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Hydrophobicity and antibacterial property of silica sol-chitosan-HDTMS treated silk fabric

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The present work aims to develop a hydrophobic surface with anti-bacterial capability on silk fabric with the help of silica sol gel, chitosan and hydrolysed hexadecyltrimethoxysilane (HDTMS) and to study the durability of coating in aqueous media (*p*H 7.4). Silica sol is produced by hydrolysing and condensing of tetraethoxysilane under alkaline conditions and then applied on silk fabric. This is followed by hydrophobization on the silica sol-treated surface of the fabric by applying hydrolysed HDTMS. Further, chitosan is applied on the silica sol-coated surface prior to HDTMS hydrophobization for its antibacterial property. The fabric is immersed in aqueous medium for 30 days to test the stability of the coating. The hydrophobicity of the fabric has been assessed by measuring the water contact angle and the antibacterial property is measured by using the AATCC-147 test protocol. The developed fabric exhibits antibacterial and hydrophobic properties. It is observed that there is a change in contact angle from 152.7˚ to 90.18˚ after 30 days of wetting and an increase in anti-bacterial activity, i.e. the zone of inhibition from 1 mm to 5 mm. The wicking properties of treated and untreated fabrics are measured to observe the impact of coating on wicking. SEM analysis has been carried out to determine the change in surface morphology of the fabric, and FTIR is used to determine the changes in the surface functional groups after coating and after wetting of coated fabric for 30 days in aqueous media. Such hydrophobic silk fabric with an anti-bacterial property may be useful for the development of natural biomaterial and other technical textile products.

Keywords: Antibacterial property, Chitosan, Hexadecyltrimethoxysilane, Silk, Tetraethoxysilane

1 Introduction

Silk is a very popular natural protein fibre derived from the silk cocoon. This is the most preferred high-quality textile material because of its different unique properties, which include delicate appearance, softness, toughness, breathability, superior wear warmth, comfort, and biodegradability. Silk fibre has numerous uses in various fields, like apparel, home furnishings, aircraft seats, and sofa covers[1–3]. Moreover, silk protein can also be used in biomedical applications, including sutures, prosthetic arteries, and bandages, as its amino acid backbone is similar to that of human skin[4].

However, silk has some disadvantages, like staining, creasing, and the growing tendency of fungi and bacteria. This is because, the silk is hydrophilic in nature due to the presence of carboxyl, hydroxyl and amine groups on the silk polypeptide backbone[1,5–9]. In addition to that, fabrics manufactured of natural fibre yarn have micropores within the yarn's three-dimensional construction. Since bacteria may easily hide and multiply within the micropore, these pores enhance the chances of bacterial colony formation[10–13]. All the problems can be avoided by developing a hydrophobic surface with antibacterial properties. Moreover, silk biomaterial exhibits highly unfavourable cellular reactions than synthetic fibres and the cellular response decreases after about a week[14]. This cellular response is due to the high hydrophilicity of silk fibre, because the hydrophilic surface adsorbs plasma proteins, like coagulation factor XII, HMWK and prekallikrein after implantation within the body[15,16]. The problem associated with cellular response can also be avoided by developing a surface which is highly hydrophobic and has a contact angle greater than 120˚[16–18]. Hence, there is a need to develop a hydrophobic surface with anti-bacterial properties on silk fibres to overcome the problems.

Surfaces can be classified as hydrophilic or hydrophobic, depending on how a water drop interacts with it. Measurements of the water contact angle are frequently used to describe the wettability of the surface[19,20]. When the water contact angle is less than 90˚, it is known that the surface is hydrophilic;

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when it is greater than 90°, it is hydrophobic[19,21]. Hydrophobic surfaces can be created on silk fabric using a variety of techniques, including sol-gel, plasma treatment, layer-by-layer deposition, and chemical vapour deposition[1–5,22–25]. In this regard, novel methods like sol-gel techniques have been developed to make new environment-friendly chemical modifications of textiles. The sol-gel procedures are a straightforward and adaptable method for creating inorganic or organic-inorganic coatings that are very homogeneous at the molecular level and have exceptional physical and chemical properties[23]. In addition to that, the sol-gel method, using silica nanoparticle, has many other advantages over other methods, such as non-toxicity, biocompatibility, environmental friendliness, non-fluorinated nature, good hemocompatibility, etc. and lowers the risk of encrustation when it comes in contact with body fluids. It shows a strong experimental repeatability[26–28].

Natural, organic, and inorganic antibiotics can be used to create antibacterial characteristics on natural fibres. Chitosan and its derivatives are well-known natural antibiotics[29]. Apart from other antibiotics, chitosan is the most common polysaccharide in nature. Chitosan has many advantages over others including its adaptability, non-toxicity, biodegradability and antibacterial properties[30–32].

Furthermore, for utilization in medical applications, the coating stability in aqueous media (*p*H 7.4) is crucial. Because wounds typically take four to six weeks to heal[33]. The *p*H of physiological body fluid is usually around 7.4. Hence, textile materials used for implant devices will come into touch with physiological body fluid for a longer period (at least four weeks)[34]. According to the literature, there is hardly any research which has been done to show how exposure to such aqueous media affects the surface's antibacterial activity and hydrophobicity over time after coating.

Therefore, in this study, silica sol, HDTMS (hexadecyltrimethoxysilane), and chitosan were used to create a hydrophobic surface with antibacterial activity on degummed woven fabric. The coating durability was evaluated by immersing the developed fabric in aqueous media (*p*H 7.4). Moreover, the developed fabric has shown good hydrophobic and antibacterial properties and thus there is a possibility of using this developed fabric in implantable devices.

2 Materials and Methods

2.1 Materials

The work was carried out by utilizing silk woven fabric manufactured by local industry. The details of fabric used for the study are given below:

Property	: Value
Silk yarn linear density, tex	: Warp 20 and weft 28
Weave structure	: Plain
Fabric areal density, g/m^2	: 113
Fabric density, epcm×ppcm	: 21× 23

Tetraethoxysilane (TEOS), ethanol, NH_4OH, HDTMS, chitosan, and acetic acid are the chemicals used in this work. All the chemicals were procured from Sigma-Aldrich. Moreover, the silk fabric was first degummed using $NaHCO_3$ and detergent to remove sericin.

2.2 Methods

2.2.1 Synthesis of Silica Sol

The Stober method[35] was used to synthesise the silica sol. The silica sol was prepared by hydrolysis and condensation of tetraethoxysilane (TEOS) using ammonium hydroxide (NH_4OH) as a catalyst in ethanol as a solvent. Firstly, ammonium hydroxide (5 mL) was dropwise mixed with 100 mL of solvent (ethanol) and stirred for 30 min at 60°C. After that, tetraethoxysilane (6 mL) was added drop by drop in the solution and further, the solution was stirred for 90 min to prepare silica sol[35–37].

2.2.2 Alkylsilanol Solution Preparation of HDTMS

HDTMS (0.75%) was mixed with 100 mL of ethanol and the solution was stirred for 60 min to prepare alkylsilanol solution of HDTMS. The solution *p*H was maintained at 5.0 during alkylsilanol solution preparation by using acetic acid[36].

2.2.3 Chitosan Solution Preparation

Chitosan solution was made by adding chitosan (2g) into 1000 mL of distillate water with acetic acid (2%) in it. After that, the solution was sonicated for 30 min to form a homogeneous mixture.

2.2.4 Treatment of Silk Fabric

The degummed silk woven fabric was dipped in silica sol solution for 20 min. After that, a padding mangle was used to pad it, while maintaining 80 - 100% expression. The padded silk fabric was then dried at 80°C for 3 min. Eventually, the silica sol-treated fabric was again soaked with prepared chitosan solution. After that, the fabric was dried at 80°C for 5 min and cured at 140°C for 3 min. Following this, the chitosan-treated silk fabric was immersed into alkylsilanol solution of HDTMS for 1 h. Thereafter, the immersed silk fabric was dried at room temperature and subsequently cured at 120 ºC for 1 h. The process of developing hydrophobic and antibacterial properties on silk fabric is given in Fig. 1.

The durability of fabric coating was investigated by dipping the fabrics in aqueous media (*p*H 7.4) for 3 days, 12 days, and 30 days respectively. After that, the changes in anti-bacterial properties and contact angle were observed. Table 1 shows the various fabric samples along with their codes for both before and after treatment.

2.2.5 Characterization Technique

The following techniques were used to characterize the developed fabric samples:

Contact Angle Measurement — The surface wettability of the uncoated, coated, and dipped fabrics were

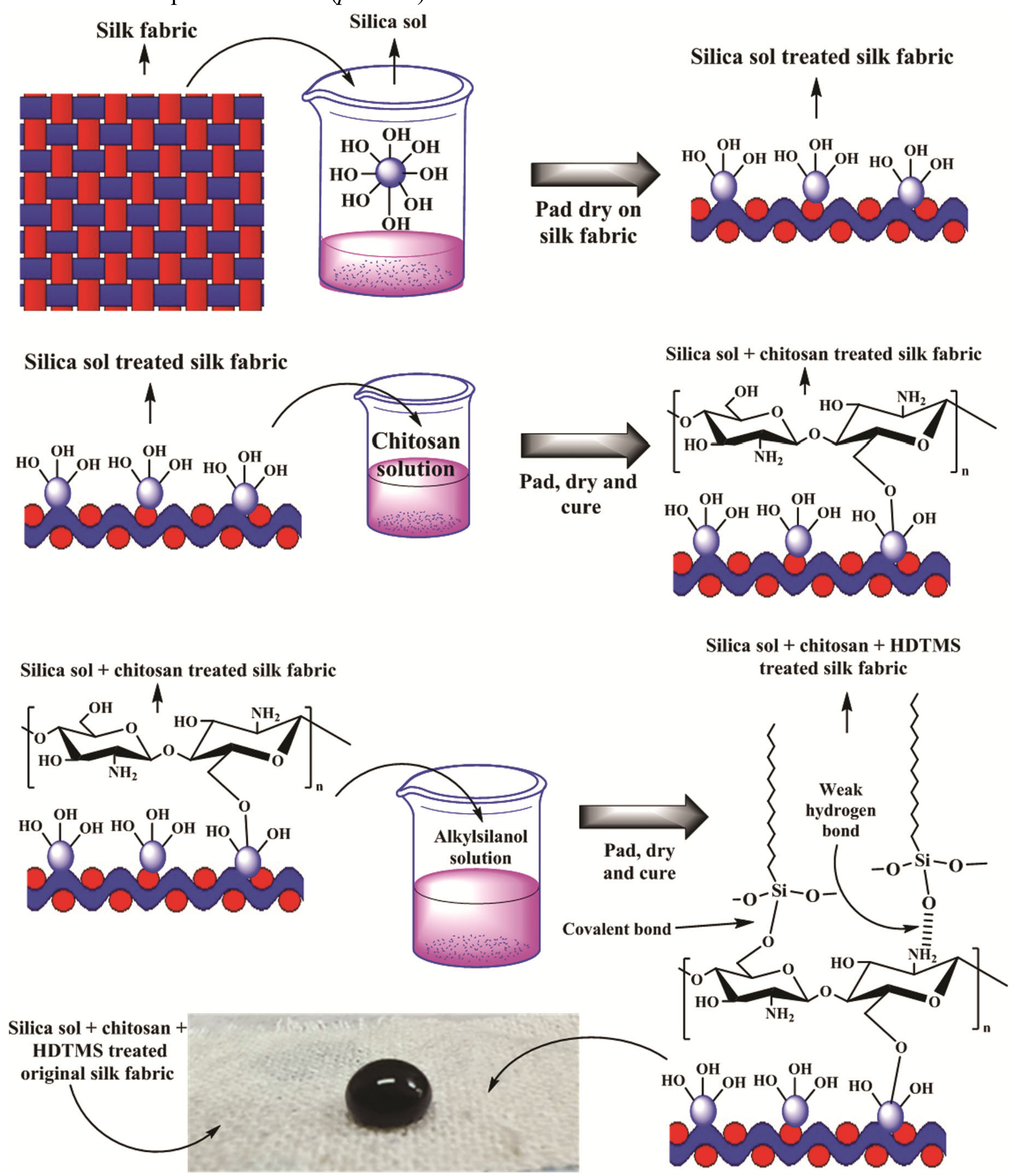

Fig. 1 — Development of hydrophobicity and anti-bacterial property on silk fabric based on silica sol, chitosan and HDTMS

Table 1 — Different silk fabrics before and after coating

Sample code	Fabric
S1	Uncoated silk fabric
S2	Coated fabric (silica sol + chitosan (0.2%) + 0.75 % (V/V) HDTMS)
S3	Coated fabric dipped in aqueous media for 3 days
S4	Coated fabric dipped in aqueous media for 12 days
S5	Coated fabric dipped in aqueous media for 30 days

assessed by measuring the contact angle. The drop shape analyzer (Kruss, Germany) is used to measure the contact angle.

Determination of Wicking Properties — The wicking properties of coated and uncoated fabric were measured by the reported method[38]. The fabric of 200 mm × 25 mm was cut for use to measure the wicking property. Thereafter, a stainless-steel scale was mounted to the clamp. Then a glass reservoir was put at the bottom of the frame. Care was taken to prevent the fabric from twisting along its axis. Using a stopwatch, the rise of liquid along the yarn was measured. When there was no change in the height of the liquid in the fabric, the test was terminated.

Measurement of Anti-bacterial Property — The AATCC-147 test protocol was used to assess the antibacterial activity of silk-coated fabric for *E. coli* bacteria [6,39,40]. Agar-Agar (2%) and Luria broth (2%) were used to make culture media in distilled water. An autoclave was used to sterilise the petri dish, tip, forcep, L rod, and media for 15 min at 120°C and 1.05 kg/cm^2 (15 psi) pressure. Petri dishes, L rods, forceps, 1 mL tip pipette and fabrics were once again sterilised by using UV for 15 min after the cultured media has been placed into the plates. After UV sterilisation, 100 µL of bacteria were distributed in the petri dish with agar media by using an L rod. The 23 mm diameter fabric was then positioned in the middle of the petri plate. Paraffin paper was used to cover the petri plates. Petri plates were then incubated for a further 24 h at 37 ºC. The zone of inhibition was calculated to test the antibacterial activity of the fabric sample.

SEM Analysis — The surfaces of the uncoated, coated, and dipped silk fabrics were studied using SEM, ZEISS, Sigma 500 VP, Germany. Silk fibres are nonconductive, that is why, the silk fabric was coated with a thin gold layer before SEM measurement.

FTIR Analysis — The changes in functional groups of uncoated, coated, and dipped fabric samples were identified by FTIR, BUKER, ALPHA II, Germany.

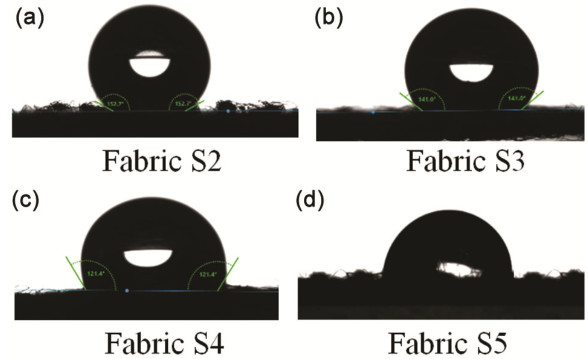

Fig. 2 — Contact angle of treated without dipping (S2), 3 days of dipping (S3), 12 days of dipping (S4) and 30 days of dipping (S5) samples

The measurements were carried out from 600 cm^{-1} to 4000 cm^{-1} wavelengths.

Measurement of Tensile Strength — The tensile behaviour of the yarns unravelled from coated, 12 days dipped, and 30 days of dipped fabric was evaluated using a Tinius Olsen Universal Testing Machine (UTM). For measuring the tensile strength of the unravelled yarn, the standard test norm ASTM D2256/D2256M − 21 was used.

3 Results and Discussion

The hydrophobicity of different silk fabric surfaces (before and after coating) is determined by measuring the water contact angle (WCA) using drop shape analyser. The measured values of contact angles for samples S1, S2, S3, S4 and S5 are 'not measurable', 152.7˚, 141.0˚, 121.4˚ and 90.18˚ respectively (Fig. 2). It is observed that the contact angle of uncoated degummed silk fabric is not measurable because the droplet of water has been disappeared immediately after dropping on the degummed silk fabric surface and the instrument indicated as 'not measurable'. This is due to the presence of a large number of hydroxyl, carboxyl and amine groups on the surface of uncoated silk fabric[2,5], as well as the water being wicked through the capillaries of silk yarn. It is evident that the contact angle value of silica sol, chitosan and HDTMS treated silk fabric sample (S2) is enhanced to 152.7˚, which is classified as highly hydrophobic surfaces (WCA > 90°)[3,19,21].

The surface roughness of treated silk fabric sample (S2) is enhanced at the nano level due to the application of silicon sol and followed by the subsequent HDTMS treatment which reduces the surface free energy by blocking polar groups. This combined effect i.e. blocking of polar groups and increase of surface roughness in the nano level result in the creation of a highly hydrophobic property for fabric sample S2[2,3,22,36,41]. The original images from drop shape analyser are provided in Fig. 2 for the surfaces of fabric samples S2, S3, S4 and S5. It is observed that the contact angle is decreased after dipping in aqueous media. The contact angle after 3 days dipping and 12 days dipping are 141.0˚ and 121.4˚ respectively. However, the contact angle is reduced to a large extent after 30 days of dipping and reached a value of 90.18˚, indicating its hydrophobic nature even after one month. The reduction in contact angle after dipping in aqueous media may be due to the crack of silica coating on the silk fibre surface as a result of swelling of the inner silk fibre. It is supported by past studies which observed the contact angle reduction after 25 to 30 washings, and the cause of reduction has been mentioned as the formation of cracks in the silica coating induced by the swelling of the fibre inside[36,42,43]. Secondly, the cause of the decrease in contact angle may also be due to the leaching out of HDTMS from the fibre surface owing to the breaking of weak bonds[42,44].

The anti-bacterial activity of fabrics S2, S3, S4, and S5 is determined using the agar-agar diffusion test method by utilizing *E. coli* bacteria. The zone of inhibition (ZOI) is measured for all samples (Fig. 3). The ZOI of fabric S2 (treated) is around 1 mm. However, the ZOI of dipped fabric samples S3, S4, and S5 is enhanced as compared to treated fabric sample S2. The increase of ZOI in the dipped fabric may be attributed to the exposure of amide groups of chitosan after dipping. The exposure of amino groups is probably due to the cracks in the coating on the fabric surface and the breakage of weak bonds[36,44]. Moreover, the exposure of chitosan after dipping can also be confirmed by observing peaks of FTIR spectra at 1508 cm^{-1} and 881 cm^{-1} (Fig. 4).

The presence of chitosan in the treated fabric sample (S2) and dipped fabric samples (S3, S4, and S5) exhibit antibacterial activity with varying extents. There are two suggested mechanisms for chitosan's antibacterial effects, viz. (i) the polycationic structure of the amino group (NH_2) in chitosan interferes the bacterial metabolism by anchoring on the bacterial cell and (ii) chitosan binds to DNA and inhibits mRNA synthesis[45,46].

The wicking behaviour of the uncoated degummed silk fabric sample (S1) and coated silk fabric sample (S2) are observed by measuring the wicking height. The maximum wicking height of uncoated fabric (S1) and coated silk fabric (S2) are 14 ± 1 cm and 0 cm respectively (Fig. 5). It is observed that the uncoated silk fabrics have shown a wicking effect (wicking height 14 ± 1 cm). This may be due to the presence of capillaries inside the yarn structure and between the yarn surfaces[47,48]. However, the silk fabric coated with silica sol, chitosan and HDTMS has shown almost zero wicking.

Pores and capillaries inside yarns as well as fabric structure are blocked due to the coating on the fabric surface[49]. Therefore, the blocked pores inside the yarn as well as the fabric surface are restricted the capillaries action in coated fabrics. The blocked pore/capillaries of spun yarn silk fabric can be beneficial for medical applications because the bacteria will not be able to proliferate and hide inside the yarn structure due to zero wicking of the coated fabric.

SEM images are used to examine the surface morphology of silk fabric after coating and dipping in aqueous medium. The surface images of the silk fabric before coating (S1), after coating (S2) and dipping (S5) are shown in Fig. 6. The treated silk

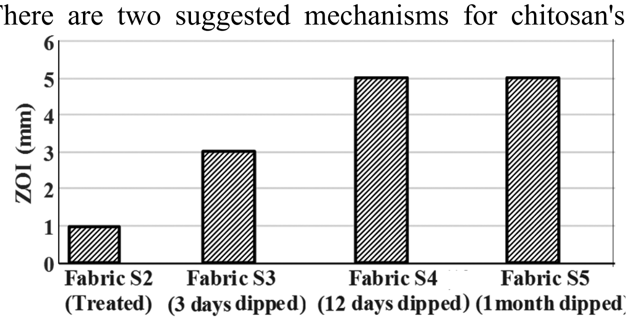

Fig. 3 — ZOI of treated and dipped fabric

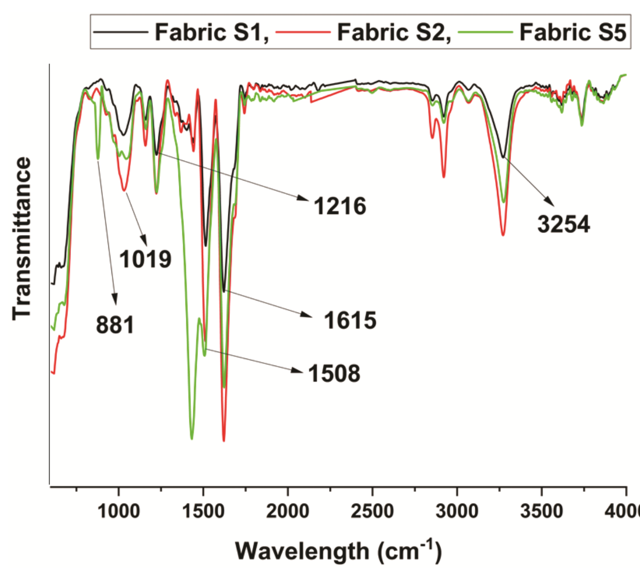

Fig. 4 — FTIR spectra of uncoated silk fabric sample (S1), silk fabric coated with silica sol, chitosan, and HDTMS (S2), and coated fabric sample after 1 month of dipping (S5)

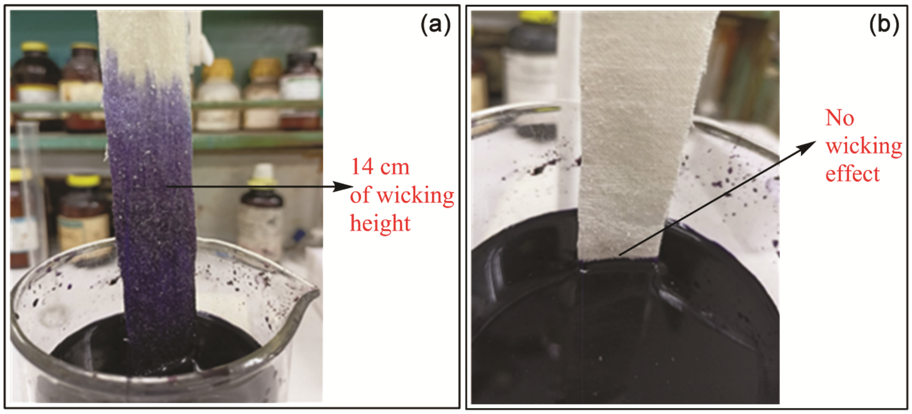

Fig. 5 — Wicking of (a) uncoated and (b) coated fabrics

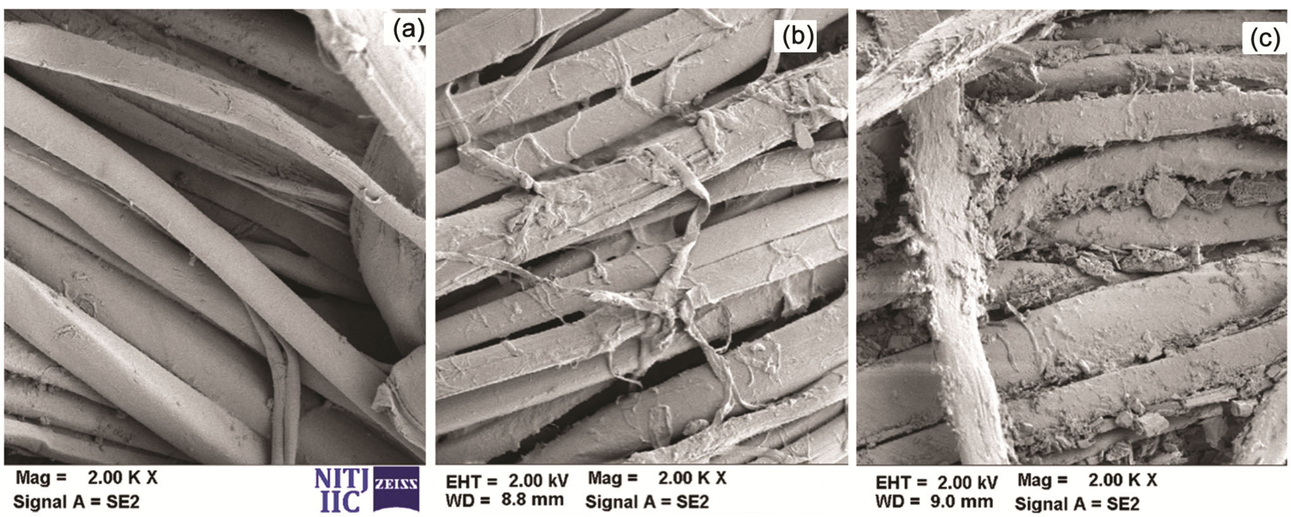

Fig. 6 — SEM images of fabric (a) S1 (uncoated), (b) S2 (coated), and (c) S5 (after 30 days of dipping)

fabrics have shown more roughness than the untreated silk fabrics, which supports the attachment of the silicon and chitosan nanoparticles on its surface [3,24]. Furthermore, the 30 days dipped sample (S5) has shown excessively increased surface roughness. This may be due to fibre degradation of silk after 30 days of dipping in aqueous media[50].

FTIR spectra of the uncoated silk fabric sample (S1), silk fabric sample coated with silica sol, chitosan, and HDTMS (S2), and coated fabric sample after 30 days of dipping (S5) are shown in Fig. 4 with baseline correction. It is observed that the absorption bands of amide I, amide II and amide III appear at 1615 cm^{-1}, 1508 cm^{-1} and 1216 cm^{-1} respectively for samples S1, S2 and S5. The fundamental stretching vibration of various hydroxyl groups is shown by the broad band between 3000 cm^{-1} and 4000 cm^{-1} for samples S1, S2, and S5 respectively. The peak intensity at 1019 cm^{-1} is enhanced for the treated fabric sample S2 as well as for sample S5, may be due to the presence of the Si-O-Si functional group. Moreover, the peak intensity of spectra 1216 cm^{-1}, 1508 cm^{-1}, 1615 cm^{-1} and 3254 cm^{-1} are changed after coating with silica sol, chitosan, and HDTMS, which is similar to the previous finding[3]. It is also found that the peak intensity of the spectra 1508 cm^{-1} is increased after 30 days of dipping in aqueous medium (*p*H 7.4), may be due to exposure of the NH_2 group of chitosan after the leaching of surface coating material. In addition to that, the spectra at around 881 cm^{-1} appear in dipped sample S5 for the saccharide structure of chitosan[51]. Therefore, the spectra at 1508 cm^{-1} and at 881 cm^{-1} are supporting the exposure of amino groups of chitosan[52]. Moreover, the exposure of amino groups may be due to the cracks on the coated surface. The increase in ZOI data for the S5 sample also supports this observation.

The changes on the silk fibre surface as observed from SEM after dipping can be explained by comparing the tensile strengths of yarn unravelled from treated fabric S2, S4 (12 days dipped fabric), and S5 (30 days dipped fabric). The tensile strength of samples S2, S4 and S5 are 1.93, 1.80 and 1.40 N respectively. The tensile strength of unravelled yarn from sample S4 is reduced to 1.80 N from 1.93 N, representing a 7% loss of tensile strength. For sample S5, the strength loss is increased further, indicating a value of 1.40 N, a loss of approximately 27% strength. This reduction in tensile strength may be due to the deterioration of silk fibre in aqueous media[50].

4 Conclusion

The silica sol gel-chitosan-HDTMS coated silk fabric has exhibited a highly hydrophobic nature, with a contact angle value of around 152.7˚. However, after 3 days, 12 days and 30 days of dipping in aqueous media, the contact angle value is lowered down to 141.0˚, 121.4˚ and 90.18˚ respectively, i.e. from highly hydrophobic to hydrophobic in nature. The reduction in contact angle is probably due to the cracks in the silica coating. Moreover, the treated fabric indicates no wicking due to the blocking of pores because of coating. In addition to that, the deterioration in the fibre surface is noticed from SEM analysis and yarn tensile strength values. In terms of antibacterial activity, coated fabric has a low antibacterial activity with a ZOI of approximately 1 mm. However, the antibacterial activity has enhanced with the number of days dipped in aqueous media for 3 days, 12 days, and 1 month; the ZOI is 3 mm, 5 mm, and 5 mm respectively. The increase in ZOI is observed as a result of the exposure of amino groups of chitosan. The amino groups of chitosan that are exposed, may be due to cracks in the coating caused by swelling. Hence, the developed hydrophobic silk fabric, with antibacterial capabilities, may be beneficial to develop natural biocompatible textile materials, which may be used as implant materials.

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