

## Antimicrobial Activity of Silk Treated with Aloe-Vera

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**Abstract:** Natural Silk in its native form is made up of the filament fibroin, coated with sericin - gummy matter which is antimicrobial in nature. However, sericin is removed during the pretreatment process. Silk being a natural and hygroscopic, fiber gets attacked by microbes easily. Hence antimicrobial treatment can add value to the wear and care of silk textiles. Synthetic antimicrobial agents may be toxic and even carcinogenic and hence the natural and eco-friendly antimicrobial agents are good substitutes for imparting the desired properties. In the present paper, ready for dyeing (RDF) silk fabric was treated with Aloe-Vera using 1,2,3,4-butanetetracarboxylic acid (BTCA) as crosslinking agent and sodium hypophosphite (SHP) as catalyst. The treated fabric with a concentration of 15 % of Aloe-Vera showed excellent antimicrobial properties. Since BTCA was used as a crosslinking agent, crease recovery angle, improved with minimal loss in breaking and tearing strength. The mechanism of treatment of Aloe-Vera is found to be chemical binding with silk and not simply of coating or impregnation. FTIR studies showed that the carboxyl side groups and short chain amino acids side groups act as sites for BTCA crosslinking interalia chemical binding of Aloe-Vera. SEM studies revealed that no coating or tangible impregnation on the surface of the fiber is visible substantiating the chemical binding phenomenon. This is further substantiated by the durability of the finish to dry cleaning of treated silk. Since Aloe-Vera is a natural product and BTCA is an eco-friendly resin, the treatment of silk with Aloe-Vera is eco-friendly in nature.

**Keywords:** Aloe-Vera, 1,2,3,4-Butanetetracarboxylic acid, Sodium hypophosphite, Silk fabric

### Introduction

Silk is a natural product that is mainly composed of sericin and fibroin proteins. The sericin constitutes 20-30 % of silk protein and it covers the filament fibroin. Silk is the strongest natural fiber having luster, inherent affinity for dyes, high absorbent and light weight qualities, excellent drape and elastic properties [1]. Silk sericin, a natural protein obtained from silkworm cocoon has a combination of many unique properties such as biodegradability, non-toxicity, oxidation resistance, antimicrobial activity, UV resistance, and moisture absorption [2]. Sericin is insoluble in cold water; however, it is easily hydrolyzed to smaller fractions, which are easily dispersed, or dissolved in hot water [3].

Since silk fibroin is a natural protein having hydrophilicity, it is susceptible to microbial attack during storage and usage. On the other hand, silk sericin possesses antimicrobial properties, thereby protecting the fiber. This property of sericin cannot be exploited as it is removed from the fiber in the pretreatment processes to improve absorbency essential for better uniform wet processing ahead. In order to retain the nature as well as for proper care of silk, antimicrobial finishing is necessary.

Efforts to finish silk with formaldehyde based resins did not enhance the easy care properties to the desired level apart from yielding poor dyeability. Due to ecological considerations, formaldehyde based finishing is less preferred in the industry and hence, alternate eco-friendly approaches for finishing of textiles are being researched. In this regard, it is reported that polycarboxylic acids may replace traditional formaldehyde

reagents for durable press finish of cotton fabrics [4-9]. A number of chemical substances are used in the textile industry to impart antimicrobial activity to textiles-antibiotics and biocides of both organic and inorganic origin, which reduce or inhibit completely the growth of microorganisms. Cationic dyes and chemical finishing agents for textiles exhibit antimicrobial properties [10]. Natural dyes are used for deodorizing and antimicrobial performance of textiles including silk. Lee *et al.* reported that treatment with *Punica granatum L.* extracts displayed outstanding deodorizing performance against ammonia gas and excellent antimicrobial performance against *S. aureus* and *K. pneumoniae* [11].

Since bifunctional aldehyde like formaldehyde as crosslinking agent has been banned due to the non-ecofriendly nature, the polycarboxylic acids as an alternative are being investigated for modification of textiles. Due to hydrophilicity and abundant side groups available in silk, it has been contemplated that treatment of silk with polycarboxylic acids is quite promising similar to cotton. Irvin [12] carried out durable press finishing of silk using BTCA(1,2,3,4-butanetetracarboxylic acid) and other cheaper polycarboxylic acids and showed them to act as good crosslinking agents on silk improving both wet and dry crease recovery angles. Of them, BTCA has been reported to be more effective than others such as citric acid. Hence, the BTCA route was considered in the present studies. On similar lines, Yang *et al.* [13] reported the durable press finishing of silk fabrics with BTCA. The effect of the treatment variables namely concentrations of SHP (sodium hypophosphite) and BTCA, curing temperature, and curing time was investigated. The results showed that BTCA is very effective to the durable press finishing of silk fabrics.

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Aloe-Vera (*Aloe barbadensis*, Miller) belonging to family *Liliaceae* is known as 'Lily of the desert'. It is being used in skincare products for over 2000 years. In modern times, scientific research has shown that the Aloe leaf contains over 75 nutrients and 200 active compounds, including 20 minerals, 18 amino acids, and 12 vitamins. These rich constituents give the Aloe-Vera gel special properties as a skin care products. Aloe-Vera also possesses antifungal and antibacterial properties, which can be exploited for medical and textile applications, such as wound dressing, sutures, bioactive textiles, etc. There are different polysaccharides in Aloe-Vera, such as glucomannan, galactogalacturan, glucogalactomannan with different compositions as well as acetylated mannan or acemannan.

The antimicrobial activity of A. Vera inner gel against both Gram-positive and Gram-negative bacteria has been demonstrated by several methods. Acemannan is considered as the main functional component of Aloe Vera [14-16], which is a long chain polymer consisting of randomly acetylated linear D-mannopyranosyl units and has immunomodulation, antibacterial, antifungal, and antitumor properties as shown in the Figure 1 [17]. Many investigators have identified partially acetylated mannan (or acemannan) as the primary polysaccharide of the gel, while others found pectic substance as the primary polysaccharide [18,19]. The acemannan is found in Aloe Vera is structurally unique that makes it a characteristic compound of aloe species amongst other well known plant mannans which have distinct side-chains or are un-acetylated and insoluble in water [20].

This complex carbohydrate accelerates wound healing and reduces radiation induced skin reactions [21,22]. Aloe Vera

gel was bactericidal against *Pseudomonas aeruginosa* and *acemannan* prevented it from adhering to human lung epithelial cells in a monolayer culture [23,24]. Glucomannan and acemannan have been proved to accelerate wound healing, activating macrophages, stimulating immune system as well antibacterial and antiviral effects [15,25-30]. The chemical composition of Aloe Vera has been published with a view to exploit therapeutic applications. The Chemical composition of Aloe Vera has been documented as reproduced in the following Table 1 [20,31-33].

Literature survey has revealed that no studies on finishing of silk are reported to impart simultaneously multifunctional properties viz; antimicrobial and anticrease properties using Aloe-Vera. Therefore, the present studies carried out on treatment of mulberry silk fabric using Aloe-Vera as antimicrobial agent and BTCA as crosslinking agent are reported.

## Experimental

### Materials

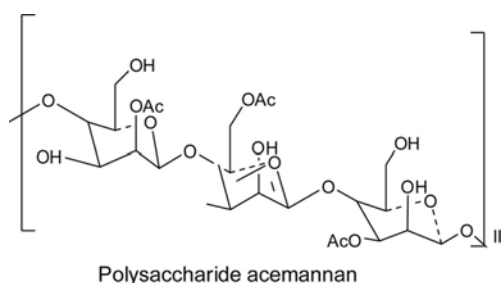
Pure Mulberry silk ready for dyeing (Denier warp - 34.7, denier weft - 65.7, EPI - 114 and PPI - 86 and GSM - 60) was prepared using organzine warp and tram as weft on power loom. Pure Aloe-Vera juice (pharmaceutical grade) was procured from the reputed and quality supplier. BTCA and SHP were procured from Sigma Aldrich. Methanol was procured from Merck India Ltd.

### Pretreatment

Mild scouring was done using soap (1 %) and soda (0.5 %) with material to liquor ratio (MLR) 1:30 at 60 °C to

**Table 1.** Chemical Composition of Aloe Vera [20,31-33]

Class	Compounds
Anthraquinones/anthrones	Aloe-emodin, aloetic-acid, aloin A and B (or collectively known as barbaloin), isobarbaloin, emodin, ester of cinnamic acid
Carbohydrates	Pure mannan, acetylated mannan, acetylated glucomannan, glucogalactomannan, galactan, galactogalacturan, arabinogalactan, galactoglucoarabinomannan, pectic substance, xylan, cellulose
Chromones	8-C-glucosyl-(2'-O-cinnamoyl)-7-O-methylaloeal A, 8-C-glucosyl-(S)-aloesol, 8-C-glucosyl-7-O-methyl-(S)-aloesol, 8-C-glucosyl-7-O-methyl-alediol, 8-C-glucosyl-noreugenin, isoaloesin D, isorabaichromone, neoaloesin A
Enzymes	Alkaline phosphatase, amylase, carboxypeptidase, catalase, cyclooxygenase, cyclooxygenase, lipase, oxidase, phosphoenolpyruvate carboxylase, superoxide dismutase
Inorganic compounds	Calcium, chlorine, chromium, copper, iron, magnesium, manganese, potassium, phosphorous, sodium, zinc
Miscellaneous including organic compounds and lipids	Arachidonic acid, $\gamma$ -linolenic acid, steroids (campesterol, cholesterol, $\beta$ -sitosterol), triglycerides, triterpenoid, gibberillin, lignins, potassium sorbate, salicylic acid, uric acid.
Non-essential and essential amino acids	Alanine, arginine, aspartic acid, glutamic acid, glycine, histidine, hydroxyline, isoleucine, leucine, lysine, methionine, phenylalanine, proline, threonine, tyrosine, valine.
Proteins	Lectins, lectin-like substance
Saccharides	Mannose, glucose, L-rhamnose, aldopentose
Vitamins	B1,B2,B6,C, $\beta$ -carotene, choline, folic acid, $\alpha$ -tocopherol



**Figure 1.** Chemical structure of acetylated mannan or acemannan.

remove lubricants and contaminants deposited on silk fabric during twisting and weaving processes.

### Preparation of Aloe-Vera Solution

Aloe-Vera juice (5 %, 10 %, 15 %) was prepared with Methanol by keeping the mixture soaked for three days at ambient conditions.

### Aloe-Vera Treatment

Aloe-Vera treatment was imparted to silk fabric with MRL of 1:20. BTCA and SHP were added to the Aloe-Vera juice solution in methanol prior to treatment. Mulberry silk was soaked for 30 min in this liquor kept in a beaker. Mulberry silk was subsequently squeezed using padding mangle under pressure of 1 kg/cm<sup>2</sup> and oven-dried at 60-65 °C for 30 min. The dried fabric was cured at a temperature 145-150 °C for 120 s. Treated silk fabric sample was washed using mild

**Table 2.** Characterization carried out for treated silk and the standard test methods

S. No	Parameter	Standard test method
01	Thread density (EPI/PPI)	IS:1963-1981
02	GSM	IS: 1964-2001
03	Tensile strength	IS:1969-1985
04	Crease recovery	IS: 4681-1981
05	Dimensional stability-shrinkage (%)	ISO 5077:2007
06	Martindale abrasion resistance	ISO:12947-1:1998
07	Moisture regain	IS: 199-1989
08	Fabric stiffness	ASTM D 1388-08
09	Colorfastness to dry cleaning	AATCC Test Method 132-2009
10	Morphological properties (SEM)	The SEM photomicrographs were recorded using JOEL JSM 6380 LA, Japan
11	Structural properties (FTIR)	KBr pellet of dispersed fiber powder of different treated silk samples was made and transmission FTIR spectra were recorded
12	Antimicrobial properties	AATCC Test Method 100-2012

soap solution at 30 °C followed by cold water wash to remove unfixed finishing chemicals.

### Evaluation of Physical and Functional Properties of Silk

Silk fabrics after finishing treatment were tested for various properties namely Add-on (%), EPI/PPI, GSM, tensile strength, crease recovery angle, abrasion resistance, moisture regain, and fabric stiffness as per standard methods given in Table 2.

### SEM and FTIR Analysis

SEM studies were carried out using SEM model-JEOL JSM 6380 LA, Japan. The samples of various treated silk fabrics were prepared on the metallic stub and coated with platinum by vacuum coating unit JEOL 1600 to make the surface conducting. These samples were observed under SEM for evaluating the morphological changes due to the treatment at an accelerating voltage of 10 kV at different magnifications.

FTIR analysis was done using an FTIR Spectrophotometer model-ABB Bomen Instrument Canada MB3000. The treated and control silk fabric samples were cut and sieved into fine powder and dried at 100 °C. 2 mg of sample powder was dispersed in 198 mg of spectroscopic grade dried KBr and made into pellet. The KBr pellet was used for recording the FTIR spectra.

### Antimicrobial Activity Measurement

Antimicrobial properties of Aloe-Vera treated silk fabric have been evaluated qualitatively by AATCC 147 method and quantitatively by AATCC 100 method for both *Staphylococcus aureus* ATCC 6538 (gram positive) and *Klesbsilla pneumonia* ATCC 4352 (gram negative) bacteria. Percent reduction of bacteria due to the specimen treatments was calculated by the following formulas:

$$R (\%) = 100 [(A - B)/A]$$

where  $R$ =% reduction,  $A$  is the number of bacteria in the control sample at time  $t=0$  (cfu/sample),  $B$  is the number of bacteria in the treated silk sample after time  $t=24$  h (cfu/sample). The reduction over than 99 % was considered as a higher level.

## Results and Discussion

### Antimicrobial Properties

The antimicrobial properties of untreated and Aloe-Vera treated silk samples were evaluated with respect to gram positive (*Staphylococcus aureus* ATCC 6538) and Gram negative (*Klebsiella pneumoniae* ATCC4352). Table 3 gives the data on antimicrobial properties of untreated and treated silk fabric. Untreated silk fabric showed no reduction in the count of the bacteria and hence it is not antimicrobial. On the other hand, Aloe-Vera finished silk fabric with 15 % concen-

tration exhibited excellent antimicrobial properties as a biocide. It is well known that silk fiber loses its strength by 30-40 % and becomes more ductile under wet condition [34]. Hence poor dimensional stability will be persisting and any mechanical force applied on the fabric during wet

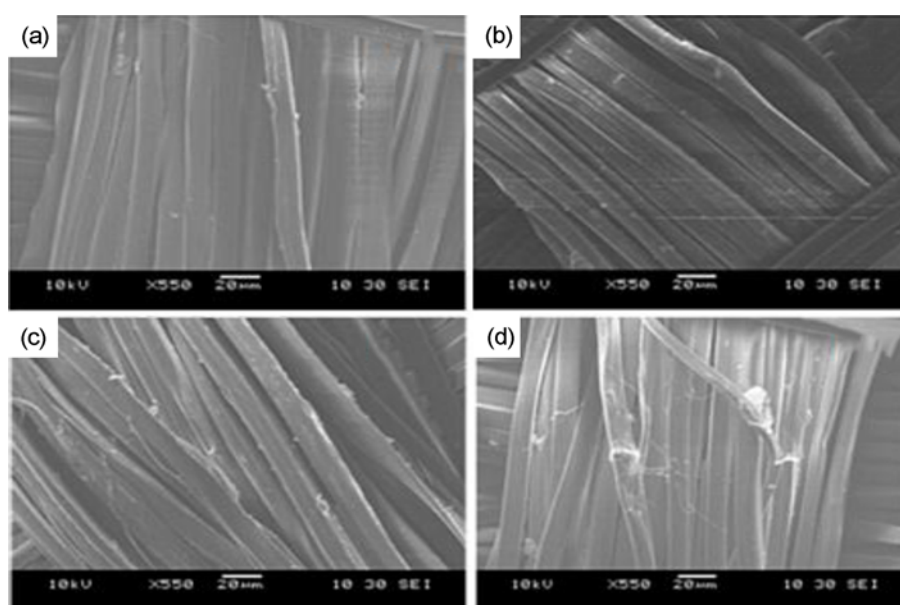
**Table 3.** Antimicrobial properties of different treated silk fabrics

S. No.	Sample	Washing cycles	% Reduction	
			<i>S. aureus</i>	<i>K. pneumoniae</i>
1	5 % Aloe-Vera treated	-	97	97
2	5 % Aloe-Vera treated	1 wash	95	90
3	5 % Aloe-Vera treated	2 wash	93	89
4	5 % Aloe-Vera treated	3 wash	89	82
5	5 % Aloe-Veratreated	4 wash	80	79
6	5 % Aloe-Vera treated	5 wash	60	64
7	10 % Aloe-Vera treated	-	98	97
8	10 % Aloe-Vera treated	1 wash	92	92
9	10 % Aloe-Vera treated	2 wash	90	93
10	10 % Aloe-Vera treated	3 wash	90	90
11	10 % Aloe-Vera treated	4 wash	80	84
12	10 % Aloe-Vera treated	5 wash	83	80
13	15 % Aloe-Vera treated	-	98	98
14	15 % Aloe-Vera treated	1 wash	98	96
15	15 % Aloe-Vera treated	2 wash	95	94
16	15 % Aloe-Vera treated	3 wash	95	93
17	15 % Aloe-Vera treated	4 wash	93	90
18	15 % Aloe-Vera treated	5 wash	92	90

washing can mechanically deform the fiber and can lead to loss of wear and tear properties. Further, silk is by and large dyed with acid, basic, and reactive dyes. It is seen that the wash fastness of silk is vulnerable when the silk is dyed with acid or basic dyes due to acidic/basic properties of soaps/detergents. Hence, the wet washing is not advisable for silk textiles dyed with acid dyes. Even in the case of silk dyed with reactive dyes it is not advisable to have washing with water as mechanical deformation may lead to loss of wear and tear properties. In the view of the above facts, the silk textiles are dry cleaned in the industry and at consumer level. Since silk is dry-cleaned during its usage, the finishing treatment was assessed for its durability up to 5 cycles of dry cleaning. It may be observed from Table 3 that Antimicrobial properties of the treated fabric were retained after 5 cycles of dry cleaning for 15 % Aloe-Vera finished fabric indicating thereby the treatment is durable.

### Morphological Studies (SEM)

Figure 2(a) to (d) show the SEM micrographs of control silk fabric, 5 % Aloe-Vera finished silk fabric, 10 % Aloe-Vera finished silk fabric, and 15 % Aloe-Vera finished silk fabric treated, respectively, at  $\times 550$  magnification. As could be seen in the figures, the surface of the fabric is smooth and no coating is seen even after the treatment. Further, tangible impregnation of the finishing recipe is also not visible. Marginal change in the surface morphology shows that the fiber has undergone some physico-chemical changes due to the crosslinking of BTCA and bridging of Aloe-Vera onto the fibroin. It is observed that the concentration of Aloe-Vera in the antimicrobial treatment has a direct bearing on the



**Figure 2.** SEM micrographs of (a) control silk fabric, (b) 5 % Aloe-Vera finished silk fabric, (c) 10 % Aloe-Vera finished silk fabric, and (d) 15 % Aloe-Vera finished silk fabric.

chemical binding as the effect of treatment is predominant with increase in add-on% of Aloe-Vera. It may be pointed out that the higher add-on% of the treatment conditions indicates higher percentage of crosslinking of BTCA and inter-alia chemical binding of Aloe-Vera with silk fibroin. Crosslinking of BTCA is in accordance with the earlier studies reported by Irvin [12] and Yang *et al.* [13]. However, the present studies lead to binding of Aloe-Vera with silk fibroin with BTCA Bridge. Based on the SEM photographs, it is inferred that the treatment is predominantly chemical binding with no impregnation within the interstitials of microfibrils of the fiber or coating. Further, the SEM observations are supplementing the results obtained in terms of weight add-on% due to Aloe-Vera finish on the silk fabrics.

### FTIR Spectral Analysis

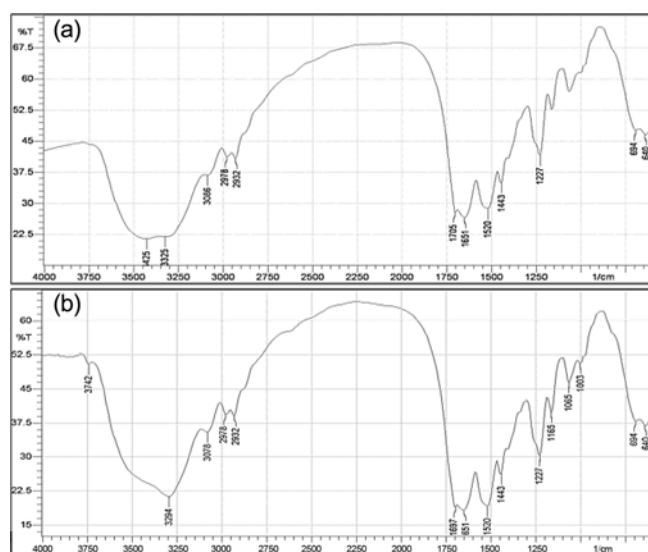
Figure 3(a) and 3(b) show the FTIR spectra of untreated and Aloe-Vera treated silk fabrics. Since the predominant bands of Aloe-Vera, BTCA and silk fibroin overlap each other, no new band due to the finishing treatment is observed in FTIR spectra. However, SEM photographs and add-on% values have revealed that the finish is not a physical coating and it may be chemical binding with no impregnation within the interstitial of the fibrils of fibroin. In view of this, FTIR-ATR technique to investigate the surface analysis has not been used. KBR pellet technique has been used to record the FTIR transmission spectra. It is reported by Irvin that BTCA crosslinking enhances the intensity of the band at 1700-1750  $\text{cm}^{-1}$  which is attributed to carboxylic bonding. However, the author has not made quantitative analysis due to the changes in the concentration of the substance in the KBr pellet. Similar observation is also made by Yang *et al.* [13]. Yang *et al.* [35] have described an improved silk textile

which exhibits more desirable wet wrinkle-recovery and tear strength than untreated silk. The silk textile contains polycarboxylic acid (BTCA) crosslinks which are relatively strong and resistant to hydrolysis. The finishing agent used by them was an aqueous solution containing 6 % BTCA and 3.3 % sodium hypophosphite [35]. Since the focus of our current studies was to impart the antimicrobial properties using Aloe-Vera through BTCA crosslinking to bind the acemannan a polysaccharide on to the silk fibroin, the above recipe of BTCA and SHP was taken into consideration. The results are substantiated by FTIR studies.

In order to quantitatively assess the observed binding of Aloe-Vera, an attempt is made to define the index of binding by normalizing the effect of concentration of the silk fibroin in the FTIR spectra. On close observation of the FTIR spectra of different treated samples, it is found that the band at 1700-1710  $\text{cm}^{-1}$  is due to carboxylic groups owing to the crosslinking of BTCA and inter-alia binding of Aloe-Vera with the silk fiber. Since the concentration of the dispersion of the sample is likely to vary between the samples, a band at 2978  $\text{cm}^{-1}$  which is  $\text{CH}_3$  symmetric vibration was taken as internal reference band. The optical density of the band at 1700-1710  $\text{cm}^{-1}$  was estimated. Likewise, the optical density for the band at 2978  $\text{cm}^{-1}$  was also estimated. Index of binding ( $I_1$ ) as the ratio of the optical density at 1700-1710  $\text{cm}^{-1}$  and optical density for the band 2978  $\text{cm}^{-1}$  was determined to assess the chemical binding of Aloe-Vera with silk through crosslinking of BTCA.

The Index binding ( $I_1$ ) is given in Table 4. It is seen that the binding index ( $I_1$ ) reduced initially (5 %) to a large extent showing that the crosslinking of BTCA and chemical binding of Aloe-Vera has led to reduction in the molecular vibrations as the side groups attached constrain the amplitude of vibration. However, there is slight increase in the binding index ( $I_1$ ) subsequently (10 % and 15 %) as the Aloe-Vera concentration increased. This may be due to enhanced crosslinking of BTCA along with excess of Aloe-Vera available for reaction. In view of the above, it may be opined that one of the prominent sites for binding of Aloe-Vera is the carboxylic group present in the silk and affinity of the same with that of BTCA and Aloe-Vera and hence is used to quantitatively assess the reaction mechanism.

Further, silk fibroin has different amino-acids including acidic, basic, and neutral acids having either long side chain or short side chain. Due to easy access of side groups attached



**Figure 3.** FTIR spectra of (a) control silk fabric and (b) 15 % Aloe-Vera.

**Table 4.** FTIR spectroscopic analysis

S. No.	Sample	Index of binding $= A_{1065}/A_{2978 \text{ cm}^{-1}}$ $I_2$	Index of binding= $A_{1700-1705}/A_{2978 \text{ cm}^{-1}}$ $I_1$
1.	Control silk fabric	1.767	11.83
2.	15 % Aloe-Vera finished silk fabric	1.305	9.39

to short side chains, it is likely that these are probable alternate sites for chemical binding of Aloe-Vera. It is reported [36] that the absorption bands of the non-crosslinked regenerated silk fibroin (SF) showed a band at  $1040\text{ cm}^{-1}$ . Further the band at  $1065\text{ cm}^{-1}$  corresponds to the gly-ala linkage (wagging) and due to stretching vibration of C-O [37]. However, the band at  $1040\text{-}1065\text{ cm}^{-1}$  after crosslinking with saccharide weakened higher level of crosslinking of the saccharide in regenerated fibroin. It may be noted from this study that the band at  $1040\text{-}1065\text{ cm}^{-1}$  corresponds to binding of saccharide on fibroin. On similar analogy, it is found that the binding of Aloe Vera (Saccharide) on silk fibroin is elucidated. Binding index decreases as function of add-on% of Aloe Vera indicating the higher extent of saccharide (Aloe Vera) binding. It may further be seen that the band at  $1065\text{ cm}^{-1}$  does not become extinct as the binding is only in the amorphous region and as such the crystalline component is least affected. Hence, the band at  $1065\text{ cm}^{-1}$  which is the contribution of short chain amino acids such as glycine and serine present in silk fibroin was considered along with band at  $2978\text{ cm}^{-1}$  for estimation of the ratio of the optical density as another index of Binding ( $I_2$ ). Since the carboxylic groups and the short chain amino acids present in the silk are predominant sites for crosslinking of BTCA followed by binding of Aloe-Vera, the Indices of Binding thus calculated were analyzed.

Table 4 gives the data on the spectral analyses. It may be observed that the Index of binding corresponding to band at  $1065\text{ cm}^{-1}$  decreased as the add-on% increased. It may be pointed out that total of short chain (SC) amino acids is 87.4 % in Mulberry as against total of long side chain amino acid (LC) is 11.8 % [38]. The probability of crosslinking of BTCA with short chain amino acids is higher than that of long chain amino acids and the index of binding ( $I_2$ ) corresponding to FTIR band  $1065\text{ cm}^{-1}$  and  $2978\text{ cm}^{-1}$  should decrease as Aloe-Vera add-on increased. This should also reflect the extent of crosslinking of BTCA inter-alia binding of Aloe-Vera with silk. The data thus obtained confirms the above hypothesis.

Likewise the index of binding ( $I_1$ ) corresponding to FTIR band at  $1700\text{-}1710\text{ cm}^{-1}$  is maximum in the control sample and it decreased initially at 5 % Aloe-Vera concentration and further showed an increasing trend due to increase in the Aloe-Vera concentration. Since BTCA concentration is kept invariant, the crosslinking of BTCA with C=O and other short side chain groups of amino acids present in silk fibroin

cannot be firmly established. However, it appears that the crosslinking of BTCA to bind Aloe-Vera is mainly at carboxyl sites or the short chain amino acids. Hence, the initial decrease in the ratio and subsequent increasing trend indicates higher percent of crosslinking to block carboxyl groups thereby reducing its intensity. Based on the above analysis, it may be inferred that the finishing recipe facilitates crosslinking of BTCA and creates sites for binding of Aloe-Vera rather than impregnation or coating. This observation substantiates the findings of morphological properties by SEM studies.

### Physical Parameters of Finished Silk Fabric

Table 5 gives the weight add-on% (o.w.f.) on different treated fabrics treated with different Aloe-Vera concentrations. It may be seen that add-on% increased as the Aloe-Vera concentration increased in the recipe, although not proportionately. The moisture regain% decreased marginally with increase in Aloe-Vera concentration. It may be due to blocking of hydrophilic end groups because of crosslinking and chemical binding by BTCA inter-alia Aloe-Vera. It implies that the durable antimicrobial property in the treated fabrics is due to chemical binding rather than coating or impregnation. Further, the decrease in moisture regain is marginal and implies that the comfort properties are not affected by the finishing treatment. Overall decrease in flexural rigidity also indicates that the fabric has become more pliable as compared to the control. It may be seen that the crosslinking of BTCA has facilitated the fabric to get the pliable attributes which are related to durable press behavior. There was overall increase in loss due to abrasion which may be attributed to high add-on, at higher concentration of Aloe-Vera. The crease recovery angle increased in both warp and weft directions due to the treatment. This could be attributed to the addition of BTCA as crosslinking agent and also facilitate binding of the Aloe-Vera with silk fibroin. When one considers both flexural rigidity and crease recovery angle of various treated fabrics, it has led to improvement in the easy care properties of silk. The overall decrease in the breaking strength in both warp and weft directions were observed. It is reported that the finishing in certain cases reduce the strength by about 20 %. The loss of strength due to the treatment ranges from 12 % to 18 % and as such the observed loss in strength is well within the values acceptable in the industry. The decrease in breaking strength in the weft direction was high at low concentration treatment and it increased due to further

**Table 5.** Physical properties effect of finishing of silk on different physical parameters

Sample	Add-on (%)	Moisture regain (%)	Flexural rigidity (G)	Abrasion resistance (%)	Crease recovery (°)	Breaking strength (N)	
						Warp	Weft
Control silk	0	9.64	93.7	6.5	193	668	445
5 % Aloe-Vera treated	5.2	8.03	24.5	6.03	207	623	347
10 % Aloe-Vera treated	6.5	7.86	29.6	6.9	211	615	391
15 % Aloe-Vera treated	7.2	7.8	31.9	7.8	212	565	389

increase in add-on% of Aloe-Vera. The observed phenomenon may be attributed to high porosity in the weft yarn as compared to warp thereby facilitating migration of the liquor in high quantity contributing to reduction in inter filament friction as compared to warp.

### Conclusion

1. BTCA (1,2,3,4-butenetetracarboxylic acid) as a crosslinking agent with SHP (sodium hypophosphite) catalyst contributes as a bridge for binding of Aloe-Vera on to silk fibroin. Since Aloe-Vera is chemically bound on to the silk fibroin, it does not exhibit coating or impregnation as revealed by SEM studies.
2. FTIR spectral studies showed that carboxylic group and short chain amino acids side group are the potential sites for crosslinking of BTCA. This facilitates binding of Aloe-Vera on to the silk fibroin to exhibit Antimicrobial properties.
3. Loss in breaking strength is minimal in warp direction while it is marginally high in weft direction. This is attributed to the yarn structure in the warp and weft direction. Due to crosslinking of BTCA, Crease recovery angle improved due to the treatment. Further, the flexural rigidity decreased due to finishing treatment, however, increased marginally at 15 % concentration of Aloe-Vera.
4. The fabric exhibited Antimicrobial properties at different concentrations of Aloe-Vera. However, durability of the finish was found to be better at 15 % of Aloe-Vera concentration as revealed by Antimicrobial properties of dry cleaning of finished fabric. Antimicrobial properties of the finished fabric were found to be satisfactorily for both gram positive and gram negative bacteria. Since Aloe-Vera is a natural plant product and BTCA is a non formaldehyde resin, the finishing of silk may be accredited as eco-friendly finishing.

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### References

1. S.-W. Myung, I.-H. Choi, S.-M. Lee, J.-Y. Park, and K.-H. Lee, *Desalination*, **234**, 158 (2008).

2. J. H. Wu, Z. Wang, and S. Y. Xu, *Food Chem.*, **103**, 1255 (2007).
3. M. L. Gulrajani, "Department of Textile Technology", p.63, Indian Institute of Technology, New Delhi, 1988.
4. C. M. Welch, *Text. Res. J.*, **58**, 480 (1988).
5. C. M. Welch and B. A. Kottes Andrews, *U.S. Patent* 4,307,820 (1989).
6. W. L. Xu and Y. Li, *Text. Res. J.*, **70**, 588 (2000).
7. W. L. Xu and Y. Li, *Text. Res. J.*, **70**, 957 (2000).
8. C. Q. Yang, X. Wang, and I. S. Kang, *Text. Res. J.*, **67**, 334 (1997).
9. B. A. K. Andrews, *Text. Chem. Color.*, **22**, 63 (1990).
10. B. Gutarowska, W. Machnowski, and L. Kowzowicz, *Fiber Polym.*, **14**, 415 (2013).
11. Y.-H. Lee, E.-K. Hwang, Y.-M. Back, M.-S. Lee, D.-J. Lee, Y.-J. Jung, and H.-D. Kim, *Fiber Polym.*, **14**, 1445 (2013).
12. A. Irvin, Ph.D. Dissertation, University of Leeds, Leeds, 1999.
13. Z. Yang and J. Hu, *RJTA*, **10**, 46 (2006).
14. E. Femenia, S. Sanchez, S. Simal, and C. Rossello, *Carbohydr Polym.*, **39**, 109 (1999).
15. A. Djeraba and P. Quere, *Int. J. Immunopharmacol.*, **22**, 365 (2000).
16. J. K. Lee, M. K. Lee, Y. P. Yun, Y. Kim, J. S. Kim, Y. S. Kim, K. Kim, S. S. Han, and C. K. Lee, *Int. Immunopharmacol.*, **1**, 1275 (2001).
17. M. Joshi and S. W. Ali, *Indian J. Fibre Text. Res.*, **34**, 295 (2009).
18. Y. Ni, D. Turner, K. M. Yates, and I. Tizard, *Int. Immunopharmacol.*, **4**, 1745 (2004).
19. S. Choi and M. H. Chung, *Semin. Integr. Med.*, **1**, 53 (2003).
20. Y. Ni, K. M. Yates, and I. R. Tizard, "Aloe Polysaccharides. In Aloes The Genus Aloe", pp.75-87. CRC Press, Boca Raton, 2004.
21. M. Castleman, "The Healing Herbs", pp.42-44, Rodale Press, Em-maus, 1991.
22. P. de Witte, *Pharmacology*, **47**, 86 (1993).
23. A. O. Azghani, I. Williams, D. B. Holiday, and A. R. Johnson, *Glycobiology*, **5**, 39 (1995).
24. L. M. Cera, J. P. Heggors, M. C. Robson, and W. J. Hagstrom, *J. Am. Anim. Hosp. Assoc.*, **16**, 768 (1980).
25. V. Visuthikosol, B. Chowchuen, Y. Sukwanarat, S. Sriuiraratana, and V. Boonpucknavig, *J. Med. Assoc. Thailand*, **78**, 403 (1995).
26. R. H. Davis, J. M. Kabbani, and N. P. Maro, *J. Am. Podiatr. Med. Assoc.*, **77**, 165 (1987).
27. R. H. Davis, M. G. Leitner, and J. M. Russo, *J. Am. Podiat. Med. Assoc.*, **78**, 60 (1988).
28. T. Kaufman, A. R. Newman, and M. R. Wexler, *Plast. Reconstr. Surg.*, **83**, 1075 (1989).
29. N. Pugh, S. A. Ross, M. A. Elsohly, and D. S. Pasco, *J. Agric. Food Chem.*, **49**, 1030 (2001).
30. K. Tan and J. Vanitha, *Curr. Med. Chem.*, **11**, 1423 (2004).

31. E. Dagne, D. Bisrat, A. Viljoen, and B.-E. Van Wyk, *Curr. Org. Chem.*, **4**, 1055 (2000).
32. A. Femenia, E. S. Sanchez, S. Simal, and C. Rosello, *Carbohydr. Polym.*, **39**, 109 (1999).
33. S. Choi and M. H. Chung, *Semin. Integr. Med.*, **1**, 53 (2003).
34. T. N. Sonwalkar, "Handbook of Silk Technology", pp.170-173, Wiley Eastern Limited, New Delhi, 1993.
35. Y. Yang and S. Li, *U.S. Patent* 5,296,269 (1994).
36. Y. Baimark and Y. Srisuwan, *Int. J. Chem. Appl.*, **4**, 259 (2012).
37. M. Bhattacharjee, S. Miot, A. Gorecka, K. Singha, Markoloparic, S. Dickinson, A. Das, N. S. Bhavesh, A. R. Ray, I. Martin, and S. Ghosh, *Acta Biomater.*, **8**, 3313 (2012).
38. G. S. Nadiger and V. G. Halliyal, *Colourage*, **31**, 23 (1984).