



Multi-functionalization of linen fabric using a combination of chitosan, silver nanoparticles and *Tamarindus Indica L.* seed coat extract

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Abstract Development of multifunctional textile materials is a quest of research today and multifunctional fabrics particularly based on cellulose are highly demanded. Green synthesis of nanoparticles is widely reported; however, the reports on green in situ synthesis of nanoparticles on textile materials is quite limited. Another issue which needs to be addressed through research is the durability of functional properties in case of nanoparticle-containing fabrics. In the present work, linen fabric was first coated with a chitosan-based formulation and the modified linen was utilized as a backbone for in situ synthesis of silver nanoparticles using tamarind seed coat extract. The modified fabric was characterized and further evaluated for change in appearance using reflectance spectroscopy. The functional properties of the modified linen viz. wrinkle recovery, antibacterial activity, ultraviolet protection, flame retardancy and antioxidant activity were evaluated. The retention of such functional properties against subsequent laundering treatments was evaluated. The brown-coloured modified linen showed efficient functional properties, with bacterial reduction of 100%, UPF rating of 50 + and antioxidant activity of 97% and LOI of 23. The functional properties except flame retardancy were retained to a satisfactory level even after 50 washes.

The results obtained were promising enough to claim the resulting material as multifunctional textile material suitable for various technical applications.

Keywords Multifunctional linen · Antibacterial · UV protection · Wrinkle resistance · Durability

Introduction

Textiles are a basic need of humans and textiles with added functions are now in high demand. Nanotechnology is one of the most important sciences used in the modification of textile materials and extensive research is available in the literature regarding such applications. The metal and metal oxide nanoparticles are known to possess various significant functional properties that can be imparted to textiles. Various nanoparticles like silver, copper, zinc oxide, titanium dioxide, cerium oxide and silica were widely researched and explored for functional modification of textiles (Aksit et al. 2017; Attia et al. 2017; Deshpande and Chavan 2013; El-Hameed El-Ebissy et al. 2016; Farouk et al. 2013; Mowafi et al. 2017; Tripathi et al. 2019; Zhou et al. 2018; El-Naggar et al. 2017). Silver nanoparticles (AgNPs) are the prime choice among the nanoparticles owing to its lower toxicity even at higher levels of ppm and different health benefits (Faunce and Watal 2010). Researchers have used silver nanoparticles for imparting the

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functionalities like antimicrobial activity, UV protection, flame retardancy, etc. to textiles (Jeon et al. 2008; Sadu et al. 2014; Sharma et al. 2009; Zhang et al. 2017; Zhou et al. 2018; Hassabo et al. 2019; Mohamed et al. 2016; Rehan et al. 2018). Some reports regarding the application of AgNPs for imparting flame retardancy are also available in the literature (Li et al. 2018; Taghiyari 2012).

Durability is an important aspect of functional finishing of textiles as these materials are frequently washed during their usage. Even though AgNPs can be utilized in imparting various functional properties to textiles; the efficacy of the functionalities would be lost due to alkaline launderings. Hence, the wash fast immobilization of nanoparticles on textiles is necessary and number of reports regarding utilization of polymers to act as anchorage for nanoparticles are available in literature (Sheikh and Bramhecha 2018; Abdelgawad et al. 2017; Danko et al. 2013; Haji et al. 2016; Haji 2017; Naebe et al. 2016). Some polymers like polyacrylic acid, polyacrylamide and their blends which are able to hold nanoparticles (Bai et al. 2007; Ding et al. 2018) could be the best solution to immobilize silver nanoparticles on textiles (Teli and Sheikh 2012, 2013). However, these polymers are synthetic and non-biodegradable. To overcome these shortcomings, we can look forward to chitosan owing to its characteristics i.e. biodegradability, bio derivative, film forming and functional property. Chitosan can also be utilized for functionalization of textile materials (El-Tahlawy et al. 2005; Gouda and Keshk 2010; Hebeish et al. 2013; Teli et al. 2013; Mohamed et al. 2016).

Chitosan is a film-forming polymer and fabrics finished with chitosan can be used as templates for immobilization of nanoparticles (Sheikh and Bramhecha 2018; Danko A 2013; Haji et al. 2016; Haji 2017; Rehan et al. 2018). Many researchers have combined chitosan with different materials like silica, clay and other nano particles (Mohamed et al. 2016; Rehan et al. 2018). The conversion of silver ions to silver nanoparticles requires the use of reducing agents like sodium borohydride which is a non-ecofriendly chemical (Mulfinger et al. 2007; Song et al. 2009). Plant extracts are rich in tannins, flavonoids, polyphenolic compounds, etc. which can act as reducing agent for the synthesis of silver nanoparticles. The green methods of nanoparticle-synthesis are available in literature (Sharma et al. 2009). Tamarind seed coat contains numerous unsaponifiable hydrocarbons such

as β -amyrin, campesterol, and β -sitosterol (Kumar and Bhattacharya 2008) along with condensed tannins (soluble polyphenolic compounds) which are also known to possess functional properties. The reports regarding application of tamarind seed coats in natural dyeing of textile materials are available in the literature (Prabhu and Teli 2014; Teli et al. 2012).

The treatment of linen fabric with chitosan-based formulations can help to bond chitosan with cellulose and the further treatment with silver ions followed by *Tamarindus Indica L.* seed coat (TSC) extract can help in getting functionalization of linen. Apart from this during the treatment, tannin will also be attached to chitosan and can give rise to additional functional properties. The combined effect of chitosan, silver nanoparticle and TSC extracts could be synergistic.

The present work explores the application of chitosan film as a template to immobilize silver nanoparticles on linen. The utilization of tamarind seed coat tannin in in situ synthesis of silver nanoparticle on linen is explored for imparting multiple functions. The efficacy of the functional properties of the modified linen is reported. The modified material is projected as a multifunctional textile.

Materials and methods

Materials

Linen fabric with plain weave having fabric cover of 0.84, 45 ends per inch (epi) and 41 picks per inch (ppi), and 241.5 grams per square meter (gsm) was obtained from Jayshree Textiles (Kolkata, West Bengal, India). Lab grade silver nitrate, sodium hypophosphite (SHP), 1,2,3,4-Butanetetracarboxylic acid (BTCA) was procured from Sigma Chemicals, India. Above material were used without any pretreatment. Chitosan (Molecular weight— 150,000, Degree of deacetylation— 90.1%, nitrogen content— 7.2%) and tamarind seeds were purchased from market.

Methods

Preparation of tamarind seed coat extract

The tamarind seed coat powder (5 g) was added to 100 ml distilled water and the dispersion was heated at

95 °C for 1 h. The extract was filtered using nylon cloth (200 mesh) and was made to 100 ml using water.

Finishing of linen fabric

The fabric was padded with 1% wt/vol chitosan in 8% wt/vol BTCA, and 3% wt/vol sodium hypophosphate, dried at 80 °C for 4 min and cured at 140 °C for 4 min. The finished fabric (sample 1) was treated with 0.25% [on weight of fabric (owf), 31.75 ppm of silver on liquor basis] silver nitrate at room temperature for 20 min and further treated with tamarind seed coat extract (5% wt/vol) for 60 min under sonication. The fabric is then washed with cold water for 5 min and dried (sample 2).

Characterization of modified linen

The FTIR spectra of samples were recorded using FTIR spectrophotometer (Nicolet 6700, USA) using ATR sampling technique by recording 45 scan in %T mode in the range of 4000–600 cm^{-1} . Analysis of the morphology was carried out using scanning electron microscope (Zeiss EVO 50, Germany). The elemental analysis of surface was carried out using EDS technique (Hitachi High Technol tabletop SEM/EDAX-element Analysis system). The size of in situ synthesized nanoparticles was evaluated by transmission electron microscope imaging (JEOL JEM-1400 TEM, USA).

Colour value by reflectance method

The change in colour of the fabric after treatment was measured on Gretag Macbeth Color-Eye 7000A (X-Rite, USA) equipped with reflectance accessories (Haji et al. 2018).

Evaluation of functional properties

Crease recovery angle (CRA) was measured as per the standard ASTM D-1296. The antibacterial activity of the treated fabrics was estimated as per AATCC Test Method 100-2004. The Ultra violet protection factor (UPF) and limiting oxygen index (LOI) of the linen fabrics was evaluated as per AS/NZS 4399:1996 and ASTM D-2863 standards respectively. Antioxidant activity of the modified linen was evaluated using DPPH radical as per the procedure available in the

literature (Sheikh and Bramhecha 2018). The durability of multifunctional properties towards repeated launderings was measured using washing conditions as per ISO 105-CO6-1M test methods.

Results and discussion

Mechanism of in situ synthesis of silver nanoparticles on linen fabric

Linen is a cellulose rich fabric which was modified with chitosan-based formulation containing chitosan, BTCA and SHP. Chitosan is a film forming polymer which was dissolved in BTCA solution. The availability of four carboxylic acid groups was considered to be the vital aspect as these groups can react with hydroxyl groups of cellulose in presence of esterification catalyst (SHP) at elevated temperatures and crosslink it. This was expected to give wash-fast chitosan layer on the fabric which can act as a template for immobilization of in situ generated nanoparticles. When such chitosan-treated sample was treated with silver nitrate solution, the absorption of silver ions would take place due to the presence of functional groups like carboxyl, amino and hydroxyl. The further treatment with tamarind seed coat extract under sonication resulted in reduction of silver ions to silver nanoparticles which gets dispersed in the chitosan film. The TSC extract contains tannins (Sinchaiyakit et al. 2011) which were reported to show reducing power. The preparation of silver nanoparticles from silver nitrate using tannin based extracts is reported in the literature. The mechanism of modification of linen fabric is presented in Figs. 1 and 2.

Mechanism of the similar kind of complex molecule was also studied by the researcher to propose the chemistry involved in the reaction mechanism (Coordination compound, Britannica.com 1998; Gordon et al. 2010; Tripathi et al. 2019). Similarly, the predicted mechanism for in situ silver complex formation in this system is as shown in Fig. 1. Tannins play dual roles in the modification of fabric i.e. as reducing agent and as crosslinking agent.

During the treatment of preparation of sample 2 (treatment with TSC extract), tannins were also absorbed by the chitosan containing fabric as a result of interaction between positively charged chitosan and negatively charged tannins. This interaction of tannin

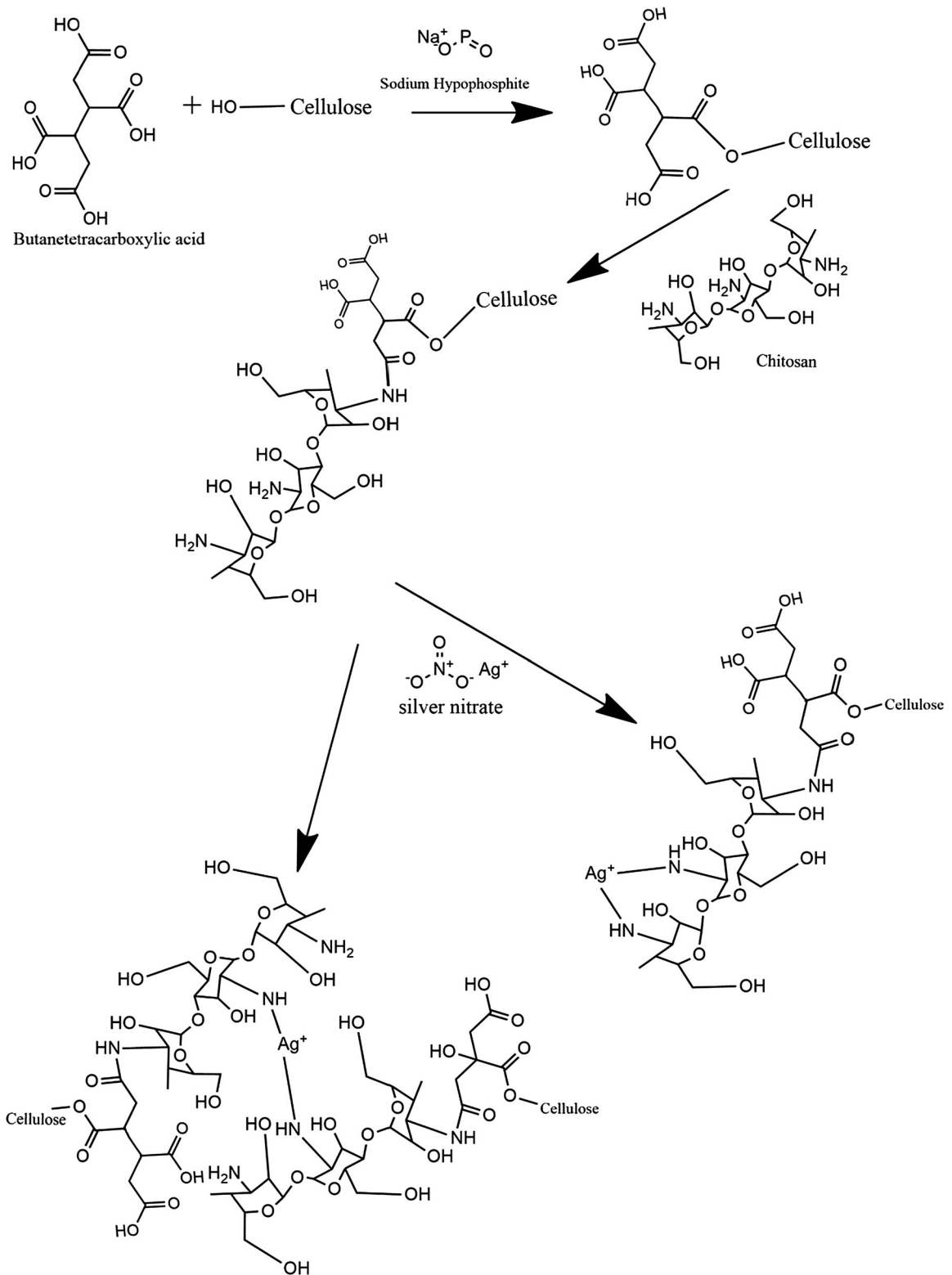
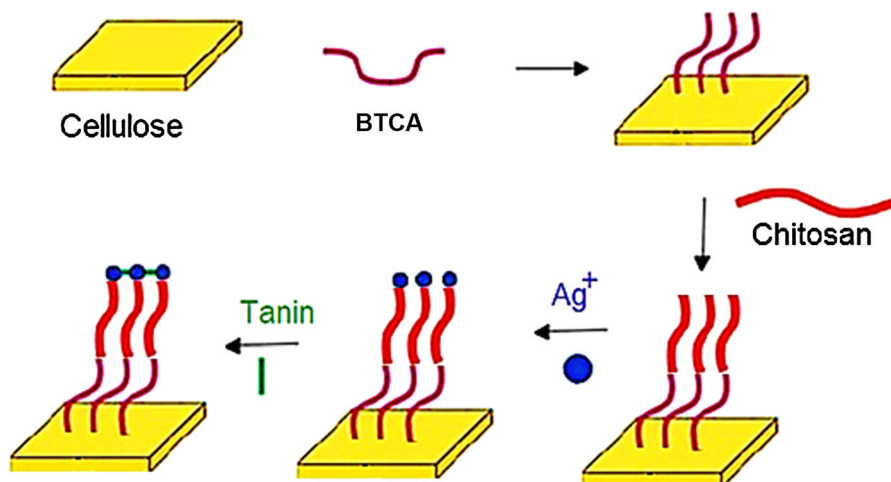


Fig. 1 Mechanism of holding silver ions on the chitosan-treated fabric

Fig. 2 Mechanism of in situ synthesis on chitosan-treated linen fabric Silver



with positively charged chitosan doesn't allow the loss of tannin and nanoparticles during the subsequent washing.

The mechanism is well supported by the change in appearance of the linen fabric in the subsequent stages (Fig. 3) and UV–visible analysis of the treatment liquor.

The fabric turned yellow after chitosan deposition due to the deposition of chitosan film and the ester formation between cellulose hydroxyl groups and carboxylic acid groups of BTCA. The appearance of the fabric showed negligible change after treatment

with AgNO_3 solution. However, the treatment with TSC extract resulted in drastic change in the appearance of the fabric. The fabric turned brown indicating the formation of silver nanoparticles as well as absorption of tannins from TSC extracts. The treatment liquor was analyzed using UV–VIS spectroscopy (Fig. 4). It indicates the colour change with time indicating the formation of nanoparticles within 15 min of sonication (Fig. 5).

As evident from Fig. 5, TSC showed absorption peaks in both UV and visible regions. Absorption near 300 nm is due to the presence of 3,4-Dihydroxyphenyl acetate according to literature (Tsuda et al. 1994). When modified fabric was treated with TSC extract, an additional peak at 420 nm appeared indicating the formation of silver nanoparticles. This can further help in stabilization of nanoparticles on the fabric. It would be interesting to note that the presence of tannins on the modified linen can also impart additional functional properties to the linen fabric. The combined effect of chitosan, silver nanoparticles and TSC extracts resulted in coloured multifunctional linen fabric.

Characterization of the modified linen fabrics

The modified linen fabrics were characterized in order to bridge the gap between structure–property relationships.

Figure 6 shows the FTIR spectra of control linen and sample 2. The presence peak at 1720 cm^{-1} was assigned to C=O stretching vibration which confirms the presence of ester linkage in modified linen. The

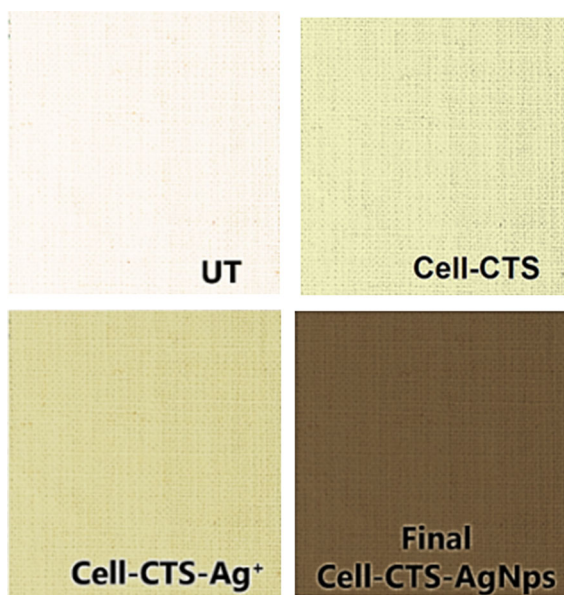
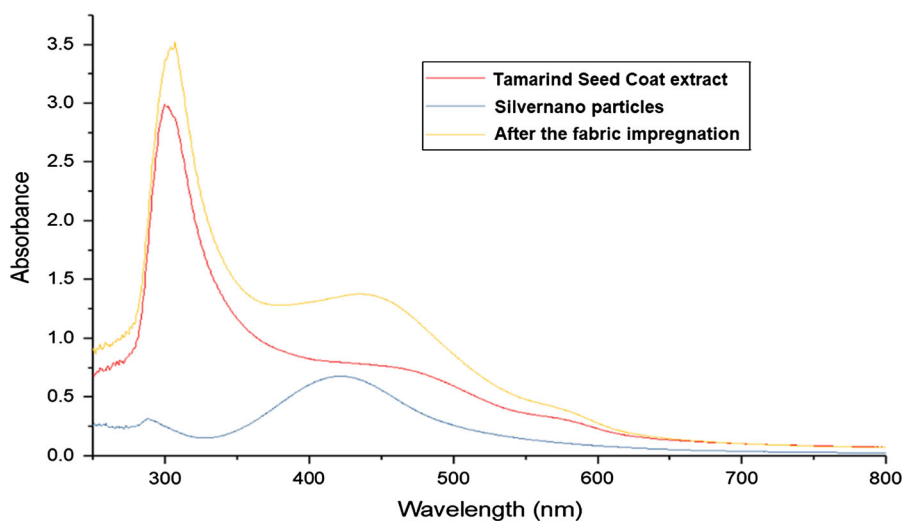


Fig. 3 Appearance of fabric after subsequent modification



Fig. 4 Change in colour of treatment liquor after subsequent process

Fig. 5 UV-visible spectra of treatment liquor



peak for N–H stretching vibration was observed at 3329 cm^{-1} which was attributed to the presence of chitosan. No significant changes were observed in FTIR spectrum of control and modified linen (sample 2).

The thermal stability of linen was analyzed using thermogravimetric analysis and the thermograms are presented in Fig. 7.

As visible from Fig. 7, initial stage of weight loss (till $250\text{ }^{\circ}\text{C}$) was similar for both the samples. The modified sample showed early start of degradation; however, the residue formation was higher at $800\text{ }^{\circ}\text{C}$. This might be attributed to the presence of nitrogen due to the treatment with chitosan and the silver nanoparticles (El-Shafei et al. 2015). The change in degradation pattern after modification can be thus correlated to the flame retardant behavior of modified linen. In general, flame retardant cellulose starts getting degraded at lower temperatures resulting

higher quantity of ash formation. This prevents further burning of material and lowers the damages caused during fire accidents. This needs to be further analyzed for the flame retardant behavior of modified linen.

The changes in morphology of the fabric after modification was analyzed using SEM technique (Fig. 8). The surface of unmodified linen was clean while sample 2 showed surface deposition which might be attributed to deposition of chitosan layer. The presence of nanoparticle was also evident from Fig. 5. After treatment with TSC extract under sonication (sample 2), the nanoparticles were immobilized which are visible in SEM images. The size of some nanoparticles was measured and those were found in the range 20–50 nm.

The EDX analysis (Fig. 9) confirmed the presence of silver (0.2 wt%) on the sample 2. The other characterization techniques confirmed the presence of silver in the form of nanoparticles (Figs. 8 and 10).

Fig. 6 FTIR spectra of control and treated linen (sample 2)

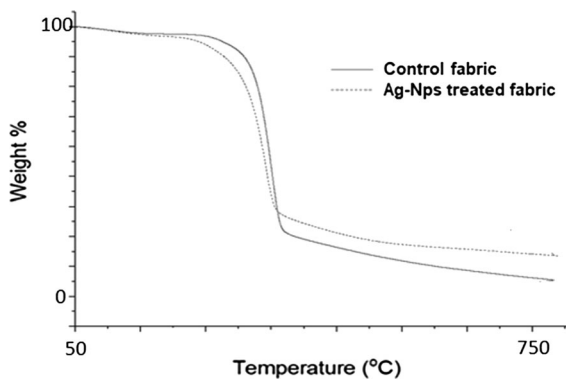
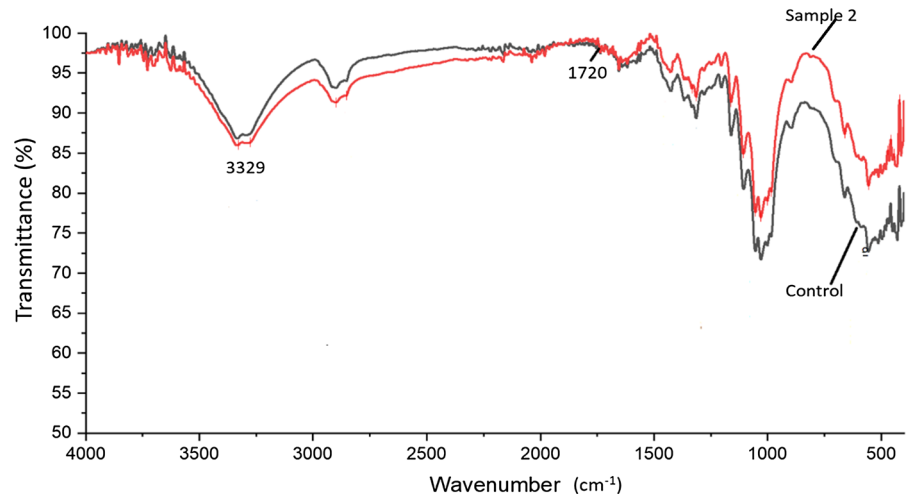


Fig. 7 Thermograms of control and treated linen (sample 2)

In order to further confirm the presence of nanoparticles, the TEM analysis was carried out at 2.56 lac magnification and the TEM image is presented as Fig. 10.

The presence of silver nanoparticles was confirmed through TEM image and the particles were found to be of much smaller size (less than 5 nm range).

Functional properties of modified linen fabrics

The functional properties of the modified fabric was evaluated and the results are summarized in Table 1.

As evident from Table 1, the finished fabric displayed improvement in crease recovery angle. This might be attributed to crosslinking of cellulose by BTCA and the deposition of chitosan through crosslinking. BTCA contains four carboxylic acid groups available for reaction with cellulose, one of which was consumed while dissolution of chitosan. The rest can react with cellulose in presence of esterification catalyst i.e. sodium hypophosphite thus preventing probability of H-bond formation between cellulose chains resulting in better crease recovery.

The modified linen also displayed efficient antibacterial activity against *S. aureus* and *E. coli* bacteria

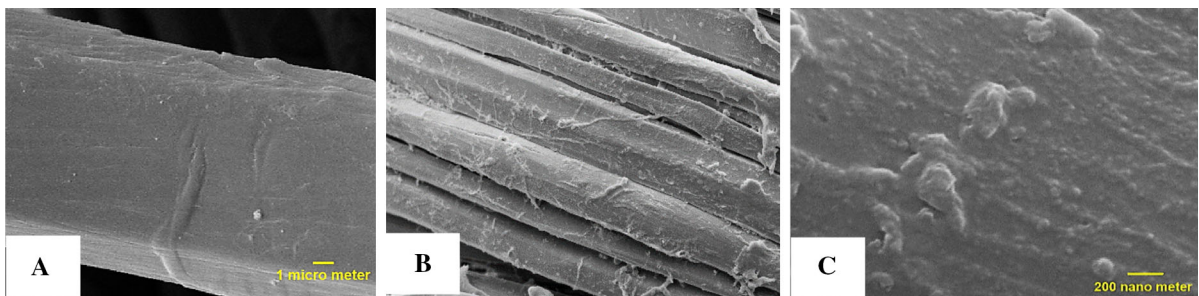
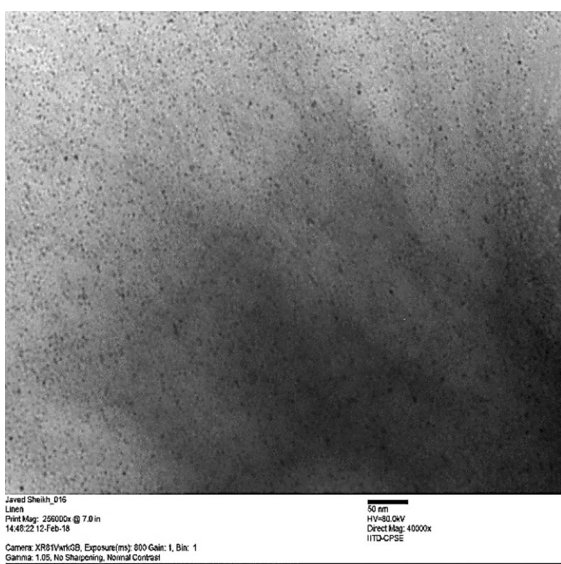
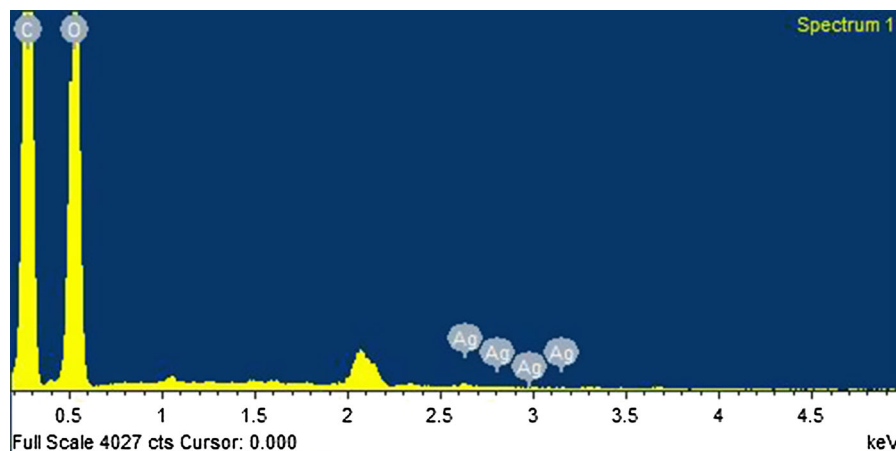


Fig. 8 SEM images of control (A) and sample 2 (B-2000X and C-65000X)

Fig. 9 EDX of sample 2**Fig. 10** TEM image of fabric sample

which was due to the presence of three bactericidal materials in the modified fabric. Chitosan is known to possess antibacterial properties against both gram positive and gram negative bacteria. The presence of primary amino group, which makes chitosan a highly basic polysaccharide, which gets protonated and

prevents growth of bacteria mainly through leaking of their cell wall and prevention of multiplication of their genetic material. The immobilization of silver nanoparticle on such layer resulted in further increase in antibacterial activity showing complete killing of bacteria. The nanosilver is a strong oxidation catalyst which kills bacteria. Apart from this, the availability of greater surface area in case of nanoparticles offer further advantage of better activity at sufficiently lower dosages. The linen fabric after synthesis of nanoparticle showed brown coloration which may be attributed to silver nanoparticle as well as absorption of tannins by cellulose structure and chitosan film. Tannins are also known for their astringent and antimicrobial activity. The combined effect of chitosan, silver nanoparticles and tannins resulted in superior antibacterial activity. The bacteria selected are the representatives of gram negative and gram positive category and modified fabric displayed efficient antibacterial activity against both *S. aureus* and *E. coli*.

The modified fabric also displayed efficient UV protection with the UPF rating of 50 + . The presence of chitosan film along with UV absorbing materials like silver nanoparticles and tannins resulted in enhanced UV protection property of linen. Limiting

Table 1 Properties of finished fabric

Sample no.	CRA	Bacterial reduction ^a (%)		UPF ^a	LOI ^a	Antioxidant activity ^a (%)
		<i>S. aureus</i>	<i>E. coli</i>			
Control	96	N	N	9.85	18.2	N
Sample 1	156	82.50	84.50	22.13	21.4	10.5
Sample 2	160	100	100	75.80	23.0	97

N Negligible

^aAverage value of three determinations

Table 2 Durability of colour and functional properties

	No. of washes	K/S	CRA ^a	Bacterial reduction ^a (%)		UPF ^a	LOI ^a	AO ^a
				<i>S. aureus</i>	<i>E. coli</i>			
	0	13.65	160	100	100	75.8	23	97
	10	13.54	155	99.9	99.0	72.6	21.2	95.1
AO antioxidant activity	20	13.44	152	99.9	99.0	65.5	21	88.5
^a Average value of three determinations	50	13.25	145	99.5	98.5	60.8	21	80.0

oxygen index (LOI) was also improved for modified fabric. This might be attributed to the presence of tannins along with nitrogen containing chitosan and silver nanoparticles. Some reports regarding application of tannins in getting leveraging flame retardant are available in literature. The presence of nanoparticles, N-containing chitosan and tannins play a synergistic role in flame retardancy. Although LOI of 23 does not indicate superior flame retardancy, the improvement of LOI value from 18.2 to 23 is quite remarkable with such recipe of finishing which can be optimized for further improvement in flame retardancy of linen. Generally, metals can absorb heat, have very high ignition temperature and doesn't burn easily. In case of nano metals, specific surface area is very high and can cover the substrate evenly in small quantities avoiding the propagation of flame. The increase in the LOI values may be due to presence of silver nanoparticles as well as chitosan (El-Shafei et al. 2015; Taghiyari 2012).

In case of sample 1, no significant radical scavenging activity was found which indicates the limited extent of radical scavenging by the functional groups of linen and chitosan. However, sample 2 displayed efficient antioxidant activity which might be attributed to the presence of the different polyphenolic compounds supplied by TSC extract. TSC extract contains 2-hydroxy-3',4'-dihydroxyacetophenone, methyl 3,4-dihydroxy benzoate, 3,4-dihydroxyphenyl acetate and epicatechin. Among these 3, 4-dihydroxyphenyl acetate is major content of TSC extract (Tsuda et al. 1994). These compounds can easily scavenge radicals responsible for aging and become stable free radical giving antioxidant property to the substrate on which these are present (Rice-Evans et al. 1997). The presence of nanoparticles can also contribute to radical scavenging resulting in synergistic antioxidant activity. Antioxidant textile can be ideal option to cosmetics as textile materials cover most part of human body

and the development of cosmeto-textile could be done using the method reported in the present work.

As evident from Table 2, the sample 2 displayed durability of functional properties till 50 washes; however, the extent of retention varied among the various functionalities. It must be noted that the colour values of the modified fabric was also retained to a significant extent. This indicates the wash-fast attachment of various components, especially tannins and nanoparticles. The antibacterial activity was retained in the excellent scale even after 50 washes. UV-protection and antioxidant activities of the modified fabric decreased after subsequent washings but retained in the satisfactory levels of protection. LOI values showed a decrease; however, but it is interesting to note the retention is this case. It must be noted that control linen sample showed LOI of 18.2 while, the LOI displayed by sample 2 after 50 washes was 21. It need detailed investigation where the optimization of such recipes of finishing can provide superior flame retardancy to cellulosic fabrics. Antioxidant properties are durable enough up to 50 washes. This is mainly due to proper cross linking of polyphenolic groups with silver nanoparticle and chitosan film grafted on cellulose. Other reason could be ionic interaction between chitosan and tannin from TSC extracts.

Conclusion

The successful development of multifunctional linen fabric was achieved using green in situ synthesis of silver nanoparticle on chitosan modified backbone. The use of TSC extract for development of nanoparticles is explored for first time on textile. The modified fabric displayed efficient crease resistance, antibacterial activity, antioxidant activity and UV protection along with moderate level of flame retardancy. The functional properties, displayed by modified linen,

showed excellent retention on subsequent washing. The successful wash-fast immobilization of in situ synthesized silver nanoparticles was thus confirmed through the facile method reported in the present work. This work provides an ecofriendly, bio-based and sustainable direction to the emerging sector of functionalization of textile materials.

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