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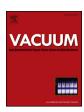
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Enhanced anti-microbial response of commercial face mask using colloidal silver nanoparticles

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ABSTRACT

The present article highlights, improvement in the quality of commercially available face mask with nanoparticles (NPs) coating to protect against infectious agents has been discussed. For this purpose, we first prepared a stock solution of colloidal Ag (nano) with 10,000 ppm concentration by facile and cost-effective synthetic approach using biodegradable and human friendly reagent as capping agent at room temperature. The commercial face mask was then treated with colloidal silver (Ag) solution with two different concentrations *viz.* 50 ppm and 100 ppm. The characterisation of the treated face mask fibre was performed prior to its testing for the antimicrobial activity for Gram positive (*Staphylococcus aureus*) and Gram negative (*Escherichia coli*) bacteria. The results showed that the face mask fibre treated with 100 ppm colloidal Ag solution shows highest antibacterial activity against *Escherichia coli* (*E. coli*) bacteria. The UV–Visible spectrum confirmed the formation of Ag nanoparticles in colloidal solution supported by photoluminescence (PL), Fourier transform infrared (FTIR) and Raman spectroscopy, X-Ray diffraction analysis (XRD), Field emission scanning electron microscopy (FESEM), Energy dispersive analysis X-Ray (EDAX) and Particle size analysis (PSA).

1. Introduction

Nanoscience and nanotechnology is field growing interdisciplinary and moving rapidly towards industrialisation. In the recent days, nanomaterials showed significant advantages in many areas like materials science, optics, electronics, mechanics, polymer and textile industry, with respect to chemical, physical and biological properties compared to bulk materials. Nanoscience and nanotechnology gained tremendous attention in textile and biomedical field for varied applications like sensors, optical displays, water repellence, wrinkle resistance, strength enhancement, UV blocking, computing and antimicrobial [1a,b]. In textile industry, cotton is broadly used as traditional material which exhibits more softness, high absorbency and easy to breathability. But the fibres of cottons have some disadvantages like relatively very low strength, low durable and easy to flammable [1c]. Therefore, synthetic textile with improved properties gathered valuable attention in biomedical field.

In the field of medical textile, large amount of medical equipment like, surgical gowns, curtains in hospital, gauze, arm and knee braces, surgical face masks etc. are used [2]. While, face mask is one of the

important equipment basically used to trap respiratory secretions (bacteria and viruses presented in air), one cannot compromise with the quality and protective effect of face mask. The protective quality of such face mask is depending on the hydrophobic and dryness of outer layer called protective layer. If protective layer is not resistive toward microorganisms, it may cause health risk to the user. In day-to-day environment, the microorganisms and bacterial species are profoundly found and can easily multiply and grow in presence of moisture, temperature and nutrients. The growth and presence of microorganisms or bacteria on face has not only adverse effect on textiles itself but also on wearer. Therefore, to reduce bacterial population in biomedical field and minimize the pathogenic infections caused by the face mask textile materials, the use of antimicrobial textiles is considered valuable attention [3] because of its bacterial resistance increases quality and durability of the product [4,5a]. The use of antimicrobial textile has advantages which can prevent the spreading of bacterial infections through textile materials to another person. Therefore, the antimicrobial textile (e.g. Face mask) has rapidly increasing market value in consumers demand due to its valuable hygienic and comfort properties.

Nowadays, the face mask made from synthetic fibres and textiles

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have more resistance to attacks by bacteria due to their high hydrophobic nature. However, use of certain nanomaterials may enhance the anti-microbial property by killing the bacteria [5b]. Various nanomaterials with varied antimicrobial properties like titanium dioxide, zinc oxide, copper, gold, magnesium, chitosan and alginate have been widely reported in recent years [6–14]. However, among all silver (Ag) nanoparticles due to their unique optical, physico-chemical and biological properties [15] have been used for wide range of potential applications in various fields like biomedical devices, cosmetics, health care products and appliances, environmental therapies and renewable energies etc. [16-20]. Likewise, Ag nanoparticles show significant antimicrobial as well as antifungal properties and therefore widely explored for diverse range of applications in commercial products [21-23]. Group I element, silver has many technologically important properties which can be beneficial for human beings. Ag nanoparticles, because of its small size and diverse applications in the bio-medical field especially antimicrobial property have attracted a considerable attention of researchers.

In view of the above, applicability of Ag nanoparticles for better outcome in desired field is totally dependent on the purity of Ag. There are various reported methods for the synthesis of Ag nanoparticles e.g. sol-gel, chemical reduction method, light assisted method, photo-physical method, photochemical method, electrochemical method, sonoelectrochemical method etc. [24]. Amongst them chemical reduction of silver salt is most popular method because of easy synthetic procedure as less time is required for synthesis and formation of high purity silver nanoparticles [25]. Literature revealed use of various reducing agents such as hydroxylamine hydrochloride, dimethylformamide, polyvinyl pyrrolidone (PVP), sodium borohydride, trisodium citrates for synthesis of Ag nanoparticles resulting in size controlled properties in the range of 1-10 nm [26-29]. However, there are few reports describing use of biologically and environmentally non-hazardous precursors [30]. There is a growing need to develop such environmental friendly, green yet efficient synthetic approaches for synthesis of Ag nanoparticles. Therefore, eco-friendly agents like starch, glucose, chitosan etc. have attracted researchers as an alternative source for toxic chemicals [31-33]. Amongst them starch has become widely used as both reducing as well as capping agent because of the hydroxyl groups present in it. Additionally, it has other significant advantages e.g. a) it is available abundant on earth b) cheap in cost compared with other reducing and capping agents and c) it shows size controlling and stabilizing properties etc. [34].

In the present work, qualitative work has been done as we successfully improved the quality of commercial face mask with respect to its antimicrobial behaviour with treatment of Ag nanoparticles. Here, we first synthesized 10,000 ppm starch stabilized colloidal stock solution of Ag nanoparticles and diluted it to various concentrations for further use. Commercially available face mask (surgical mask) was treated with colloidal Ag nanoparticles solutions with two different concentrations i.e. 50 ppm and 100 ppm. Antimicrobial activity of treated and untreated mask against both gram-positive and gram-negative bacteria was performed. The presence of Ag nanoparticles was confirmed and characterized by analytical tools such as, UV-Visible, photoluminescence (PL), Fourier transform infrared (FTIR) and Raman spectroscopy, X-Ray diffraction analysis (XRD) and Particle size analysis (PSA). The treated as well as untreated masks were also characterized in order to confirm the loading of Ag nanoparticles over mask face mask.

2. Materials and methods

Ag nitrate (98.5%) was commercially purchased from Sigma Aldrich Co. India Ltd. Hydrazine hydrate (80%) was commercially purchased from SRL, Pvt. Ltd. India. Starch was purchased from Molychem Chemical Pvt. Ltd. Mumbai, India. De-ionized water (DI) was used wherever required. Sterile face mask (non-woven) multi layered was

commercially purchased from Ambay medical devices, Mumbai, India. The micro-organisms gram positive bacteria (*S. aureus*) and gram-negative bacteria (*E. coli*) were used as the subcultures for antibacterial studies.

UV-Visible absorption spectra of colloidal solution as well as treated face mask were recorded by using SPECORD 210 PLUS (Analytikjena, Germany) UV spectrophotometer in the wavelength range of 200-800 nm. Photoluminescence (PL) measurements were recorded using Cary Eclipse Fluorescence Spectrophotometer G9800A (Agilent Technologies, USA). The excitation wavelength for measurement of PL spectra was set to 350 nm. X-ray diffraction (XRD) pattern of colloidal solution (drop casted on glass substrate) analysed by using Cu Kα radiation ($\lambda = 0.1546 \text{ nm}$) at room temperature at a generated voltage of 40 kV and current of 120 mA at scanning rate of 2°/min between 20 range of 10-80°. Fourier transform infrared (FTIR) spectra of samples were recorded using Perkin Elmer Spectrum two (USA) in the range of 4000 to 400 cm⁻¹. Scanning electron microscope (SEM) measurement and elemental analysis (EDAX) were performed using Bruker Advanced operated at 300 kV. Raman spectra of the samples were recorded using Enwave Optronics EZ Raman spectrometer. The particle size distribution (DLS) of colloidal solution was recorded using Sympatech (France) particle size analyzer at a laser wavelength of 780 nm in the range of 1-100 nm.

2.1. Synthesis of colloidal (nano) silver (Ag) solution

The starch capped 10,000 ppm concentrated colloidal Ag solution using DI water was synthesized by chemical reduction method. In a typical procedure, starch (0.32 g m) was added in a beaker containing 200 ml DI water and stirred at room temperature for complete dissolution. Ag nitrate (3.2 g m) was introduced in the same beaker and the reaction mixture was stirred for another 15–20 min at room temperature. Above reaction mixture was then treated with 2–3 drops of diluted (1 ml in 100 ml DI water) hydrazine hydrate solution at room temperature and mixture was left for stirring for about 30 min. The formation of colloidal Ag was visibly noticed by formation of golden yellow colour which was further confirmed by UV–Visible spectroscopy recording Surface Plasmon Resonance (SPR).

2.2. Preparation of antimicrobial face mask

The colloidal Ag (Ag) with two different concentrations viz. 50 ppm and 100 ppm were prepared from 10,000 ppm stock solution of colloidal Ag solution. The commercially purchased face mask (approximately 5×5 cm size) were soaked in as-prepared solutions of 50 ppm and 100 ppm for 5–7 h at room temperature and dried at room temperature for 4–5 h. Finally, the treated face masks with colloidal Ag solutions were further utilized for antimicrobial study against grampositive and gram-negative bacteria. The mask treated with 50 ppm solution is labelled as A50 while mask treated with 100 ppm is labelled as A100.

2.3. Antibacterial assay

The antimicrobial effect of face mask samples treated with colloidal Ag solution with different concentrations were studied using a well-diffusion assay against the gram-negative bacterium *Escherichia coli* and the gram-positive bacterium *Staphylococcus aureus* [35]. Each of the petri-plates containing autoclaved, solid Luria-Bertani agar (Himedia Laboratories, India) were inoculated with 106 colonies forming units (CFU) of respective bacterial strain per plate employing a glass spreader. Well-like cavities of uniform area were bored using a sterilized cork borer and incorporated with A50 and A100 samples treated with colloidal Ag solution. Three samples of identical weight viz. untreated mask as reference sample (REF), A50 and A100, each measuring 100 mm² were inoculated in all the petri-plates, aseptically.

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