEM). The SEM images of cotton fabrics coated with coating formulation containing 20g/kg TiO_2, Ag, ZrO_2 and ZnO nanoparticles and 100g/kg binding agent, are presented in figure 4.

Figure 4(b), (c), (d), and (e) microscopic images illustrate that the cotton substrate coated with 20g/kg TiO_2, Ag, ZrO_2,and ZnONps, respectively, is covered with well dispersed quite uniform NPs / binding agent film. Also, there are noticeable aggregates composed of smaller particles.

Also figure 4 (g, h, i and j) illustrated the EDX spectrum of a coated sample demonstrating the presence of titanium, silver, zirconium and zinc element on the surface of the fabric treated with TiO_2, Ag, $ZrO_2$and ZnO NPs (20g/kg coating formulation) respectively, it confirms the presence of the nominated nanoparticles on the treated fabrics surface as peak of titanium, silver, zirconium and zinc was observed. The existence of silicon element in addition to carbon and oxygen peaks in the EDX spectrum is related to the softener and crosslinking agent added to the coating film.

2.7 Tentative mechanism

The enhancement in the coating as well as the remarkable improvement in the antibacterial functionality and UV protection ability of the obtained coated cellulosic fabrics via inclusion of any of the nominated nanoparticles into coating layer could be explained as given in Scheme 1

I. Reaction between the binder and the cellulose component:

$$\underset{\text{Cotton Viscose linen/cotton}}{\text{Cell.OH}} + \text{ROH}_2\text{C-Binder-CH}_2\text{COOR} \xrightarrow[\Delta]{H^+} \underset{\text{self-crosslinking polyacrylate binder}}{\text{Cell.O.H}_2\text{C-Binder-CH}_2\text{COOR}} \quad (1)$$

II. Fixation of metal nanoparticles:

$$\text{Cell.OH} + \text{ROH}_2\text{C-Binder-CH}_2\text{COOR} + \text{NPs} \xrightarrow{H^+} \text{NPs-containing binder film-loaded onto fabric surface} \quad (2)$$

III. Enhancing the extent of crosslinking:

$$\text{Cell.OH} + \text{ROH}_2\text{CN-DMDHEU-NCH}_2\text{OR} + \text{ROH}_2\text{C-Binder-COOR} + \text{NPs} \xrightarrow[\Delta]{H^+} \text{crosslinked three-dimensional network-encapsulated nanoparticles} \quad (3)$$

IV. Enhancing the flexibility of the binder film to give crosslinked/softer film:

$$\text{Cell.OH} + \text{ROH}_2\text{CN DMDHEU NCH}_2\text{OR} + \text{ROH}_2\text{C Binder COOR} + \text{H}_2\text{N silicone softener} + \text{NPs} \xrightarrow[\Delta]{H^+} \text{crosslinked softer film-containing nano particles/loaded onto the fabric surface} \quad (4)$$

Scheme 1. Tentative reactions mechanism

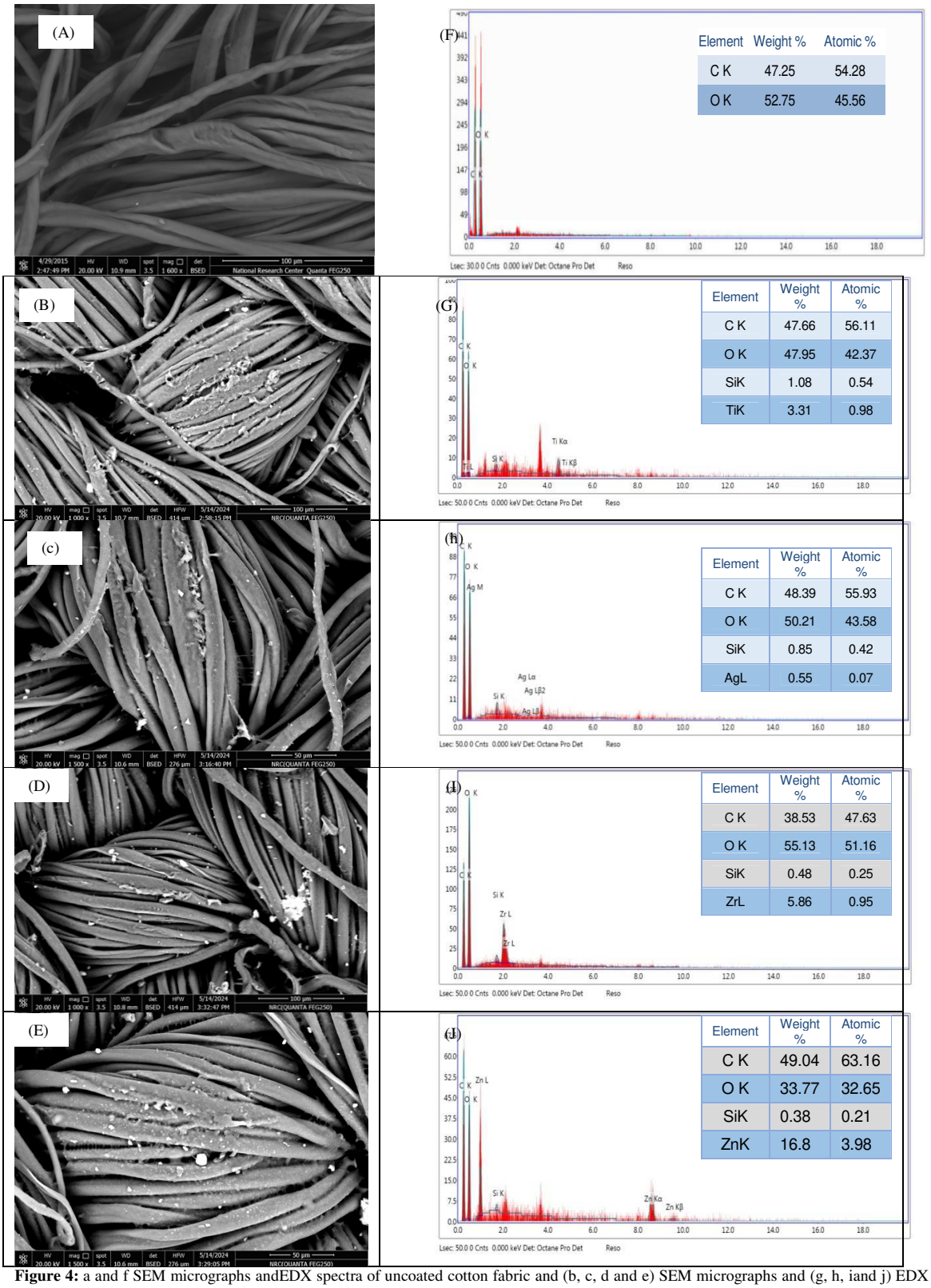

Figure 4: a and f SEM micrographs andEDX spectra of uncoated cotton fabric and (b, c, d and e) SEM micrographs and (g, h, iand j) EDX spectra of coated cotton fabric respectively.

4. Conclusion

Smart coating layer for various cellulosic substrates have been produces utilizing different metal nanoparticles in the coating layer, the mass ratio of the binder and nanoparticles has been adjusted and optimized. Compared to untreated textiles, cellulosic substrates treated with a developed coating layer gained multifunctional properties including durable antibacterial properties as well as UV-protection.

Fundamental variations in the type and concentration of nanoparticles and the inclusion of binder in the coating formulation governed the amount of nano-particles loaded / fixed on the fabrics' surfaces, during the microwave fixation step.

5. Fund

The authors have no fund.

6. Conflict of interest

The authors have no conflict of interest.

7. Acknowledgment

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