APPLICATION OF NANOSILVER ON TEXTILES SYNTHESIZED USING CHEMICAL REDUCTION METHODS

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Abstract: *Silver nanoparticles easily interact with other particles and increase their antibacterial efficiency, Moreover, they have received considerable attention due to their attractive physical, chemical and optical properties. Silver nanoparticles were prepared by the chemical reduction method. Silver nitrate was taken as the metal precursor, poly [N-vinylpyrolidone] (PVP) as capping agent and three different reducing agents. Three kinds of nanoparticles were synthesized from glucose ($C_6H_{12}O_6$), formaldehyde and sodium borohydride ($NaBH_4$). The formation of the silver nanoparticles were monitored using ultraviolet-visible spectroscopy. The UV-Vis spectroscopy revealed the formation of silver nanoparticles by exhibiting the typical surface Plasmon absorption maxima at 440-460nm from the UV-Vis spectrum. The antibacterial activity of silver nanoparticles were assessed by determination of their zones of inhibition against the Gram negative (Escherichia coli) bacteria.*

Keywords: *Antimicrobial agent, Chemical reduction, Formaldehyde, Glucose, Nanosilver, Sodium borohydride, Viscose nonwoven.*

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I.INTRODUCTION

The use of silver and other metal ions for their sustained anti-fungal, anti-bacterial and antiviral effects have been practiced for a long time. [1] Because of their high reactivity due to the large surface to volume ratio, nanoparticles play a crucial role in inhibiting bacteria growth in aqueous and solid media. Recently, silver nanoparticles exhibiting antibacterial activity have been synthesized. The antibacterial activity of the silver-containing material can be used, for example, in medicine to reduce infection as well as to prevent bacteria colonization on prostheses, catheters, vascular grafts, dental materials, stainless steel materials and human skin.

Silver nanoparticles have been synthesized using different physical, chemical and biological methods such as milling, electrochemical method, thermal decomposition, laser ablation, microwave irradiation and sonochemical synthesis. The simplest and the most commonly used bulk-solution synthetic method for metal nanoparticle is the chemical reduction method. In fact, production of nanosized metal silver particles with different morphologies and sizes using chemical reduction of silver has been reported. [2]

In the present work on the preparation of nanosized silver nanoparticles from aqueous solution of silver nitrate, formaldehyde, glucose and sodium borohydride were employed as reducing agents. PVP employed as a capping agent.

2. MATERIALS & METHODS

2.1. Materials

Silver nitrate, Glucose, Formaldehyde, Sodium borohydride, Polyvinyl pyrrolidone (PVP) were used. Distilled water was used for synthesis of nanosilver.100% Viscose spunlace nonwoven fabric were used.

2.2. *Synthesis of silver nanoparticles*

Three colloidal forms of silver nanoparticles were synthesized by one-step synthesis method using glucose, formaldehyde and sodium borohydride as reducing agents. Uniform silver nanoparticles were obtained by reduction of silver nitrate at temperature of 50°C using polyvinyl pyrrolidone (PVP) as a stabilizer.

2.2.1. Synthesis of Ag nanoparticles using glucose as a reducing agent

Silver nanoparticles were synthesized by dissolving $AgNO_3$ (0.68 g) and PVP (5 g) were dissolved in 100 ml of 50% (w/w) of glucose solution. Silver nanoparticle or colloidal silver are synthesized in the presence of PVP according to the following redox reaction:

$$2\,AgNO_{3\,(aq)} + C_6H_{12}O_{6\,(aq)} + H_2O_{(l)} \longrightarrow 2Ag_{(s)} + 2HNO_{3\,(aq)} + C_6H_{12}O_{7\,(aq)}$$

In this reaction, glucose ($C_6H_{12}O_6$) reduces the silver cation from the silver nitrate. As the silver metal forms, PVP coats the outside of the particles, preventing them from aggregating and forming larger particles.

2.2.2. Synthesis of Ag nanoparticles using formaldehyde as a reduing agent

Silver nanoparticles were synthesized using $AgNO_3$ (0.34 g) and PVP (7.5 g) in 100 ml of 99.9% formaldehyde.

2.2.3. Synthesis of Ag nanoparticles using sodium borohydride as a reducing agent

One of the most popular methods to synthesize silver nanoparticles is by the use of ice-cold sodium borohydride to reduce silver nitrate. A large excess of sodium borohydride is needed to reduce the ionic silver and further to stabilize the nanoparticles formed. Add 30 ml of 0.002M sodium borohydride ($NaBH_4$) on a stir plate. Ice bath is used to slow down the reaction and give better control over final particle size/shape. Stir and cool the liquid for about 20 minutes. Drip 8 ml of 0.001M silver nitrate ($AgNO_3$) into the stirring $NaBH_4$ solution at approximately 1 drop per second. The solution turned light yellow after the addition of 8 mL of silver nitrate and brighter yellow when all of the silver nitrate had been added. Stop stirring as soon as all of the $AgNO_3$ is added. By mixing both solutions (i.e. $NaBH_4$ and $AgNO_3$), Ag ions were reduced and clustered to form monodispersed Nano particles as a transparent sol in the aqueous medium. Add a drop of 0.3% polyvinylpyrrolidone (PVP), PVP prevents aggregation. The addition of a few drops of 1.5M sodium chloride (NaCl) solution causes the suspension to turn darker yellow, then grey as the nanoparticles aggregate.

2.3. Application of antimicrobial agents

100% viscose spunlace nonwoven fabric was padded using 30 gpl colloidal solutions of nanosilver along with 100 gpl binder. The fabric was padded through padding mangle by 2-dip-2-nip method by keeping 85% expression, dried and cured at 80°C for 15 minutes.

2.4. *Characterization of nanoparticles*

UV-Vis spectrophotometer was used for detection of nanosilver. The antimicrobial finished samples were observed visually and the topography or morphology of the fabric samples was analysed using high resolution SEM (JEOL- JSM 6360) with suitable accelerating voltage and magnification.

2.5. *Antimicrobial Assay:*

The antimicrobial susceptibility of silver nanoparticles was evaluated using the disc diffusion or Kirby-Bauer method (AATCC 147). To examine the bactericidal effect of silver nanoparticles on Gram-negative bacteria, approximately 10^5 colony forming units (CFU) of *E. Coli* strain was cultured on agar plates supplemented with nanosized silver particles. Zones of inhibition were measured after 24 hr of incubation at 35°C.

3. RESULTS AND DISCUSSION

Silver nanoparticles were synthesized according to the method described in the previous section; the colloidal solution turned pale brown, pale yellow, pale red and clear yellow indicating that the silver nanoparticles were formed. Fig. 1 shows the photographs of samples obtained by different reducing agents. The colourless glucose slurry turned pale yellow- brown in addition of silver nitrate precursor (Fig. 1A) and in the case of formaldehyde, the colour changed to pale red (Fig. 1B). In the case that we used ice cold bath for $NaBH_4$, the colourless Ag^+ solution turned to a clear yellow solution (Fig. 1C).

Fig. 1A Fig. 1B Fig. 1C

3.1. Silver nanoparticles synthesized using glucose as a reducing agent

UV-visible spectroscopy is one of the most widely used techniques for structural characterization of silver nanoparticles. The absorption spectrum (Fig. 2) of the pale yellow-brown, silver colloid prepared by glucose reduction showed a surface Plasmon absorption band with a maximum of 440 nm. The peak obtained is very strong and broad and can be assigned to the hydroxyl groups, either from glucose or gluconic acid. Almost this peak appears at the nanosilver sample.

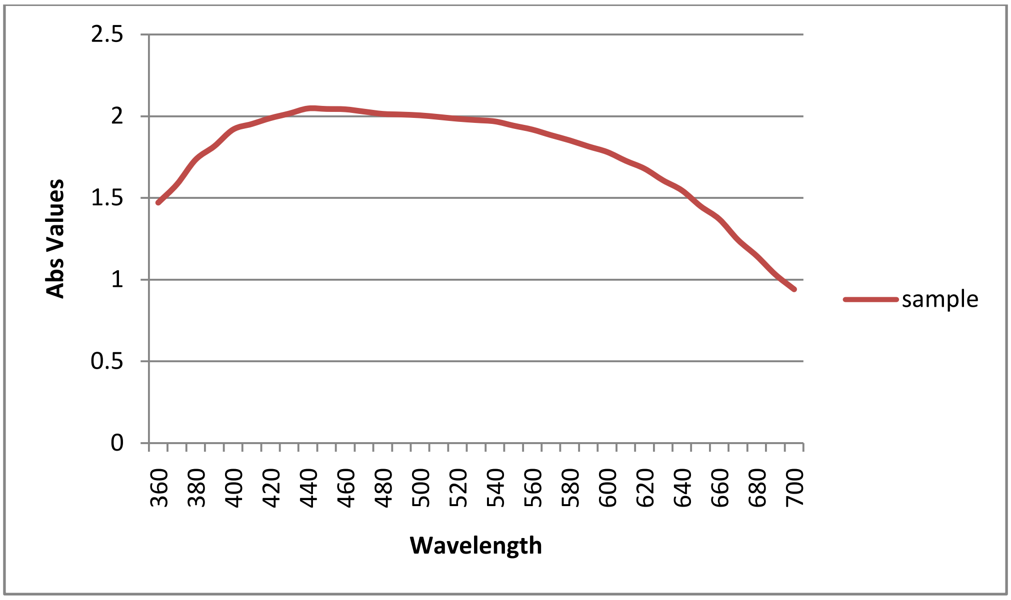

Fig. 2. UV- Visible absorption spectra for Silver nanoparticles synthesized using glucose as a reducing agent

3.2. Silver nanoparticles synthesized using glucose as a reducing agent

The UV-Vis absorption spectrum of silver nanoparticles synthesized using formaldehyde as a reducing agent and PVP as capping agent as shown in figure 3. UV-Vis absorption spectrum (Fig. 3) reveals the formation of silver nanoparticles by showing surface Plasmon absorption maximum at 460nm. The position and shape of the Plasmon absorption depend on the particles size, shape and the dielectric constant of the surrounding medium.

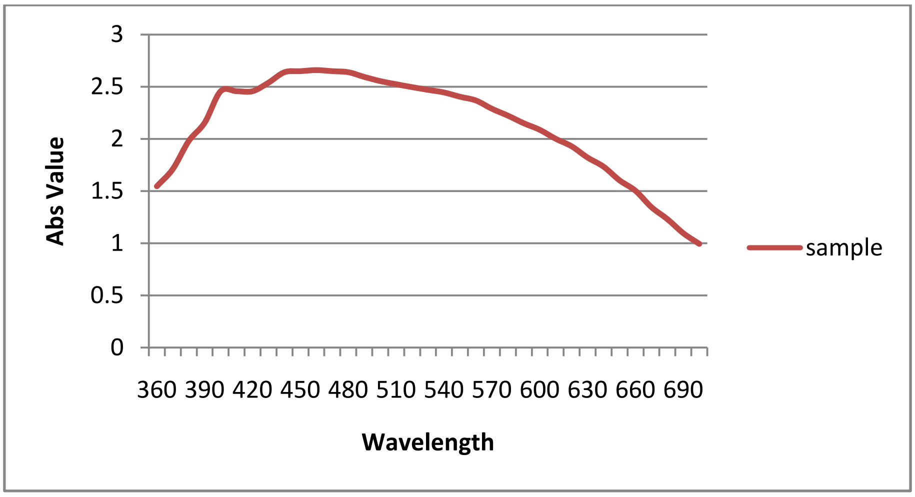

Fig. 3. UV- Visible absorption spectra for Silver nanoparticles synthesized using formaldehyde as a reducing agent

3.3. Silver nanoparticles synthesized using glucose as a reducing agent

The UV-Vis absorption spectra of nanoparticles prepared using sodium borohydride as reducing agent were obtained (Fig. 4). The UV-Vis spectra reveal the formation of silver nanoparticles by showing surface Plasmon resonance at 400nm.

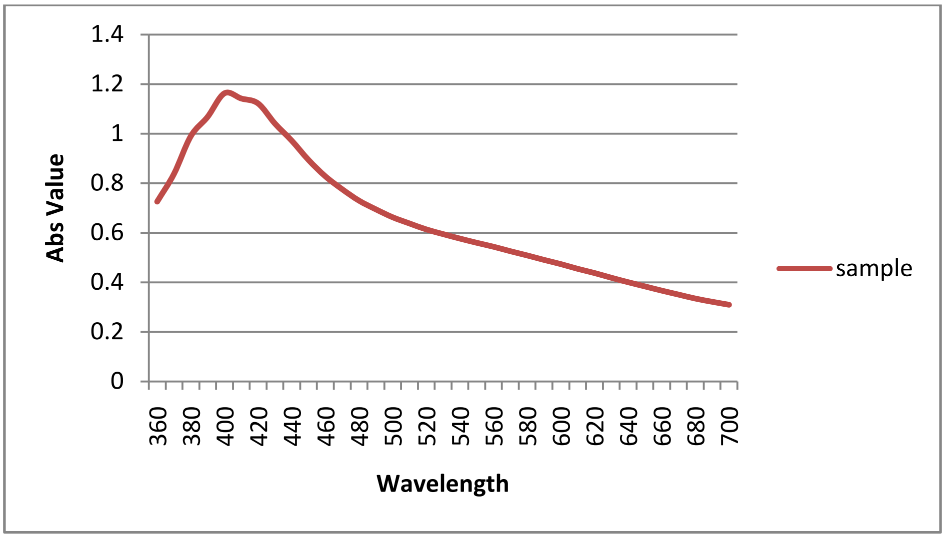

Fig. 4. UV- Visible absorption spectra for Silver nanoparticles synthesized using sodium borohydride as a reducing agent

In all the above cases samples did not show any clear absorption peaks in UV-Visible range. This may be due to agglomeration of the particles observed in the SEM (Fig. 6,7,8).

During the chemical reduction, the reducing agent donates electrons to the silver ions causing silver to revert to its metallic form. In such cases using PVP as protecting agent, the stabilization of silver was highly effective. Silver nanoparticles, known to possess inhibitory and bactericidal effects, have a high surface area to volume ratio along with a high fraction of surface atoms that gives elevated antimicrobial activity compared to the silver metal as a whole.

3.4. Antimicrobial assay:

Finally, the antimicrobial susceptibility of silver nanoparticles synthesized was investigated. The *Kirby-Bauer* diffusion method was used as antimicrobial susceptibility testing method. Petri dishes inoculated with the tested Gram-negative bacteria, including highly pathogenic *E. Coli* at a concentration of 10^5 to10^6 CFU/ml were used for the test. Zones of inhibition were measured after 24hr of incubation at 35°C.

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Figure 5 shows plates to which a bacterial suspension (approximately 10^6 CFU/ml) was applied. The diameter of inhibition zones (in millimetre) around the different silver nanoparticles sols with against test strain are shown in table 1.

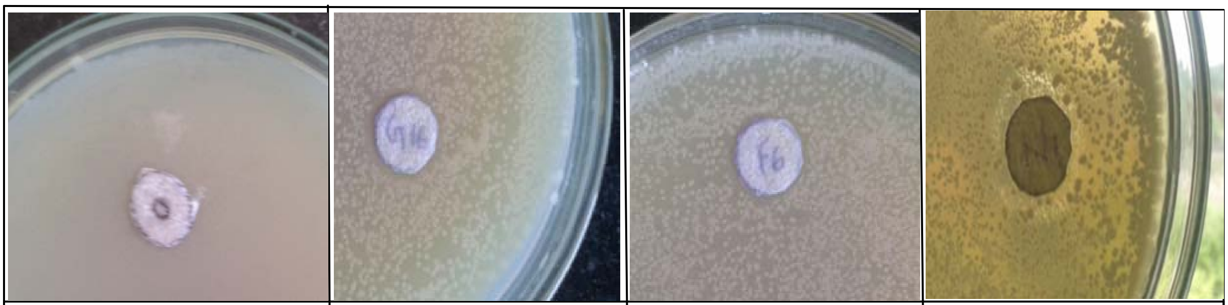

Fig. 5.a	Fig. 5.b	Fig. 5.c	Fig. 5.d

Fig. 5.a zone of inhibition of untreated sample

Fig. 5.b zone of inhibition of Silver NPs synthesized using glucose as a reducing agent.

Fig. 5.c zone of inhibition of Silver NPs synthesized using formaldehyde as reducing agent

Fig. 5.d zone of inhibition of Silver NPs synthesized using sodium borohydride as reducing agent

Table no. 1. Zones of inhibition (MM) of silver nanoparticles sols prepared by different reducing

Agent against test strains

Silver NPs sol	Bacteria	Zone of inhibition (diameter in millimetre)
Liquor	*E. coli*	0
Glucose silver nanoparticles	*E. coli*	16
Formaldehyde silver nanoparticles	*E. coli*	16
Sodium borohydride silver nanoparticles	*E. coli*	18

Silver nanoparticles synthesized using sodium borohydride shows has considerable antibacterial activity.

The mechanism of the bactericidal effect of silver colloid particles against bacteria is not very well known. Silver nanoparticles may attach to the cell membrane and disturb its power function such as permeability and respiration. It is reasonable to state that the binding of the particles to the bacteria depends on the surface area available for interaction. Smaller particles having the larger surface area available for interaction will give more bactericidal effect than the larger particles.

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3.5. Scanning Electron Microscopy (SEM)

The antimicrobial finished samples were observed visually and the topography or morphology of the fabric samples was analysed using high resolution SEM with suitable voltage (10kv) and accelerating magnification (95 to 2000). The photographs are shown in Fig. 6. From this figure, it is clear that continuous polymer film has been formed on the finished fabric.

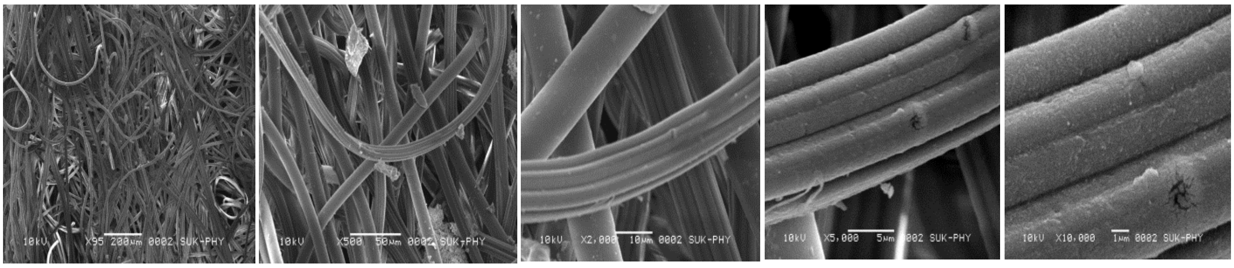

Fig. 6 Surface characteristics of nanosilver synthesized using Glucose

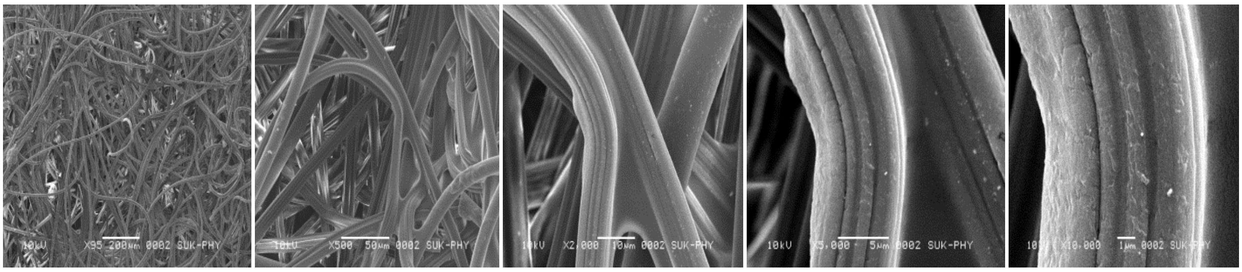

Fig.7. Surface characteristics of nanosilver synthesized using Formaldehyde

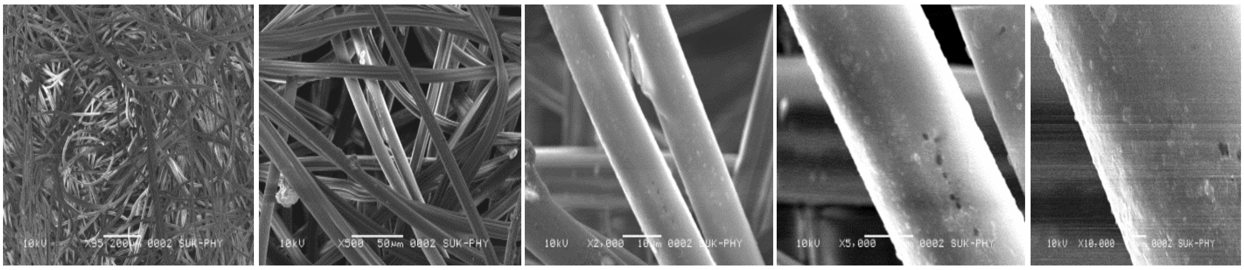

Fig. 8. Surface characteristics of nanosilver synthesized using Sodium Borohydride

4. CONCLUSION

Nanosilver can be used effectively as an antimicrobial agent. The UV-Vis spectrum shows the characteristics Plasmon absorption peak for the silver nanoparticles ranging from 420 to 460 nm. SEM study of antimicrobial finished fabric reveals that a continuous polymer film has been formed on the fabric.

In the case of viscose rayon spunlace fabric treated with silver nanoparticles using glucose, Formaldehyde and Sodium borohydride the zone of inhibition of Gram-negative was a maximum of 16 mm, 16 mm and 18 mm respectively. The results of the present study suggest that silver nanoparticles, which were synthesized using sodium borohydride as the reducing agent showed superior antibacterial action compared to the nanosilver particles synthesized by using glucose and formaldehyde as a reducing agent.

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