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Psidium Guajava Leave Extract as Reducing Agent for Synthesis of Zinc Oxide Nanoparticles and its Application to Impart Multifunctional Properties for Cellulosic Fabrics

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Abstract: Nowadays, cotton fabric treatment with nanoparticles has been increasingly high because of its unique properties. In this study, using the bioactive compounds in *Psidium guava* leave extracts, zinc precursors were reduced to obtain ZnO nanoparticles using two extracts, namely, water extract and ethanol extract. A comparative evaluation was carried out to analyze and develop a wider understanding of the effect of processing parameters on the ZnO nanoparticles. The ZnO nanoparticles have been characterized using a particle size analyzer, Transmission Electronic Microscopy (TEM), X-ray diffraction, and Thermal Gravimetrical Analysis (TGA) to determine their structure. The cotton fabric was treated with ZnO nanoparticles in the presence and absence of different natural biopolymers, namely; alginate, chitosan, and carboxymethyl cellulose (CMC). The treated cotton samples were characterized by atomic absorption and scanning electron microscopy (SEM), UPF value, antimicrobial activity, and self-cleaning properties. It was found that the cotton fabric treated with ZnO-chitosan nanocomposites gives the higher UPF values and antimicrobial activity against *Staphylococcus aureus*, *Escherichia coli*, and *Candida albicans* of all other treated fabrics.

Keywords: cotton fabric; *Psidium guajava* leave extract; nanotechnology; zinc oxide nanoparticles; antibacterial; self-cleaning; ultraviolet protection.

1. Introduction

In modern materials science, nanotechnology is one of the most active research domains. Nanoparticles are of distinct properties compared with bulk types of the same substance based on their particular characteristics such as size, distribution, and morphology. A nanoparticle (nano-powder, nanocluster, or nanocrystal) is a microscopic particle of less than 100 nm in at least one dimension [1].

Much work has been devoted in recent years to adding nanoparticles of metal oxide to textile materials for the production of multifunctional fabrics with new and improved properties or removing the heavy metal from wastewater [2-14]. The cotton fabric was commonly used in garments and other products of everyday use because of its softness and comfort, but it also has some low performance, such as proneness to bacteria, vulnerability

to UV, hydrophilic, etc. with the enhancement of the health concept, the interests of multifunctional cotton were followed by more and more scientists [15-26].

ZnO nanoparticles are among the metal oxides used to treat materials because of their uniqueness of characteristics such as chemical stability, non-toxicity, antibacterial and UV protection, high photocatalytic activity, and high clarity of the visible wavelength spectrum [27-29].

The ZnO nanoparticles can be synthesized through various routes, such as chemical reduction, microwave, and green route procedures. [30] But there are several problems associated with chemical methods. Therefore, much attention was given to the green route method of nanoparticle synthesis as an alternative method for synthesizing nanoparticles due to its cost-effectiveness and reducing the dangerous chemical compound's use [31]. The biosynthesis of ZnO NPs through the green route was a promising domain in nanotechnology due to their uses of active Phyto-compounds, which employ as a reducing and capping agent [32].

Psidium guajava leaves can be used as a biocatalyst / chelating agent to synthesize ZnO NPs from zinc acetate precursors because they have high polyphenols, carotenoids, terpenoids, flavonoids, and triterpenes in the crude water and ethanol extracts of the leaves [33]. These biomolecules have functional groups and can coordinate with Zn(II) ions, thereby facilitating and stabilizing the formation of ZnO NPs [34].

Researchers recently tried to functionalize cotton fabrics with bio–Nanocomposites, i.e., natural polymer matrix components combined with nano-scale organic/inorganic metal particles because of their simple accessibility and low toxicity. [35-37] For example, the metal oxide Nanoparticles-chitosan Nanocomposite, as mentioned in earlier research, is used in antibacterial cotton fabric working and also in hydrophobic surfaces to minimize sweat absorption [28].

The goal of this study was to investigate the Characterization of the ZnO NPs that synthesized using psidium guava leaves extracts Through changing the processing parameters and provide an excellent functional property to cotton fabric by treatment the fabric using ZnO NPs and its composites

2. Materials and Methods

2.1. Materials.

Bleached scoured cotton fabric (220 g/m^2) supplied by Ghazel El-Mahala for Textile Industry Co., Egypt, was used during this research. *Psidium guajava* leaves (*Psidium guajava* L.) were purchased from an agricultural farm in Egypt in Egypt. Ethanol (95%), sodium carbonate (Na_2CO_3), citric acid, sodium hypophosphite (SHP), and sodium carbonate were provided from Fluka. Zinc acetate was purchased from El Nasr pharmaceutical chemicals Co. Alginate purchased from Fluka BioChemica GmbH Co., carboxymethyl cellulose purchased from Carl Roth GmbH Co. chitosan low molecular weight (100000-300000) was purchased from ACROS Co. All the chemicals and reagents were used as received without purification.

2.2. Methods.

2.2.1. Preparation of *Psidium guajava* leaves (*Psidium guajava* L.) extract.

The freshly *Psidium guajava* leaves were collected, washed, and cleaned with water to remove the dust. The dried leaves were cut into small pieces. The extraction of *Psidium guajava* L was done using two solvents, namely water and ethanol. For aqueous extraction, 100 g from the leave pieces was placed in 1 L boiled water for 60 min. The extract was subjected to vacuum filtration with the Whatman Filter Paper no. 1. Extraction was then stored at a temperature of 4°C and used without any further purification. For ethanolic extraction, 200g from the leave pieces were placed on the Soxhlet device in 1 L ethanol. The temperature was adjusted to 70°C and kept for four hours. The extract was subjected to vacuum filtration with the Whatman Filter Paper no. 1. The extraction was then stored at a temperature of 4°C and used without any further purification.

2.2.2. Synthesis of zinc oxide nanoparticles (ZnO NPs) using *Psidium guajava* L. extract.

Zinc oxide nanoparticles (ZnO NPs) have been synthesized using the modified method from the reported one by Rajendra *et al.* [38] from zinc acetate as precursors and reduced in the presence of *Psidium guajava* L extract in water or ethanol as a capping agent by use of sodium carbonate to adjust the pH medium to (8, 10, and 12).

In this modified procedure, 70 ml of the *Psidium guajava* L extract in water or ethanol (as a capping agent) was placed in a beaker. The pH medium was adjusted to (8, 10, or 12) using sodium carbonate (10%). After that, the solution temperature was raised to 70°C, that 1 N zinc acetate in 30 ml distilled water was added to the solution of *Psidium guajava* L extract drop by drop under stirring for 30 minutes. Then the solution was kept at this temperature for 90 min under stirring. The produced powder was filtered and dried at 90°C for 24 h. The produced powder was combinations of $Zn(OH)_2$ and $Zn(CO_3)_2$ or combinations between them as $Zn_5(OH)_6(CO_3)_2$ [6]. So, the calcination step is important to forming ZnO NPs at it was done at 400°C for 4 hr. The calcinated ZnO NPs were used for further use.

2.2.3. Treatment of cotton fabric with *Psidium guajava* L. extract.

Cotton fabrics were pre-treated with citric acid (10 g/l) and sodium hypophosphite (5 g/l) using the pad-dry method through immersing in the treated bath for 5 min and squeezed with 100 % wet pickup, then dried at 100°C for 3 min.

ZnO NPs emulsion for fabric treatments was prepared in distilled water to obtain 10 % in the presence of 10 g/l different polymeric materials (alginate, carboxymethyl cellulose, and chitosan) according to the following procedure: 1 g polymeric material was dissolved in 100 ml distilled water under stirring. At 80°C, 1 g calcinated ZnO NPs was added under vigorous stirring, the stirring was continued for 30 min for good distribution of the nanoparticle inside the polymer network. After that, the solution was homogenized using a homogenizer for 3 min at 20000 rpm to ensure a completely homogenous distribution.

Pre-treated cotton fabric was immersed in the final produced emulsion for 10 min, then squeezed with 100 % wet pickup and dried at 100°C for 5 min and cured at 140°C for 3 min. The treated fabric was used for further investigation.

2.3. Analysis and Measurements.

2.3.1. Transmission Electronic Microscopy (TEM).

Transmission Electron Microscopy (TEM) images for synthesized ZnO NPs were investigated with a JEOL JEM-1200 EX transmission electron microscope operating at 120 kV. For TEM characterization, the ultrasound bath was used to diffuse a small volume of ZnO NPs into distilled water. One drop of the dispersion was placed on a copper grid.

2.3.2. Scanning Electron Microscopy (SEM).

Scan electron microscopy was used to analyze the surface and internal structure of the treated cotton fabrics with ZnO NPs using SEM; JEOL, JSM-6360LA, Japan. For the indication of contents or distribution of metals on the fabric surface, SEM is connected with Energy Dispersive Spectroscopy (EDX).

2.3.3. Particle Size Analyzer.

The particle size measurements of the synthesized ZnO NPs were investigated by the laser particle size analyzer MasterSizer/2000; MALVERN Instruments, UK, according to standard test methods ASTM E11:61 [39], ISO 3310-1:2016 [40], and ISO 565: 1990 [41].

2.3.4. Atomic absorption.

A multi-element flame atomic absorption spectrometer, SpectrAA Varian Type220, was employed to determine the treated fabric Zn content. A hollow cathode multi-element lamp was used for zinc determination at 213.9 nm wavelength with bandwidths of 1 nm. The flame composition was the air with 13.5 L/min.

2.3.5. X-ray diffractometry.

The crystallinity of the synthesized ZnO NPs after calcination was studied using X-ray diffractometry. The macro-structural characteristics of materials like the crystallinity, the crystal size, and the crystals' orientation are important for X-ray diffractometry.

2.3.6. Thermogravimetrically Analysis (TGA).

TGA thermographs of treated textiles were analyzed for a temperature range from room temperature to 600°C with a heating rate of 10°C/min at in air atmosphere using NETZSCH STA 2500 Regulus (German). The weight of the fabric sample was 4-9 mg

2.3.7. Contact angle and wettability time.

The contact angle was reported at atmospheric conditions (25°C) on Dataphysics GmbH (OCA-15EC) according to ASTM D7334-08 standard form [42]. Wettability time has been investigated to drop water on the surface of treated fabric until completely penetrated through the fabric surface [43].

2.3.8. Self-cleaning action.

The self-cleaning action of the treated cotton fabrics was demonstrated following the suitable described method by Ibrahim *et al.* [44], which can be described as follow: The untreated and treated fabrics with ZnO NPs with/without biopolymer were immersed in methylene blue solution (0.02 %) for 30 min and dried overnight in air. After that, the samples were exposed to UV light for 24, 48, and 72 h. Colour strength values (K/S) of the dyed fabrics were measured using a Hunter lab's Ultra Scan PRO spectrophotometer (USA).

2.3.9. The UV-Protection Factor (UPF).

The UV-protection factor (UPF) for untreated and treated samples of cotton fabric was determined by the Australian/New Zealand standard (AS/NZS 4366-1996) [45]. The ultraviolet transmission through the fabric was determined by a Cary Varian 300 UV-Vis spectrophotometer [5,8,9,19,46,47].

2.3.10. Color strength (K/S).

The color strength (K/S) and whiteness degrees of the untreated, treated, and dyed fabrics were evaluated using Hunter Lab Ultra Scan PRO (USA, 2007). The relative color strength (*K/S*) of the cotton fabrics was measured and assessed by applying the Kubelka–Munk equation as following [48-57]:

$$K/S = \frac{(1-R)^2}{2R} - \frac{(1-R_o)^2}{2R_o}$$

where *K* is the absorption coefficient, *S* is the scattering coefficient, R_o is the reflectance of the uncolored (white) sample, and *R* is the reflectance of the colored sample.

2.3.11. Mechanical and physical properties of the treated fabric.

Tensile strength and elongation at break are conducted on a tensile strength apparatus type FMCW 500 (Veb Thuringer Industrie Werk Rauenstein 11/2612 Germany) at 25°C and 65 % relative humidity according to the ASTM Test Method D1682-59T. [58] The dry crease recovery angle (CRA) was measured according to AATCC Test Method 66 – 2014. [59] Fabric roughness was measured using Surface Roughness measuring instrument SE 1700 a using ASTM Test Method D 7127 – 13 [60]. Stiffness was performed using the cantilever apparatus according to ASTM test method D 1388-14e1 [61].

2.3.12. Antibacterial activity.

The antibacterial activity was quantitatively examined using the AATCC 100-2012 (bacterial reduction method) [24,62,63] and quantitively tested using disc diffusion to analyze the antimicrobial activity of treated fabrics according to the AATCC Test Method (147-2016) [8,10,64,65]. Both examined methods were done against *Staphylococcus aureus* (ATCC 29213) as a gram-positive bacteria and *Escherichia coli* (ATCC 25922) and *Candida albicans* (ATCC 10231) as a fungus.

2.3.13. Durability.

The durability of treatment and imparted properties to the fabric were evaluated by washing the treated cotton fabric with 2 g/l Hostapal (non-ionic detergent) for 10 min at 40°C

and drying at 100°C for 3 min (for each washing cycle). After that, the investigated properties were re-examined again after selected washing cycles.

3. Results and Discussion

In the presence of *Psidium guajava* L extract as a capping agent, the preparation of nanoparticles of zinc oxide was studied through hydrolyzed with sodium carbonate at various pHs. Also, the produced ZnO NPs were characterized using a TGA, XRD, and TEM.

3.1. Characterization of synthesized ZnO NPs.

Guava leaves consist of several biological molecules that offer desirable health benefits, such as phenols, flavonoids, and essential oils. Guava leaf compounds are bio-active and solvent-soluble. The difference in solvent polarity is influencing chemical components' solubility and extraction production. Therefore, choosing the right solvent is one of the main steps to enhance the extraction process.

Guava leaves were eventually extracted in two solvents, namely water and ethanol. Both extracts in water and ethanol have been used to synthesize ZnO NPs at different pH (8, 10, and 12).

The particle size of synthesized ZnO nanoparticles using *Psidium guajava* L. extract was observed and illustrated in Figure 1. The particle size of ZnO NPs synthesized with *Psidium guajava* L in various pH media (8, 10, and 12) is shown in Figure 1. Extract from various pH media in water and alcohol (8, 10, and 12). It is apparent that, by growing the pH medium, the particles in synthesized ZnO NPs decrease. The deposited effect of *Psidium* components on the ZnO NPs surface could be attributed to this decreasing.

It is further noted that the particle size of the synthesized ZnO NPs with *Psidium guajava* L. extract in water is higher than those in alcohol in different pH (8, 10, and 10) medium. It is clear that the particle size of synthesized ZnO NPs using both *Psidium guajava* L. extract provides a three size at both pH 8 and 10; these sizes are around (5-7, 12-18, and 110-170 nm) and (< 1, 5-7, and 80-90 nm) for *guajava* L. extract in water and ethanol respectively. The main particle size was in the second peak (12-18 and 5-7) for *guajava* L. extract in water and ethanol, respectively. At pH 12, the particle size of synthesized ZnO NPs using both *Psidium guajava* L. extract provides two sizes. These sizes are around (3 and 12 nm) and (8 and 9 nm) for *guajava* L. extract in ethanol and water, respectively, with the main particle size in the second peak for both extracts.

There can be no doubt that at pH 8 ZnO NPs are very close to synthesis in both extracts, while the pH medium increases to 10 causes ZnO NPs to start to develop. Therefore, the synthesized ZnO NPs have been increased as the pH medium in both extracts increases. Furthermore, in the ethanolic extract, the formation of ZnO NPs is smaller than in water extract. Moreover, for shaping ZnO NPs in both extracts, pH 12 proved to be an excellent medium.

The coordinated ZnO NPs have been studied to decompose reduced zinc oxide with *Psidium guajava* L by thermo-gravimetrical analyses (TGA). Figure 2 indicates water loss accompanied by capping agents' decomposition at approximately 290, 310°C in the first stage of the thermal decomposition profile, respectively, in water and ethanol extract. However, the decomposition step ends around 310-320°C except for ZnO NPs from ethanol extract at

pH 12; thus, the decomposition step end at 385°C. This step also enhances the weight loss percent from 20 to 58 % and from 35 to 55 % for water and ethanol extract, respectively.

Last phase stopping at 600°C offers an improvement of 15% to 30% and 25 to 40% for water and ethanol extract, respectively, for final weight loss percent. In brief, a temperature of 0–400°C is enough to completely break down zinc compounds with a good weight loss ratio.

ZnO NPs essentially consists of a 3D space that is periodically structured. X-rays must be dispersed by atoms of the sample. Basically, the dispersal of X-rays from arranged atoms creates diffractions in several directions, which are common for these atoms. D-spacing values from the achieved peaks enable mineral identification since every element has a set of d-spacings that do not match. This is simply accomplished by contrasting d-spacings with regular reference patterns.

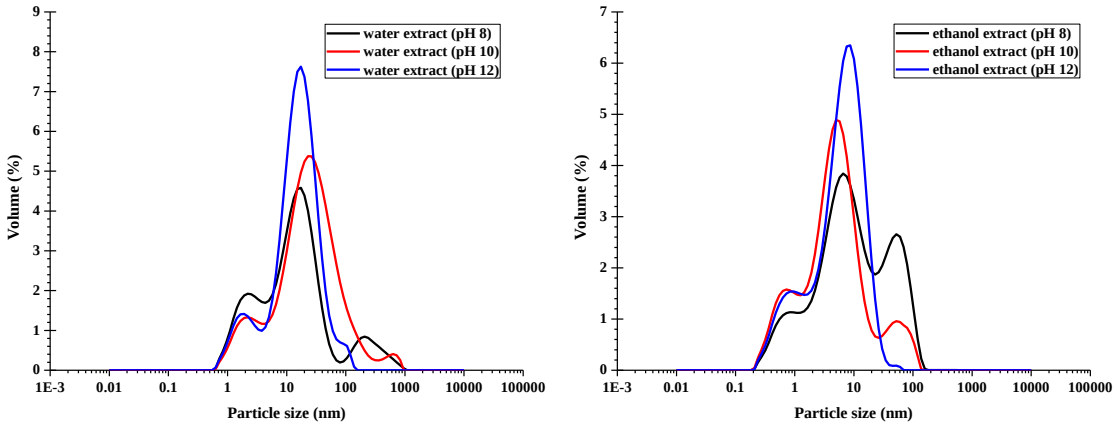


Figure 1. Particle size for synthesized ZnO NPs using both *Psidium guajava* L. extracts in water and ethanol at different pH.

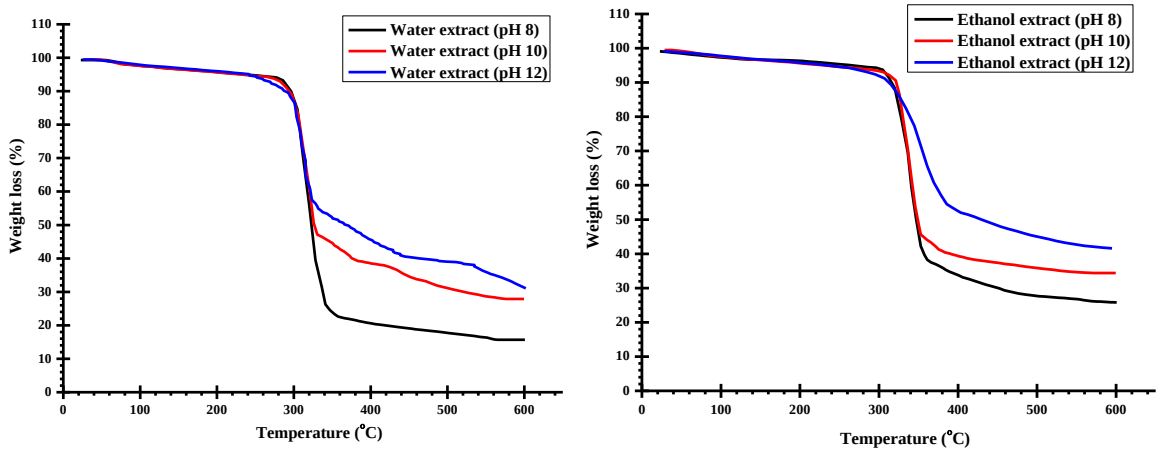


Figure 2. TGA for synthesized ZnO NPs using both *Psidium guajava* L. extracts in water and ethanol at different pH.

The X-ray diagrams in Figure 3 display the same high intensity and width sequence for all the examined synthesized ZnO NPs. It reveals all prepared ZnO NPs with the same patterns under all pH medium, using both *Psidium guajava* L extract in water and alcohol. d-Spacing values at 2 Theta positions (2θ; 32.09-32.21, 34.73-34.81, 36.53-36.69, 47.87-48.07, and 56.99-57.03) for synthesized ZnO NPs using *Psidium guajava* L extract in water and alcohol as a capping agent coincided with the results published by the many researchers in literature in terms of values 2.7, 2.5, 2.5, 1.8, and 1.6, respectively [3,5,6,44].

The TEM images of *Psidium guajava* L extract in water and alcohol and synthesized ZnO NPs on various pH media (8,10, and 12) are shown in Figure 4. TEM images are shown

as a good distribution spherical shape. For *Psidium guajava* L, the TEM analysis indicates that the particles have small scatter sizes of 80 and 60 nm, respectively, in the water and ethanol extract at pH 8.

By comparison, in *Psidium guajava* L extracts in both water and ethanol, ZnO NPs were produced in nanoform. TEM images show that thin, spherical nanoparticles approximately 5 nm are agglomerates of small clusters during synthesis steps by increasing pH (until pH 12), as seen in Figure 4.

3.2. Characterization of functionalized cotton fabric.

3.2.1. Zinc content (%), and the ultraviolet protection factor (UPF)

The total content of zinc metal percent per 1 g treated cotton fabrics with synthesized ZnO NPs with/without biopolymers has been quantitatively determined by the flam atomic absorption method.

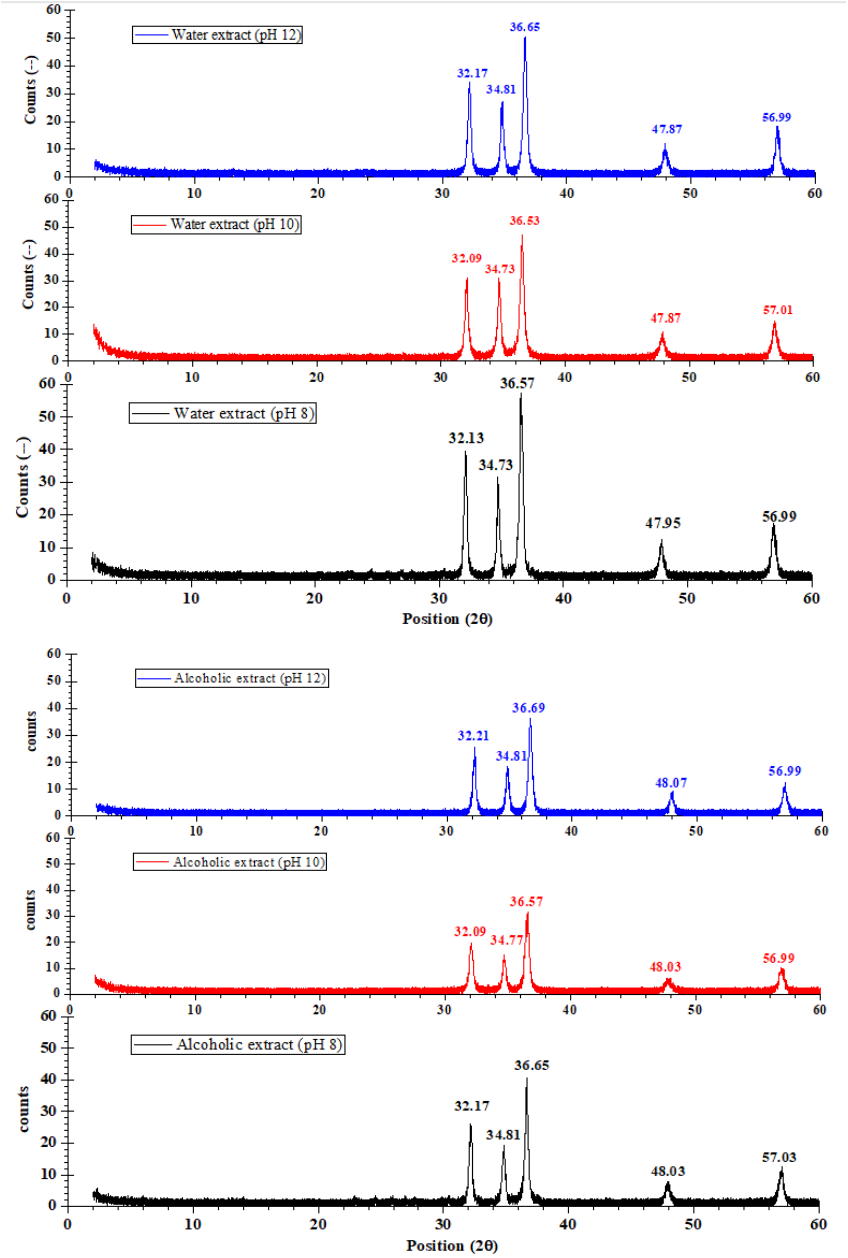


Figure 3. XRD for synthesized ZnO NPs using both *Psidium guajava* L. extracts in water and ethanol at different pH.

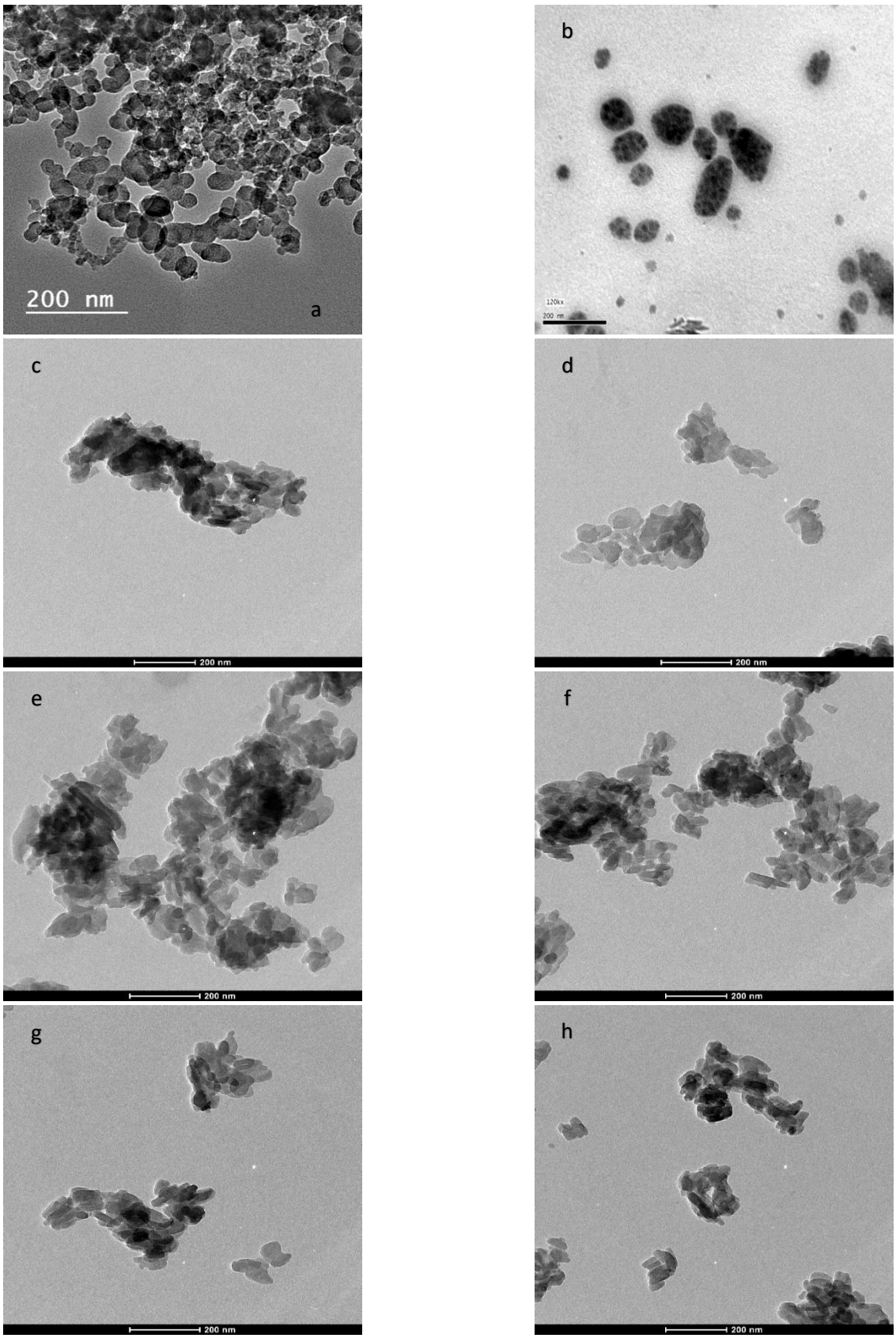


Figure 4. TEM image for synthesized ZnO NPs using both *Psidium guajava* L. extracts in water and ethanol at different pH. (**a**) *Psidium guajava* L.in water extract; (**b**) *Psidium guajava* L.in ethanol extract; (**c**) ZnO NPs in water extract at pH 8, (**d**) ZnO NPs in ethanol extract at pH 8; (**e**) ZnO NPs in water extract at pH 10; (**f**) ZnO NPs in ethanol extract at pH 10; (**g**) ZnO NPs in water extract at pH 12; (**h**) ZnO NPs in ethanol extract at pH 12.

The data show that the better binding efficiency was recorded to treated fabrics in the presence of chitosan over the pre-treated fabric with citric acid and sodium hypophosphite due to the ability of ZnO NPs to bind to the negative charge in the biopolymers. Therefore,

data provide that zinc percent uptake onto the treated fabric with ZnO NPs in chitosan presence is much higher than the treated fabrics with ZnO NPs only or with the other biopolymers. This is back to charge attraction between metals and negative charges of biopolymers molecules.

Further investigation for the treated fabric would determine the treated cotton fabric's ability to protect against ultraviolet waves by measuring the Ultraviolet protection Factor as ZnO NPs improve this property.

Therefore, the UPF values of untreated and treated cotton fabrics with synthesized ZnO NPs in the presence or absence of different biopolymer compounds were listed in Table 1. All treated fabrics have UPF values higher than untreated ones, which confirmed that ZnO NPs enhance the ability of cotton fabrics to protect from ultraviolet radiation and saving human skin from the harmful ultraviolet radiation

Data have shown that, compared to the untreated fabric, the higher the Zn content, the greater the UPF values. Results further demonstrate that the UPF values for fabrics treated with ZnO NPs in the presence of the biopolymer compound are better than those for those treated with ZnO NPs only, and UPF values typically increase as the Zn content % increased.

The UPF limits for UV-protection of the treated fabrics should be at least 40–50+, according to the report [47]. Thus, all treated fabrics with different formulations are associated with excellent UV protection even after washing.

Table 1. Zn content (%) and ultraviolet protection factor (UPF) for treated fabrics with synthesized ZnO NPs with/without biopolymers.

	extract	Zn content (%)/1 g fabric	UPF	
			Before washing	After washing
Blank		0	0.1	0.1
ZnO NPs	Water	0.52	53.5	52.6
	Ethanol	0.62	61.2	57.9
ZnO NPs/Alginate	Water	0.24	52.4	42.2
	Ethanol	0.25	56.6	45.6
ZnO NPs/CMC	Water	0.20	49.8	42.5
	Ethanol	0.23	53.4	42.3
ZnO NPs/chitosan	Water	0.44	56.3	46.3
	Ethanol	0.46	59.7	56.2

3.2.2. Morphological behavior of treated fabric.

Figure 5 illustrates the SEM images and EDX spectra of the treated cotton fabrics with synthesized ZnO NPs with/without different biopolymers.

SEM images showed depositions of ZnO NPs onto the cotton surface, which provide the presence of the nanoparticles only or inside the deposited thin film of the biopolymer network. The SEM photos showed good deposition for the ZnO NPs used. However, the distribution of the ZnO NPs, homogeneousness, and thickness depends on the form of the biopolymers used.

In addition, the Zn elements on the treated materials were verified by the EDX spectrum. The Energy-Dispersion X-ray spectroscopy (EDX) technique was used to determine the Zn element's presence on the treated cotton surface. The EDX patterns detected carbon and oxygen elements on all the samples spectra, as well as Zn elements due to the presence of ZnO NPs. The spectra verified that the ZnO NPs were fixed to the surface of treated cotton fabrics.

It is observed that fabrics treated using the synthesized ZnO NPs acquire much roughness surfaces than those treated in the presence of any biopolymers. This reflects the

importance of biopolymer in producing homogeneous distribution and penetration for the nanoparticles on treated fabrics' surface. Thus, treated fabric with synthesized ZnO NPs in the presence of any biopolymers provide a clear and smooth surface.

Furthermore, treated fabric with synthesized ZnO NPs in chitosan presence confirms the presence of more ZnO NPs content than the other two biopolymers (alginate or CMC). This observation was confirmed through EDX for treated fabric. The same result line is used to monitor Zn content for treated fabrics. SEM micrographs of treated fabrics with biopolymers indicate that surface morphology appears as a coated layer on the cotton surface and is characterized by being smoother and homogenous.

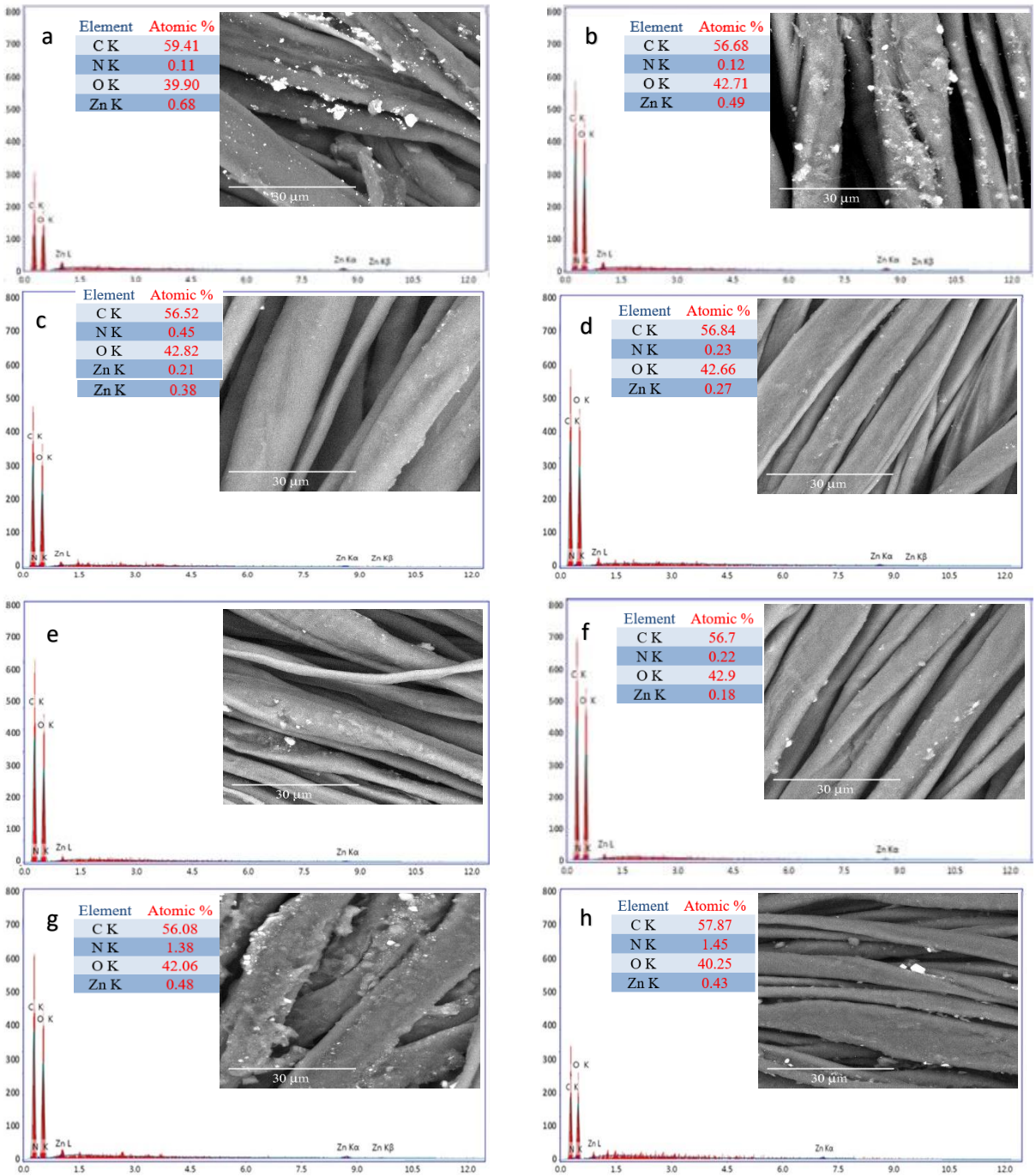


Figure 5. SEM and EDX images for treated cotton fabrics with synthesized ZnO NPs with/without biopolymer. (**a**) fabric with ZnO NPs (ethanol extract); (**b**) fabric with ZnO NPs (water extract); (**c**) fabric with ZnO NPs (ethanol extract) and Alginate; (**d**) fabric with ZnO NPs (water extract) and Alginate; (**e**) fabric with ZnO NPs (ethanol extract) and CMC; (**f**) fabric with ZnO NPs (water extract) and CMC; (**g**) fabric with ZnO NPs (ethanol extract) and chitosan; (**h**) fabric with ZnO NPs (water extract) and chitosan.

3.2.3. Physical and mechanical properties.

Treated cotton fabrics with *Psidium guajava* L. extracts and synthesized ZnO NPs with/without biopolymer have been checked for their mechanical and physical properties such as tensile strength, elongation at a break, bending length, air permeability, crease recovery angle, and surface roughness, and the results are recorded in Table 2.

From Table 2, it was clear that the crease recovery angle (CRA) increases dramatically as the roughness of the surface, air permeability, tensile strength, and elongation at break reduces after treatment with *Psidium guajava* L. extracts and ZnO NPs with/without biopolymer. This reveals that the examined ZnO NPs have been deeply absorbed into the microstructure of cotton fiber and responsible for these modifications, a thin film from the biopolymer used, deposited on the cotton's surface [5,18,66,67]. This deposited film cause was decreasing in ruggedness and increasing in air permeability values.

Also essential in determining the utility of the properties mentioned above was a pre-cross-linking treatment with citric acid and sodium hypophosphite. The formation of covalent cross-linking links among nearby cellulosic chains would provide rigidity to the cotton structure during this pre-treatment. The citric acid and reaction would meanwhile chemically degrade the cellulosic chain in the cotton fiber.

Further investigation was going for investigation the crease recovery angle for treated fabrics in both warp and weft direction and bending length. The results provide that the crease recovery angle and bending length of all treated fabrics in presences of biopolymers have increased values compared with the untreated ones or treated fabrics in the absence of biopolymer materials. These results confirm that the biopolymer materials play an important role in enhancing the mechanical or physical properties. The crease recovery angle's key enhancement was possibly due to creating an extreme cotton structure network closely cross-linked by covalent chemical linkages between the cellulose chain and biopolymers [5,18,66,67].

Table 2. Physical and mechanical properties for treated fabrics with synthesized ZnO NPs with/without biopolymer.

	extract	**R (µm)**	**Tensile strength**	**Elongation at a break (%)**	**CRA (W+F°)**	**Bending length (cm)**	**Air permeability ($cm^3/cm^2/s$)**
Blank		21.45	152.12	39.21	198.19	2.05	21.87
ZnO NPs	Water	23.24	123.51	34.81	223.17	2.36	95.53
	Ethanol	23.61	121.34	44.04	225.84	2.38	95.46
ZnO NPs/Alginate	Water	20.23	161.27	45.45	267.72	2.31	14.57
	Ethanol	20.31	162.31	58.91	272.74	2.32	15.94
ZnO NPs/CMC	Water	20.56	162.34	45.76	270.62	2.25	16.89
	Ethanol	20.54	162.45	58.96	273.57	2.28	16.59
ZnO NPs/chitosan	Water	19.78	164.65	46.41	285.45	2.41	18.78
	Ethanol	19.67	164.78	59.81	291.04	2.52	20.43

Cotton fabric was treated at room temp for 10 min.

3.2.4. Self-cleaning activity.

Methylene blue (MB) is a popular thiazine dye used for photo-catalytical activity evaluation. The photo-catalytical transformation of MB by irradiated ZnO NPs can take place through two mechanisms: (1) oxidation process: complete mineralization of the substrate to carbon and mineral ion and (2) reduction process gives the leuco Methylene Blue shape (LMB) which is rapidly oxidized by the dissolved oxygen. (see below equations) [68]

$$MB + n\ h_{vb} + (^{\cdot}OH) \rightarrow CO_2 + NO_3^- + SO_4^{-2} + NH_4^+ \quad (1)$$

$$MB + 2\ e_{cb}^- + H^+ \rightarrow LMB \quad (2)$$

$$2\ LMB + O_2 \rightarrow MB + H_2O \quad (3)$$

Furthermore, the presence of oxygen reduces the leuco MB formation due to two reasons: *i*) atmospheric O_2 scavenged almost entirely the conduction band electrons; (*ii*) the possible MB leuco form reacted immediately with O_2 to give the original-colored MB

Remove MB stains in the biopolymer matrix's presence and absence to evaluate the self-cleaning properties of the cotton textiles treated with the ZnO NPs was evaluated. The ZnO NPs-coated cotton fabrics' photocatalytic activity was investigated through their photodegradation of MB dye by measuring the stained fabric's color strength compared to the untreated one. By meaning, untreated and treated cotton fabrics have investigated their self-cleaning ability by adjusting methylene blue (MB) color intensity (K/S) before or after exposure to sunlight (UV).

The ZnO NPs are well known for their ability to create free radicals due to light photons' exposure, which are known as a self-cleaning property. The role in the color decomposition (or fading color) with the formed free radicals is due to its ability to changing the unsaturated chromophore to a saturated undyed molecule. This action is called color decomposition.

Figure 6 displays the K/S and K/S reduction (%) of the untreated and treated cotton fabric with *Psidium guajava* L. extracts and synthesized ZnO NPs with/without biopolymer after UV exposure for 12, 24, and 48 hrs. Data shows that the discoloration in the treated fabrics is greater than in the untreated ones. Treated fabrics with ZnO NPs only without biopolymer were more discolored than those with either *Psidium guajava* L. extracts only or biopolymer. This may be due to the impregnation of ZnO NPs into the polymer network, which the main reason for increasing the coloring adsorption of the basic colorant onto the fabric surface and decreasing the effect of nanoparticle activity.

Also, treated fabrics with *Psidium guajava* L. extracts only increase the dying uptake and didn’t provide self-cleaning activity as expected; thus, the dye discoloration values are almost identical with the untreated fabrics, while the presence of biopolymer with ZnO NPs on the cotton surface enhance the self-cleaning activity over the exposure time to UV. Furthermore, the impregnation of ZnO NPs in chitosan provides the highest dying uptake than the other two biopolymers (Alginate or CMC) due to the presence of two functional groups in the main structure of chitosan (hydroxyl and amine).

On the other hand, chitosan's presence shows higher values in dye discoloration, which is attributed to chitosan's role in the distribution of the nanoparticles in the treatment solution are better than the other two polymers. This homogenous distribution causes good distribution and covers the composite on the fabric surface, leading to more color decomposition efficiency.

Due to a photocatalytic degradation activity of the ZnO NPs deposited on the fabric surface, the MB stains' color intensities on the ZnO NPs-coated fabrics decreased dramatically. On the surface of the untreated fabric, the MB stains remained easily visible. The photocatalytic degradation properties depended on the presence and type of biopolymer used to impregnate ZnO NPs.

3.2.5. Antimicrobial properties.

The antimicrobial activities of the treated fabrics with ZnO NPs with/without biopolymer coating were qualitatively and quantitatively demonstrated by using three types of microbes; gram-positive bacteria (*Staphylococcus aureus)*, gram-negative bacteria (*Escherichia coli*), and fungus *(Candida albicans)*.

The antimicrobial activity of treated cotton fabrics was shown in Figure 7 and Table 3. The results show that there are no inhibition results on two types of bacteria and fungus tested on untreated cotton fabric (blank). Compared to the three types of studied microbes, treated fabrics with both *Psidium guajava L.* extracts in water and ethanol show substantially increased antibacterial activity, which is provided by measuring the inhibition zone area. The data provides good inhibition zone area (including tested fabric area (78.6 mm^2)) recorded for treated fabric with water extract as 187.76±0.15, 280.72±0.23, 278.96±0.17 mm^2 and for treated fabric with ethanol extract as 189.73±0.21, 327.37±0.42, 204.63±0.36 mm^2 for *E. coli*, *S. aureus*, and *C. albicans*, respectively.

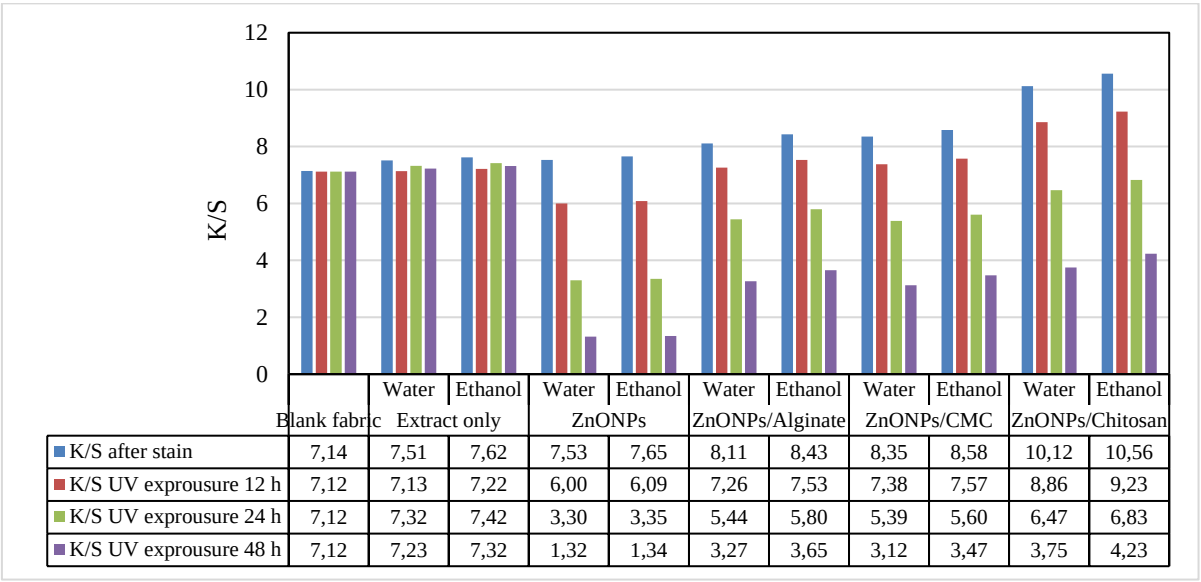
The presence of phenolic acid and flavonoids in *Psidium guajava* L extract is the cause for this successful activity. Compared to gram-positive bacteria, treated fabrics are higher effective than Gram-negative bacteria, owing to the variations in the composition of both the studied bacteria strain's cell walls. These compounds also serve as an antifungal substance since they suppress ergosterol, the fungal cell membrane's key component [69-71].

Treated fabrics with synthesized ZnO NPs with/without biopolymer against both bacteria and fungi provide antimicrobial behavior greater than untreated fabric. The antimicrobial effect increased base on the biopolymer used. The presence of chitosan with nanoparticles provides more antimicrobial effect than other both polymer and nanoparticles only. That is because chitosan provides a good antimicrobial due to amino groups, which plays an excellent role in attacking the cell membrane of microbes [66]. The carboxyl group present in alginate provides antimicrobial properties but less than the amino group present in chitosan. Furthermore, ZnO NPs provide antimicrobial properties which imparted to cotton fabrics and confirmed by this method.

The difference in the influence on various microbes in each of these materials was further due to the cell walls' composition differences. The gram-positive bacteria are distinguished because each cell membrane, enclosed with a thick and porous cell wall, allows some bioactive components to spread through this. Instead, gram-negative bacteria consist of three different layers containing several bioactive elements.

ZnO NPs are successfully interacted by the coating process with the cells of the bacteria. The biopolymers engaged in the coating have good diffusion and stabilization of ZnO NPs on the cotton surface, reducing the capacity to tie bacterial cells onto the cotton surface [6,19,20,72].

Besides, treated cotton textiles with synthesized ZnO NPs with/without biopolymer have excellent antibacterial properties than the untreated ones. The antibacterial reduction percent decreased as the treated fabrics' washing cycles were increased (5, 10, 15, and 20 cycles) (Table 3). This decrease in reduction percent is varied regarding the biopolymer used in the treatment. The durability of the fabrics being treated in the process provides an excellent antimicrobial property against tested microbes even after different washing cycles. The bacterial reduction percent provides a reduction of up to 93 % after 20 washing cycles for treating fabrics with ZnO NPs/chitosan.



	Blank fabric	Extract only		ZnONPs		ZnONPs/Alginate		ZnONPs/CMC		ZnONPs/Chitosan	
		Water	Ethanol	Water	Ethanol	Water	Ethanol	Water	Ethanol	Water	Ethanol
K/S after stain	7,14	7,51	7,62	7,53	7,65	8,11	8,43	8,35	8,58	10,12	10,56
K/S UV exprousure 12 h	7,12	7,13	7,22	6,00	6,09	7,26	7,53	7,38	7,57	8,86	9,23
K/S UV exprousure 24 h	7,12	7,32	7,42	3,30	3,35	5,44	5,80	5,39	5,60	6,47	6,83
K/S UV exprousure 48 h	7,12	7,23	7,32	1,32	1,34	3,27	3,65	3,12	3,47	3,75	4,23

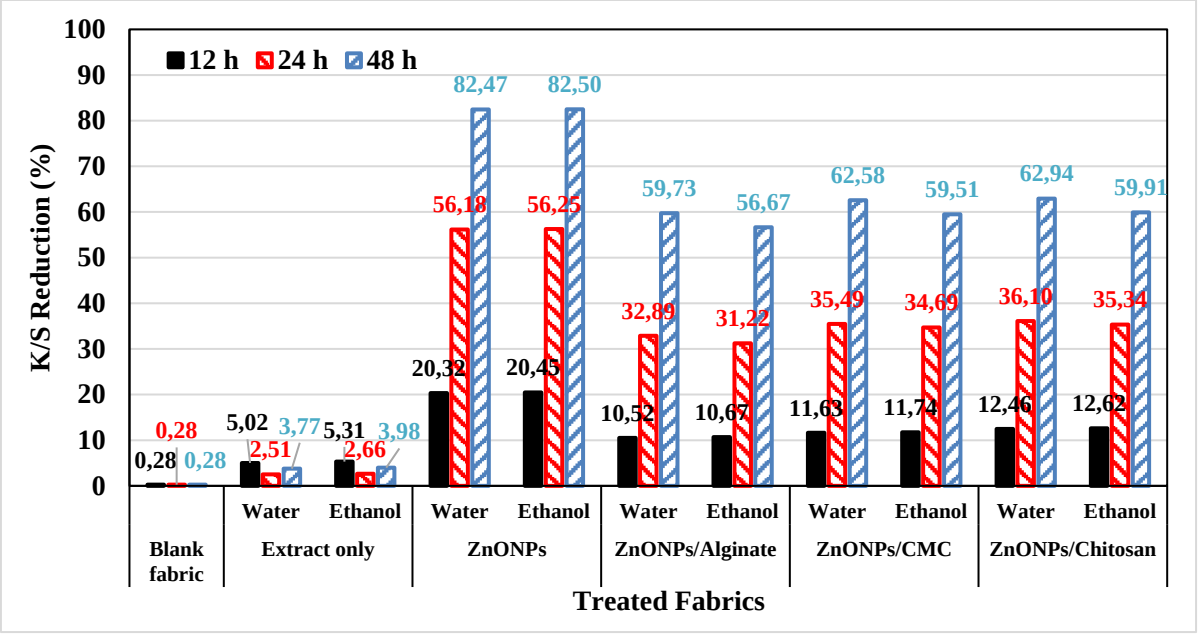


Figure 6. Self-cleaning activity expressed as K/S (a) and K/S reduction (%) (b) for treated fabrics with *Psidium guajava* L. extracts and synthesized ZnO NPs with/without biopolymer after UV exposure for 12, 24, and 48 hrs.

3.2.6. Contact angle and wettability.

There are two parameters analyzed to explain the interactions of fabric surface with water, i) the contact angle and ii) the wettability time. The angle of contact between a droplet and the fabric surface can be described as the contact angle. When the water droplet obtains a small contact angle at the surface, the surface is then considered hydrophilic and quickly moisturized, and the water droplet spreads. The surface area between the droplet and the surface of the fabric is thus rising. On the other hand, the surface is called hydrophobic. A water droplet on the fabric surface forms a large contact angle; the surface tends to repel water. Treated fabrics were analyzed by calculating the contact angle and wettability time, and the results are listed in The biopolymer's presence as a carrier for good distribution of ZnO NPs in their network decreased the contact angle than treated fabric with ZnO NPs only. This is due to the thin layer of film on the treated cotton surface, which relatively filled the superficial fibers' cavities and slightly enhanced the hydrophobicity. As both biopolymers

(alginate and CMC) are soluble in water, they improve the hydrophilicity property more than chitosan (water-insoluble biopolymer).

Table 4.

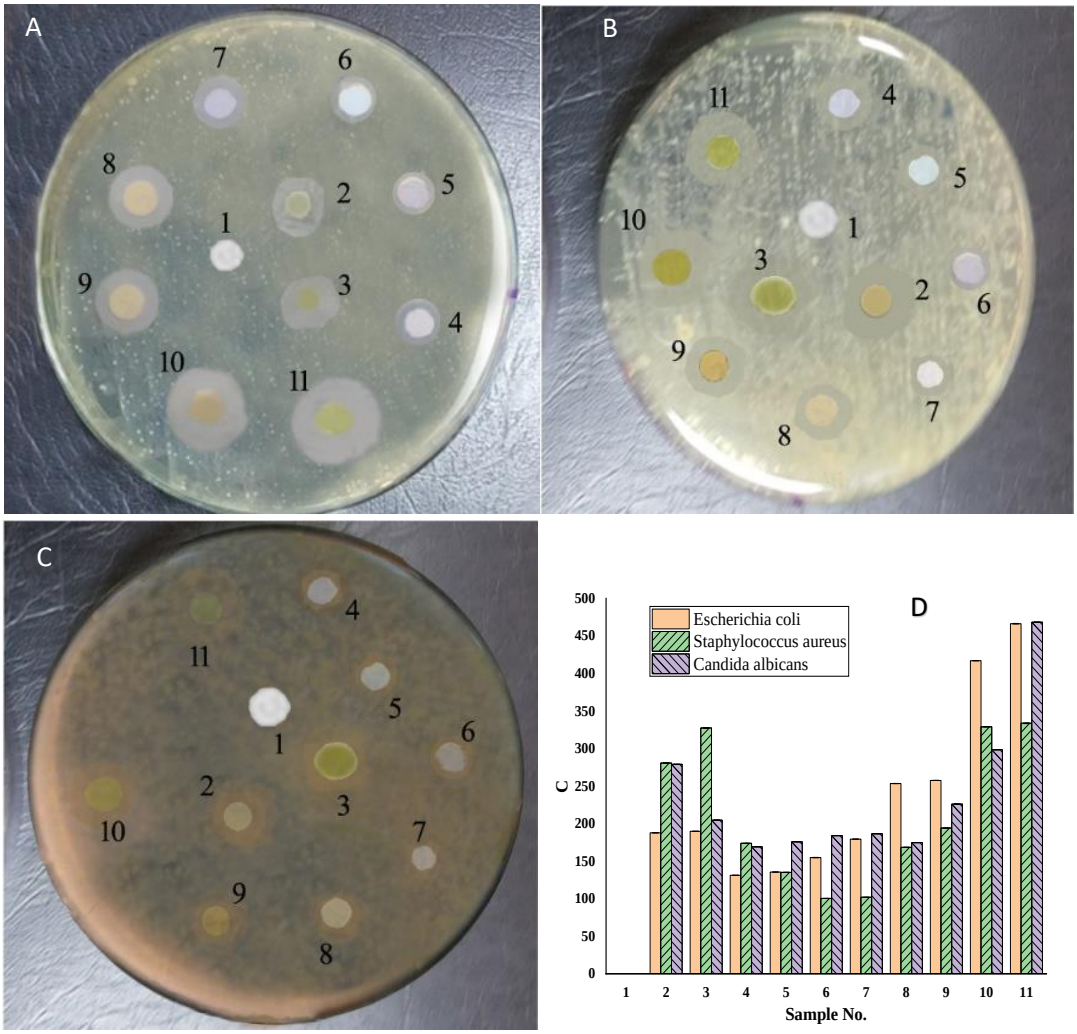


Figure 7. Antibacterial activity of treated fabrics with *Psidium guajava* L. extracts and synthesized ZnO NPs with/without biopolymer against (**a**) *E. coli* (gram-negative bacterium); (**b**) *S. aureus* (gram-positive bacterium); (**c**) *C. albicans* (fungus); (**d**) calculated area of inhibition zone (1) untreated fabric; (2) treated fabrics with *Psidium guajava* L. in water extract; (3) treated fabrics with *Psidium guajava* L. in ethanol extract; (4) treated fabrics with ZnO from water extract; (5) treated fabrics with ZnO from the ethanol extract; (6) treated fabrics with ZnO/Alginate from water extract; (7) treated fabrics with ZnO/Alginate from the ethanol extract; (8) treated fabrics with ZnO/CMC from water extract; (9) treated fabrics with ZnO/CMC from the ethanol extract; (10) treated fabrics with ZnO/Chitosan from water extract; (11) treated fabrics with ZnO/Chitosan from the ethanol extract.

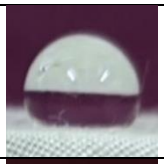
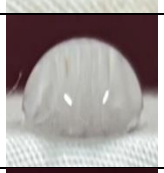
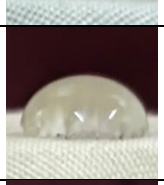
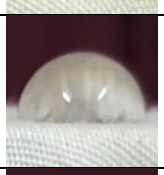
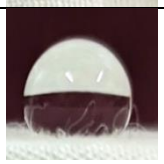
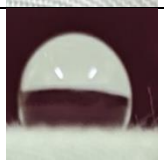
Table 3. Microbial reduction % of untreated and treated cotton fabrics.

Treated fabric with	Extraction process	Microbial Reduction %														
		E. coli (ATCC 25922)					*S. aureus* (ATCC 29213)					*C. albicans* (ATCC 10231)				
		before washing	after washing cycles				before washing	after washing cycles				before washing	after washing cycles			
			5	10	15	20		5	10	15	20		5	10	15	20
Blank fabric		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Extract only	**Water**	62.91	51.27	31.13	29.80	28.23	76.18	62.08	37.66	36.05	34.14	56.53	46.08	27.98	26.79	25.38
	Ethanol	66.77	54.42	40.20	38.49	36.45	87.21	71.06	62.48	59.80	56.64	61.31	49.97	43.94	42.07	39.85
ZnO	**Water**	84.09	78.27	68.19	67.53	66.74	90.72	83.67	71.46	70.65	69.70	80.90	75.67	66.62	66.02	65.32
	Ethanol	86.02	79.84	72.73	71.87	70.86	96.23	88.16	83.87	82.53	80.95	83.28	77.62	74.60	73.66	72.55
ZnO/ Alginate	**Water**	84.67	81.76	76.73	76.39	76.00	87.99	84.46	78.36	77.96	77.48	83.08	80.46	75.94	75.64	75.29
	Ethanol	85.64	82.55	79.00	78.57	78.06	90.75	86.71	84.56	83.90	83.11	84.27	81.44	79.93	79.46	78.91
ZnO/ CMC	**Water**	84.38	80.02	72.46	71.96	71.37	89.36	84.07	74.91	74.30	73.59	81.99	78.07	71.28	70.83	70.30
	Ethanol	85.83	81.19	75.86	75.22	74.46	93.49	87.44	84.22	83.21	82.03	83.78	79.53	77.27	76.56	75.73

ZnO/ Chitosan	**Water**	96.48	95.09	90.45	82.69	82.61	99.74	97.41	94.49	92.58	90.32	96.20	94.41	93.54	91.97	90.10
	Ethanol	98.48	98.24	91.40	89.00	86.16	99.66	99.33	98.43	97.48	96.99	98.41	97.94	96.65	94.13	93.16

The biopolymer's presence as a carrier for good distribution of ZnO NPs in their network decreased the contact angle than treated fabric with ZnO NPs only. This is due to the thin layer of film on the treated cotton surface, which relatively filled the superficial fibers' cavities and slightly enhanced the hydrophobicity. As both biopolymers (alginate and CMC) are soluble in water, they improve the hydrophilicity property more than chitosan (water-insoluble biopolymer).

Table 4. Contact angle, wettability time, and drop photo image for treated cotton fabrics.

Treated fabric with		**Synthesized ZnO NPs with**					
		Water extract			**Ethanol Extract**		
		Wettability time (sec)	**Contact angle (°)**	**Drop photo image**	**Wettability time (sec)**	**Contact angle (°)**	**Drop photo image**
Blank		2	0		2	0	
ZnO NPs	Before washing	90	126.52 ± 2.38		90	112.54 ± 6.63	
	After washing	30	118.50 ± 2.02		60	102.42 ± 5.64	
ZnO NPs/Alginate	Before washing	30	133.32 ± 5.51		60	107.35 ± 1.82	
	After washing	15	128.66 ± 4.96		15	100.09 ± 1.64	
ZnO NPs/CMC	Before washing	90	136.88 ± 3.09		60	89.86 ± 4.25	
	After washing	30	132.54 ± 2.78		15	80.85 ± 3.83	
ZnO NPs/chitosan	Before washing	210	134.03 ± 3.18		120	111.39 ± 9.81	
	After washing	60	131.73 ± 3.02		30	107.96 ± 9.32	

Furthermore, as the contact angle of treated fabric increases compared to blank fabric, the wettability time also increases. The excellent observation and the wetting performance for treated fabric with ZnO NPs in chitosan presence provide a higher time than other treated fabrics even with ZnO NPs. This phenomenon is due to the thin layer of film on the treated fabric surface, which relatively filled the superficial fibers' cavities and enhanced the fibrous surface with higher rough morphology, causing increased hydrophobicity.

The contact angles for treated fabrics were reported after washing to analyze the treated cotton fabrics' resilience. It is observed that the contact angle and wettability time was decreased significantly. Another investigation treated fabrics with ZnO NPs in the presence of chitosan biopolymer, providing a good wettability performance even after washing. From this analysis, a composite coating on the fabric's surface was implemented to achieve a specific water repellence, weather resistance. Therefore, depending on the coating substance, superhydrophobic fabrics can be used in water-oil applications.

4. Conclusions

Psidium guava leaf extract can be used as a reducing agent to synthesize zinc oxide nanoparticles as a green route method instead of a chemical method that uses hazardous chemicals because it has many bioactive compounds such as phenols, flavonoids. Psidium

Guava Leave was extracted using two solvents (water and ethanol). Both of them can be used as reducing agents for the synthesis of zinc oxide nanoparticles.

The synthesized ZnO NPs was studied at various pHs. The particle size of synthesized ZnO NPs was analyzed. It was found that, by increasing the pH medium, the synthesized ZnO NPs particles decreased. It is also noted that the particle size of the synthesized ZnO NPs with *Psidium guajava* L. extract in water is higher than those in alcohol at different pH (8, 10, and 12) medium. Moreover, for shaping ZnO NPs in both extracts, pH 12 proved to be an excellent medium. TEM images show that thin, spherical nanoparticles approximately 5 nm are agglomerates of small clusters during synthesis steps by increasing pH (until pH 12).

The treated cotton fabric with ZnO NPs and ZnO NPs impregnated with biopolymers (Alginate, chitosan, and carboxymethyl cellulose) showed that the higher the Zn content, the greater the UPF values. The UPF values and antimicrobial activity for fabrics treated with ZnO NPs in the presence of the biopolymer compound are better than those treated with ZnO NPs only.

Untreated and treated cotton fabrics have investigated their self-cleaning ability by adjusting methylene blue (MB) color intensity (K/S) before or after exposure to sunlight (UV), and it was found that the discoloration in the treated fabrics is greater than in the untreated ones. Treated fabrics with ZnO NPs only without biopolymer were more discolored than those with either *Psidium guajava* L. extracts or biopolymer.

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Conflicts of Interest

The authors declare no conflict of interest.

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