Egyptian Journal of Chemistry
http://ejchem.journals.ekb.eg/

Fabrication of Multifunctional Nanoparticles As Surface Modification System For Textile Fabrics

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In Loving Memory of Late Professor Doctor ""Mohamed Refaat Hussein Mahran""

Abstract
The wastes of various untreated and uncolored textile materials resulting from research and service activities such as cotton, wool, and polyester fabrics in the Textile Research and Technology Institute were given added value by treating them with synthesis silver nanoparticles and chitosan in the present or absence of extracted natural oils from plants like basil, lemon (Citrus aurantifolia) and mint. Using the method of immersion, squeezing, and thermal stabilization in certain conditions selected from time and temperature to improve its antimicrobial activity. Transmission Electron Microscopes (TEM) were used for the determination of the size, shape, and size distribution of Synthesis of silver nanoparticles (AgNPs) using different oils. The treated fabrics morphological changes were characterized using (SEM) and energy-dispersive X-ray analysis. The color intensity and the ultraviolet protection (UPF) of the printing-treated fabrics in comparison to the untreated ones were investigated. Thermogravimetric Analysis (TGA) is performed to investigate the changes in the polymeric structure of fabrics. The changes in tensile strength and elongation at break have been studied. The results of this investigation displayed significant improvement of treated fabrics against the antimicrobial Staphylococcus aureus (G+) (S. aureus) and Escherichia coli (G+) compared to the untreated ones.
Keywords: silver nanoparticles, (TEM), printing, natural oils, and fabrics

1. Introduction

The significant advancements in Nanoscience and nanotechnology have an impact on most industries worldwide, particularly the textile and everyday sectors. The response from the laboratory has allowed nanotechnology to solidify its position in the global market. There are still a lot of unsolved questions in this discipline despite the tremendous progress made in it. Putting nanotechnology into reality on a wide scale to enable the commercial manufacturing of nanomaterials and their related products presents additional challenges. The textile industry soon realized the benefits of having experience in well-established fields and the potential for both technical and financial success. As a result, during the past 10 years, many research groups worldwide have focused on finishing textile materials with Ag, TiO2, and ZnO NPs [1]. Cotton is the most widely used natural cellulosic fiber in textile and clothing production. As consumers demand more sophisticated characteristics from traditional textile products, cotton is being altered more and more [2]. On the other hand, cotton fibers are hydrophilic and naturally nourishing, providing an excellent environment for the growth of fungi and germs [3]. And, when left undyed or dyed in light hues, they lack an effective UV radiation barrier [4]. So, Zinc oxide nanoparticles (ZnO-NPs) were created by using sodium hydroxide as an alkali and sodium alginate as an anti-agglomeration agent in a basic chemical synthesis method. Next, ZnO-NPs were surface-modified with SiO2 nanoparticles using the sol-gel method. By adding aminopropyltriethoxy-silan (APTES) and vinyltriethoxysilan (VTES), which boost the SiO2, ZnO-NPs nanocomposite's affinity towards the glycosidic chains of cotton textiles, it was successful in enhancing the multifunctional capabilities of the nanocomposite. These nanocomposites of ZnO-NPs, SiO2, ZnO-NPs, SiO2, ZnO-NPs/APTES, and SiO2, ZnO-

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Receive Date: 23 December 2023, **Revise Date:** 02 February 2024, **Accept Date:** 0 February 2024
DOI: 10.21608/ejchem.2024.257379.9032

NPs/VTES produced exceptional antibacterial and UV protection in cotton fabrics that were highly durable even after 20 washing cycles. ZnO nanoparticles are surface-modified and SiO2-coated to be used in cotton textiles [5]. Nanotechnology has also invented several ways to enhance the ultraviolet protection factor (UPF) of the fabric, which increases with increasing environmental pollution. Ultraviolet-resistant (UPF) cotton fabrics were developed by coating with semiconductor metal oxide nanoparticles such as ZnO and TiO2 [6]. Moreover, silver nanoparticles represent a significant advancement in nanotechnology because of their increased durability and decreased chemical reactivity as compared to other metals. Given their special physicochemical characteristics, silver nanoparticles have drawn much interest in biological applications. Researchers have become interested in the use of biosynthesized AgNPs in several domains, such as agronomic, antifungal, antibacterial, and antiviral industries [7]. Cotton is a significant cellulose fiber, but its limited applications in various industries are typically hindered by its lack of antibacterial properties, self-cleaning nature, UV protection, and electric conductivity. So, silver nanoparticles have been successfully incorporated into cotton textiles as an antimicrobial agent because of their potent antibacterial and inhibitory effects on a wide range of bacteria, fungi, and viruses at low human toxicity. And to create cotton textiles with excellent performance and added value that is self-cleaning, UV-protected, and electrically conductive [8]. Green synthesis of silver nanoparticles (AgNPs) functionalized with seaweed (Padina gymnospora) extract was investigated as a Simple, affordable, and compatible way of synthesizing seaweed functionalized. These synthesized AgNPs were coated on cotton fabric by pad try-cure method using citric acid as a cross-linking agent. The chemical linkage of colloidal AgNPs on AgNPs coated fabric was getting greater UPF (UV Protection Factor) values than they compared the surface roughness (Ra) of the coated sample showed a lower roughness value obtained in 2 μm than the untreated cotton fabric. AgNPs-coated cotton fabric provided better UV protection values than Seaweed Extracts (SWE) coated and uncoated cotton fabric. the obtained results exhibited the AgNPs coating did not largely affect the UV protective and antimicrobial activity even after 10 times repeated washings. Therefore, AgNPs coated cotton fabric can be a promising durable material for biomedical and textile garment finishing applications [9].Researchers led to that Moringa oleifera leaf extract (ME) has been enhanced as an eco-friendly antibacterial agent for fabrics made from cotton or its modified forms, carboxymethyl cellulose, and cationic cotton. Furthermore, to increase the antibacterial activity of cellulosic fabric treated with Moringa extract, silver nanoparticles (AgNPs) were created using polyvinyl alcohol and glucose as a reducing agent matrix. Excellent antibacterial activity was demonstrated by the silver nanoparticle and moringa extract finished fabric, indicating the potential for these materials to be employed as safe and biocompatible antibacterial finishing agents for cellulosic fabric [10]. Furthermore, a Colloidal silver nanoparticle solution syntheticed from herbal extracts (H-AgNPs) was produced using Tulsi extract (Ocimum tenui florum) as a reducing and capping agent. The anionic surfactant sodium dodecyl sulfate (SDS) was used to create 10 layers of a colloidal solution of H-AgNPs for the cotton woven sample utilizing the pad-dry-cure process. Additionally, a different sample was created using the same concentration of HAgNPs colloidal solution combined with a blend of natural oils. Cotton textiles treated with AgNPs have strong antibacterial properties and superior UV protection. Additionally, the mechanical qualities of cotton fibers, such as elongation and tensile strength, were greatly improved by the nanocoating. The herbal reduction of AgNP is therefore highly promising and may be regarded as a possible contender to satisfy the real need for UV protection antimicrobial cotton fabric finishing in the textile industry [11]. Wool fiber among natural fibers, which are still preferred for usage even though there are many synthetic fibers available nowadays [12]. Wool is a unique natural material that has several benefits, such as excellent warmth and flame retardant, stain, and antistatic properties. Wool fibers are used to create clothing and suits in addition to floor coverings [13, 14]. Other than that, original wool fiber has many important disadvantages, such as being a great substrate for the growth and development of bacteria at the right temperature and humidity, having a naturally hydrophobic outer surface due to the fatty acid layer, and having a rough surface due to the cuticle structure [15]. The use of AgNPs to provide antimicrobial effects and their functional properties to wool textile fibers has been the main focus of recent research in the textile industry [16]. Pomegranate peel extract was used as a reducing agent in the biosynthesis of silver nanoparticles for treating wool using the exhaustion method. The adsorption of nanoparticles on wool was based on electrostatic interactions and it was improved at lower pH conditions in which there are fewer anionic groups on the wool fiber. Also, higher temperatures during treatment led to increased nanoparticle adsorption on wool. When samples were tested for antibacterial activity against E. coli bacteria, the textiles treated with AgNPs showed good antibacterial capacity even after many cycles of washing [17]. Citrullus colocynthis (C. Colocynthis) was utilized as a reducing and

stabilizing agent to create silver nanoparticle colloidal solutions (AgNPs). Wool fabric was treated with the produced colloidal AgNPs solution by padding method to enhance its antibacterial activity. Treated wool fabrics showed antibacterial activity, especially in Staphylococcus aureus (G+), Enterobacter cloacae (G-), and Candida albicans (Yeast)compared to untreated ones [18]. Trisodium citrate (TSC) was used as a reducing and capping agent while treating wool textiles with silver (Ag) nanoparticles in a single step to create multifunctional wool fabrics. The fibers treatment showed extremely good antistatic, UV radiation absorption, and antibacterial characteristics. Additionally, the created treatment is quite durable in washing since the treated fibers are still intact after 20 washes [19]. The synthesis, characterization, and application of various chitosan (Ch) derivatives containing Ag, Cu, and Zn nanoparticles (NPS) on wool yarn were investigated. the results indicated the successful surface modification of wool fibers with Ch-NPs. The treated samples showed excellent antimicrobial activities (~ 100%) against Staphylococcus aureus (S. aureus) and Escherichia coli (E. coli). The treated samples had generally very high antimicrobial activities (> 80%) even after 10 washing cycles. The surface modification of wool fibers with Ch-NPs can be considered an environmentally friendly method for antimicrobial finishing [20]. On the other hand, lemon peel oil (oil) and its nanoemulsion, as well as encapsulated with nano clay, were created and applied to wool fabrics to increase their resistance to microbes. Fabrics treated with oil (Citrus aurantifolia) and its derivatives showed significant antibacterial effects against Staphylococcus aureus (G+) when compared to untreated textiles [21]. Polyester (PES) textiles boast remarkable strength, chemical resistance, processability, quick drying times, and dimensional stability, making them extensively used across several industries. However, it produces a hydrophobic surface where microorganisms can thrive due to the substantial adsorption of metabolic wastes from the skin's sebaceous glands and sweat [22]. Through the use of chemicals such as calcium chloride, sodium alginate, chitosan, and nanoparticles of aluminum oxide and silver nitrate at different stages of finishing, the most important expected properties of hospital wear were realized on polyester fabrics. The results showed that chitosan treatment significantly accelerated the rate of water absorption in nonwoven polyester fabric. The research also showed that chitosan and silver nitrate generated good antibacterial activity fabric. Each sample's deodorizing effect was further observed. Thus, it may be possible to successfully produce medical polyester textiles with the required properties by mixing alginate and chitosan with silver nitrate and aluminum oxide nanoparticles [23]. The alteration of textiles to generate materials for human performance (sports, medicinal, and protective) is one of the suggestions for future textile development. Antioxidant and antibacterial protection were enhanced by adding silver nanoparticles to a polyester surface. To do this, polyester textiles were made using ethylenediamine aminolysis as ligands, which trapped silver ions to further decrease silver nanoparticles (AgNPs). Dopamine (PDA) was utilized to give the polyester textile antibacterial and antioxidant qualities by transforming silver ions into AgNPs via its phenolic hydroxyl groups. Improved tensile strength, antimicrobial and antioxidant qualities, and PDA-AgNPs composite nanocoating showed that polyester with a PDA-AgNPs overlay might be employed for long-term biomedical textiles [24]. Three knitted textiles were chosen and investigated whose composition and structure were insufficient to provide effective UV protection**.** The surface textures and fabric weights of the chosen textiles varied greatly**.** There were more opportunities for adjustments because the three structures used different knit types. To increase the UV protection factor and reduce the quantity of ultraviolet radiation passed through the fabric, the cover factor of wearable textiles was changed using the textile modification process of calendaring. The exposed area was observed to reduce by a factor of two after one pass, while the UV protection factor rose by 200%. With repeated calendaring, the treated fabric's thickness and air permeability were reduced. The mechanical parameters were not significantly modified by the fabric compression since the bending stiffness was almost constant [25]. Environmentally friendly finishing methods for imparting functional properties of various textiles are very desirable. So, this article aims to treat cotton, wool, and polyester fabrics, with silver nanoparticles/ oil composite in the presence of chitosan as a crosslinker. The oil is extracted from natural plants like basil, lemon (Citrus aurantifolia), and mint to enhance their functional properties. Such as raising resistance to microorganisms, and enhancing UPF protection as well as colour strength. It is highly desired to apply functional qualities to different fabrics using environmentally friendly finishing techniques.

2. Materials and methods

2.1. Fabrics

The waste of untreated textile materials was collected from different textile companies for clothing in Egypt such as cotton, wool, and polyester fabrics.

2.2. Chemicals

In this work, oil of basil, mint, and lemon (Citrus aurantifolia) which is grown in Egypt was used where: Basil, as shown in Figure 1, (Ocimum basilicum), is one of the most important crops with essential oils as well as polyphenols, phenolics, flavonoids, and phenolic acids, The most important pharmacological uses of basil are anti-cancer activity, radioprotective activity, anti-microbial activity, anti-inflammatory effects, immunomodulatory activity, anti-stress activity, anti-diabetic activity, antipyretic activity, anti-arthritic activity, anti-oxidant activity, as a prophylactic agent and in cardiovascular disease. It has been reported that important essential oil components are terpenes, phenylpropanoids, alcohols, and aldehydes, and essential Phenolic acids and flavanol-glycosides are the main phenolic components in basil [26].

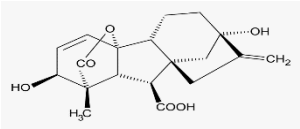


Figure 1: Basil

Mint, as shown in Figure 2, is terpene alcohol with a strong minty, cooling odor and taste. It is obtained from peppermint oil or is produced synthetically by hydrogenation of thymol. Menthol is used medicinally in ointments, cough drops, and nasal inhalers. It is also used as flavoring in foods, cigarettes, liqueurs, cosmetics, and perfumes. Menthol is a cyclic monoterpene alcohol with well-known cooling characteristics and a residual minty smell of the oil remnants from which it was obtained. Because of these attributes, it is one of the most important flavoring additives besides vanilla and citrus. Due to this reason, it is used in a variety of consumer products ranging from confections such as chocolate and chewing gum to oral-care products such as toothpaste as well as in over-the-counter medicinal products. These plants exhibit biological activity in vitro and in vivo such as antibacterial, antifungal, antipruritic, anticancer, and analgesic effects, and are also effective fumigants. In addition, menthol is one of the most effective terpenes used to enhance the dermal penetration of pharmaceuticals. This review summarizes menthol's chemical and biological properties and highlights its cooling effects and toxicity [27].

Figure 2: Mint

The valuable biological activity of C. Limon as shown in Figure .3 is determined by its high content of phenolic compounds, mainly flavonoids (e.g., diosmin, hesperidin, limo citrin) and phenolic acids (e.g., ferulic, synapic, p-hydroxybenzoic acids). The essential oil is rich in bioactive monoterpenoids such as D-limonene, β-pinene, and γ-terpinene. Recently scientifically proven therapeutic activities of C. Limon include anti-inflammatory, antimicrobial, anticancer, and antiparasitic activities [28].

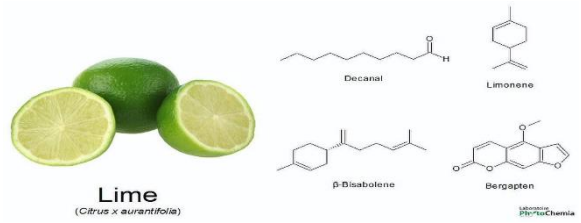


Figure 3: Limon

Silver nitrate ($AgNO_3$), ethyl alcohol, ethyl acetate, acetone, surfactant (tween 80), and ammonium persulphate were purchased from Sigma Aldrich Co. (Germany). Acetic acid was purchased from El Nasr Pharmaceutical Chemicals- Abou Zabel - Egypt. Extra pure anhydrous citric acid was supplied by (Loba Chemise, India). Non-ionic detergent (nonyl phenol ethoxylate, was supplied from Starch & Detergent Company. Alexandria, Egypt). Sodium hypophosphite monohydrate (SHP) obtained from Duksan Chemical Co. Thickening agent: Daico thick 1600, synthetic thickener was kindly supplied by Daico Chemical Industry, Cairo, Egypt. Pigment MD Red BB was kindly supplied by Daico Company, Cairo, Egypt. Printofix Binder MTB was kindly supplied by Clarinet. All chemicals and reagents were of laboratory grade.

2.3. Methods

2.3.1. Synthesis of silver nanoparticles (AgNPs)

First, oil in water microemulsion of different oils (basil, mint, and lemon oils) was prepared by adding 10 ml of each oil into 90 ml of water and 2 ml of tween 80 then the mixture was stirred for 30 min, and then the mixture was ultrasonicated for another 30 min. after that,10 ml from the microemulsion oil were added to aqueous $AgNO_3$ (90 ml; 0.02 M) for 10 min at 80°C. The pH medium was adjusted to 10. In the beginning, the color of the solution changed to pale yellow and then converted to dark brown, indicating the formation of AgNPs.

2.3.2. Treatment of the fabric using silver nanoparticles/ oil composite

The fabrics were cut into 20 × 20 cm, and then the fabric was washed using a non-ionic detergent 2 g/L and then air-dried. The fabrics were immersed in a solution containing 10 g/L citric acid and 5 g/L sodium hypophosphite for 5 min at 50°C, then padded with 100 % wet pickup to remove excess solution, and air-dried. Chitosan aqueous solution (100 mL; 4%) was prepared and then 100 ml of the solution containing AgNPs/oil was added gradually under constant stirring over 30 min. Then, fabrics were immersed in chitosan/AgNPs/oil composite solutions or AgNPs/oil solution for 5 min at 50°C, then padded with 100 % wet pickup to remove excess composite, and air-dried. Then cured at 150°C for 4 min.

2.3.3. Printing

2.3.3.1. Preparation of the printing pastes

The printing paste was prepared according to the following recipe:

Pigment	50
Thickener *	x
Binder	100
Ammonium persulphate	10
Water	y

* The amount of thickener was used Dicothick 30 g / kg

2.3.4. Printing technique

The printing paste was applied to untreated and treated cotton, wool, and polyester fabrics using the silk screen printing technique. Fixation was carried out by thermal treatment at 150 ˚C for 5 minutes in an automatic thermostatic oven (Wenner Mathis Co., Switzerland).

2.4. Measurements and analysis

2.4.1. Antimicrobial activity for the treated fabric

Evaluation of the antimicrobial activity for untreated and treated fabrics with synthesis silver nanoparticles and chitosan in the present or absence of extracted natural oils from plants like basil, lemon (Citrus aurantifolia), and mint was estimated towards Staphylococcus aureus (G^{+ve}) and Escherichia coli (G^{-ve}) according to AATCC test method 100–2012 (AATCC Test Method (100-2012), 2012). The reduction of bacteria is calculated using the following equation:

$$R\% = \frac{A-B}{A} \times 100$$

2.4.2. Colorimetric measurements

Color strength (K/S Value) was measured on a Minolta Spectrophotometer Hunter Lab Universal Software Ultra scan (USA). The values were calculated using the Kubelkae– Munk equation at max 540 nm [29].

$$K/S = \frac{(1-R)}{2R} - \frac{(1-R_0)}{2R_0}$$

Where

R = Decimal fraction of the reflectance of treated printed fabric.

R_0 = Decimal fraction of the reflectance of the unprinted fabric.

K = Absorption coefficient.

S = Scattering coefficient.

Also, Color measurements of treated textiles were made according to the CIE (L*, a*, b*) system to evaluate the color coordinates using the Hunter-Lab spectrophotometer (model: Hunter Lab DP-9000). Where: CIE (L*, a*, b*) between two colors each given in terms of L*, a*, b* is calculated from: $\Delta E^* = \sqrt{(\Delta L *)2 + (\Delta a *)2 + (\Delta b *)2}$ Here, ΔL* (L* sample minus L* standard) = difference in lightness and darkness. Δa* (a* sample minus a* standard) = difference in red and green.

L* value indicates lightness, (+) if the sample is lighter than the standard, (-) if darker., a* & b* values: indicate the relative positions in the CIE Lab space of the sample and the standard, from which some indication of the nature of the difference can be seen.

2.4.3. Fastness Testing

The color fastness of the printed fabrics to wash before and after treatment was determined by the AATCC test method 69, 23 (993)

2.4.4. Evaluation of UV Protection

The transmittance and UPF values of the original fabrics unprinted and printed treated fabrics were measured using AS/NZ S4399:1996-UPF, according to AATCC Test Method 183:2010-UPF.

2.4.5. Tensile strength and elongation %

Mechanical properties of treated fabrics as well as untreated fabrics using an Instron Tensile Tester (USA) according to ASTM D 76 Standard Specification for Textile Testing Machines to measure the tensile and elongation for untreated and treated fabrics.

2.4.6. Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Analysis (EDX)

The treated fabrics with silver nanoparticles (AgNPs) encapsulated with mint oil solutions were investigated under (SEM) by (type QUANTA FEG 250, HOLLANDE) as well as untreated fabrics. In addition, EDX was utilized to display the presence of new elements in treated fabrics compared to untreated fabrics.

2.4.7. Transmission Electron Microscopy (TEM)

The shape and size of the prepared synthesis of silver nanoparticles (AgNPs) using different oil solutions were practically determined using TEM; JEOL-JEM-1200-Japan. Samples for TEM measurements were intended by placing a drop of colloidal solution on 400 mesh copper grids coated by an amorphous carbon film and evaporating the solvent in the air at room temperature.

2.4.8. Thermogravimetric Analysis (TGA)

TGA analysis was carried out using thermal analyzer 7 series (Perkin Elmer, USA) with an attached TG unit. A movable thermostatically controlled electric furnace was used to heat the sample at the desired rates. The sample was heated under a normal atmosphere at a rate of 10^{o} C/ min, and the loss in the weight of the samples was recorded against temperatures from 30^{o} to 500 ^{o} C.

3. Results and Dissection

3.1. Antimicrobial activity for the treated fabric

The antibacterial activity was quantitatively examined using the AATCC 100-2012 (bacterial reduction method) as shown in Table 1. The results showed that the activity of cotton, wool, and polyester fabrics against S aureus (G^{+}) and E. coli (G^{-}) is better when treated with silver nanoparticles composite in the presence of mint, better than lemon and better than basil, respectively than untreated. Due to the presence of active phenol groups in silver nanoparticles/ oil composite that bound to fabrics via chitosan. On the other hand, the strong smell of mint, its effectiveness, and stability compared to lemon oil, which follows it, then basil, which leads to creating a space that keeps microbes away from attacking fabrics. It has been demonstrated that silver nanoparticles exhibit antibacterial action when combined with conventional antibiotics. It is most frequently used for antibacterial action and can be used to treat diseases like skin ulcers [30]. It can stop germs from developing on or adhering to the surface. These days, bandages and patches containing silver nanoparticles are utilized to aid in the healing of some burns and wounds [31]. Thus, silver nanoparticles are incorporated onto textile surfaces utilizing a variety of oils in the presence of chitosan, a potent binding polymer, and are used in medical textiles like children's clothes and bed sheets.

3.2. Color Characteristics

Color strength is pointing to K/S for the untreated and treated fabrics with silver nanoparticles/composite in the presence and absence of oils is illustrated in Table 2. The results indicate that printed-treated fabrics with silver nanoparticles/ basil oil composite enhance the color strength of the untreated one. This may be due to the presence of -COOH and OH groups. Also, the washing fastness of printing on cotton, wool, and polyester fabrics pretreated with silver nanoparticles and chitosan in the presence and absence of oils solution was evaluated using the grayscale according to AATCC Test Method 61 as shown in Table 2 with fastness rating 3-5 on the grayscale, respectively.

Table 1: Antimicrobial activity for the untreated and treated fabrics

Samples	**Bacterial Reduction %**			
	Escherichia coli (G−) (E. Coli)	Staphylococcus saureus(G+) (S. aureus)	Escherichia coli (G−) (E. Coli)	Staphylococcus saureus(G+) (S. aureus)
	Before washing		After washing	
Untreated cotton fabric	0	0	0	0
Treated cotton fabric with silver nanoparticles/ oil of basil and chitosan	80.28	78.33	78.12	77.14
Treated cotton fabric with silver nanoparticles/ oil of Lemon and chitosan	85.27	82.78	81.35	80.75
Treated cotton fabric with silver nanoparticles/ oil of mint and chitosan	93.25	91.33	88.90	88.90
Untreated wool fabric	0	0	0	0
Treated wool fabric with silver nanoparticles/ oil of basil and chitosan	82.31	80.14	81.00	79.14
Treated wool fabric with silver nanoparticles/ oil of Lemon and chitosan	87.13	83.57	85.10	80.50
Treated wool fabric with silver nanoparticles/ oil of mint and chitosan	95.82	93.21	90.11	90.00
Untreated polyester fabric	0	0	0	0
Treated polyester fabric with silver nanoparticles/ oil of basil and chitosan	83.22	80.9	80.10	78.30
Treated polyester fabric with silver nanoparticles/ oil of lemon and chitosan	89.42	88.46	87.00	80.50
Treated polyester fabric with silver nanoparticles/ oil of mint and chitosan	99.37	96.8	91.13	90.00

The results show that the washing fastness of the pretreated printed fabrics is better than untreated fabrics. This result is consistent and confirms the ability of oils that contain phenolics and flavonoids to increase the color value, especially in the presence of chitosan as a support crosslinker agent. Color measurements of treated textiles were investigated according to the CIE (L*, a*, b*) system to esteem the color coordinates. In this method, L* indicates lightness/darkness values from 100 to 0 appearing white to black, a* values run from negative (green) to positive (red), and b* values run from negative (blue) to positive (yellow). It is observed that the fastness properties of fabrics against washing are not influenced.

3.3. This indicates that oil composite is fixed to the fabrics by permanent. Evaluation of UPF Protection

UVR (ultraviolet radiation) can have both short-term and long-term effects; including premature aging of the skin. So, wearing appropriate clothing is the most often advised method of UV protection. In a similar vein, it was shown that the UV-protective properties were highly influenced by the natural dyes' UV radiation absorption properties [32]. This is what was confirmed by the results, which the improvement of the X-ray results is for treated and printed fabrics over untreated fabrics, especially treatment in the presence of basil oil, which gave the best results. This is may be due to the stronger color of basil than others and because it contains many active groups, such as its counterparts in mint and lemon, respectively**.** This also leads to its stronger attachment to fibres, especially wool and then cotton than polyester. This is due to the active groups in wool, such as many amino acids, compared to those of other fibres **.** These results are consistent with the results of color strength, as the color intensity increased when treated in the presence of basil oil compared to untreated and printed fibres only in general, and the results were also better for treated and printed wool than other fibres in particular as shown in Table 3.

3.4. Tensile strength and elongation

The results of mechanical properties, as shown in Table 4 for untreated fabrics cotton, wool, and polyester and treated fabrics using silver nanoparticles/ composite in the presence or absence of oils, indicate that the treatment causes the decrease of tensile strength and the elongation increase. This might be explained by the existence of oils and silver components, which are crucial to this transformation.

3.5. Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Analysis (EDX)

Previous results indicated that the best results were for treated cotton, wool, and polyester fabrics using silver nanoparticles/ mint oil composite so it was confirmed by examining under (SEM) and (EDX). The results of (SEM) explained that nanoparticle homogeneous distribution on the surface for the treated fabrics than the untreated one which is shown in Figure 4. The results of (EDX) analysis for untreated and treated fabrics using silver nanoparticles/ mint oil composite are shown in Figure 5, it is investigated that the presence of Ag in treated samples as compared to untreated samples suggests that materials with positive charges have potent antibacterial activities, which cause bacterial surfaces to shift charge and, ultimately, cause bacterial cells to autolyze. This is consistent with anti-microbial outcomes.

Table 2: The difference between k/s and washing fastness for untreated and treated fabrics

Sample	K/S	L*	a*	b*	Washing fastness	
					St.	Alt.
Untreated cotton fabric	0.02	92.55	-0.22	1.28	-	-
Printed untreated cotton fabric	12.06	34.01	47.90	20.64	3-4	3-4
Printed treated cotton fabric with silver nanoparticles and chitosan	16.06	32.46	44.32	18.77	4-5	4-5
Printed treated cotton fabric with silver nanoparticles/ oil of lemon and chitosan	18.67	34.45	48.56	19.84	4-5	4-5
Printed treated cotton fabric with silver nanoparticles/ oil of mint and chitosan	19.71	35.16	48.31	19.18	4-5	4-5
Printed treated cotton fabric with silver nanoparticles/ oil of basil and chitosan	21.0	34.46	48.79	78.35	4-5	4-5
Untreated wool fabric	0.10	85.19	-0.44	11.90	-	-
Printed untreated wool fabric	13.68	36.15	35.78	11.50	3-4	3-4
Printed treated wool fabric with silver nanoparticles and chitosan	12.54	30.35	43.98	18.29	4-5	4-5
Printed treated wool fabric with silver nanoparticles/ oil of lemon and chitosan	15.10	34.42	40.53	15.50	4-5	4-5
Printed treated wool fabric with silver nanoparticles/ oil of mint and chitosan	20.11	34.30	40.93	15.02	4-5	4-5
Printed treated wool fabric with silver nanoparticles/ oil of basil and chitosan	25.32	33.16	42.83	17.22	4-5	4-5
Untreated polyester fabric	0.05	88.93	-0.13	0.63	-	-
Printing untreated polyester	16.11	34.51	46.94	18.81	3-4	3-4
Printed treated polyester fabric with silver nanoparticles and chitosan	16.04	33.84	46.90	20.09	4-5	4-5
Printed treated polyester fabric with silver nanoparticles/ oil of lemon and chitosan	16.82	34.51	48.01	19.75	4-5	4-5
Printed treated polyester fabric with silver nanoparticles/ oil of mint and chitosan	17.03	34.28	47.47	20.65	4-5	4-5
Printed treated polyester fabric with silver nanoparticles/ oil of basil and chitosan	17.07	34.24	46.83	20.57	4-5	4-5

Table 3: The difference between UPF for treated and treated fabrics

Samples	UPF
Untreated cotton fabric	5.3
Treated cotton fabric with silver nanoparticles and chitosan	7.2
Treated cotton fabric with silver nanoparticles/ oil of lemon and chitosan	7.7
Treated cotton fabric with silver nanoparticles/ oil of mint and chitosan	8.4
Treated cotton fabric with silver nanoparticles/ oil of basil and chitosan	10.4
Printed untreated cotton fabric	33.4
Printed treated cotton fabric with silver nanoparticles and chitosan	36.9
Printed treated cotton fabric with silver nanoparticles/ oil of lemon and chitosan	38.8
Printed treated cotton fabric with silver nanoparticles/ oil of mint and chitosan	40.2
Printed treated cotton fabric with silver nanoparticles/ oil of basil and chitosan	45.6
Untreated wool fabric	55.8
Treated wool fabric with silver nanoparticles and chitosan	60.0
Treated wool fabric with silver nanoparticles/ oil of lemon and chitosan	66.2
Treated wool fabric with silver nanoparticles/ oil of mint and chitosan	72.5
Treated wool fabric with silver nanoparticles/ oil of basil and chitosan	78.8
Printed untreated wool fabric	14.2
Printed treated wool fabric with silver nanoparticles and chitosan	90.0
Printed treated wool fabric with silver nanoparticles/oil of lemon and chitosan	94.5
Printed treated wool fabric with silver nanoparticles/ oil of mint and chitosan	98.9
Printed treated wool fabric with silver nanoparticles/ oil of basil and chitosan	101.5
Untreated polyester fabric	2.9
Treated polyester fabric with silver nanoparticles and chitosan	4.2
Treated polyester fabric with silver nanoparticles/ oil of lemon and chitosan	8.3
Treated polyester fabric with silver nanoparticles/ oil of mint and chitosan	9.1
Treated polyester fabric with silver nanoparticles/ oil of basil and chitosan	9.4
Printed untreated polyester fabric and printed	16.1
Printed treated polyester fabric with silver nanoparticles and chitosan	26.4
Printed treated polyester fabric with silver nanoparticles/ oil of lemon and chitosan	30.1
Printed treated polyester fabric with silver nanoparticles/ oil of mint and chitosan	32.4
Printed treated polyester fabric with silver nanoparticles/ oil of basil and chitosan	36.8

Table 4: Tensile strength and elongation of untreated and treated fabrics

Samples	Tensile strength (kg f/mm2)	Elongation %
Untreated cotton fabric	31	30
Printed untreated cotton fabric	30	20
Printed treated cotton fabric with silver nanoparticles and chitosan	38	22
Printed treated cotton fabric with silver nanoparticles/ oil of basil and chitosan	31	20
Printed treated cotton fabric with silver nanoparticles/ oil of mint and chitosan	29	20
Printed treated cotton fabric with silver nanoparticles/ oil of lemon and chitosan	27	24
Untreated wool fabric	30	20
Printed untreated wool fabric	35	25
Printed treated wool fabric with silver nanoparticles and chitosan	40	30
Printed treated wool fabric with silver nanoparticles/ oil of basil and chitosan	35	40
Printed treated wool fabric with silver nanoparticles/ oil of mint and chitosan	40	46
Printed treated wool fabric with silver nanoparticles/ oil of lemon and chitosan	45	32
Untreated polyester fabric	9	30
Printing untreated polyester	11	23
Printed treated polyester fabric with silver nanoparticles and chitosan	24	40
Printed treated polyester fabric with silver nanoparticles/ oil of basil and chitosan	24	45
Printed treated polyester fabric with silver nanoparticles/ oil of mint and chitosan	23	40
Printed treated polyester fabric with silver nanoparticles/ oil of lemon and chitosan	23	80

3.6. Transmission electron microscopy (TEM)

TEM of the prepared synthesis of silver nanoparticles without mint oil composite (m) and the prepared synthesis of silver nanoparticles /mint oil composite (n) as shown in Figure 6. The results indicated that a range between 11.10 to 16.66 nm and between 19.10 to 27.70 nm was determined for each respectively.

3.7. Thermogravimetric Analysis (TGA)

TGA shows the relationship between mass loss and the temperature change, allowing the study of the thermal stability of the Ag-loaded cotton samples, onset degradation temperature (Tei) and Char Residue has been obtained. Derivative Thermogravimetry (DTG) is the first derivative curve of TGA, which shows the relationship between the rate of mass change and temperature change.

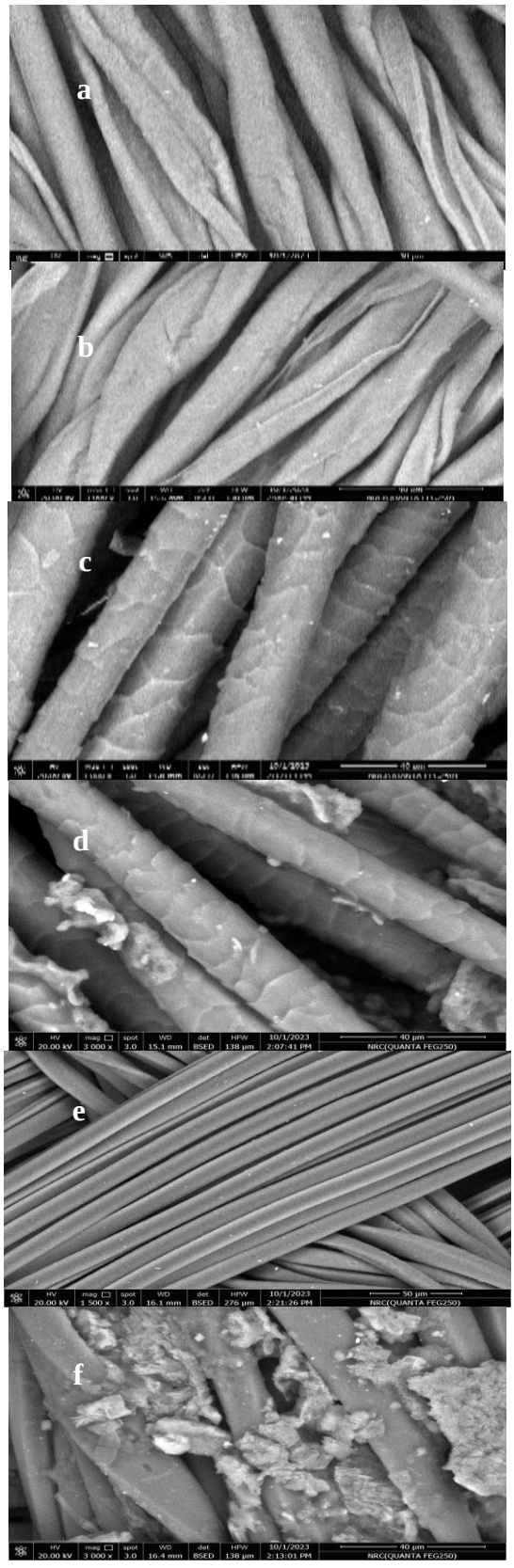


Figure 4: (SEM)for treated fabrics using silver nanoparticles/ mint oil composite a) untreated cotton fabric, b) treated cotton fabric, c) untreated wool fabric, d) treated wool fabric, e) untreated polyester fabric, f) treated polyester fabric,

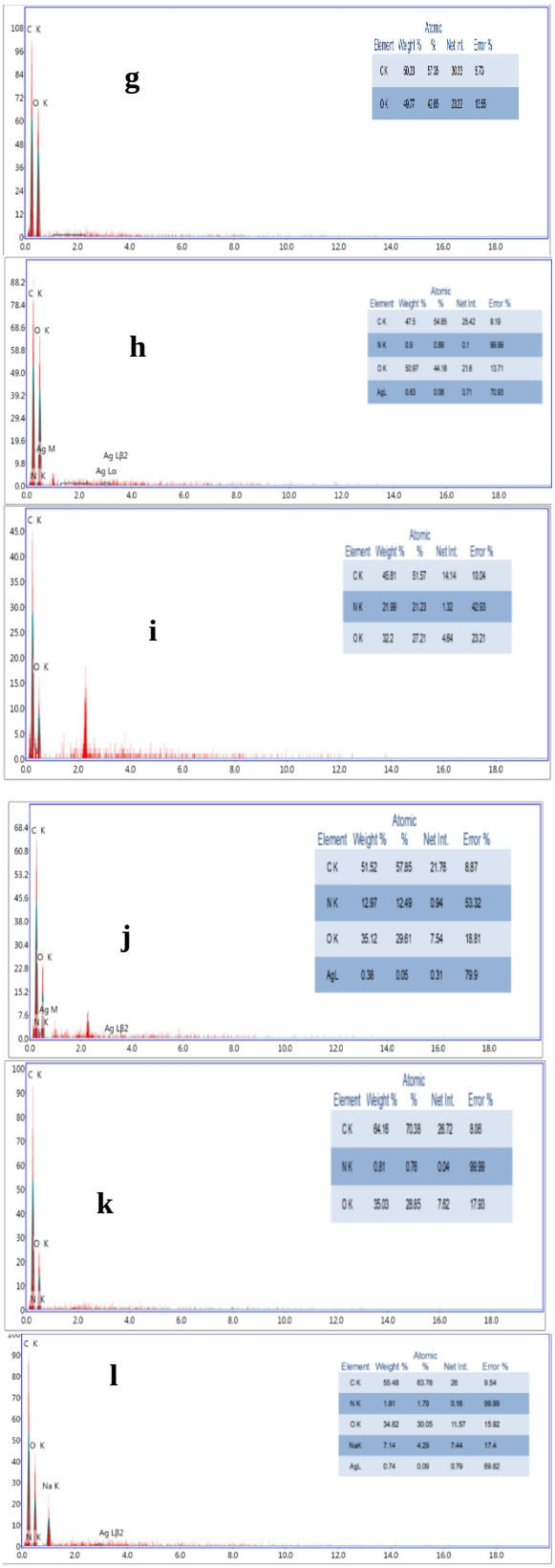


Figure 5: EDX) analysis for untreated and treated fabrics using silver nanoparticles/ mint oil composite g) untreated cotton fabric, h) treated cotton fabric, i) untreated wool fabric, j) treated wool fabric, k) untreated polyester fabric, l) treated polyester fabric

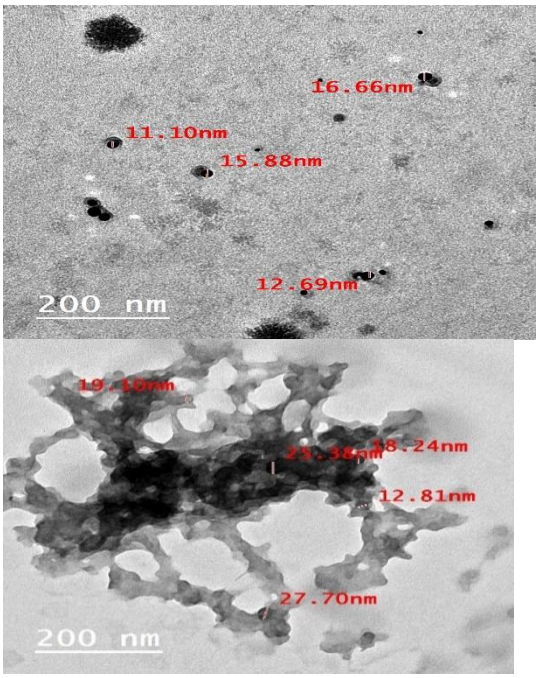
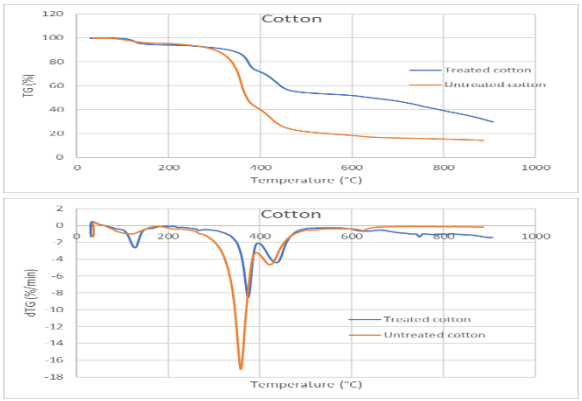


Figure 6: TEM of the prepared synthesis of silver nanoparticles without mint oil composite (m) and The prepared synthesis of silver nanoparticles /mint oil composite (n)

The weight-loss temperature is very close and not obvious in the TGA curve; however, it is apparent, and the maximum decomposition temperature can be obtained by DTG. The effect of treatment on the thermal properties of cotton fabrics was analyzed by TGA. Figures 7 and 8 show the degradation profiles of the cotton fabrics. It is quite clear that TGA curves indicate a slower initial degradation due to the evaporation of water from fabrics, and then a rapid degradation process, attributed to serious degradation of the main molecular chains in the range of 300–400_C.

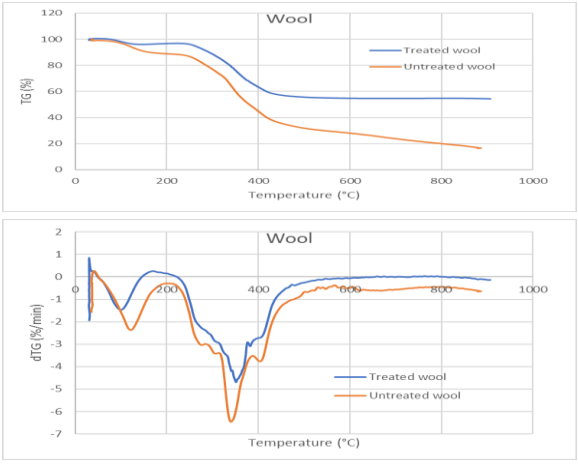
to the intersection of tangents drawn at the temperature point of the maximum weight loss rate with the extended baseline of the TGA curve. The maximum decomposition temperature (Tmax) corresponds to the temperature peak in the DTG curve. Tei and Tmax of the pretreated cotton fabric were lower than those of the untreated sample and Ag-loaded cotton fabric because the intermolecular forces are weakened by the reaction of potential reducing end aldehyde groups in cellulose with NO3 in solution. Meanwhile, there was an obvious peak in the DTG black curve at about 200_C which was attributed to the decomposition of Ag2O on cotton fabric. The TG and DTG parameters of the Ag-loaded cotton fabric were higher than those of the untreated due to the existence of AgNPs on the cotton fabric. The size of the Ag on the surface of cotton fabric was nanometer, resulting in a large specific surface area and high surface energy, which can block the degradation of the fabric to a certain extent and improve the maximum decomposition temperature. Therefore, it can be concluded that AgNPs will improve the thermal properties of cotton fabrics.



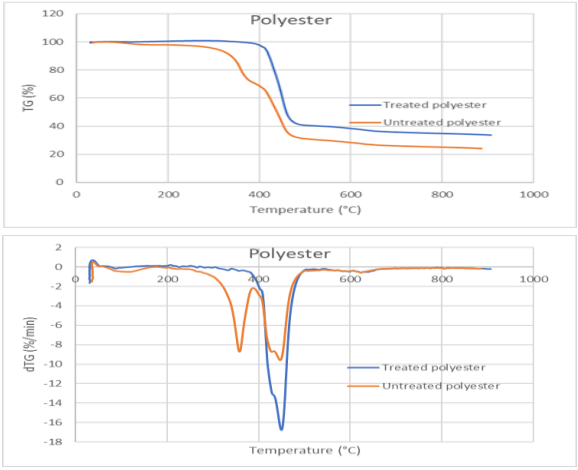
Figures 7 and 8 show the degradation profiles of the cotton fabrics

On the other hand, the thermal properties of the treated wool fibres are very important as these properties have scientific and technological significance. The thermal analysis permits the detection of changes in physical (melting point, crystallisation temperature, the degree of crystallinity, etc.) and chemical (crosslinking, degradation, and other reactions) structure brought about by the interactions between the Ag nanoparticles and wool fabrics, the thermogravimetric analysis and DTG curves of untreated wool fabric and also Ag nanoparticles loaded wool fabrics as shown in figures 9,10. The Ag-loaded wool fabric samples showed slightly better thermal stability than the untreated wool fabric. It can be seen that the weight loss occurs at several stages for both untreated and Ag-loaded wool fabrics, from room temperature to 108 °C, 229 to 437 °C, and 437 to 600 °C. For the untreated wool fabric, in the first stage, approximately 10.5% of the weight is lost, and this weight loss is mainly due to the loss of moisture absorbed by wool fibres. Subsequently, up to 229 °C, the weight loss is quite stable and only 1.8% of weight is lost and some volatiles are removed at this stage. The highest weight loss occurred at the third stage from 229 to 437 °C and approximately 57.5% weight loss was lost at this stage due to the formation of various volatiles due to the thermal degradation of wool fabrics. In the last stage, another 6.5% of the weight is lost up to 600 °C, and char yield is 23.7%. On the other hand, in the case of the treated wool fabric, in the first stage, 7.6% weight is lost compared to the 10.5% weight loss observed for the control wool fabric indicating that the treated wool will reduce its moisture content. In the second stage up to 229 °C,

only 1.1% of the weight was lost. In the third stage, up to another 57% weight was lost. In the final stage, another 8.5% weight was lost producing a char yield of 27.7%, much higher than the char yield produced by the control fabric at that temperature. The results indicate that the Ag nanoparticles loaded onto wool fabric reduced the fabric's thermal degradation increasing the char yield. The DTG curve shows that the peak degradation temperature for the untreated and treated wool is almost the same but the temperature for the 10% weight loss of the control and the Ag-loaded fabric is quite different, 98.0 and 238.1 °C respectively. The corresponding figure for the 50% weight loss is 340.7 and 345.8 °C respectively. Therefore, it can be concluded that the Ag nanoparticles loaded onto wool affected its thermal stability.



Figures 9 and 10 show the degradation profiles of the wool fabrics



Figures11 and 12 show the degradation profiles of the wool fabrics

According to Figures 11,12, three stages of degradation were seen in polyester fabrics. The first occurred between ambient temperature and 360 ◦C alongside a mass reduction of around 5% caused by the elimination of absorbed moisture. With an average mass loss of 70%, the second stage of polymer degradation occurred between 360 and 417 ◦C. The third stage occurred between 490 and 600 ◦C. The thermal stability variation between specimens found in the second stage up to 417 ◦C shows that stability rose as follows: The third stage, which occurs between 490 and 600 ◦C, exhibited rapid deterioration for the untreated than the ag nanoparticles loaded polyester fabrics

4. Conclusion

The treatment of the cotton, wool, and polyester fabrics using silver nanoparticles/ oils composite gives a progress of surface functionalization to the fabrics. It was investigated that this treatment increases the percent reduction of bacteria for treated and treated printed cotton, wool, and polyester fabrics, especially in the presence of the mint oil against S. aureus (G+) and E. coli (G-) as well as a durable antibacterial activity after 5 washing cycles. The results reveal that the treated and treated printed fabric nanoparticles have enhanced the UPF protection as well as the color strength. Also, the washing fastness for the printed samples of fabrics is good to excellent with ratings 3–4 and 5. Moreover, the treatment causes the decrease of tensile strength and the elongation increase**.** Also (SEM) explained that nanoparticle homogeneous distribution on the surface for the treated fabrics than the untreated one. Moreover, EdX measurement indicates the presence of Ag for treated samples as compared to untreated samples. This leads to an increase in the antimicrobial effect for treated fabrics than the untreated ones.

5. Conflict of interest

The authors declare that there is no conflict of interest.

6. Acknowledgments

This paper was supported by an internal project at National Research Centre Egypt entitled: " Optimal utilization and recycling of textile materials waste resulting from research and service activities" Code number: 13010201(2022 -2023).

The authors are gratefully thankful to acknowledge the Central Labs Services (CLS) and Centre of Excellence for Innovative Textiles Technology (CEITT) in Textile Research and Technology Institute (TRTI), National Research Centre (NRC) for the facilities provided.

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