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Cotton based bioactive wound dressing material with high absorbency and antibacterial activity

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Cotton gauze fabrics with improved absorbency through yarn twist optimization and antimicrobial property using nanosilver have been prepared. The absorbency, mass loss (%), dehydration rate, wicking rate, surface morphology, surface chemical nature and antibacterial activity of cotton gauze fabrics have been evaluated. The gauze fabric with low twisted yarn shows better absorbency and vertical wicking rate as compared to that with higher twisted yarn. The cotton gauze fabric with optimized twist multiplier (3.0 TM) is then treated with silver nitrate precursor to form *in-situ* nanosilver on the surface of cotton by applying elevated pressure and temperature. The surface morphology has been studied by SEM and chemical nature by FTIR. The *in-situ* technique produces an average nanosilver particle size of 120 ± 48 nm and shows 100% reduction for *Klebsiella pneumoniae* (Gram-negative bacterium) and 99.99% for *Staphylococcus aureus* (Gram-positive bacterium).

Keywords: Absorbency, Bioactive material, Cotton, Gauze fabric, Nanosilver, Wicking, Wound dressing

1 Introduction

The wound is a break in the continuity of the tissue on the lining of the skin or mucosa, resulting from a blow, cut or other impacts. The main objective of wound management is to promote healing and protect the wound from secondary infection. The dressing should have an ability to absorb all types of exudates from the wound, exchange gas between the wound and the environment, and resist the bacterial infection. The dressing material should not adhere to the wound while removal. There are currently a plethora of absorbent wound dressing materials available in the market with the claim to encourage wound healing. But, some of these absorbent dressing materials are not meeting their claims when applied in a clinical setting[1]. Collagen, non-collagenous proteins, proteoglycans, chitosan and alginate are the major biopolymers used in the bioactive wound dressing materials[2,3]. The synthetic materials are dominated by polyurethanes, polyethylene glycol and silicones. The major problems with the synthetic materials are impermeability to drugs and proteins, non-absorbent, allowing the accumulation of would exudate which results in secondary infection and non-biodegradability[4]. In the present study, the development of cotton-based gauze fabric with high absorbency and antimicrobial properties is attempted.

Imparting antibacterial properties on cotton fabrics using nanosilver by various techniques has been reported, viz grafting technique[5], surface plating[6], cationic & exhaustion method[7], biomass method[8], UV radiation[9], Nanocomposite[10], *in-situ* technique[11-15], and microencapsulation of alginate with nanosilver[16]. Angélica *et al.* [17] studied the grafting of cotton gauze using poly (methacrylic acid) and loaded with ZnO nanoparticles to impart the antibacterial property. Most of the reported methods, such as impregnation of nanosilver on cotton are time-consuming, tedious and not suitable for scaling up. Other problems of nanosilver on cotton include the improper attachment of nanosilver on cotton surface and blackening of nanosilver due to the formation of silver oxide by oxidation in air.

It is also well known that the cotton fibre has good swelling and absorbency properties; however, once the fibre is spun into yarn, the absorbency and swelling properties reduce significantly. This is mainly due to the compact arrangement of fibres and helical structure formation owing to the twist in yarn. These yarn twists play a vital role that affects swelling, softness and absorbency characteristic of both the yarns and fabrics. Hence, in addition to wet chemical processing of cotton fibres, yarn structural

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engineering is required to enhance the absorbency of fabrics. Based on the above issues, the goal of this work was to obtain a simple and scalable technology for the production of nanosilver impregnated cotton gauze fabrics for wound management, by retaining the inherent absorbency of cotton fibres. The produced material was tested as per the standard requirements of primary wound dressing and also the antibacterial property was evaluated against both Gram-positive and Gram-negative bacteria.

2 Materials and Methods

2.1 Raw Material

The cotton (MCU-5, India) fibre of length 30 mm (2.5% span length), fineness 3.4 Mic (1.2 denier), tenacity 25 g/tex and elongation 6.7 % was used for the preparation of yarn and subsequently gauze fabrics. The cotton fibre was spun into yarns using a compact spinning system to a nominal count of 19.68 tex (30s Ne combed) with eight different twist factors, viz 2.6, 2.8, 3.0, 3.2, 3.4, 3.6, 3.8 and 4.0 Twist Multiplier (TM) to provide different tightness of fibres in the yarn.

The properties (strength and elongation %) of eight different TM cotton yarns are shown in Table 1. The yarn was then woven into gauze fabric with 18 ends per inch (epi) and 16 picks per inch (ppi). Analytical grade sodium hydroxide, hydrogen peroxide, trisodium phosphate, sodium chloride and calcium chloride dehydrate were purchased from Fisher Scientific® India Pvt. Ltd and silver nitrate ($AgNO_3$) was purchased from Merck® India; these were used as such without further purification.

2.2 Methods

2.2.1 Preparatory Processes of Cotton Gauze Fabrics

The grey cotton gauze fabric was subjected to the mercerization and bleaching process. Initially, the fabric was treated with 23% sodium hydroxide solution at 112 °C using laboratory model beaker dyeing machine for 60 s. The temperature of the bath was reduced to 80 °C, the fabric was removed and then washed under the stretched condition with hot and normal water. After that, the fabric was bleached in a bath which contains 3% hydrogen peroxide solution, 1.5% sodium hydroxide and 0.5% trisodium phosphate for 45 min. The bleached fabric was then washed with hot and cold water (two times) followed by neutralization with dilute acetic acid.

Table 1 — Yarn lea strength property for different twist multiplier yarns

Twist multiplier (TM)	Twists/m	Cotton strength product (CSP)	Elongation %
4.0	862	3025	6.4
3.8	820	3036	6.4
3.6	776	2948	5.9
3.4	733	2853	5.8
3.2	690	2881	5.2
3.0	646	2602	5.4
2.8	603	2445	5.4
2.6	560	2177	4.8

2.2.2 Absorbency and Mass Loss % of Cotton Gauze Fabrics

The absorbency of cotton gauze fabric was determined as per the standard method BS EN 13726-1:2002 free swell absorptive capacities. For this test, 5 cm × 5 cm dressing specimens with four-layer of gauze fabric was prepared. The test solution (A) was prepared using 2.298 g of sodium chloride and 0.368 g of calcium chloride dihydrate in 1.0 L of de-ionized water. All the eight different cotton gauze fabric dressing specimens were weighed and recorded before testing and placed in petri dishes. The solution A was warmed to 37 ± 1°C and 40 times the mass equivalent of the specimen was dispensed slowly and gently onto the specimens in the petri dishes. The petri dishes were then placed in an incubator for 30 min at 37 ± 1°C (body temperature) along with the specimen. After 30 min of conditioning, the dishes were removed from the incubator and suspended by one corner by using tweezers to allow the excessive solution to drip off for 30 s and reweighed for wet mass. The calculation was carried out using the following equation:

$$\text{Mass loss upon drying (\%)} = (B\text{-}A) \times 100/B \quad \ldots (1)$$

where B is the mass of specimens before testing; and A, the mass of specimens after testing.

2.2.3 Vertical Wicking

The vertical wicking is one of the important properties for fibrous dressing materials and this test can be applied only to fibrous dressings due to unique nature. The test specimens were prepared to 25 mm width and 165 mm length as per the standard test method, AATCC TM197:2013. Eosin B was added into the solution A. The specimens were slowly immersed into the solution vertically up to 5 mm length and the wicking time was noted for different intervals of 50 mm, 100 mm and 150 mm. Vertical wicking rate of dressings was determined as given in following equation:

$$W = d/t \quad \ldots (2)$$

where W is the wicking rate in mm/s; d, the wicking distance in mm; and t, the wicking time in second.

2.2.4 In-situ Nanosilver Deposition on Cotton Gauze Fabric

For the *in-situ* nanosilver deposition technique[11] cotton fabric (2 m length) was immersed in 1.0 mM silver nitrate solution and the material-to-solution ratio was maintained at 1:20 (w/v). The samples were placed in a rotary digester at 15 psi pressure and 121 ºC temperature for 15 min. After the treatment of cotton samples with silver nitrate, the fabric's colour turned to yellow and then to dark brown colour. The fabrics were washed with copious amount of water and air-dried.

2.2.5 SEM Analysis

The surface morphology of nanosilver treated fabrics through *in-situ* technique was studied using the scanning electron microscopy (SEM) (Model: Philips XL30, Philips, Netherlands). The samples were coated with a thin layer of gold using a plasma sputtering apparatus, prior to the scanning to avoid charging of the samples. The observations were made at an accelerating voltage of 10 kV with two different magnifications (×5000 and ×12000).

2.2.6 FTIR Spectra Analysis

The Fourier transform infrared (FTIR) spectra of the control cotton gauze and the nanosilver treated fabric were recorded using a Shimadzu FTIR (Shimadzu Corporation, Japan) with a scan rate of 4 cm^{-1} in the wavenumber range 4000 - 700 cm^{-1} for studying the chemical changes occurred due to nanosilver treatment.

2.2.7 Silver Release Measurement

Silver release analysis of treated fabrics was carried out using atomic absorption spectro-photometer (AAS, GBC scientific equipment, Avanta PM, Australia). The nanosilver treated fabric was suspended in deionized water in the ratio of 1:50 (ref. 16). The released silver from the fabric in deionized water was analysed at an interval of 60 min, up to 1440 min and expressed in percentage.

2.2.8 Antibacterial Activity

Antibacterial activity was performed according to AATCC TM 100: 2019 standard method. This is a quantitative procedure for the evaluation of antibacterial activity in textile materials. In this method, *Staphylococcus aureus* and *Klebsiella pneumonia* were used as target pathogens.

3 Results and Discussion

3.1 Relationship of Yarn Twist and Absorbency

When the number of twists increases in the cotton yarn, the fibres present in the yarn will be compactly arranged in the yarn cross-section, as shown in Fig. 1(a). The pore volume and porosity of the yarn will be reduced substantially. In the case of low twisted yarn, the fibre is loosely arranged in the yarn cross-section, as shown in-Fig. 1(b). The pore volume and porosity of the yarn will be more in this case. Generally, swelling of fibres leads to changes in its dimension like a diameter (d), length(*l*), area (A) and volume (V), as shown in Fig. 1(c)[18]. Cotton fibres show a large transverse swelling, with a smaller axial swelling, so that the swelling anisotropy is high[19,20]. In the case of highly twisted yarn, transverse swelling will be low when compared to the low twisted yarn, due to low porosity of inter-fibres in the yarn. This subsequently results in the reduction of absorbency of the yarn.

The surface morphology of 2.6 TM and 4.0 TM with ×350 magnification is shown in Figs 2 (a) and (b).

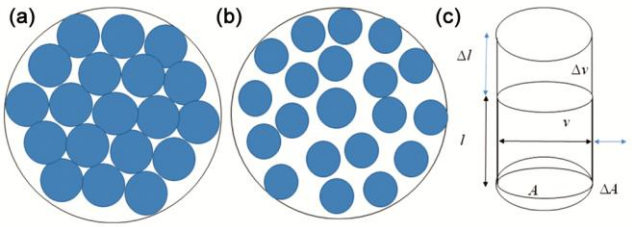

Fig. 1 — Schematic representation of (a) high twist yarn fibre packing, (b) low twist yarn fibre packing, and (c) fibre swelling[18]

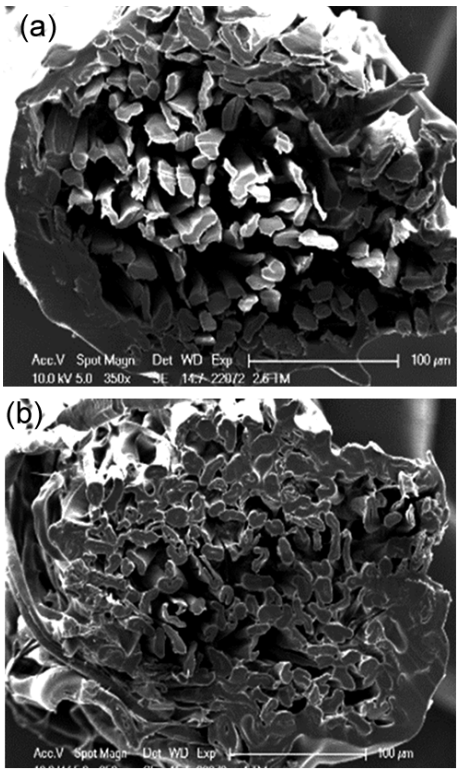

Fig. 2 — SEM micrographs of yarn cross-section view for (a) 2.6 TM and (b) 4.0 TM cotton yarns (scale bars represent 100 µm)

In Fig. 2 (a), the air voids and inter-fibre space are seen clearly in the yarn cross-section. In the case of 2.6 TM yarn cross-section, the fibre is closely packed at the outer surface of the yarn, but the core has more inter fibre space as observed in Fig. 2 (a). It is also observed in Fig. 2 (b) that 4.0 TM yarn cross-section has closely packed fibres with less inter-fibre spaces. As the twist of fibres level increases, fibre compression stress in the yarn will be more, which tends to reduce the pore space between the fibres and increases the cohesion of the fibres.

The absorption and mass loss % of the cotton gauze fabrics prepared from different TM are given in Table 2. The absorption capacity of gauze fabrics is decreasing with the increase in twist of the yarn, with a linear relationship. The water absorption is 29% higher for 2.6 TM of cotton fabric as compared to that of 4.0 TM. A significant difference is found between the lower TM and higher TM gauze fabrics in terms of absorption. In the case of mass loss upon drying (%), there is no significant difference with respect to the TM of cotton gauze fabrics.

Table 2 — Absorbency, mass loss (%) and vertical wicking rate of the cotton gauze fabrics

Twist multiplier (TM)	Absorbency g/g	Mass loss upon drying, %	Vertical wicking rate mm/s		
			50mm	100mm	150mm
2.6	11.2	1	1.5	0.4	0.15
2.8	10.4	1	1.1	0.3	0.15
3.0	10.2	1	1	0.3	0.14
3.2	9.5	2	0.89	0.28	0.1
3.4	9.3	2	0.89	0.26	0.05
3.6	9.2	2	0.71	0.23	0.05
3.8	8.6	3	0.71	0.23	0.05
4.0	7.6	3	0.69	0.22	0.05

3.2 Vertical Wicking Rate

The wicking rate is also an important characteristic of gauze fabric to transport the liquid from the wound surface. The vertical wicking rate of cotton gauze fabric (Table 2) decreases with increase in the TM of yarn. The lower yarn twist (2.6 TM) shows 50% higher wicking rate than higher yarn twist (4.0 TM) in the fabric for 50 mm of wicking height. This is mainly due to the better capillary action and high porosity between the yarn that helps to wick the liquid quickly in the low twisted yarn, which is conformed from Fig. 2 (a). In case of high twisted yarns, such as 3.6 - 4.0 TM (Table 2), the low porosity and compact packing of fibres will restrict the capillary action in the yarn. Twist in the yarn influences the wicking rate of cotton gauze fabric as reported earlier[21,22]. However, the movement of fluid flow into the fabric is proportional to the square root of time, i.e. the wicking rate decreases with respect to the time, but the flow continues, till presumably, the rate of evaporation equals the rate of absorbency[20]. Table 2 shows no significant effect due to TM on wicking rate in the higher distances, such as 100 mm and 150 mm; might be due to the balancing of capillary force of fabric by the gravitational force. The selection of TM yarn is done as per the standard IS 13683:2006, that the lea strength of 30s Ne combed yarn should be 2500 CSP. Accordingly, 3.0 TM yarn cotton gauze fabric is selected as optimum TM for further studies.

3.3 *In-situ* Nanosilver Formation on Cotton

Cellulose chain consists of reducing and non-reducing ends. In the non-reducing end, the anomeric carbon is involved in a glycosidic bond, where the covalent chemical bond holds together a glycoside[23]. The anomeric carbon in reducing end is free to convert to an aldehyde structure. Silver nitrate ($AgNO_3$) solution absorbed into the cellulose structure at increased temperature (121ºC) and pressure (15 psi) during processing is reduced by an extensive number of the hydroxyl groups and the hemiacetal reducing ends to convert Ag^+ to Ag^0. The growth of the metallic form of silver is restricted by the size of pores present in the cellulosic fibres. The nano-size range of pores in the cellulose fibres results in the formation of nanosilver, exhibiting reddish colour due to surface plasmon resonance property. The temperature and pressure play a vital role in the reduction process. The deposition of nanosilver on the surface of cotton fibres is confirmed in the SEM analysis (Fig. 3).

The SEM analysis of the cotton gauze fabric treated with nanosilver (Fig. 3) shows the uniform deposition of nanosilver throughout the yarn surface. Due to good deposition of silver nanoparticles, the surface of fabric develops high reactivity against bacteria and shows good antimicrobial properties. The average size of nanosilver present on the cotton fabric is analyzed to be around 120 ± 48 nm. This method is simple and scalable for industrial adaption. There is no need for any chemical reducing / stabilization agent or any grafting process required for the fixation of nanosilver particle on the cotton gauze fabric. The absorbency and wicking rate of control and nanosilver

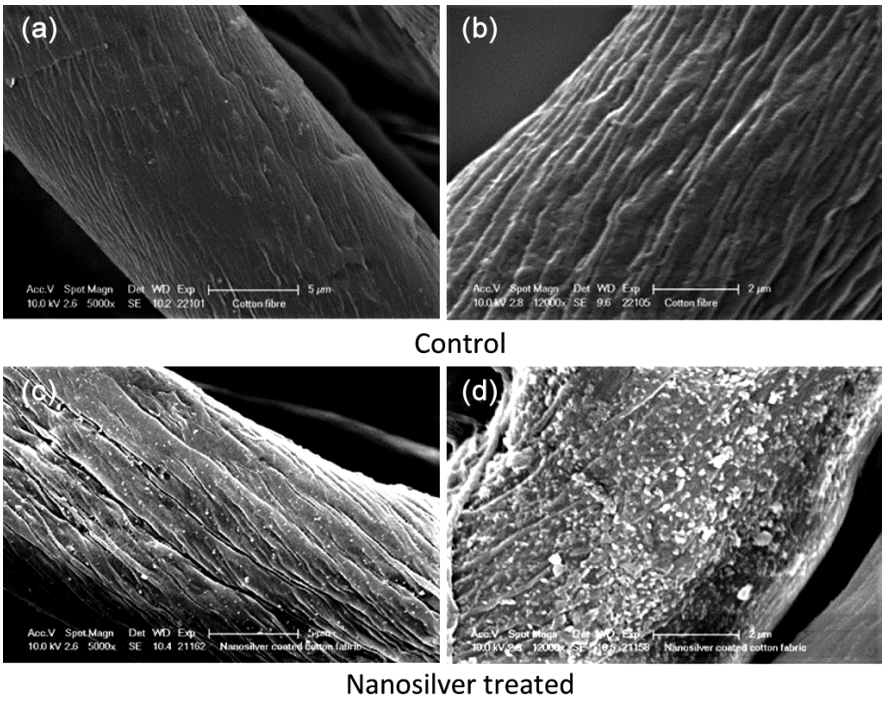

Fig. 3 — SEM micrographs of control and nanosilver treated fabrics (a) & (c) scale bar 5 µm, and (b) & (d) scale bar 2 µm

Table 3 — Absorbency, wicking rate and antimicrobial property of the control and nanosilver treated cotton gauze fabric

Cotton gauze fabric	Absorbency g/g	Wicking rate, mm/s			Antimicrobial property, % reduction	
		50 mm	100 mm	150 mm	*K. pneumonia*	*S. aureus*
Control	10.18	0.99	0.30	0.14	0	0
Nanosilver treated	10.24	0.93	0.30	0.14	100	99.99

treated results are shown in Table 3. It is observed that there is no significant difference between the control and the nanosilver treated cotton gauze fabric, in terms of absorbency and wicking rate. It confirms that the nanosilver particles present in the fabric do not affect the capillary action in the yarn and the inter-fibre space between the fibres.

3.4 FTIR Analysis

The FTIR analysis is carried out for both control and nanosilver treated cotton gauze fabrics. There is no significant difference in various peaks obtained for both control and nanosilver treated fabrics. The results confirm that the nanosilver treatment does not change or modify the chemical nature of cotton, and thus supports the entrapment of nanosilver particles on the fibre by physical means[11,8].

3.5 Silver Release Study

The silver content of nanosilver treated cotton gauze fabric is found 0.2 % (wt/wt) as analyzed by AAS. The release kinetics of the treated fabrics in water is analyzed at an hourly interval for a period till 24 h. The initial release during the first 1 h is found 0.35%, while the maximum amount of release is 0.47% of the total silver available in the fabrics. This finding confirms that the nanosilver is better entrapped in the cotton fibres and not easily released during suspension in water.

3.6 Antimicrobial Test

The nanosilver treated cotton gauze fabrics are tested against *K. pneumoniae* (Gram-negative) and *S. aureus* (Gram-positive) bacteria. From Table 3, it is observed that nanosilver treated fabric shows excellent activity with 100% bacteria reduction for Gram-negative and 99.99% reduction for Gram-positive bacteria. This activity might have been occurred due to the release of silver as analyzed by AAS and also by contact killing of the bacteria by nanosilver. Various mechanisms reported in the literature include the formation of pit by attachment of nanosilver in bacterial cell membrane, formation of free radicals and reactive oxygen species due to silver ions, interaction of silver ions with the thiol groups of cell molecules, and destruction of DNA molecules[24]. Due to the combinations of various mechanisms,

nanosilver on the cotton gauze fabrics might have exhibited antibacterial activity in this work.

4 Conclusion

The cotton gauze fabrics prepared from optimized twisted yarn (3.0 TM) show better absorbency property and wicking rate as compared to that of regularly high twisted yarn fabrics. The *in-situ* technique of nanosilver treated cotton fabric shows a 100% reduction for Gram-negative bacterium and 99.99% for Gram-positive bacterium. The reported *in-situ* method for nanosilver deposition on cotton fabrics is easily scalable for industrial adoption, as it does not require any chemical agents for reduction or stabilization. SEM analyses demonstrate the uniform deposition of nanosilver on the surface of cotton gauze fabric. Using the above optimum TM (3.0) with nanosilver treatment, the use of cotton in bioactive wound dressings could be improved and also will not affect the environment during its disposal.

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