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Effect of silver nano particles synthesized by milkweed (Calotropis) leaves extract on antibacterial activities of bio-medical composites textile materials

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Abstract: Polyester-Viscose (PV) non-woven is widely used bio medical textile material for bandages, gloves, gowns, aprons etc. On the other end, Milkweed (Calotropis) leaves have inherently inbuilt high level of antibacterial properties, well proven from its wide span use made in rural areas for treating burns, fungal attack and wound healing mainly. Thereby a novel PV/ green synthesized silver nano composite was engineered by using medicinally recognized milkweed leaves extract in association with silver nitrate ($AgNO_3$). Apart from this PV/ milkweed leaves extract composite was also prepared to identify extent of antibacterial proficiency of green agent in absence of established silver.

An antibacterial activity potential and moisture management properties of bio-medical composites textile materials (treated using milkweed leaves extract with and without silver nano particles-AgNPs) were studied and compared. Chemically synthesized silver nano particles (AgNPs) were given its consistent preference as well as reported premier effectiveness as an antibacterial component. Parallel streak method (AATCC-147) was used for the conformation of antibacterial activities of the samples against the two most common gram positive and gram negative types of bacterial cultures viz. Staphylococcus aureus and Escherichia coli. The remarkable antibacterial activities were observed for the green synthesized AgNPs incorporated composite. This significant modification was mainly attributed to the green capping agent role played by the milkweed leaves extract in the present study. The presence of AgNPs and uniformly distributed nano particles in the composite structure was observed by Environmental scanning electron microscopy (ESEM) Elemental Mapping and Energy dispersive x-ray analysis (EDAX) results. The average nano particle size (160 nm) statistical distribution was done by using 'ImageJ' Software. The Moisture management test grade (AATCC-195) indicates

"good" overall Moisture management capabilities (OMMC) of the green synthesized AgNPs incorporated composite textile material. Such green synthesized AgNPs incorporated composite textile material is very useful in disposable medical applications.

Key words: Milkweed (Calotropis) leaves extract, Silver nano particles, Green synthesis, Composites, Bio-medical, Antibacterial activities

Abbreviations

PV – (Polyester-Viscose)
AgNPs – (Silver nano particles)
$AgNO_3$ – (Silver nitrate)
DDW – (Double Distilled Water)
ESEM – (Environmental Scanning Electron Microscopy)
EDAX – (Energy Dispersive X-ray Analysis)

Nomenclature

g – (Gram)
GSM – (Grams per Square meter)
g/mol – (MW, molecular weight in gram)
nm – (nanometer)
mL – (millilitre)
mm – (millimeter)
% – (Percentage)
°C – (Centigrade)

4.1 Introduction

Biomedical is the term related to the technology and engineering design employed for medical equipments used by living beings. Whereas term "Biomedical textile", represents textile products and structures developed for medical and biological applications. Such products have been found very useful in first aid, clinical as well as hygienic purpose (Ali et al. 2019; Guarino et al., 2018; Purwar et al, 2004; Pacelli et al., 2006). Hygiene characteristics of the material used for the medicinal purpose like gloves, mask, caps, etc., coming in direct contact with the human skin, is of prime importance (Abou et al.,2012; Patel et al., 2016; Tessier et al., 2005; Zahedi et al., 2010).

The AgNPs have established their recognition for their exceptional antibacterial properties on the account of much higher surface active area (Ahmed et al., 2016; Carbone et al., 2016; Tessier et al., 2005; Shaikh et al., 2017). The antibacterial activity of silver or AgNPs have been found effective till date against more than 650 types of bacteria (Boroumand et al., 2015; Perelshtein et al., 2008). That's why till date AgNPs are preferably used in various antimicrobial products and dominated medical sector. Although efficient enough, this antibacterial agent when chemically synthesized deferred in eco friendliness. However, with the development of 'green chemistry' approach, use of undesirable toxic components gets debarred from the nanomaterial synthesis (Duan et al., 2015; Krishnamoorthy et al., 2012; Murali et al., 2016; Radadiya et al., 2020; Vigneshwaran et al., 2007). Several reports advocating used of plant extract based green synthesized nanoparticles in place of chemical synthesized have been found in the literature (Patel et al., 2015; Siddiqi et al., 2018; Duan et al., 2015; Karthik et al., 2016) The extracts have been obtained from the different parts of the plants; well proven for their medicinal characteristics (Ahmed et al., 2016; Boroumand et al., 2015; Mantle et al., 2001; Sharma et al., 2018; Siddiqi et al., 2018; Velmurugan et al., 2016). The phytochemicals exists in the extracts act as reducing as well as capping agents for the formation of AgNPs (Akhter et al., 1992; Duan et al., 2015; Erdman et al., 1981).

Hence the textile materials used in medical clothing can serve as a nutrient media for the growth of microorganisms, advisable to endow with antibacterial treatment (Abou et al. 2012; Atiyeh et al., 2007; Kannon et al., 1995; Purwar et al., 2004). Thereby in recent time, an increased interest has been noticed in developing textile composites enriched with the antibacterial capabilities (Abbasi et al., 2011; Yetisen et al., 2016; Vigneshwaran et al., 2007). Woven, Non-woven, yarn as well as fibre form of textile materials made up of cotton, PV (Polyester-Viscose), Polypropylene etc. have been used in preparing bio medical textile products like bandages, gloves, gowns, aprons, suture threads etc. with inherent antibacterial efficacy (Chattopadhyay et al., 2014; Gao et al., 2019; Zahedi et al., 2010). Different techniques such as layer-by-layer coating, pad and dry cure, sol-gel, pulsed laser ablation, sonochemical techniques and cold dipping technique etc. have been used for the purpose (Chattopadhyay et al., 2009; Abbasi et al., 2011; Boroumand et al., 2015; Fan et al., 2016; Karthik et al., 2016; Guarino et al., 2018; Montazer et al., 2014; Perkas et al., 2007; Ugur et al., 2010; Zhao et al., 2014). Even practices for simultaneous synthesis and deposition of the nanoparticles on the textile materials have also been realized. The cold dipping technique has been used popularly for AgNPs under in situ deposition on textile materials

in a simple and effective way (Patel et al., 2015; Chattopadhyay et al., 2014; Radadiya et al., 2020).

Green synthesis of AgNPs with Calotropis gigantean (Milkweed) leaves extract and their in situ deposition with cold dipping technique on PV non-woven textile materials has been practiced in the present study. Medicinal plant Calotropis gigantean (Milkweed) is found in abundance and its leaves enriched with antibacterial properties have been used widely for instant treating of burns, fungal attack and wound healing since decades in tribal areas (Akhter et al., 1992; Erdman et al., 1981; Jain et al., 1996; Mantle et al., 2001; Pant et al., 1989). Thereby the bio medical composites were produced by treating the PV non-woven once with Milkweed (Calotropis gigantea) leaves extract only, and secondly by green synthesized (in association with Calotropis gigantea leaves extract) AgNPs. Their antibacterial efficacy against the two most common gram positive and gram negative bacterial cultures viz, Staphylococcus aureus and Escherichia coli was worked out.

4.2 Materials and methods

4.2.1 Materials

i. Polyester-Viscose (70:30) non-woven fabric with 40 GSM used for manufacturing mask, gloves and gowns
ii. Silver nitrate ($AgNO_3$) with molecular weight 169.87 $g.mol^{-1}$
iii. Milkweed (Calotropis) leaves
iv. Double distilled water (DDW)
v. Whatman filter paper (Grade 1 qualitative)

4.2.2 Methods

4.2.2.1 Preparation of milkweed leaves extract

Milkweed leaves (around 1 kg) were collected from nearby river bank area in Gujarat region of India with staggered time interval for the preparation of random sample. They were washed twice with tape water to remove debris, dirt and other contaminations, followed by final washing with DDW. After that the leaves were oven dried at 70°C ± 05°C. Such dried leaves were than crushed and grind into fine powder.

The Milkweed leaves extract with 10% concentration (Milkweed leaves powder (g): DDW (mL) = 1:10) was prepared by dissolving milkweed powder (100 g) into DDW (1000 mL) heated and agitated continuously for two hours

at 70°C ± 05°C temperature. The solution was then cooled, and filtered by Whatman filter paper to remove undissolved particles (Patel et al., 2015; Shaikh et al., 2017).

4.2.2.2 Synthesis of silver nano particles (AgNPs) colloidal

Selected $AgNO_3$ has the molar mass of 169.87 g.mol^{-1}, for preparation of 10 mM solution 0.169 g of silver nitrate solution was added drop by drop in 100 ml of 10% concentration milkweed leaves extract. Then colloidal solution was heated at 80°C along with continuous magnetic stirring till its colour has changed from light to dark brown on the completion of the AgNPs synthesis (Chattopadhyay et al., 2014; Shaikh et al., 2017).

4.2.2.3 Preparation of composite textile materials

Totally three types of samples were considered during the experimentation, viz; (i) Sample 00: Untreated fabric, (ii) Sample 01: Fabric treated with AgNPs colloidal solution (10% concentration of milkweed leaves extract with 10 mM $AgNO_3$), and iii) Sample 02: Fabric treated with 10% concentrated milkweed leaves extract only (see Table 4.1). Both the treated samples were made by using cold dipping technique. The sample was dipped continuously for 02 hours at room temperature into the respective solution bath by maintaining constant bath ratio of 1:25 (1 g of the sample: 25 ml solution) (Tessier et al., 2005; Patel et al., 2015). The samples were then dried at the room temperature for 24 hours and preserved in sealed plastic bag for further analysis.

Table 4.1 Details of composite textile materials

	Fabric	Milkweed leaves extract	Silver nitrate ($AgNO_3$)
Sample 00 (Untreated)	↑ Polyester-viscose (polyester – 70%, viscose – 30 %) non-woven ↓	–	–
Sample 01 (Green synthesized AgNPs composite)		100 mL (10% conc.)	0.169 g
Sample 02 (Only milkweed leaves extract treated)		100 mL (10% conc.)	Not used

4.2.2.4 Test methods

Untreated and treated textile materials were evaluated for their physical, low stress, comfort associated and antibacterial characteristics, important for

biomedical application. Standard methods were followed for testing and the samples in each category were tested only after due conditioning at the standard atmospheric conditions (Temperature: 27°C ± 2°C and Relative humidity: 65% ± 2%) for 24 hours. The average of ten independent evaluations of each test except moisture management capabilities, morphological studies and antibacterial assessment was considered.

Physical parameters

(a) GSM (Gram per square meter): Sample of 10 cm × 10 cm was cut precisely and weighted on an electronic balance (0.01 gm accuracy) and GSM value was calculated (equation 1).

$$GSM = \frac{w \times (100)^2}{10 \times 10} \quad \text{...(i)}$$

(b) Thickness was measured by using Thickness gauge at 1 lbs/inch^2 pressure.

Low stress properties

(a) Fabric stiffness: Average fabric bending length (c) was measured on Shirley stiffness tester from samples each of 6 × 1 inch size and bending modulus was calculated (equation 2).

$$\text{Bending Modulus } q \text{ (kg/cm}^2) = \frac{12G \times 10^{-6}}{g^3} \quad \text{...(ii)}$$

Where, g is the cloth thickness in cm. and G is flexural rigidity (equation 3)

$$\text{Flexural rigidity } G \text{ (mg/cm)} = wc^3 \times 10^3 \quad \text{…(iii)}$$

Where w is cloth weight in gm/cm^2

(b) Crease recovery angle: It was measured as per ISO 2313 using Crease recovery tester.

Comfort associated properties

(a) Air permeability: It was measured as per ASTM D737 using METEFEM Air permeability tester.

(b) Overall moisture management capability (OMMC) was measured as per AATCC-195 on SDL-ATLAS Moisture management tester.

Morphological and elemental assessment of the nano-composite textile material

The Morphological assessment of the synthesised Nano-composite textile

material was done by using ESEM (Environmental Scanning Electron Microscopy, Model: XL-30, Philips, Netherlands) and Elemental analysis and elemental mapping of the synthesised Nano-composite textile material was done by EDAX (Energy Dispersive X-ray Analysis, Model: XL-30, Philips, Netherlands). The average nano particle size of the AgNPs was calculated by using 'ImageJ 1.53a' Software.

Antibacterial qualitative assessment of nano-composite textile material

The qualitative evaluation was done as per AATCC 147(2004), Parallel streak method. This method works best for the leaching type antibacterial agents (AATCC Test Method 147, 1998; EN ISO 20645:2004, 2004)

Control sample that is untreated was compared with the treated samples using both gram positive and gram negative types of bacterial cultures viz., Staphylococcus aureus (NCIM 2654) and Escherichia coli (NCIM 2832) (AATCC Test Method 147, 1998; EN ISO 20645:2004, 2004). Comparison was made on the ground of measurement obtained for average width Zone of Inhibition (ZOI) that means clear zone without bacterial growth. All the samples were cut into strip of 10 mm fixed width for this purpose and placed on the surface of nutrient agar medium with the bacterial cultures. The plates were incubated at 37°C for 24 hours to check the bacterial inhibition.

4.3 Results and discussion

Table 4.2 relates results for various physical, low stress and comfort related properties of the untreated and treated textile materials.

4.3.1 Physical properties

It can be observed from the results (see Table 4.2), GSM and Thickness values of treated samples have shown minute rise in comparison to reference untreated sample. Critical difference $[CD\ (\%) = \frac{\text{Difference in two values}}{\text{Average of two values}} \times 100]$ for GSM observed was 2.74% for AgNPs treated and 1.21% for milkweed extract treated samples. However, the value of CD% drops to 0.72% and 0.24% respectively for thickness. Hence uniformity of the nonwoven fabric produced for the medical textiles should be maintained higher. Under such condition these changes noticed in fabric physical measures are expectedly reflecting addition of the functional matters into the light weight fabric structure.

Table 4.2 Physical properties of untreated and treated textile materials

Physical properties						
Properties	**Sample 00**		**Sample 01**		**Sample 02**	
GSM (gram per sq. meter)	40.904		42.043		41.403	
Thickness (mm)	0.415		0.418		0.416	
Low stress properties						
	Length wise	**Width wise**	**Length wise**	**Width wise**	**Length wise**	**Width wise**
Bending modulus (g/cm^2)	9.43	0.87	6.70	1.28	7.34	0.99
Crease recovery angle (°)	116.4	111	112.3	107.7	114.4	110.3
Comfort related properties						
Air permeability ($m^3/m^2/h$)	4753		4658		4705	

4.3.2 Low stress properties

Low stress properties are relating to the handle (capability to recover) of the fabric against the low magnitude stresses subjected at the point of end use. Textile material undergoes more or less bending deformation on the movement of body part where medicinal clothing was worn. It was required that material should acquire quickly shape as per new body contour and that was possible only if it possesses low bending modulus. No doubt there are arbitrary changes noticed in the length wise as well as width wise bending modulus of the samples under consideration (see Table 4.2). But these changes didn't reflect marginal rise or drop in bending modulus which will not allow the fabric fit for bio medical use (Ali et al., 2019; Montazer et al., 2014).

As such crease recovery for the disposable bio medical nonwoven fabrics is not so much important. But the fabric should possess some resilience to recover from crease during first and last use. Almost all the samples have executed crease recovery angle more or less equals to 110°, sufficient to recover from undesirable wrinkle formed on use (Booth J.E, 1996; Kothari V.K., 1999).

4.3.3 Comfort related properties

Comfort of the wearer should be given a prime importance especially when a medical textile material is worn next to the skin for a prolonged time interval.

The air permeability and moisture management capabilities of the fabric play crucial role in this regards. Treated samples have not shown critically different (≥ 5%) drop in air permeability value on loading the functional elements.

The Overall moisture management capability (OMMC) represents an index for the overall ability of the fabric to manage the transport of liquid moisture. This measure includes three aspects of performance, viz; spreading speed in other words drying speed, moisture absorption rate of bottom surface and accumulative one-way liquid transport ability. Higher OMMC is indicative for better comfort to the wearer or moisture transport ability of the fabric.

Test results for OMMC and their grading defined as per AATCC 195-2009 are given in table 4.3 and table 4.4 respectively. The graphs and charts for the OMMC (see Fig. 4.1), accumulative one-way transport index (%) (see Fig. 4.2), finger print test analysis (see Fig. 4.3, a-c), Water content vs Time (see Fig. 4.4, a-c), and Water location vs Time (see Fig. 4.5, a-c) for the all three samples computed by the MMT interfaced computer are given here for ready reference.

Table 4.3 Moisture management test results of samples

	Sample 00	Sample 01	Sample 02
Wetting Time Top(sec)	1.217	1.217	21.341
Wetting Time Bottom(sec)	5.335	5.429	5.616
Top Absorption Rate (%/sec)	71.626	25.531	14.739
Bottom Absorption Rate (%/sec)	29.486	5.660	16.570
Top Max Wetted Radius (mm)	30.000	5.000	10.000
Bottom Max Wetted Radius (mm)	25.000	0.000	0.000
Top Spreading Speed (mm/sec)	13.036	3.561	0.332
Bottom Spreading Speed (mm/sec)	7.415	0.000	0.000
Accumulative one-way transport index (%)	–1117.024	388.447	–82.663
OMMC	0.304	0.487	0.018

Table 4.4 Grading specification of MMT test as per AATCC 195-2009

Parameter	Surface	Grade				
		1	2	3	4	5
Wetting time, s	Top	> = 120	20 – 119	5 – 19	3 – 5	<3
		No wetting	Slow	Medium	Fast	Very fast
	Bottom	> = 120	20 – 119	5 – 19	3 – 5	<3
		No wetting	Slow	Medium	Fast	Very fast

Contd...

Contd...

Parameter	Surface	Grade				
		1	2	3	4	5
Absorption rate %/s	Top	0 – 10 Very slow	10 – 30 Slow	30 – 50 Medium	50 – 100 Fast	>100 Very fast
	Bottom	0 – 10 Very slow	10 – 30 Slow	30 – 50 Medium	50 – 100 Fast	>100 Very fast
Max. wetted radius, mm	Top	0 – 7 No wetting	7 – 12 Small	12 – 17 Medium	17 – 22 Fast	>22 Very fast
	Bottom	0 – 7 No wetting	7 – 12 Small	12 – 17 Medium	17 – 22 Fast	>22 Very fast
Spreading speed, mm	Top	0 – 1 Very slow	1 – 2 Slow	2 – 3 Medium	3 – 4 Fast	>4 Very fast
	Bottom	0 – 1 Very slow	1 – 2 Slow	2 – 3 Medium	3 – 4 Fast	>4 Very fast
One way transport capacity	-	< -50 Very poor	-50 – 100 Poor	100 – 200 Good	200 – 400 Very good	>400 Excellent
OMMC	-	0 – 0.2 Very poor	0.2 – 0.4 Poor	0.4 – 0.6 Good	0.6 – 0.8 Very good	>0.8 Excellent

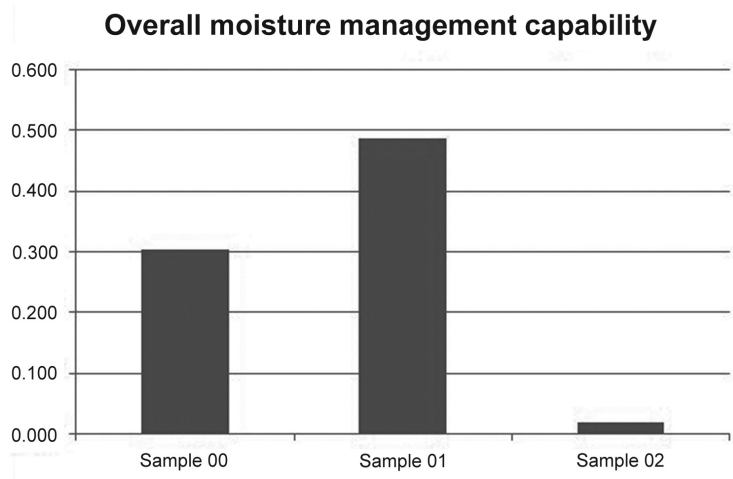

Figure 4.1 Overall moisture management capability

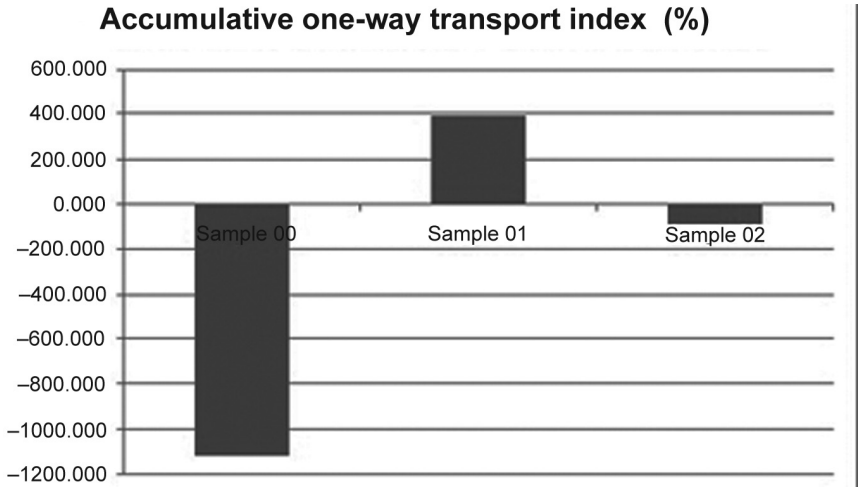

Figure 4.2 Accumulative one-way transport index (%)

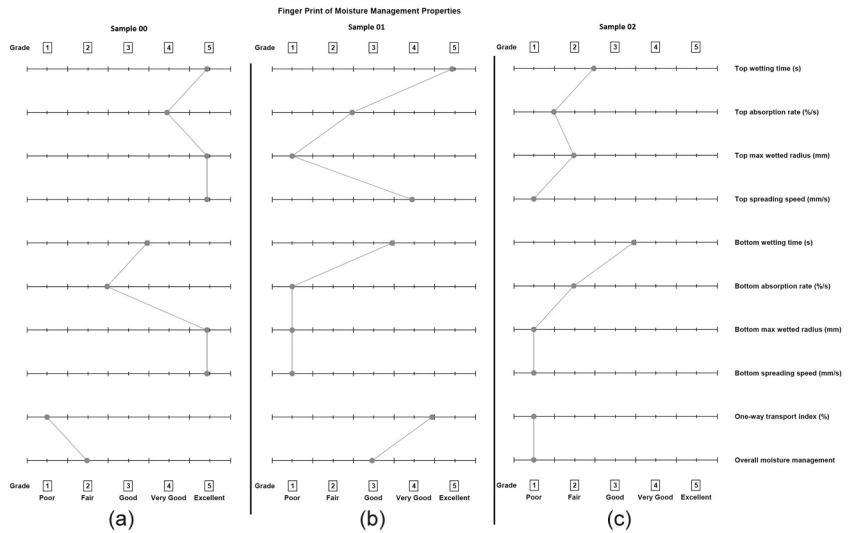

Figure 4.3 Finger print test analysis results (a) Sample 00, (b) Sample 01 and (c) Sample 02

According to the OMMC value "0.4872" observed for sample 01, it was characterised as good (0.4-0.6) moisture manager than reference sample 00 (0.3041; poor = 0.2 - 0.4) and sample 02 (0.0183; very poor = 0 - 0.2) in the group. This observation was further substantiated by the accumulative one-

way transport index (388.447 %) of the sample 01, rated it as “Very good” (200-400).

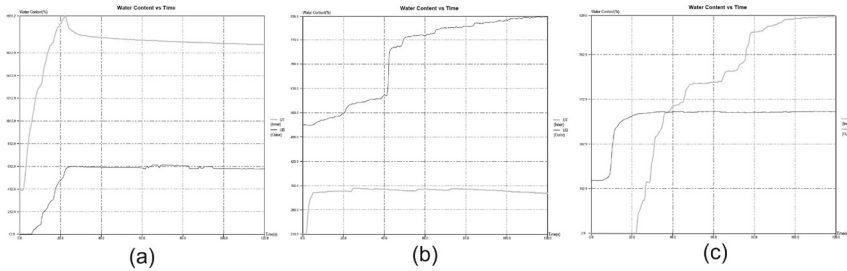

Figure 4.4 Water content vs Time (A) Sample 00, (B) Sample 01 and (C) Sample 02

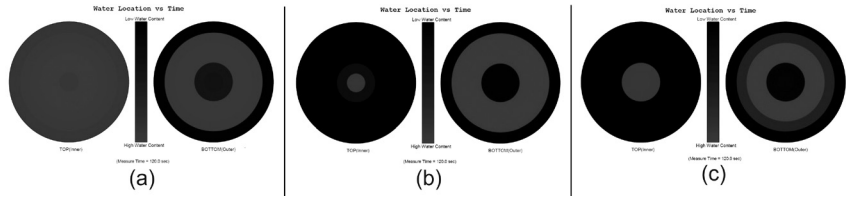

Figure 4.5 Water location vs Time (a) Sample 00, (b) Sample 01 and (c) Sample 02

4.3.4 Morphological assessment of the nano-composite textile material

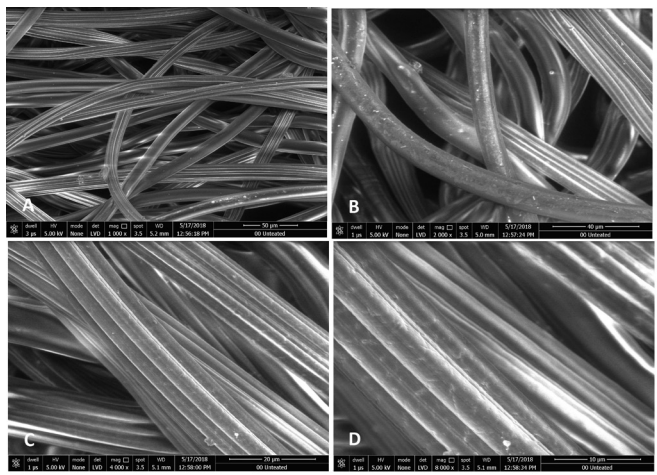

Figure 4.6 ESEM images at different magnification (a-d) of Sample 00

The ESEM images taken for all the three samples (00 -02) at four magnifications levels are shown in Fig. 4.6-4.8 (a-d) respectively. Hence sample 00 represents the untreated base material only and sample 02 only Milkweed leaves extract treated sample, execution of apparently similar structure is likely. Sample 01 has validated occurrence of the uniform deposition of AgNPs on the constituent fibers for the selected deposition technique. Some agglomerations of primary particles were also observed.

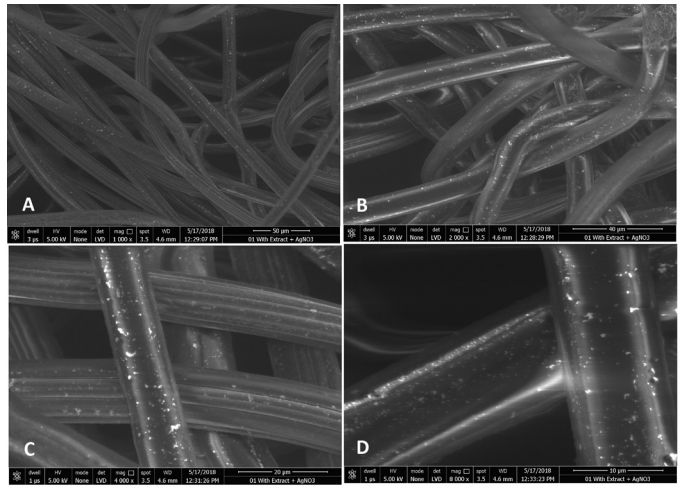

Figure 4.7 ESEM images at different magnification (a-d) of Sample 01

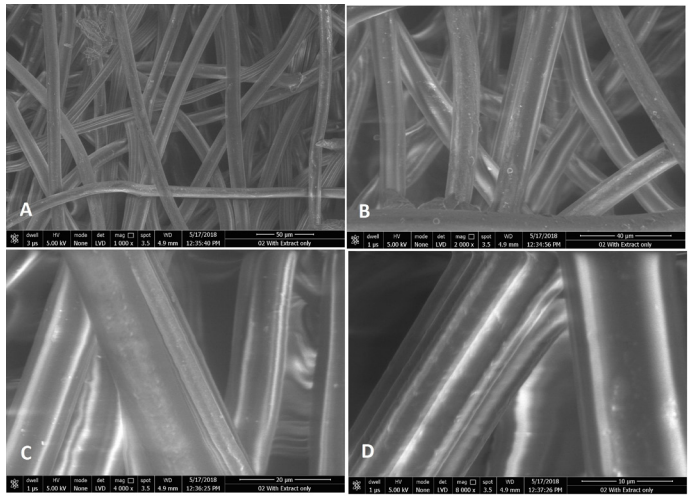

Figure 4.8 ESEM images at different magnification (a-d) of Sample 02

4.3.4.1 Nano particle size statistical distribution

Using "ImageJ 1.53a" software the nano particle size was determined. Employing "Image 1.53a" the area of the nanoparticles was determined in pixel. Pixels were converted to nanometers by applying scale on picture by the software. Diameter of the nanoparticles was calculated with Microsoft excel. The particle size normal distribution and histogram are illustrated in Fig. 4.9 (a-b) respectively.

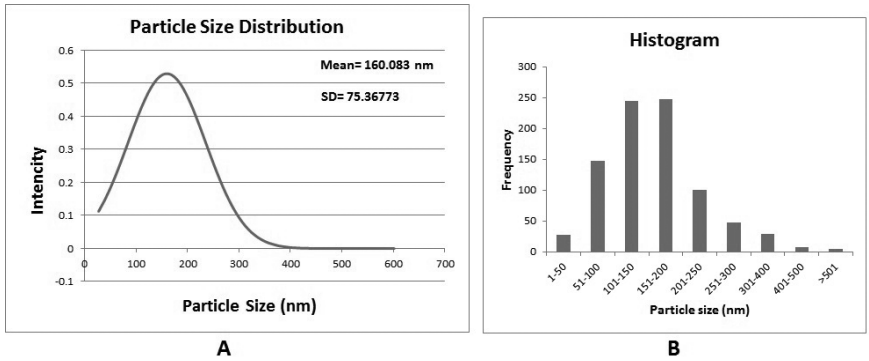

Figure 4.9 Nano particle size distribution (A) and, Its Histogram (B)

It can be observed that the particles were distributed uniformly throughout with an average particles size of 160.083 nm (see Fig. 4.9a). This has substantiated reducing and capping agent behaviour of the Milkweed leaves extract during the synthesis of AgNPs colloidal.

4.3.5 Elemental assessment of the nano-composite textile material

The Energy dispersive spectrums taken for the selected area of the samples (00, 01 and 02) are shown in Fig. 4.10 (a-c) respectively. Strong signals were observed from the silver atoms in the nanoparticles at around 3.0 - 3.2 keV, which is a characteristic peak of metallic silver nano crystals (see Fig. 4.10b). Further, green synthesized silver nanoparticles were also characterized by elemental mapping. Results for the elemental mapping of sample 00, 01, and 02 are shown in Fig. 4.11 (a-c) respectively. The results have revealed that in the electron micrograph region of synthesized product, silver nanoparticles are well distributed. While, their composition by weight (%) and atomic (%) of the elements are given in Table 4.5. The green synthesized AgNPs treated sample 01 in the group has shown presence of Silver (AgL, silver complex)

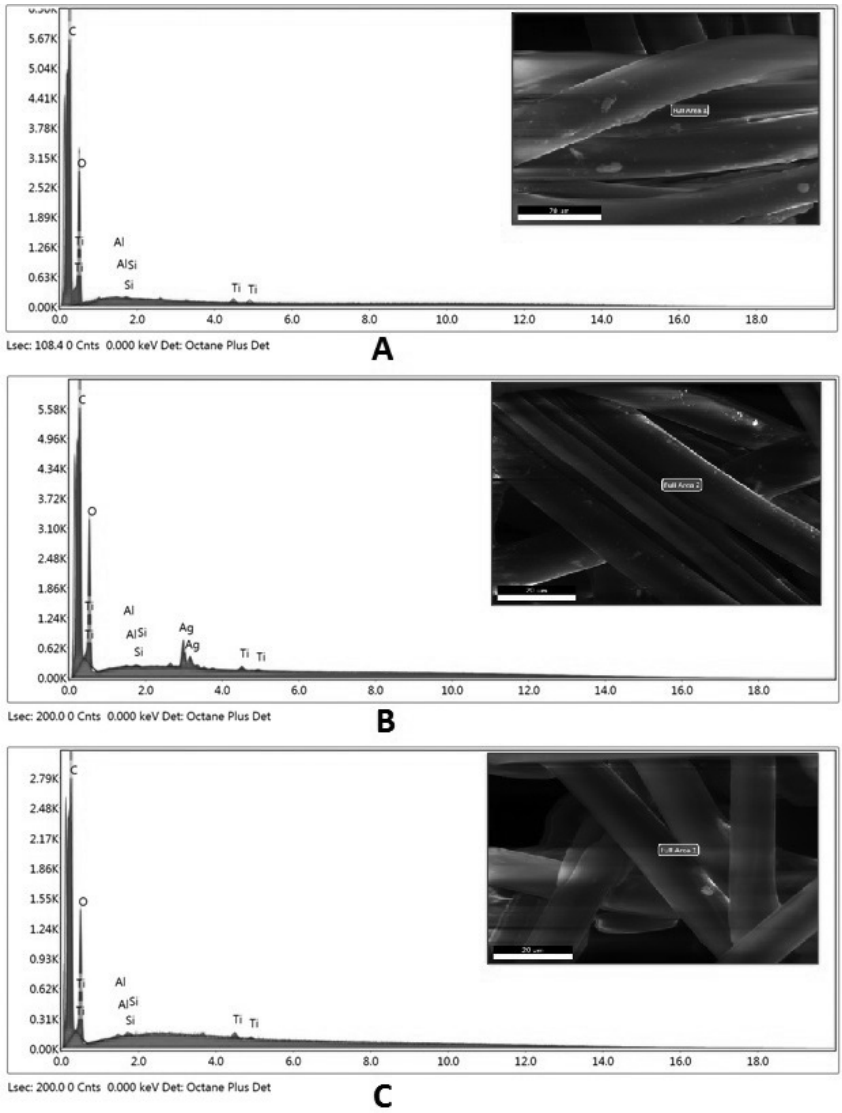

Figure 4.10 Elemental assessment of the A) Sample 00, B) Sample 01, C) Sample 02

composition 3.56 % by weight for the selected level of $AgNO_3$ add on. Apart from this, highest composition (57.80%) of the Carbon (C) along with next to highest composition (38.12%) of the Oxygen (O) by weight can be observed. The common additional elements noticed for all the samples were Aluminium

(Al), Silicon (Si), and Titanium (Ti) with very less but almost identical amount for untreated and AgNPs treated samples. However, somewhat rise in the composition values of these additional elements can be noticed for only Milkweed leaves extract treated sample 02.

Table 4.5 Composition of the elements

	Sample 00		Sample 01		Sample 02	
Element	**Weight %**	**Atomic %**	**Weight %**	**Atomic %**	**Weight %**	**Atomic %**
C K	60.97	67.72	57.8	66.47	62.5	69.25
O K	38.51	32.12	38.12	32.91	36.63	30.47
AlK	0.02	0.01	0.03	0.02	0.09	0.04
SiK	0.04	0.02	0.06	0.03	0.12	0.06
TiK	0.46	0.13	0.43	0.12	0.66	0.18
AgL	–	–	3.56	0.46	–	–

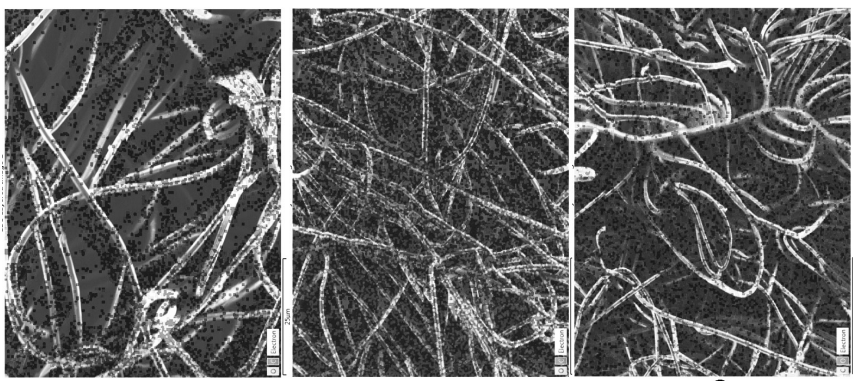

Figure 4.11 Elemental Mapping of the (A) Sample 00, (B) Sample 01, (C) Sample 02

4.3.6 Antibacterial assessment of the nano-composite textile material

Photographs for the antibacterial activity test results for sample 01 and sample 02 with reference to sample 00 are shown in Fig. 4.12 (a-b) and Fig. 4.13 (a-b) respectively. The average values for zone of inhibition (ZOI) of all the three samples against both the organisms are given in Table 4.6. It can be seen that average ZOI value against bacterial cultures S. aureus (+ve) and E. coli (-ve) for sample 01 are 23.75 mm and 24.5 mm respectively, and same 12mm for

Sample 02. As expected sample 00 has not shown any bacterial inhibition against the both tests bacterial cultures.

Table 4.6 Average zone of inhibition (mm)

Organism	Sample 00	Sample 01	Sample 02
Staphylococcus aureus (SA)	None	23.75	12
Escherichia Coli (EC)	None	24.50	12

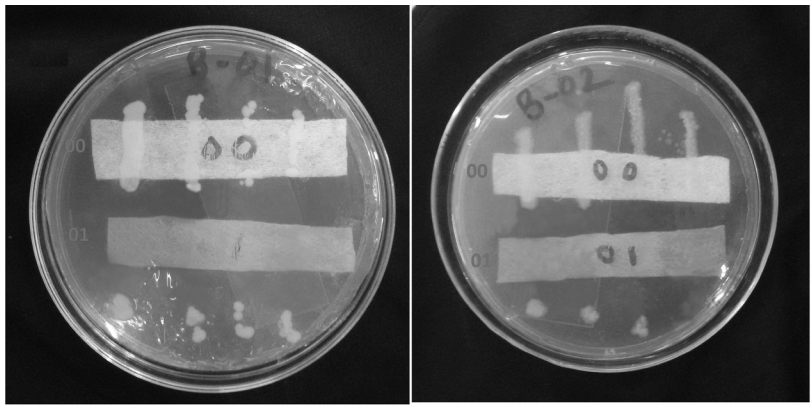

Figure 4.12 Antibacterial assessment of the Sample (00) and Sample (01) with Staphylococcus aureus (A) and Escherichia coli (B) respectively.

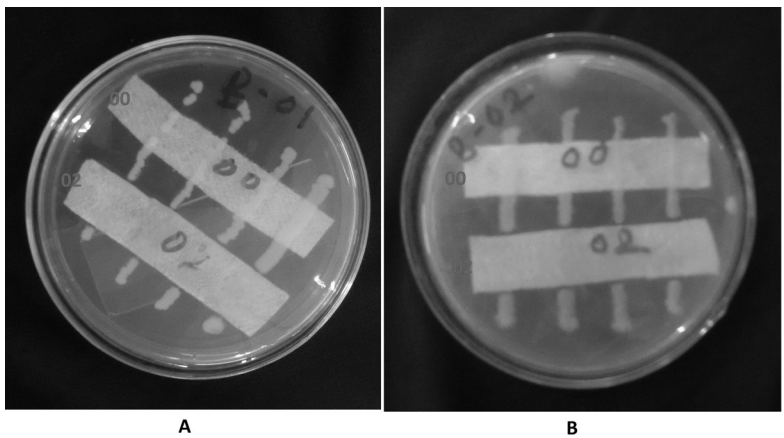

Figure 4.13 Antibacterial assessment of the Sample (00) and Sample (02) with Staphylococcus aureus (A) and Escherichia coli (B) respectively.

Thus sensible antibacterial activity against both the bacterial culture has been realized for Sample 01 mainly due to presence of active antibacterial composition of AgNPs. But in the absence of such active group in the morphological structure (see Fig. 4.8 a-d) of sample 02 has not allowed it to illustrate significant antibacterial activity. Bacterial inhibition can be seen within the range of the fabric sample only against both the bacterial culture in the present study (see Fig. 4.13, a-b).

This behaviour was mainly attributed to the capping of active silver component to the similar nano particle size in the structure of sample 01. The argument finds support from morphological study (ESEM) done for sample 02. Thus Milkweed leaves extract added during the synthesis process was not allowed nano silver particles to be get converted into macro/micro particles otherwise, thus accomplished successfully role of capping agent in the present study. However, as an individual as well as in compound, Milkweed leaves extract remain at par to show its potential for antibacterial activities for the selected concentration level.

4.4 Conclusions

Green synthesis of silver nano particles (AgNPs) was carried out by using self-sufficient antibacterial milkweed leaves extract. In situ synthesis and deposition of the nanoparticles and milkweed leaves extract on PV non-woven fabric were accomplished by cold dipping technique. The composite have not shown remarkable change in their physical parameters; GSM and thickness and executed desirable low stress properties; bending modulus and crease recovery. Comfort associated properties; air permeability and OMMC have shown positive inclination for AgNPs/ PV composite.

Nano Characterisation was done by ESEM and EDAX, Elemental mapping and statistical distribution have revealed the uniform distribution and formation of the silver nano particles on the surface of the green synthesized AgNPs/ PV non-woven composite textile material. The elemental assessment (EDAX) spectrum has confirmed presence of the composition of silver (Ag) in the green synthesized AgNPs/ PV non-woven composite (sample 01). The green AgNPs/PV composite has exhibited significant antibacterial properties against both bacterial culture viz, Staphylococcus aureus and Escherichia coli. However Milkweed leaves extract, although possess natural antibacterial potential, but at the selected concentration in the composite (Sample 02), did not show remarkable antibacterial activity against the both bacterial culture. The greatest benefit of green synthesis technique is; a simple technique without use of any surfactant, binder or any toxic chemical. Thereby it can serve well

as eco-friendly way for developing antibacterial capabilities enriched bio medical products.

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TECHNICAL ARTICLE

Reinforcing biomedical gadgets with nano-composite textiles: Present scenario at a glance

Rainish V. Radadiya, Tasnim N. Shaikh and Bharat H. Patel

Abstract

Textile materials have been given prime considerations in medicinal health care field irrespective of their external or internal application on human body. Invention of nano technology has remarkably contributed in re-engineering of textile materials used for biomedical application. Nano composites so produced offer benefits of more than one material with added higher durability and non-toxicity critically required for the protection against major culprits like; bacteria, fungi etc. prevailing in this field. The paper presents a brief summary of such nano composite textiles either used or developed by various researchers in the field.

Keywords: Biomedical, Healthcare, Nano-composite, Textile Materials

Introduction

Biomedical and biomedical engineering are same sounding but discrete streams of engineering. Biomedical is the term associated with the application of technology and engineering to living beings, especially the design and employment of medical equipment. On the other hand biomedical engineering is the application of engineering principles and design concepts to medicine and biology for healthcare purposes like diagnostic or therapeutic purpose. Biomedical engineering field is thereby bridges the gap between engineering and medicine by combining the design and problem solving skills of engineering with medical and biological sciences and facilitates in advance health care treatment. The work done in biomedical engineering confines more towards research and development, spanning a broad array of subfields or say based on well-tuned multidisciplinary approach. This can be well realized from the commercialized applications like development of biocompatible prostheses, various diagnostic and therapeutic medical devices ranging from clinical equipment to micro-implants, common imaging equipment such as Magnetic Resonance Imaging (MRIs) and Electroencephalography (EEGs), regenerative tissue growth, pharmaceutical drugs and therapeutic biological.

Biomedical textiles

Biomedical textile is one of the branch of biomedical engineering and deals with the development of textile products and constructions, for medical and biological applications. Such products have been found very useful in first aid, clinical as well as hygienic purpose. According to their application area biomedical textiles are broadly classified into four groups, viz; a) protective and health care textiles, b) External hygiene products, c) Implantable materials and d) extracorporeal devices [1,2]. Figure 1 illustrates such bifurcation of popularly used products.

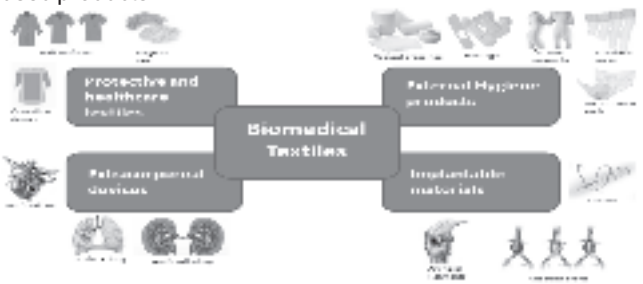

Figure 1: Bifurcation of biomedical textiles as per end use application

Collectively biomedical textile is the combination of base textile material with added bioactive properties. Form (e.g. fiber, yarn, fabric) and type (e.g. cotton, viscose rayon, lycra etc.) of base textile material differs as per the suitability in a peculiar end use region and many a times more than one material variant found suitability for a given application area. Some of the commercialized application areas along with base material employed for the purpose is summarized in Table 1. [2-12].

Table 1 : Base textile material used in commercialized application area

A. Non-implantable materials

Absorbent pad	• Non-woven made up from Cotton, viscose, or Lyocell Wound-contact layer
Wound-contact laye	• Woven, non-woven, or knitted made up from Alginate fibre, chitosan, silk, viscose, lyocell, or cotton
Simple non-elastic and elastic bandages	• Woven, or non-woven made up from Cotton, viscose, Lyocell, Polyamide fibre, or elastomeric-fiber yarns
High-support bandag	• Woven, non-woven, or knitted made up from Cotton, viscose, Lyocell, or elastomeric fiber Yarns
Compression Bandages	• Woven, non-woven, or knitted made up from Cotton, viscose, Lyocell, or elastomeric fiber Yarns
Orthopaedic Bandages	• Woven, non-woven, or knitted made up from Cotton, viscose, Lyocell, polyester, polypropylene, or polyurethane foam
Plasters	• Woven, non-woven, or knitted made up from Cotton, viscose, plastics film, polyester fibre, glass fibre polypropylene fibre
Gauze dressing	• Woven, non-woven, or knitted made up from Cotton, viscose, lyocell, Alginate fibre, Chitosan
Wadding	• Non-woven made up from Viscose, cotton linters, wood pulp
Gauze dressing	• Spun laid, or needle punched Nonwoven made up from Polylactide fibre, polyglycolide fibre, carbon

B. Implantable materials

Sutures	• Monofilament, or braided Biodegradable made up from Collagen, catgut, TC2F polyglycolide and polylactide fibre
Non-Biodegradable Sutures	• Monofilament, or Braided biodegradable made up from Polyester fibre , polyamide fibre, PTFE fibre, polypropylene fibre, polyethylene fibre
Arty mat tendon	• Woven, or braided made up from PTFE fibre, polyester fibre, silk, collagen, polyethylene fibre, polyamide fibre
Artificial ligament	• Braided made up from Polyester, carbon fibre, collagen
Artificial skin	• Nonwoven made up from Low density polyethylene fibre Artificial cartilage Chitin
Eye-contact lenses and Artificial cornea	• Poly (methyl methacrylate) fibre, silicon fibre, collagen
Artificial joints/ bones	• Silicone, polyacetal fibre, polyethylene fibre
Vascular grafts	• Woven, or knitted made up from PTFE fibre, polyester fibre
Wadding	• Non-woven made up from Viscose, cotton linters, wood pulp
Heart valves	• Woven, or knitted made up from Polyester fibre

C. Healthcare / hygiene products

Surgical gowns	• Woven, or nonwoven made up from Cotton, polyester fibre, polypropylene fibre
Surgical caps	• Nonwoven made up from Viscose
Surgical masks	• Nonwoven made up from Viscose, polyester fibre, glass fibre
Surgical drapes, cloths	• Woven, or nonwoven made up from Polyester and polyethylene
Surgical hosiery	• Knitted made up from Cotton, polyester fibre, polyamide and elastomeric fibre yarns
Blankets	• Woven, or Knitted made up from Cotton, polyester fibre
Sheets, pillow cases	• Woven made up from Cotton
Uniform	• Woven made up from Cotton, polyester fibre
Protective clothing, Incontinence diaper /sheet, Cover stock	• Nonwoven made up from Polyester fibre, polypropylene fibre

Base textile materials

Different forms of textile structures, viz; fiber, yarn, woven, nonwoven, knitted, and composites have found place in medical and healthcare applications. No doubt that the kind of textile material used for biomedical application is motivated by its end use need [1-17]. The major variants considered in their designing are briefly summarized below:

Function

The textile material needs to fulfil the particular purpose/ function for which it has been designed; e.g. swabs require good absorbency.

Biocompatibility

This criterion involves reaction of the textile material with blood and tissue present in the body. Extent of reaction differs as per degree of contact made, viz; internal or external use. An internal implantable device has more potential for reaction than an external device and is, therefore, subject to tighter regulations [17-32]. Just for an example; artificial ligament is permanent and is able to react with blood cells and the surrounding tissue, whereas external bandage that is temporary and only contacts the outer skin tissue.

Cost

This variant is affected by either one or sum of more than one of cost influencing factors like; raw materials, manufacturing process etc., but defined on the basis of product end-use performance requirements;

- Surgeons gowns and swabs should have a low production cost
- Vascular grafts and artificial skin will have a relatively high production cost

Product approval

Each country has its own regulations and standards for medical textiles.

Thus depending on the specific end-use, the product should meet the particular demands irrespective of application area; internal or external. However, they should invariably comprise of basic bioactive properties, especially antimicrobial [19 - 24].

Bio active material

Antimicrobial, bactericidal properties of biologically active metals, known as bio metals are known to human beings since long back in the form of “grandma recipe”. Silver, copper, zinc, etc. in the form of salts, complexes, colloid particles, "silver" water were commonly used house hold remedies for many dieses [2-10, 24-36].

Modern scientists offer new complex antibacterial and antiviral Nano systems on the basis of metal oxides or intermetallic oxide compounds, such as TiO_2, SnO_2, ZnO_2 and SiO_2. These compounds are functionalized by organic or organometallic

molecular structures capable to connect ions of the transition bio metals, such as Ag+ and Cu++. Such systems can be used for manufacturing of medicinal and non-medicinal means, dermatological compounds and creams, for bactericidal modification of surfaces and coatings in living quarters, industrial and specialized premises [2-29, 24-42]. The popularly used bioactive materials along with their medicinal propertis are mentioned in Table 2.

Table 2 : Bioactive materials along with their medicinal properties

Bioactive materials/ molecule	Source	Bioactivity/application
Artemisinin	Plant Artemisia annua	Malaria
Ivermectin	Soil actinomycete Streptomyces avermitilis	Parasitic helminth infections: Lymphatic filariasis and Onchocerciasis
Paclitaxel	Pacific yew tree, Taxus brevifolia	Cancer
Lovastatin	Mushroom Aspergillus terreus	Atherosclerosis/heart disease
Toxin / proteins	Bacillus Thurigiensis	Anticancer effect on leukaemic cells
Quercetin	Synthetic	Effect on diabetic vascular tissue
Astaxanthin	Algae	Anticancer and antioxidative activity
Carrageenan	Algae (Seaweeds)	Protective effect against UVR-induced toxicity and Mutagenicity
Silk	Silkworm	Drug delivery system
Streptomycin	Actinobacterium Streptomyces griseus	Antibiotic
Zingerone	Ginger	Anticancer and apoptotic
Gingerol		activity
Coumarins	Synthetic	Ant proliferative activity on breast cancer cell lines
Curacin A	Marine cyanobacterium	Antitumor activity
Monoclonal antibody	Recombinant Proteins	Diagnosis of house mite allergens
Polyclonal and monoclonal	Recombinant	Filarial antigen detection
antibodies	Molecules	assay for Brugia malayi
Textile dyes	Synthetic	Genotoxic effects on algae and animal cells
Zorbamycin	Streptomyces flavovirdis	Antitumor
Kanamycin	Streptomyces kanamyceticus	Antibacterial
Kanglemycin C (K-C)	Nocardia mediterranei var. Kanglensis	Immunosuppressive
Rapamycin	Streptomyces hygroscopicus	Antifungal
Pandavir (nigericin)	Streptomyces hygroscopicus	Affects ion transport and ATPase activity
Avermectin	Streptomyces avermitilis	Anthelminthic
Oligomycin	Streptomyces avermitilis	Cell growth inhibitor
Resormycin	Streptomyces platensis	Herbicidal, antifungal
Neihumicin	Micromonospora neihuensis	Cytotoxic

Significance of composite as biomedical

Common problem across the hospitals and healthcare institutions is microbial contamination of textile fabrics used by doctors, nurses, patient, visitors and others. These can lead to infections and consequently to cross-infections. Not only that hospital acquired infections are prolonging the healing of patients, and causing potential risks for serious illness, but are also representing the extra costs to the health service [1-5]. Therefore, it is essential to reduce transmission of harmful microorganisms and spreading of the secondary infections within a curative environment. Accordingly, it is extremely important that medical textile in all forms meet the demands for antimicrobial protection and adds to its therapeutic value. They should ensure adequate protection against microorganisms, biological fluids and aerosols, i.e. impermeability for microorganisms in wet and dry atmospheres, and also for air-borne microorganisms as disease transmission prevention is a very important consideration for intra-corporeal or implantable devices within the human body (e.g. vascular grafts and sutures) and for extracorporeal devices such as catheters and hollow fibres for dialyzers [5-12]. Controlling the undesirable effects of microorganisms on textiles is becoming an important issue, especially within the medical textile industry. Under such circumstances use of pure textile material will not serve the purpose, it should be a composite textile produced in combination with active membrane group as per the need of application area [5-14, 29-42].

Different means used for the production of textile composite with bioactive properties

Textile material's bioactive properties are either added or enhanced by different means but commonly used practices include; i) Chemical diffusion and ii) Coating and Lamination. The active membranes in the former one operates on controlled release mechanism (Leaching), whereas in the later one remain bound on the surface [17-34]. Their associated advantages and disadvantages are briefly summarized in Table 3.

Table 3 : Advantages of bioactive membrane application methods

Application mean	Advantages	Disadvantages:
Chemical Diffusion	- Effective against microbes on the fibre surface or in the surrounding environment	- 'Reservoir' depletion; finish on longer effective - Can cause health problems (in some cases) - More prone to microbial resistance - Poor washing durability
Coating and	- Acts only on microbes on the fibre surface Lamination - Developing microbial resistance is less possible - Good washing durability (depends on the material affinity and agent chemistry)	- Might become deactivated, even though present on the surface - Can be abraded away - Use of auxiliary chemicals, cross-linking agent and other might affect biocompatibility - Blocking of functional groups responsible for antimicrobial action (less effective)

The treatment given to the textile material in the course of conversion to biomedical material must be effectively permanent and covering wide spectrum of bacteria and fungi. They should not cause problems such as irritation to the wearer by the virtue of physical properties, appearance, handle, comfort and odour mainly. They should not be toxic, pollute environment, affected by surroundings over and above produced at reasonable cost [2-8].

But both the application modes were failed mainly at economy and comfort fronts during classical edge. This was attributed to the use of micro/ macro forms of bioactive materials which provides smaller active surface area required more consumption of active membrane, made product stiffer; inconvenient to wearer and also adversely impacted base fabric properties as well as cost effectiveness. Apart from these most of active membrane origin belongs to chemicals and processing of them in larger volume usually resulted in unwantedly pollution [2-8].

Nanoparticles and their importance as an active membrane

In recent years metal nanoparticles have attracted focus of many researchers in the field due to their unique mechanical, magnetic, electronic, optical and chemical properties. Nano particles are found significantly effective from those of bulk materials as an active membrane. This is mainly attributed to their very tiny sizes and due to that larger projected specific surface area although used in considerably low stuff. Both the positive ways out can overrule limitations of macro/ micro edge biomedical products in terms of cost as well as performance [22-56]. Varieties of preparation routes have been reported for the metallic nanoparticles as shown in Figure 2.

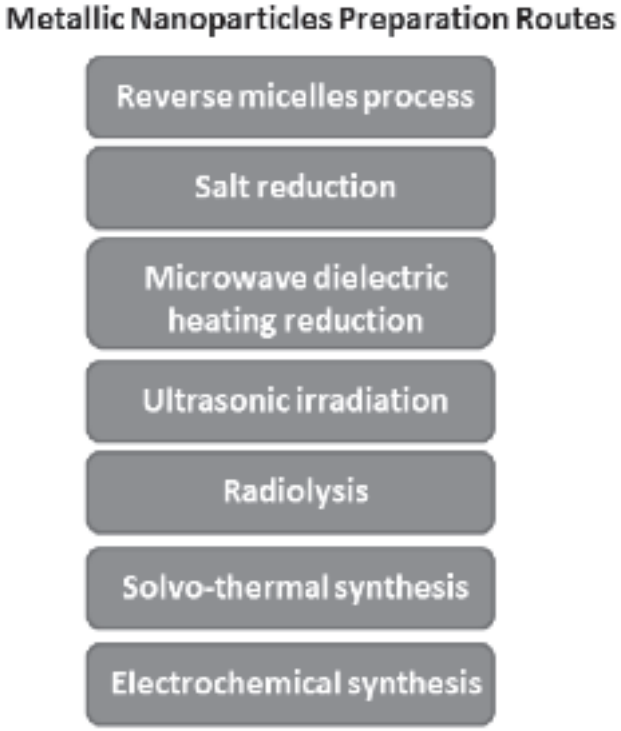

Figure 2: Metallic nanoparticles preparation routes

Bio-medical application of nano particles

The use of nanoparticles as antibacterial agent is relatively new. Because of their high reactivity due to the large surface to volume ratio, nanoparticles play a crucial role in inhibiting bacterial growth in aqueous and solid media [2].

In the case of Nano particles, the Nano crystals are usually grown from chemical (like Ag+, Cu++) solutions. The ions come from a salt (e.g silver nitrate- $AgNO_3$). The ions are first reduced to atoms by means of a reducing agent. The obtained atoms then nucleate in small clusters that grow into particles. Depending on the availability of atoms, which in turn depends on the salt to reducing agent concentration ratio, the size and shape of the nanoparticles can be controlled [2-4].

On the other hand, in recent times, there is a growing interest in the synthesis of metal nanoparticles from the nature. For this purpose, biomass or extracts of different plants have been tried with success as reducing agents [2-5, 22-50].

Dragieva et al., found that the positive charge on the silver ions released from nanoparticles is crucial for its antimicrobial action through the electrostatic attraction between positively charged nanoparticles and negatively charged cell membranes of microorganisms [51].

Sondi et al., stated that by formation of “pits” in the Gram-negative bacterial cell wall with changes in the membrane permeability leading to death of bacterial cell [52].

Danilczuk et al., found that the generation of free radicals attacking membrane lipids and subsequent free-radical induced damages of membranes [53].

Singh et al., works on the bacterial surfaces and after penetration into the bacterial cell nanoparticles react with Sulphur-containing proteins and phosphorus-containing compounds such as DNA, disturbing their functions [54].

Krutyakov et al., founds that the silver nanoparticles act as independent biocidal agents, while silver ions play only secondary role, and mechanism of action of silver nanoparticles is quite different from that of silver ions [55].

Guzman et al., found that the nanoparticles of silver showed high antimicrobial and bactericidal activity against gram positive bacteria such as Escherichia Coli, Pseudimonas aureginosa and staphylococcus aureus which is a highly methicillin resistant strain [56].

Conclusion

The importance and potential of nano-technology in medical textiles has been assessed in the last few years. The development of biotechnologically functionalised textile materials requires improved fundamental understanding of the relevant parameters like, functionality, biocompatibility, costing, product approval and the development of novel biotechnological production processes for medical textiles. Development in surface characteristics of textile materials is of fundamental importance in the production of medicinal functionalised textiles. While a lot of research focuses on modification of surfaces by chemically or physically, the introduction of functionalities using nanotechnology is a relatively unexplored and new scientific area.

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This is to certify that Prof./Dr./Mr./Ms **Mr. Rainish V. Radadiya,** Assistant Professor, Textile Engineering Department, The M.S University of Baroda *has presented a paper titled* Effect of silver nano particles synthesized by milkweed (Calotropis) leaves extract on antibacterial activities of bio-medical composites textile materials *in the '5th International Conference on Industrial Textiles – Products, Applications and Prospects - InduTech 2020' on 21st - 23rd August 2020, organized by Departments of Textile Technology and Automobile Engineering, PSG College of Technology, The M.S. University of Baroda, Gujarat, Hof University of Applied Sciences Fachhochschule, Germany; Institute for Frontier Materials (IFM), Deakin University, Australia and Northwest Composites Centre, School of Materials, University of Manchester, UK in collaboration with Office of the Textile Commissioner, Ministry of Textiles, Govt. of India.*

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