



Chemical synthesis of silver nanoparticles in its solid state: highly efficient antimicrobial cotton fabrics for wound healing properties

Mehrez E. El-Naggar ^{a*}, Hadeer A. Emam ^b, Maha I. Fatthalla ^b, Mohamed S. Abdel-Aziz ^c,
and Magdy K. Zahran ^b

^a Textile Research Division, National Research Centre, 33 El Behouth st.-Dokki, Giza, Egypt

^b Department of Chemistry, Faculty of Science, Helwan University, Egypt

^c Genetic Engineering and Biotechnology Division, National Research Centre, 33 El Behouth st.-Dokki, Giza, Egypt



CrossMark

Abstract

Nanoparticles have been used as effective platforms for treating skin wounds over the last decade. Potential therapeutic effects on wound healing have been demonstrated through metal nanoparticles, notably silver nanoparticles (AgNPs). Solid state synthesis was used in this work for the preparation of AgNPs without using any external chemicals or solvents. In this current work, dextran was used as reducing agent for silver ions and in the same time as stabilizer for the produced nanoparticles. AgNPs is readily synthesized by eco-grinding artlessly for dextran powder, sodium hydroxide beads. The efficacy of dextran as a dual function for the synthesis of AgNPs was assessed by adjusting the utilized concentration of silver nitrate. Ultra-violet visible spectroscopy (Uv-vis), transmission electron microscopy (TEM), dynamic light scattering (DLS), zeta potential, X-ray diffraction (XRD), scanning electron microscope -Energy dispersive X-ray (SEM-EDX) are tools for AgNPs characterization before and after cotton fabric treatments. The resulting AgNPs with different concentrations were added to cotton fabrics. The cytotoxicity and antimicrobial activities of the treated fabrics were examined. The findings showed that the spherical shape was confirmed with a reasonable distribution of prepared AgNPs. In addition, the stability of AgNPs has been attained and values greater than -30 mv have been obtained. The findings also revealed that the nanoparticles were effectively distributed onto the surface of fabrics and penetrated into them. The cotton fabrics treated possess superior antimicrobial properties, which are determined by the process of disc diffusion to suppress pathogenic microbes. Furthermore, for human and non-human cell lines, the cell viability of the treated cotton fabrics has no toxicity effect. On that framework, the synthesis of AgNPs with high yield is based on solvent-free is also offered by one pot synthesis of silver nanoparticles that is eco-friendly, low cost, save effort and chemicals.

Keywords: Silver nanoparticles; dextran; solid state synthesis; antimicrobial properties; cytotoxicity

1. Introduction

Recently antimicrobial fabrics gained a great attention for their potentiality to eliminate the transmission of pathogenic microbes in both medical and health care environments [1]. The antimicrobial fabrics could also reduce the unpleasant odor. Antimicrobial fabrics were used for long time in health care as hospital gowns, paint cloths, bed covers, etc. [2]. The antimicrobial fabrics should achieve the following requirement to be applicable to injured skin as wound dressing [3,4]. These requirements include the fabrics should be nontoxic, not make bacterial resistance, should not cause allergic reaction, have broad spectrum antimicrobial and should be sustainable [5–7]. Metal nanoparticles (NPs) is considered as good alternatives to some organic compounds which have been used in textile

finishing as antimicrobial agents. Nanoparticles include nanometals (e.g. silver and copper), nano oxides (e.g. titanium and zinc), carbon nanotubes and clays [6]. Antimicrobial dressings of wound include two groups; namely antiseptic or antibiotic dressings [8]. Antiseptic dressings exhibit broad spectrum activity that can kill or inhibit many pathogens such as bacteria, fungus, protozoa, viruses, and prions [9,10]. On the other hand, antibiotic dressings are nontoxic and effectively work on the target sites without damaging host tissues [11]. The perfect antimicrobial dressing must have broad spectrum activity against different microorganisms, be nonallergic and nontoxic to host cells, have be able to groove exudate and preserve a humid wound situation, should release drugs quickly in a continued manner, should decrease unpleasant odor, and be

*Corresponding author e-mail: mehrez_chem@yahoo.com (Mehrez E. El-Naggar).

Receive Date: 11 January 2021, Revise Date: 28 January 2021, Accept Date: 31 January 2021

DOI: 10.21608/EJCHEM.2021.57667.3236

©2021 National Information and Documentation Center (NIDOC)

cheap [12,13]. Silver nanoparticles (~20nm) exhibited broad spectrum antimicrobial activities against different human pathogens [14,15]. Antimicrobial fabrics containing silver nanoparticles were fabricated into cotton fabrics as wound dressing textile [16].

Due to the extreme unique and extraordinary electrical, physiochemical, and mechanical properties of Due to the extreme unique and extraordinary electrical, physiochemical, and mechanical properties of materials in nanostructured form, nanotechnology has rendered a major revolution in every aspect of human daily life [17,18]. The justification for enhancing the efficiency of nanostructures is believed to be increasing the active surface areas of the particles. They are also ideal for various biomedical applications, such as smart textiles, wound dressing products, medical devices, etc [19]. Throughout many applications, different metal nanoparticles are being used in several industry sectors, silver nanoparticles are extensively utilized. Silver nanoparticles (AgNPs) are very effective antimicrobial agent toward different microbial organisms. With their outsized surface area to volume ratio, silver nanoparticles exhibit a virtuous antibacterial property, which delivers excellent interaction with the microorganism [20–27].

AgNPs have also been frequently used in different finishing textiles including such gloves, medical coats and laboratory gowns, surgical gowns and dressing bandages, which are alleged to be capable of preventing bacterial growth. Especially in comparison with bulk particles, the synthesis of AgNPs has extraordinary chemical, optical, electrical and magnetic properties [28,29].

There many methods and huge of synthetic and hazardous materials used for the preparation silver nanoparticles (AgNPs). Most of these preparation methods are electrochemical reduction [30], sonochemical method [31,32], laser ablation technique [33–35], biological synthesis [36,37], microwave irradiation reduction [38,39], chemical reduction [40–42] and so on. It is worth noting that most of these methods are not applicable for the preparation of nanoparticles in large scale. In addition, most of the literature compounds that used for nanoparticles preparation is mainly relied on synthetic polymers that have side effect in the environmental pollution. Of these chemical synthetic polymers are sodium borohydride, hydrazine hydrate, dimethyl formamide, sodium citrate, ethanol, methanol and dimethyl sulphoxide. In our research work, an outstanding issue about the mass production of nanoparticles is environmental protection. Green approaches showed promising routes [43], such as the solvent-free process [44–49]. As in solvent-free technology, potential benefits such as accessibility, high performance and insignificant delivery of harmful waste are achieved. In order to synthesize

silver nanoparticles under ambient conditions, an innovative synthetic method has been developed. Solid state synthesis of nano particles is therefore regarded for the green approach to the development of materials with the necessary functionality. No need to utilize additional reducing agent or stabilizing agent throughout this methodology and the reaction was carried out without the need for using an organic or an aqueous solvent. AgNPs were prepared in solid-state synthesis by mixing and grinding the precursors for all the materials used; polymer, NaOH and Ag ions. The present research work was therefore intended to take the advantage of a novel solid-state green method without using any solvent to prepare silver nanoparticles (AgNPs). The outstanding green strategy for this is to obtain nanoparticles with significant properties and control the size and shape of AgNPs prepared by such method [50].

Thus, the current study documented the development of a technique that is emphasising about the environmentally sustainable one-pot green synthesis of relatively efficient dry powdered silver nanoparticles (AgNPs) utilizing dextran as both a reducing and stabilising agent in the presence of sodium hydroxide (NaOH). Sodium hydroxide is thought to be able to enhance the reduction potential of dextran. Therefore, when silver nitrate (AgNO_3) is added to the alkali-treated dextran, the alkali-treated dextran gives the well-established dual function of dextran; reduction of silver ions (Ag^+) to AgNPs and stabilising the as-formed AgNPs to prevent their aggregation and agglomeration. The assessment of AgNPs formation was carried out by examine the structural and morphological characteristics of AgNPs are investigated by structural and morphological features by UV-vis spectroscopy, transmission electron microscopy (TEM), particle size analyzer, zeta potential, and X-ray diffraction (XRD) analysis. Eventually, Cytotoxicity in terms of human and non-human cell lines and antimicrobial properties of treated cotton fabric were measured against four types of microbes; *S. aureus*, *E. coli*, *C. albicans* and *A. niger*.

2. Materials and methods

2.1. Materials

Silver nitrate was purchased from Sigma Aldrich Co (Germany). Dextran and sodium hydroxide were purchased from Across Co (Germany). Deionized water was used for dilution, analysis and application. All other chemicals are of analytical grade and used as received without further purification or modification.

2.2. Methods

2.2.1. Preparation of solid-state silver nanoparticles (Ag-NPs)

Silver nanoparticles (Ag-NPs) was prepared in its solid-state using dextran as both reducing agent

and in the same time as stabilizing agent at ambient temperature and without using any extra reductant or organic solvents. Firstly, 1 g of dextran was mixed with sodium hydroxide pellet (0.35 g/1 g dextran) and submitted for mechanical grinding for 10 min. After that, different concentrations of silver nitrate; AgNO_3 (0.5g, 1g and 2 g) as a precursor for silver ions (Ag^+) were added to the fine powder of dextran/NaOH and mechanical grinding was continued for another 20 min. The three fine powder were subjected for washing with distilled water and centrifuged to obtain pure powders of Ag-NPs stabilized or coated with dextran chains. Finally, the wet fine powders of Ag-NPs were dried using freeze drying instrument. the three different concentrations depending on the concentration of the utilized AgNO_3 are nominated as Ag-NPs-1, Ag-NPs-2 and Ag-NPs-3.

2.2.2. Preparation of Ag-NPs loaded cotton fabrics

0.1 g of each concentration was dissolved in 100 mL of deionized water under the effect of sonication for 15 min. The bleached cotton fabric (10 cm*10 cm) was immersed in each solution for 1 min and squeezed with pick up 100%. The treated cotton fabrics were then dried at 80 C for 2 min and curing at 120 C for 2 min. Through the treatments and curing, the white color of fabrics was changed to yellow and yellowish brown due to the effect of Ag-NPs.

2.3. Characterization of AgNPs and cotton fabrics treated with different concentrations of Ag-NPs

2.3.1. Ultraviolet–visible (UV-vis) spectroscopy
UV-vis spectroscopy was used in order to assess the wavelength and specific absorption for AgNPs by adding 1 ml of the three diluted samples to 20 mL of distilled water and then identified using UV-Vis spectrophotometer (T80 UV/vis spectrometer, PG Instrumentals Ltd. Germany) in the 200-600 nm wavelength range.

2.3.2. Transmission Electron microscope (TEM) for particle shape determination

The shape and distribution of AgNPs has been described through TEM (JEOL, 2100, Japan). Before the examination, the samples were first submitted to sonification. On a copper grid, the samples of the prepared colloidal solution were put and allowed to dry until examination.

2.3.3. Particle size analyzer

Dynamic Light Scattering (DLS, Nano-ZS, Malvern Instruments Ltd., UK) is an unique technique that is commonly used to describe the hydrodynamic diameter of nanoparticle suspensions based on the particles' demonstrated Brownian movements.

2.3.4. Zeta potential evaluation

The zeta potential is a key indicator of the stability of colloidal dispersions. The magnitude of the zeta potential indicates the degree of electrostatic repulsion between adjacent, similarly charged particles in a dispersion. For molecules and particles that are small enough, a high zeta potential will confer stability, i.e., the solution or dispersion will resist aggregation. When the potential is small, attractive forces may exceed this repulsion and the dispersion may break and flocculate. So, colloids with high zeta potential (negative or positive) are electrically stabilized while colloids with low zeta potentials tend to coagulate or flocculate.

2.3.5. Crystallinity evaluation of Ag-NPs by means of X-ray diffraction (XRD)

XRD can be described as a swift analytical technique used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined. All diffraction methods are based on generation of X-rays in an X-ray tube. These X-rays are directed at the sample, and the diffracted rays are collected. A key component of all diffraction is the angle between the incident and diffracted rays. Powder and single crystal diffraction vary in instrumentation beyond this.

2.3.6. Morphological structure of the prepared Ag-NPs treated cotton fabric

For the identification and examination of the surface structure of the treated cotton fabrics, the scanning electron microscope (SEM, TESCAN VEGA 3, Czech Republic)). The energy disperse X-Ray (EDX) was used to identify the elemental analysis of the evaluated sample.

2.3.7. Cytotoxicity of treated cotton fabric with AgNPs

Cytotoxicity was developed in order to investigate the protection of the treated cotton fabrics. The treated cotton fabric samples with different concentrations of AgNPs were then tested against human epithelial type 2 (Hep2) cells, the eternal cell line consisting of human liver carcinoma cells (HepG2) and human cells called Buffalo Green Monkey Kidney Cells (BGM).

These human cell lines were afforded by Holding Company for Biological Products & Vaccines VACSERA, Egypt. In order to evaluate the treated cotton fabrics, the human cells should be contacted to the treated cotton fabrics and then determined via cell viability % using MTT assay method using Tetrazolium assay kit (MTT cell proliferation assay kit, Duchefa Biochemie, Netherlands). The

absorbance was measured at 550 nm using a Varian Cary 300 ultraviolet–visible spectrophotometer. The obtained cytotoxicity results were displayed in percentage as reduction of viability of cells cultured in presence of treated compared to cells cultivated in a medium without the untreated samples. The human cells without the samples were calculated as a reference control with a cell viability of 100%. The cell viability (%) tested by MTT assay method over blank and treated samples after (24 and 48 h).

2.3.8. Antimicrobial properties of Ag-NPs treated cotton fabric

To evaluate the antimicrobial activities of treated fabrics to be used as wound dressing four pathogenic test microbes were used namely; *Staphylococcus aureus* ATCC 6538 (G+ve bacterium), *Escherichia coli* ATCC 25922 (G-ve bacterium), *Candida albicans* ATCC 10231 (yeast) and *Aspergillus niger* NRRL A-326 (fungus) as described by Collens and Lyne's [51]. Petri dishes containing nutrient agar medium were densely inoculated with both bacteria and yeast (CFU of about 10^8) whereas Potato dextrose agar medium plates were inoculated with the fungus (CFU of about 10^7). Each plate was inoculated with 100µl from stock inoculum from each test microbe and spreaded over the plate surface using sterile swabs. The treated fabric discs (1cm diameter) were placed over the surface of inoculated plates. The inoculated plates were reserved in refrigerator at 4oC for about 2-4h. The plates were then incubated at 37oC for 24h for bacteria and at 28oC for 48h for the fungus. The antimicrobial activity was investigated by calculating the inhibition zones around the tested samples expressed in millimeter (mm).

3. Results and discussion

The target of our work was designed to prepare Ag-NPs in the solid state without using any solvents; organic or aqueous. Followed by evaluate its efficiency of the cotton fabrics as antimicrobial finishing agent. In this research work, Ag-NPs was prepared using dextran as both reductant and stabilizing agent. Dextran was selected for its bioavailability, compatibility and cost-effective. The tools used for preparation are very facile and simple. The mechanical grinding of dextran with NaOH facilitate the chains of dextran to be alkaline, more active to reduce the ions of Ag to Ag-NPs through the reductant group in C6 of dextran. in the same time, there numerous hydroxyl groups connected to the backbone of dextran chains which acted as stabilizer for the obtained Ag-NPS and protect it from further agglomeration. The color of dextran is white and changed to yellow brownish with the addition of AgNO₃ while grinding as shown in **Figure 1**. It is observed that, the prepared solid was changed to deep

yellow color while dilution with deionized water and diluted to yellow color with further dilution.

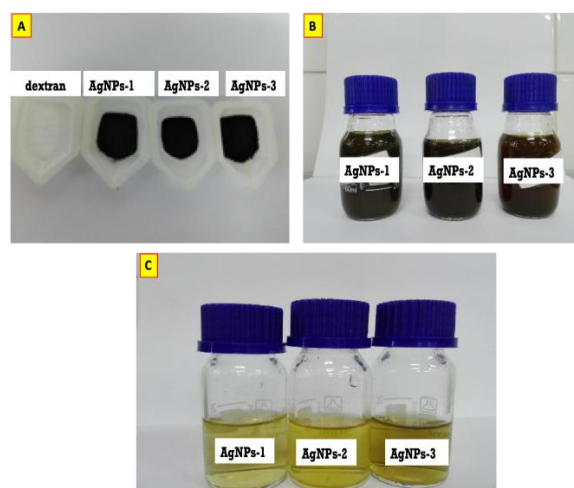


Figure 1: photo images of (A) dextran and AgNPs in the powder form, (B) colloidal solution of AgNPs (high concentration) and (C) colloidal solution of AgNPs (low concentration)

3.1. Characterization of the as-synthesized AgNPs via solid state technique

3.1.1. UV-vis spectra

After eye observation for the color of the formed Ag-NPs, UV-vis was used for further confirmation and to investigate the wavelength in nm for Ag-NPs. It is well known, the absorption band for Ag-NPs are existed between 400-430 nm. **Figure 2** shows that UV-vis absorption spectrum of dextran and dextran coated Ag-NPs of. It was found that dextran has no peak around 400 nm while the UV-vis absorption spectrum of Ag NPs showed that featureless surface Plasmon resonance band around 410 nm confirming that the formation of Ag NPs.

In details, the sample coded with AgNPs-1 which contain in its preparation 0.5 g of AgNO₃ exhibit an absorption peak around 404 nm. The value of absorption peak was shifted to high wavelength (408 nm) while increasing the concentration of AgNO₃ to 1g as presented from the graph of AgNPs-2. On the other hand, the absorption peak was significantly moved to high wavelength (423 nm) with increasing AgNO₃ concentration to 2 g. The absorbance was increased due to increasing the number of particles. It is also depicted that with the second concentration (AgNPs-2), the band is nearly sharp which affirming the uniformity of particle size and thus the particle homogeneity. On contrary, AgNPs-3 that prepared with high concentration of AgNO₃, the band is very broad confirming that the formed particles are heterogeneous.

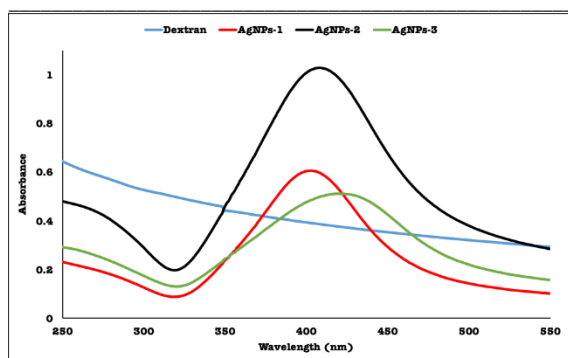


Figure 2: UV-vis of dextran and AgNPs prepared using different concentrations of AgNPs

3.1.2. Determination of particle shape for AgNPs

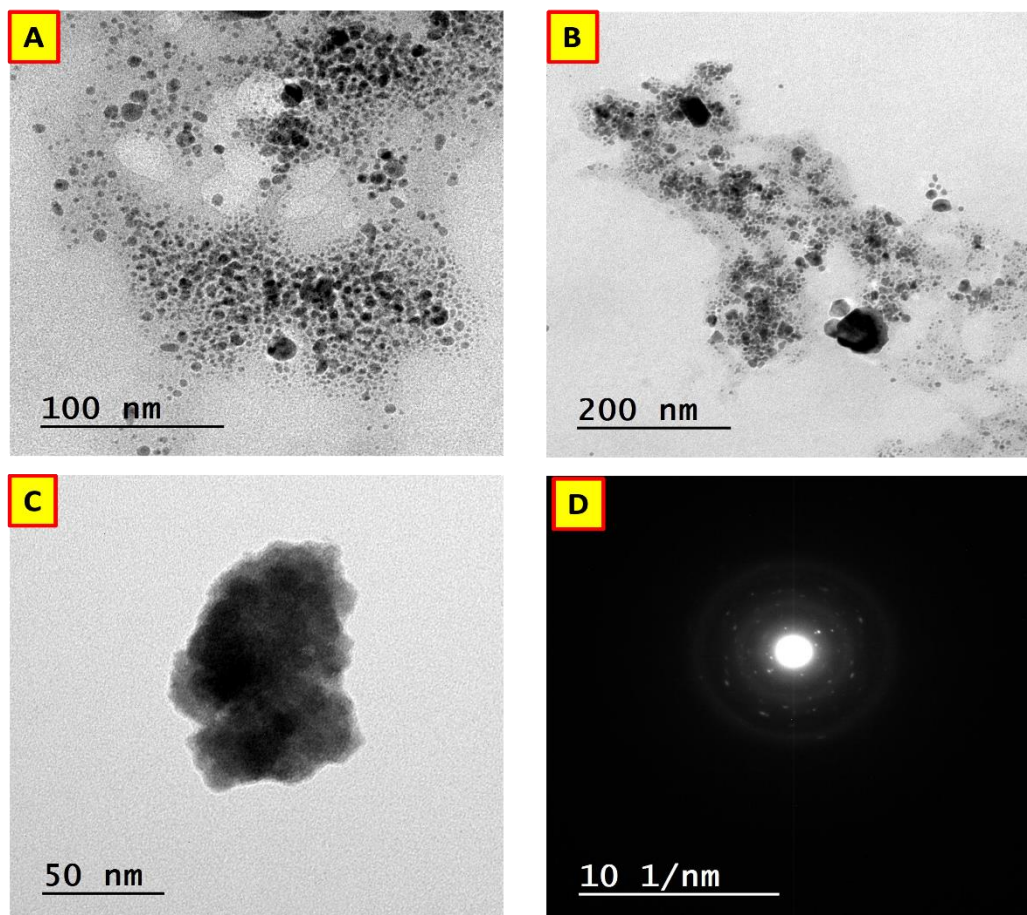


Figure 3: TEM of (A) AgNPs-1, (B) AgNPs-2 and (C) AgNPs-3 and (D) selected area diffraction of AgNPs-2

Based on the aforementioned TEM observation, AgNPs-2 was selected for further analysis to observe the selected area diffraction for the formed particles which indicated that the synthesized silver nanoparticles were formed as single crystals.

3.1.3. Particle size analyzer and zeta potential value for the as synthesized silver nanoparticles

The average hydrodynamic size of the three prepared AgNPs with different concentrations due to the different concentrations of the utilized AgNO₃

For particle shape observation, the powder of AgNPs (0.05 g) prepared with three different concentrations of AgNO₃ (0.5 g, 1 g and 2 g) was dispersed in deionized water to be ready for TEM evaluation and the obtained images are set in **Figure 3**. As shown in Figure 3 (A, B) that the particles shape of AgNPs-1 and AgNPs-2 using 0.5 g and 1 g of AgNO₃ respectively are formed with spherical shape and also with good distribution confirming that 1 g of dextran has the ability to reduce and stabilize the produced AgNPs. On the other hand, AgNPs-3 that prepared with high concentration of AgNO₃ (2 g) / (1 g) dextran has an agglomerated particle with no noticeable edges due to that the availability of functional groups of dextran are low to reduce Ag ions and to protect them from aggregation.

was determined by the means of dynamic light scattering (DLS) and the graphs were drawn in **Figure 4**. It is clearly noted that the average particle size is changed. AgNPs prepared using 0.5 g of AgNO₃ (AgNPs-1) exhibit average particle size equal to 8 nm while the obtained average size (15 nm) is ascribed for AgNPs prepared using 1 g of AgNO₃ (AgNPs-2). On the other hand, the sample coded with AgNPs-3 (prepared using 1 g of AgNO₃) demonstrates that average diameter is 58 nm. The varieties in particle size for the three samples are

attributed to the potential effect of dextran as reductant and stabilizing effect.

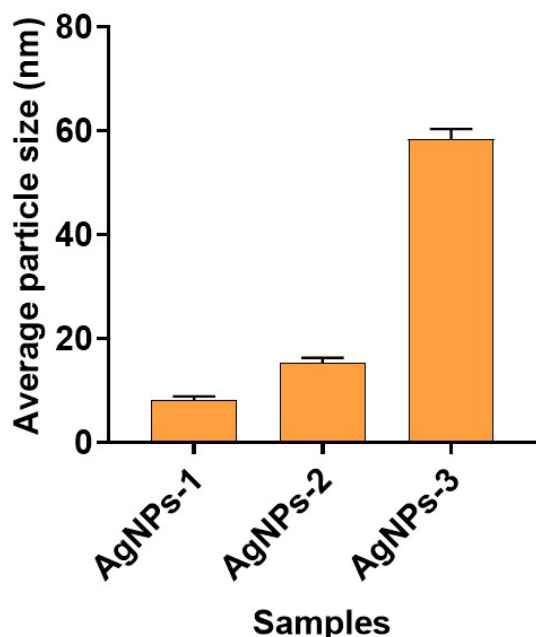


Figure 4: Average particle size using DLS for AgNPs-1, AgNPs-2, and AgNPs-3 prepared using different concentrations of silver nitrate in the presence of dextran as both reductant and stabilizing agent

At low concentration of AgNO_3 , the potential effect of dextran to reduce silver ions and protect the formed nanoparticles is maximized. dextran contains glucose units which is available for reduce all the precursor ions of silver at its low concentration. Through increasing the concentration of AgNO_3 to 1 g (AgNPs-2), the potential effect of dextran as reductant and stabilizing is partially decreased, which, in turn, leads to enlargement the formed particle size. Up on increasing the concentration of AgNO_3 to 2 g (AgNPs-3), the concentration of silver ions is sharply increased comparing to the concentration of dextran and thus, its performance to reduce and stabilize the formed nanoparticles becomes low, conclusively, leads to increasing the diameter of size to 58 nm).

The aforementioned observations and characterization provide us an exception about the zeta potential value for each sample. Figure 5 displays the Zeta potential (ζ -potential) for AgNPs. The powdered AgNPs was suspended in deionized water and sonicated for 15 min before zeta potential determination. It is expected that increasing the diameter of size has a reverse relationship with the stabilization effect. Increasing the particle size is due to the low stabilizing effect of the utilized polymer. In our study, due to the higher concentration of silver ions, the stabilization effect of dextran becomes low. Therefore, it is depicted from figure 4 that the value

of zeta potential of AgNPs-1, AgNPs-2 and AgNPs-3 is -36 mv, -32 and -26 mv respectively.

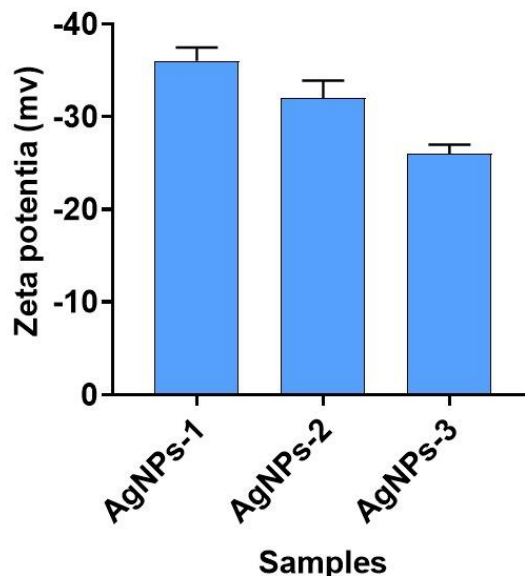


Figure 5: Zeta potential value of AgNPs (AgNPs-1, AgNPs-2, and AgNPs-3) prepared using different concentrations of silver nitrate in the presence of dextran as both reductant and stabilizing agent

The value is decreased with increasing the concentration of the utilized AgNO_3 . It is known that the value of zeta potential above -30mv or +30mv is an indication for the good stability of the formed Ag NPs, which, in turn, related to the stabilization potential of dextran. The stability of AgNPs-2 solution is higher than the other two concentrations. taking in mind that the negative signals are attributed to the negative effect of the hydroxyl groups of glucose units. The negative charge of the zeta potential value is due to the adsorption of the hydroxyl groups (OH) from dextran onto the surface of the particles.

3.1.4. X-ray diffraction (XRD) of Ag NPs

In order to clarify the purity and crystallinity of formed nanoparticles that prepared using different concentrations of AgNO_3 , XRD was carried out to scan and check the structure of the analyze compound. As shown from Figure 6 (A, B) for the samples coded with AgNPs-1 and AgNPs-2, the graph obtained from the XRD has typical pattern observed at 38.2° , 44.2° , 65° , and 77.5° which may be indexed to the (111), (200), (220) and (311) facets of silver, respectively indicating that the synthesis of AgNPs. Sample 2 (AgNPs-2) was selected for elemental analysis using EDX to identify the elements presented in the analyzed sample. As shown from Figure 6 (D) that the sample has Elements; C, O and Ag. The presence of C and O are attributed to dextran that surround the particles of Ag while the peak of Ag is to confirm that the successful

preparation of AgNPs stabilized via dextran molecules.

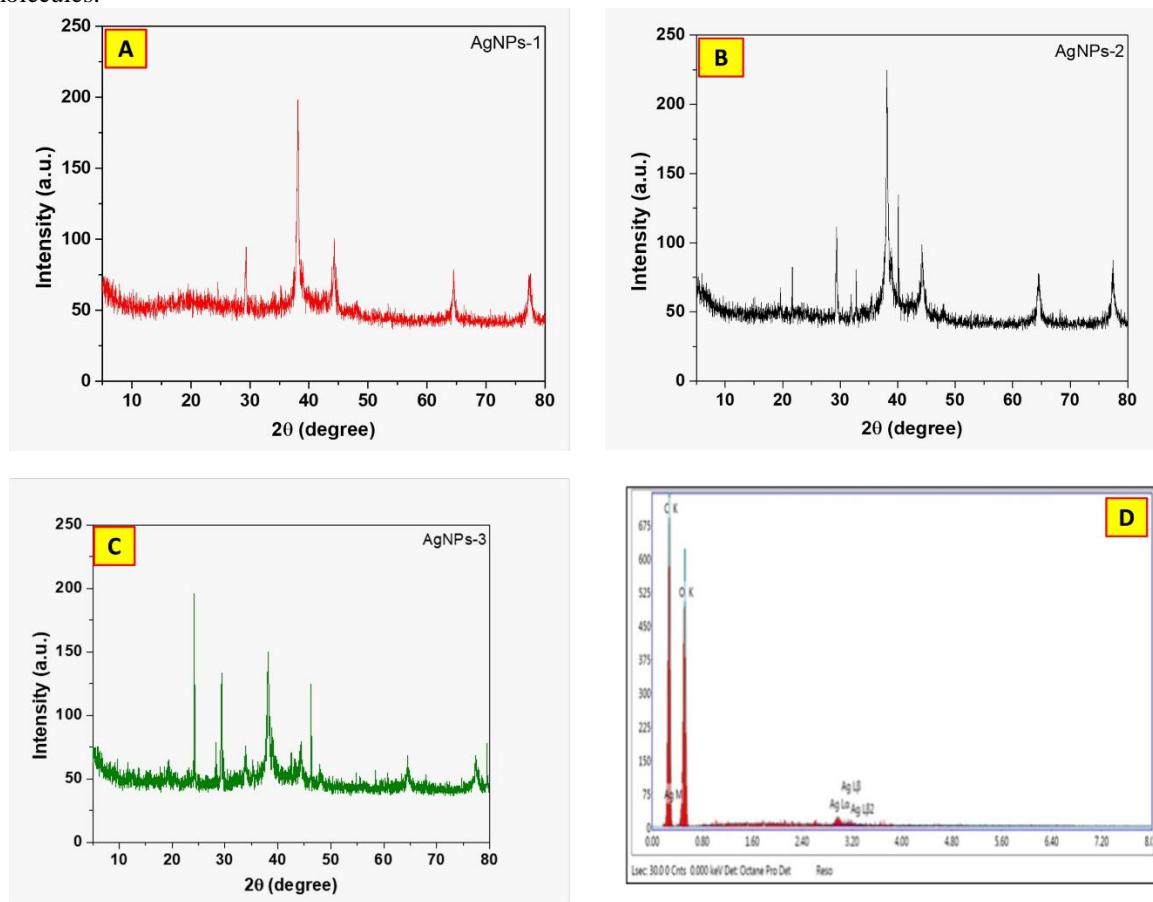


Figure 6: XRD analysis for (A) AgNPs-1, (B) AgNPs-2 and (C) AgNPs-3. (D) EDX of the synthesized AgNPs-2

3.2. Characterization of cotton fabrics treated with AgNPs

Next, the second target is to apply the prepared AgNPs as antimicrobial finishing agent for cotton fabric and evaluated the resultant treated cotton fabric

3.2.1. Scanning electron microscopy (SEM) of cotton fabrics loaded with Ag NPs

After confirming the preparation of AgNPs with full advanced characterization such as UV-vis, TEM, particle size analyzer, zeta potential, XRD and EDX tools, the three prepared different concentrations were utilized in order to impart the antimicrobial properties for cotton fabrics. 0.1 g of AgNPs from the nominated their different concentrations; AgNPs-1, AgNPs-2 and AgNPs-3 was dispersed in distilled water used for treatments. **Figure 7** displays the cotton fabric treated with AgNPs and compared with the untreated cotton fabric. Each picture for all the scanned samples are taken at low and high magnifications.

It was observed that the untreated cotton fabric (**Figure 7A**) exhibits clean and smooth surface and after treatment the surface become rough due to the

precipitation of AgNPs onto the surface of fabric (**Figure 7 B, C and D**). In addition, some of these nanoparticles are penetrated inside the surface due to the small size of nanoparticles. the color of untreated cotton fabric is white and converted to color fabric due to the treatments as shown in the inset images in each figure. It is also depicted that from the figures that the quantity of AgNPs formed on the surface of fabric increases by the increasing of concentration.

3.2.2. Cytotoxicity of treated cotton fabric

The cytotoxicity parameter of any treated fabrics is very important factor to facilitate their application in medical domains such as wound dressing materials [52,53].

Thus, in terms of the cytotoxicity, **Figure 8** represent cell viability percent of untreated cotton fabrics and the treated fabrics with the as prepared AgNPs against non-human and human cells such as Buffalo Green Monkey (BGM), Human Hep2 (HeLa derivative) and Human cervix carcinoma (HepG2). The cell viability percent was measured and calculated at different times (24 h, 48 h, 72 h and 96 h). It is observed that from **Figure 8**, the cell viability% for all tested cells is around 99% with

different times (24 h, 48 h, 72 h and 96 h) for the untreated cotton fabrics. It is also depicted cotton fabric treated with a solution of 0.1 g of AgNPs-1 (CF@AgNPs-1) has no change in the cell viability after 24, 48, 72 h and 96 h of attachment the three human and non-human cells indicating that untreated cotton fabrics and that treated with AgNPs-1 solution did not induce toxicity. It is remarkable that the cell viability percent is more than 92% for all treated cotton fabric with AgNPs-1. The cell viability percent is marginal decreases when the cotton fabric

was treated with 0.1 g of the second solution (AgNPs-2). These values are sharply decrease to a value around 80 % when the fabric was treated with the high content of AgNPs (AgNPs-3). It is worth noting that the cell viability was decreased when the treated fabric was submitted for along time. thus, the values of cell viability at 72 and 96 h are less than the value obtained at 24 and 48 of subjecting the fabric to the tested cell.

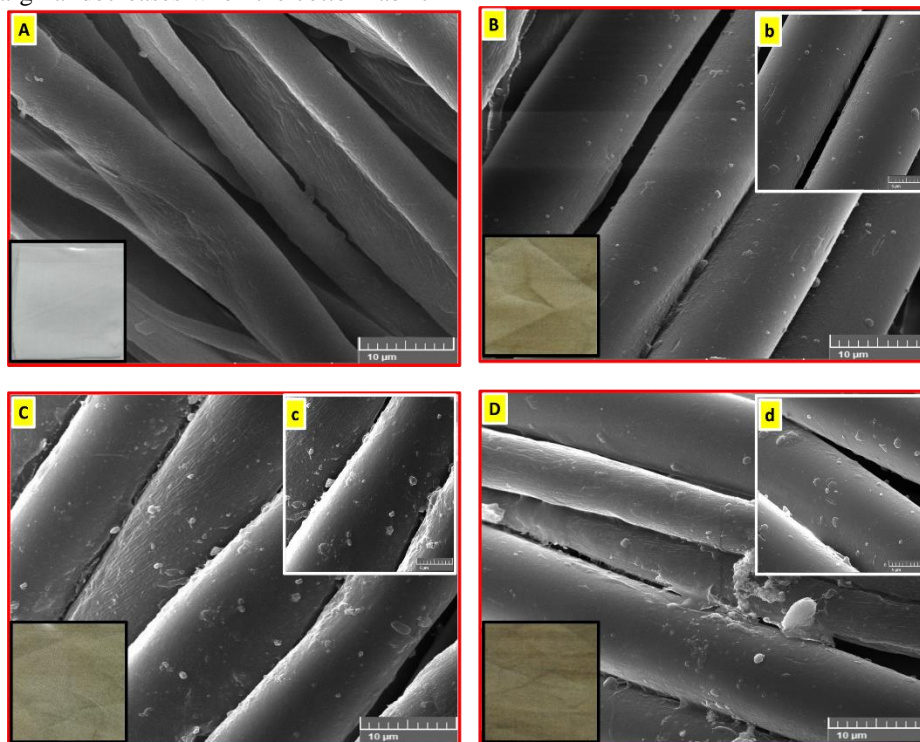


Figure 7: SEM micrographs of (A) untreated cotton fabric and treated cotton fabrics with (B, b) AgNPs-1, (C, c) AgNPs-2 and (D, d) AgNPs-3

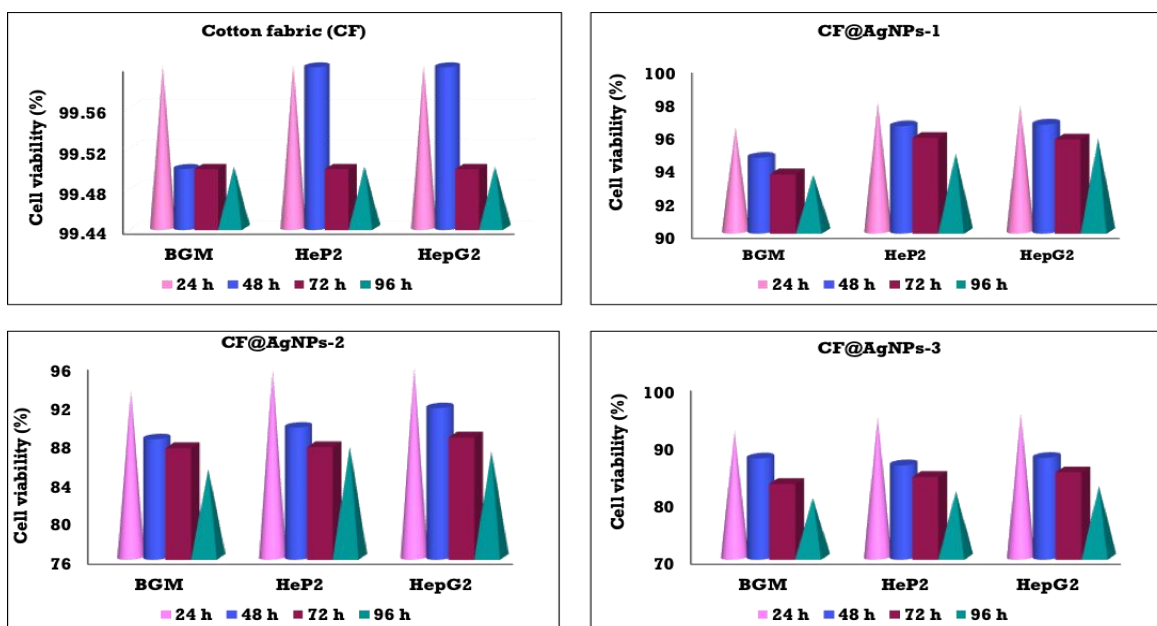


Figure 8: Cytotoxicity effect of treated cotton fabric with AgNPs against BGM, HeP2 and HePG2 cells

Overall, the average cell viability (%) of these samples is above 80%, confirming the low toxicity of these samples even using the high concentrated AgNPs for fabric treatment. It can be concluded from the above data that AgNPs-1 exhibited no cytotoxic effect. Meanwhile, AgNPs-2 and AgNPs-3 demonstrated minor cytotoxicity meanwhile, AgNPs-3 displays slight cytotoxicity even after 96 h of subjection to human and non-human cells affirms that AgNPs at these aforementioned concentrations is safe and non-toxic for human and non-human cells and valid to be used for fabric treatments.

3.2.3. Antibacterial properties of cotton fabric treated with AgNPs

Results in Figure 9 and Table 1 indicated that the untreated Fabric (blank) didn't exhibit any antimicrobial activity against any test microbe. On the other hand, Samples 1, 2 and 3 exhibited gradual increase in antimicrobial activity against all test microbes; *S. aureus* (13, 13 and 14mm, respectively), *E. coli* (13, 14 and 15, respectively), *C. albicans* (12, 13 and 16mm, respectively) and *A. niger* (11, 11 and 16mm, respectively).

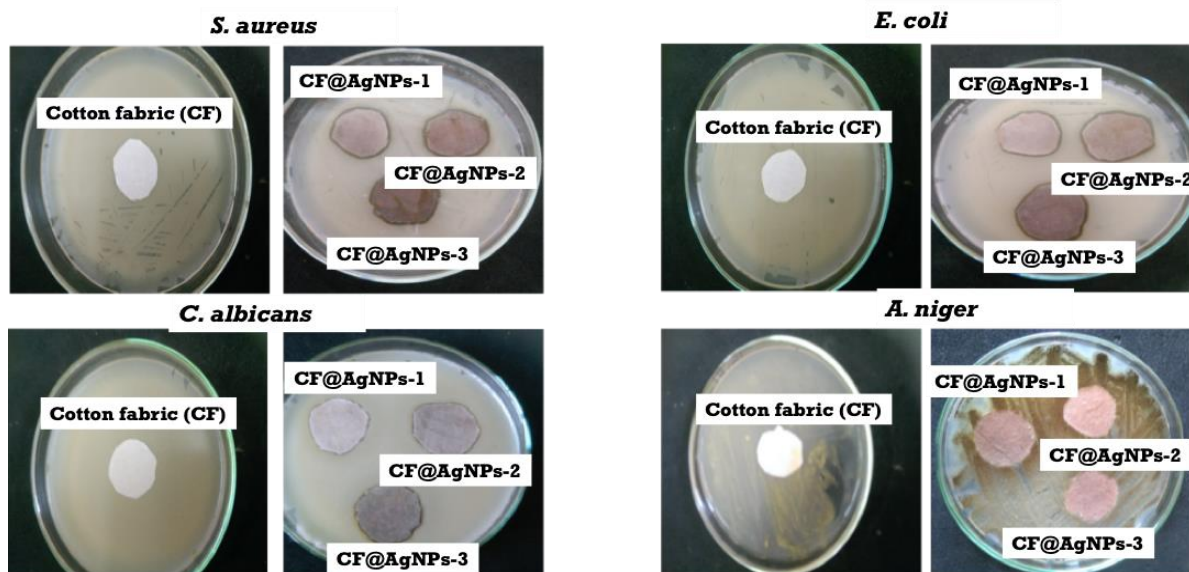


Figure 9: inhibition zone measurement of untreated cotton fabric (CF) and treated cotton fabrics with AgNPs (CF@AgNPs-1, CF@AgNPs-2 and CF@AgNPs-3) against some pathogenic microbes

Table 1: lists the measured clear zone in mm for the untreated and treated cotton fabric with AgNPs against the tested pathogenic microbes.

Sample Code	Clear zone (φmm)			
	<i>S. aureus</i>	<i>E. coli</i>	<i>C. albicans</i>	<i>A. niger</i>
CF	0	0	0	0
CF@AgNPs-1	13	13	12	11
CF@AgNPs-2	13	1	13	11
CF@AgNPs-3	14	15	15	16

The relatively lower clear zones around the silver nanoparticles may be due to that AgNPs make strong chemical bonds with the fabrics and thus not release easily into the medium. By monitoring these lower inhibition zones, it can be concluded that these treated fabrics exhibit controlled antimicrobial properties which can be resulted from its maintaining the amount AgNPs inside into fibrils for a long time which, in turn, enhance the antimicrobial efficiency for many times of wearing.

The challenge of developing cotton fabrics, which really maximise antimicrobial activity in combination with the minimization of cytotoxicity, may be ultimately contribute on the antimicrobial and cytotoxicity findings stated previously thereto.

Conclusion

It is indeed imperative to treat cotton fabrics with an antimicrobial finishing agent to kill the affected bacteria in the human body. Nevertheless, the use of traditional antimicrobial

compounds is still dangerous and costly, particularly in developing countries. Thus, we tried to design an easy method for the preparation of silver nanoparticles (AgNPs) in a solid state which is considered as a simple strategy used in no time and also without considered cost for the preparation. AgNPs was synthesized at different concentrations using dextran as natural biopolymer for the reduction of silver ions and protect the resultant nanoparticles from aggregation. The research study outputs signify that AgNPs was confirmed by the appearance of absorbance around 420 nm. Moreover, the hydrodynamic particle size was observed in very small size 8 nm to 58 nm depending on utilized concentration of AgNO₃. It was also depicted that, all three different concentrations of the resulted AgNPs are sphere-like morphology and well stable as displayed from TEM and zeta potential values. XRD states the complete conversion of Ag ions to AgNPs with no impurities such as silver oxides. The samples of bleached cotton fabrics were then padded in a solution of the three prepared AgNPs samples (AgNPs-1, AgNPs-2 and AgNPs-3; 0.1 g AgNPs/100 mL of water) followed by squeezing drying and curing and evaluated as antimicrobial finishing agent for textile fabric. It was noted from SEM that AgNPs was deposited in small size on the surface of the fabrics beside their penetration the fabric surface. The results depicted that the treated cotton fabrics with AgNPs (any prepared concentration) is safe and has no noticeable toxic effect as proved from cytotoxicity test. Ultimately, the treated cotton fabrics has superior antimicrobial properties when submitted to pathogenic microbes such as *S. aureus*, *E. coli*, *C. albicans* and *A. niger*. Based on the results obtained, the solid state process for the large-scale preparation of AgNPs is known to be safe, simple to operate and easy to transport, which is ideal for medical purposes due to the potential role of nanoparticles as an outstanding antimicrobial finishing agent.

References

- [1] El-Naggar ME, Soliman RA, Morsy OM, Abdel-Aziz MS. Nanoemulsion of Capsicum fruit extract as an eco-friendly antimicrobial agent for production of medical bandages. *Biocatal Agric Biotechnol* 2020;23:101516.
- [2] Sun G. Antimicrobial textiles. Woodhead Publishing; 2016.
- [3] Ramadan MA, Taha GM, EL-Mohr WZE-A. Antimicrobial and UV protection finishing of Polysaccharide-Based Textiles using Biopolymer and AgNPs. *Egypt J Chem* 2020;63:1–2.
- [4] El-Sayed SM, El-Naggar ME, Hussein J, Medhat D, El-Banna M. Effect of Ficus carica L. leaves extract loaded gold nanoparticles against cisplatin-induced acute kidney injury. *Colloids Surfaces B Biointerfaces* 2019;184. <https://doi.org/10.1016/j.colsurfb.2019.110465>.
- [5] Qin Y. Medical textile materials. Woodhead Publishing; 2015.
- [6] Anita S, Ramachandran T, Rajendran R, Koushik C V, Mahalakshmi M. A study of the antimicrobial property of encapsulated copper oxide nanoparticles on cotton fabric. *Text Res J* 2011;81:1081–8.
- [7] Kamal MS, Mahmoud E, Hassabo A, Eid MM. Effect of Some Construction Factors of Bi-layer Knitted Fabrics Produced for Sports Wear on Resisting Ultraviolet Radiation. *Egypt J Chem* 2020;63:5–6.
- [8] A Mohamed A, Fouda A, S Elgamal M, EL-Din Hassan S, I Shaheen T, S Salem S. Enhancing of cotton fabric antibacterial properties by silver nanoparticles synthesized by new Egyptian strain *Fusarium keratoplasticum* A1-3. *Egypt J Chem* 2017;60:63–71.
- [9] Gethin G. Role of topical antimicrobials in wound management. *J Wound Care* 2009.
- [10] Gomaa S, Hilal NM, Abofarha SH. Improvement of Dyeing and Antimicrobial Properties of Cotton Fabrics Through Pre-treatment with

- Silver Nanoparticles. Egypt J Chem 2020;63:7–8.
- [11] Lipsky BA, Hoey C. Topical antimicrobial therapy for treating chronic wounds. Clin Infect Dis 2009;49:1541–9.
- [12] Vowden P, Vowden K, Carvorsi J. Antimicrobial dressings made easy. 2011.
- [13] Cutting KF. Wound dressings: 21st century performance requirements. J Wound Care 2010;19:4–9.
- [14] Gopinath V, MubarakAli D, Priyadarshini S, Priyadharsshini NM, Thajuddin N, Velusamy P. Biosynthesis of silver nanoparticles from *Tribulus terrestris* and its antimicrobial activity: a novel biological approach. Colloids Surfaces B Biointerfaces 2012;96:69–74.
- [15] Sathishkumar G, Gobinath C, Karpagam K, Hemamalini V, Premkumar K, Sivaramakrishnan S. Phyto-synthesis of silver nanoscale particles using *Morinda citrifolia* L. and its inhibitory activity against human pathogens. Colloids Surfaces B Biointerfaces 2012;95:235–40.
- [16] Sheriff R, Thajuddin N. Fabrication of silver nanoparticles with cotton for antibacterial wound dressing. Pharm Nanotechnol 2013;1:78–82.
- [17] El-Naggar ME, Hasanin M, Youssef AM, Aldalbahi A, El-Newehy MH, Abdelhameed RM. Hydroxyethyl cellulose/bacterial cellulose cryogel dopped silver@titanium oxide nanoparticles: Antimicrobial activity and controlled release of Tebuconazole fungicide. Int J Biol Macromol 2020;165. <https://doi.org/10.1016/j.ijbiomac.2020.09.226>.
- [18] Roszek B, De Jong WH, Geertsma RE. Nanotechnology in medical applications: state-of-the-art in materials and devices 2005.
- [19] Youssef AM, El-Naggar ME, Malhat FM, El Sharkawi HM. Efficient removal of pesticides and heavy metals from wastewater and the antimicrobial activity of f-MWCNTs/PVA nanocomposite film. J Clean Prod 2019;206:315–25. <https://doi.org/10.1016/j.jclepro.2018.09.163>.
- [20] Hebeish A, El-Rafie MH, El-Sheikh MA, El-Naggar ME. Nanostructural features of silver nanoparticles powder synthesized through concurrent formation of the nanosized particles of both starch and silver. J Nanotechnol 2013;2013. <https://doi.org/10.1155/2013/201057>.
- [21] Hebeish A, El-Naggar ME, Fouda MMG, Ramadan MA, Al-Deyab SS, El-Rafie MH. Highly effective antibacterial textiles containing green synthesized silver nanoparticles. Carbohydr Polym 2011;86. <https://doi.org/10.1016/j.carbpol.2011.05.048>.
- [22] Yan Jinhua Mehrez E El-Naggar, Orlando Rojas AA. Antibacterial Activity of Silver Nanoparticles Synthesized In-situ by Solution Spraying onto Cellulose. Carbohydr Polym 2016;147:500–8.
- [23] El-Naggar ME, Shaheen TI, Zaghloul S, El-Rafie MH, Hebeish A. Antibacterial Activities and UV Protection of the in Situ Synthesized Titanium Oxide Nanoparticles on Cotton Fabrics. Ind Eng Chem Res 2016;55. <https://doi.org/10.1021/acs.iecr.5b04315>.
- [24] Hebeish A, El-Rafie MH, EL-Sheikh MA, Seleem AA, El-Naggar ME. Antimicrobial wound dressing and anti-inflammatory efficacy of silver nanoparticles. Int J Biol Macromol 2014;65. <https://doi.org/10.1016/j.ijbiomac.2014.01.071>.
- [25] El-Naggar ME, Shaarawy S, Hebeish AA. Bactericidal finishing of loomstate, scoured and bleached cotton fibres via sustainable in-situ synthesis of silver nanoparticles. Int J Biol Macromol 2018;106:1192–202.
- [26] Hassabo AG, El-Naggar ME, Mohamed AL, Hebeish AA. Development of multifunctional modified cotton fabric

- with tri-component nanoparticles of silver, copper and zinc oxide. *Carbohydr Polym* 2019;210:144–56.
- [27] Hebeish A, El-Naggar ME, Tawfik S, Zaghloul S, Sharaf S. Hyperbranched polymer–silver nanohybrid induce super antibacterial activity and high performance to cotton fabric. *Cellulose* 2019;26:3543–55.
<https://doi.org/10.1007/s10570-019-02319-x>.
- [28] Morris H, Murray R. Medical textiles. *Text Prog* 2020;52:1–127.
- [29] Petkova PS. Surface nano-structured materials to control bacterial contamination 2016.
- [30] Zhang Y, Chen F, Zhuang J, Tang Y, Wang D, Wang Y, et al. Synthesis of silver nanoparticles via electrochemical reduction on compact zeolite film modified electrodes. *Chem Commun* 2002;2814–5.
- [31] Salkar RA, Jeevanandam P, Aruna ST, Koltypin Y, Gedanken A. The sonochemical preparation of amorphous silver nanoparticles. *J Mater Chem* 1999;9:1333–5.
- [32] Talebi J, Halladj R, Askari S. Sonochemical synthesis of silver nanoparticles in Y-zeolite substrate. *J Mater Sci* 2010;45:3318–24.
- [33] Bae CH, Nam SH, Park SM. Formation of silver nanoparticles by laser ablation of a silver target in NaCl solution. *Appl Surf Sci* 2002;197:628–34.
- [34] Boutinguiza M, Comesaña R, Lusquiños F, Riveiro A, Del Val J, Pou J. Production of silver nanoparticles by laser ablation in open air. *Appl Surf Sci* 2015;336:108–11.
- [35] Valverde-Alva MA, García-Fernández T, Villagrán-Muniz M, Sánchez-Aké C, Castañeda-Guzmán R, Esparza-Alegría E, et al. Synthesis of silver nanoparticles by laser ablation in ethanol: a pulsed photoacoustic study. *Appl Surf Sci* 2015;355:341–9.
- [36] Roy S, Mukherjee T, Chakraborty S, Das TK. Biosynthesis, characterisation & antifungal activity of Silver nanoparticles synthesized by the fungus *aspergillus Foetidus mtcc8876*. *Dig J Nanomater Biostructures* 2013;8:197–205.
- [37] Honary S, Barabadi H, Gharaei-Fathabad E, Naghibi F. Green synthesis of silver nanoparticles induced by the fungus *Penicillium citrinum*. *Trop J Pharm Res* 2013;12:7–11.
- [38] Yin H, Yamamoto T, Wada Y, Yanagida S. Large-scale and size-controlled synthesis of silver nanoparticles under microwave irradiation. *Mater Chem Phys* 2004;83:66–70.
- [39] Bahadur NM, Furusawa T, Sato M, Kurayama F, Siddiquey IA, Suzuki N. Fast and facile synthesis of silica coated silver nanoparticles by microwave irradiation. *J Colloid Interface Sci* 2011;355:312–20.
- [40] Szczepanowicz K, Stefanska J, Socha RP, Warszynski P. Preparation of silver nanoparticles via chemical reduction and their antimicrobial activity. *Physicochem Probl Min Process* 2010;45:85–98.
- [41] Lee SM, Song KC, Lee BS. Antibacterial activity of silver nanoparticles prepared by a chemical reduction method. *Korean J Chem Eng* 2010;27:688–92.
- [42] Salleh A, Naomi R, Utami ND, Mohammad AW, Mahmoudi E, Mustafa N, et al. The potential of silver nanoparticles for antiviral and antibacterial applications: a mechanism of action. *Nanomaterials* 2020;10:1566.
- [43] Youssef AM, Assem FM, El-Sayed HS, El-Sayed SM, Elaaser M, Abd El-Salam MH. Synthesis and evaluation of eco-friendly carboxymethyl cellulose/polyvinyl alcohol/CuO bionanocomposites and their use in coating processed cheese. *RSC Adv* 2020;10:37857–70.
- [44] Abdelgawad AM, El-Naggar ME, Eisa WH, Rojas OJ. Clean and high-throughput production of silver

- nanoparticles mediated by soy protein via solid state synthesis. *J Clean Prod* 2017;144. <https://doi.org/10.1016/j.jclepro.2016.12.122>.
- [45] Hebeish A, Shaheen TI, El-Naggar ME. Solid state synthesis of starch-capped silver nanoparticles. *Int J Biol Macromol* 2016;87. <https://doi.org/10.1016/j.ijbiomac.2016.02.046>.
- [46] Hussein J, El-Naggar ME, Anwar M, Latif YA, Khateeb S. Synthesis of docosahexaenoic acid-loaded zinc oxide nanoparticles as a promising treatment in neurotoxicity. *Comp Clin Path* 2019;28. <https://doi.org/10.1007/s00580-019-02990-3>.
- [47] Hussein J, Attia MF, El Bana M, El-Daly SM, Mohamed N, El-Khayat Z, et al. Solid state synthesis of docosahexaenoic acid-loaded zinc oxide nanoparticles as a potential antidiabetic agent in rats. *Int J Biol Macromol* 2019;140:1305–14. <https://doi.org/10.1016/j.ijbiomac.2019.08.201>.
- [48] Hussein JS, Rasheed W, Ramzy T, Nabeeh M, Harvy M, El-Toukhy S, et al. Synthesis of docosahexaenoic acid-loaded silver nanoparticles for improving endothelial dysfunctions in experimental diabetes. *Hum Exp Toxicol* 2019;38:962–73. <https://doi.org/10.1177/0960327119843586>.
- [49] Hussein J, El Naggar ME, Latif YA, Medhat D, El Bana M, Refaat E, et al. Solvent-free and one pot synthesis of silver and zinc nanoparticles: Activity toward cell membrane component and insulin signaling pathway in experimental diabetes. *Colloids Surfaces B Biointerfaces* 2018;170:76–84. <https://doi.org/10.1016/j.colsurfb.2018.05.058>.
- [50] Hebeish A, El-Rafie MH, El-Sheikh MA, Seleem A, El-Naggar ME. More insight on characterization of nano-sized particles of silver powder and their application in antimicrobial wound dressing and antiinflammatory efficacy. *Egypt J Chem* 2013;56:169–85.
- [51] Collins CH, Lyne PM, Grange JM, Falkinham JO. Estimating microbial numbers. Collins Lyne's Microbiol Methods, 8th Edn Butterworth-Heinemann Ltd, Oxford 2004:144–255.
- [52] El-Naggar ME, Shaarawy S, Hebeish AA. Multifunctional properties of cotton fabrics coated with in situ synthesis of zinc oxide nanoparticles capped with date seed extract. *Carbohydr Polym* 2018;181. <https://doi.org/10.1016/j.carbpol.2017.10.074>.
- [53] Rehan M, El-Naggar ME, Mashaly HM, Wilken R. Nanocomposites based on chitosan/silver/clay for durable multifunctional properties of cotton fabrics. *Carbohydr Polym* 2018;182:29–41. <https://doi.org/10.1016/j.carbpol.2017.11.007>.