**Polyacrylic Acid and Chitosan Treatments on Silk Fabric for Protection Enhancements**

**ABSTRACT**

Silk is utilized as most valuable fibers based on the unique properties like shiny sheen, remarkable comfort performance and bio considerations. Silk materials possess high applications as a natural substrate in the textile/garment industry and as a bio component in the medical applications. As silk is fine, delicate and sensitive it needs more care to protect from the ill effects of insects, light and chemicals. Hence, in this work an attempt is carried out on silk fabric by treatment with polyacrylic acid and chitosan followed by dyeing using few natural dyes and one synthetic reactive dye and subsequently subjected for different tests towards physical properties, colorimetric and fastness properties, low stress mechanical properties, antimicrobial and uv protection properties, SEM and XRD studies. The results of these tests give very good improvement of the protection behavior on the silk fabric suitable for the garments and other end use products.

**KEYWORDS:** Silk, Polyacrylic acid, Chitosan, Anti-microbial, uv protection factor

## 1. INTRODUCTION

"Silk fiber substrate is regarded as the well known natural protein textiles being used as a luxurious and prestigious materials for centuries due to the unique properties of elegant appearance, soft handle, wearing comfort, air permeability, brilliant color shades of dyeing and printing together with the conventional utility of fiber strength, elasticity, resilience, drape ability, heat conductivity, and absorbency" [1,2]. Silk textiles are identified as the most attractive and lustrous of all the natural fiber categories [3,4,5].

"Silk fibers of Bombyx-mori collected from the cocoon consist generally of two components like fibroin and sericin which constitute around 70% and 30% respectively. A glue like protein called sericin contains the amino acids such as glycine, serine, aspartic acid totaling more than 60% and keeps the two fibroin fibers intact to form the environmentally stable fibroin–sericin structure" [6,7]. "As a natural material, silk finds varied applications in the textile/garment sectors and as a bio component for medical applications as scaffolds and sutures" [8,9,10]. "The wide spectrum of applications from silk is due to its basic properties of sheen, preferable comfort performance, and bio suitability. Hence, based on all these attractive and quality performances, silk has been historically categorized as the queen of all the textiles" [2,4].

Although silk contains many advantages factors some drawbacks also present in it which limit its use. As silk is delicate and costlier textile fiber, it needs more care to protect from the damage of insects, light, and chemicals. Hence, in order to protect and enhance the properties of silk material, it is decided in this work to treat the silk fabric with polyacrylic acid (PAA) and chitosan (CN). The treated silk fabrics are then dyed with few natural dyes and one synthetic reactive dye and periodically undergone with different tests for physical properties, colorimetric and fastness properties, low stress mechanical properties, antimicrobial and uv protection properties, SEM and XRD studies. The test results give a favourable outlook on the silk fabric with enhanced protection aspects suitable for the valuable garments, apparels and other end use products.

## 2. MATERIAL AND METHOD

### 2.1 Material

The 100% mulberry silk fabric in plain form was procured from the Vadavalli Sarvodaya Sangam, Coimbatore, India. The forest department, Coimbatore, India supplied the natural sources. The synthetic reactive dye was purchased from the commercial shop in Tirupur, India. The details of all these items are mentioned below in the Table (1). The Polyacrylic acid (PAA) was procured from the local scientific company, Coimbatore, India. Commercial chitosan was procured from M/s Cochin Refineries Company, Kerala, India. In this study, the chemicals and auxiliaries mentioned elsewhere were in the AR grade

**Table (1).** Details of Silk Fabric and Coloring Agents

| S. No. | Details of Silk Fabric |
|---|---|
| 1. | Warp Count → $2/80^s$ |
| 2. | Weft Count → $2/80^s$ |
| 3. | Ends / Inch → 100 |
| 4. | Picks / Inch → 60 |
| 5. | GSM → 95 |
| 6. | Cloth Width (Inch) → 44 |
| **S. No.** | **Details of Coloring Agents** |
| 1. | Kum kum (*crocus sativa*) → Yellow |
| 2. | Bar berry (*berberis vulgaris*) → Yellow |
| 3. | Annatto (*bixa orellana*) → Orange |
| 4. | Onion (*allium cepa*) → Red Orange |
| 5. | Red Sandalwood (*pterocarpus santallinus*) → Orange Red |
| 6. | Grape (*citrus paradise*) → Purple |
| 7. | Madder (rubia cardifolia) → Red |
| 8. | Reactive yellow HE6G (C.I. Name: *Reactive yellow 135*) → Yellow |

### 2.2 Method

#### 2.2.1 Degumming of the Raw Silk Fabrics

The raw silk fabric was pretreated with hydrochloric acid (10 gpl) for one hour with a MLR (material to liquor ratio) of 1:30 at 30$^o$C to remove the unwanted substrates. Then the degumming was continued on the pretreated silk fabric by sodium carbonate (2% owm) at 85$^{o}$C for two hours by the established technique mentioned earlier [11,12,13].

### 2.2.2 Treatment of the Silk Fabrics With Polyacrylic Acid

"The polyacrylic acid (PAA) (concentration of 0.5%, 1%, 1.5%, 2%, 2.5%, 3.0%, 3.5%, 4%, 4.5%, 5%, 5.5%, % 6.0% w/v) in 0.25 N NaOH was applied on degummed silk fabric for 30 minutes. After this treatment the silk samples were squeezed, dried at 105$^{o}$C for 30 minutes, and cured at 140$^{o}$C for 60 seconds. Then the silk samples were washed, dried and kept ready for the next treatments" [14].

### 2.2.3 Treatment of the Silk Fabrics With Chitosan

The chitosan was dissolved with the help of 2% acetic acid solution [15, 16], filtered and kept as the stock solution for application. The required concentration of the solution was taken in a suitable bath with material to liquor ratio of 1:30. "The silk samples were immersed into the chitosan bath; pH was maintained at 5.0±0.2 using acetic acid. The temperature of this bath was raised to 95$^{o}$C and continued at that temperature for 30 minutes. The samples after the application were taken out, washed with warm water followed by normal water and then dried" [15,16,17].

### 2.2.4 Dyeing and Measurement of K/S Value of the Dyed Silk Fabrics

"The dyeing was carried out on the silk fabric samples with the concentration of 25 gram per litre (gpl) for natural dyes and 2% on weight of material (owm) for synthetic dyes at boil for two hours with a material to liquor ratio (MLR) of 1:20 as per the established technique of dyeing" [18,19,20].

### 2.2.5 Fastness Properties of the Dyed Silk Fabrics

"The IS 764-test 3-1979 method was used for the determination of wash fastness of the dyed silk fabric; AATCC test method 16E-2004 was used for the light fastness determination; AATCC test method 8-2007 was used for the determination of fastness to crocking" [21,22,23]. To determine the rubbing fastness of dyed silk fabrics under wet and dry conditions, AATCC standardized crock meter was used [24].

### 2.2.6 Measurement of Physical Properties of the Silk Fabrics

"The standard established methods such as grab tensile test method, Elmendorf tear tester as per ASTM D1424 – 2009, Cusik Drape meter, Rth = L/k, ((m2 ºC)/W), and Shirley stiffness tester respectively were used for the measurement of the physical properties such as tensile strength, elongation, drapeability, thermal resistance, and stiffness of the silk fabrics" [25,26].

### 2.2.7 Objective Assessment of the Silk Fabrics by KES-F

The mechanical and surface properties of the silk fabrics were assessed by Kawabata evaluation system (KES-F) [27,28].

### 2.2.8 Antimicrobial Assessment of the Silk Fabrics

The agar diffusion test (SN 195920) and the modified Hohenstein test (JIS L 1902) were subjected on the silk fabrics. The Staphylococcus aureus (AATCC 6538) and Escherichia coli (AATCC 11230) were the organisms used in both the tests [29,30,31].

### 2.2.9 UV Protection Factor Assessment on the Silk Fabrics

The silk fabric samples were subjected with uv protection finishing (Super FX Anti UV) ingredients and tested subsequently according to the standard method [32,33].

### 2.2.10 SEM Study on the Silk Fabrics

The 30kV scanning electron microscope JEOL (Japan) Model JSM-6360 was used for the SEM study on the silk fabric samples [34,35].

### 2.2.11 XRD Study on the Silk Fabrics

The unknown crystalline components present in the silk fabric samples were determined using XRD technique (Shimadzu XRD6000) [36,37].

## 3. RESULTS AND DISCUSSION

### 3.1 Physical Properties of the Silk Fabrics

The basic physical properties of the silk fabrics (tensile strength, elongation, drape co-efficient, thermal resistance, and stiffness / bending modulus value) with no treatment, PAA treatment and CN treatment are presented in Table 2 and related values for the treated fabrics after dyeing are given in Table 2a. According to the trials run for the optimization of focus and time, the PAA and CN treatments on silk fabrics have been finalised. Earlier, the silk fabrics were treated with PAA and CN with the concentration started from 0.5% w/v to 6.0% w/v with different time duration from 5 minutes to 60 minutes with a step addition of 5. It was optimized that the PAA and CN treatments on silk gave favorable results at the time duration of 30 minutes. Hence, it was decided to apply the PAA and CN on the silk fabric with the concentration of 0.5% w/v to 6.0% w/v for the time duration of 30 minutes only for all the treatments and the data are presented in the Table 2. From table 2, it is seen that when the concentrations of PAA and CN treatments on the silk fabric increases the physical properties also corresponding influenced. It is well known that the tensile strength which means that the material under tensile stress in the largest deformation of homogeneous material stress. The tensile strength of PAA and CN treated silk fabrics is substantially increased compared with the silk fabric without these treatments. It is observed that there is nearly 8% and 10% increase of tensile strength as an average for both warp and weft of silk fabric treated with PAA and CN respectively over that of untreated one. Between the PAA and CN treated silk fabrics for both the average of warp and weft, the latter one has the edge of around 3% increase. When compared to the corresponding weft fabric, silk fabric has a significantly higher warp way tensile strength. As the concentration of PAA and CN increases there is a considerable increase in the tensile strength both in warp and weft way up to 4% w/v only, afterwards the increase is insignificant. In the case of elongation (%) among the PAA, CN and no treated silk fabrics the difference is only marginal and there is a decrease in the tune of maximum of average 3% only. Elongation is considered as the increase in length or deformation of a textile fiber due to stretching. The elongation is revealed to be very important, since the textile products particularly garments like corsetry, and stretch products without elasticity would hardly be usable. The drape coefficient of the silk fabric is not influenced after the PAA and CN treatment, the average decrease is around only 1.0%. It is known that drape is the term used to mention the way of a fabric hangs under its own weight. The draping qualities that required from a textile fabric will change completely depending on its end use. It is meant that the measurement of a fabric's drape to assess its ability and also its hanging aspects in graceful curves. The PAA and CN treatments on the silk fabrics do not give any impact in the thermal resistances which is almost retained around 98 ($m^2$.mk/w) in all the cases. The facts of thermal characteristics is believed important since it is directly related to clothing comfort. Compared to that of the untreated one, the bending modulus values of the PAA and CN treated silk fabrics show around one fold increase. The stiffness test determines the bending stiffness of a fabric by allowing a narrow strip of the fabric to bend to a fixed angle under its own weight. The higher the bending length, the stiffer is the fabric. The stiffness of a fabric in bending is very dependent on its thickness because thicker the fabric stiffer it is, if all other factors remain the same. From the Table 2, it is seen in the physical property values of the silk fabrics applied with PAA and CN that there is a considerable difference up to the concentration of 4% (w/v) after that there is no significant change. With a concentration of 4% w/v for 30 minutes, the application of PAA and CN on silk fabrics is therefore optimised; this factor is chosen and fixed for the later works. "After the PAA and CN treatments (4% w/v, 30 minutes) the silk fabrics were dyed with few natural dyes (kum kum, bar berry, annatto, onion, red sandalwood, grape, and madder) and a synthetic reactive yellow dye; then the corresponding physical properties were studied and the data are shown in Table 2a. The values in Table 2a follow the similar trend as discussed with the Table 2. The difference is significant only in the case of the untreated silk fabrics when compared with that of PAA and CN treated and dyed silk fabrics. Among the PAA and CN treated dyed silk fabrics, there is only insignificant difference in the physical properties" [38,39,40].

**Table 2.** Physical Properties of the Silk Fabrics

| S. No. | Concentration of Chemicals (%w/v) | Time (sec) | Tensile Strength (Kgf/mm2) | | Elongation (%) | | Drape co-efficient | Thermal Resistance ($m^2$.mk/w) | Stiffness / Bending Modulus Value |
|---|---|---|---|---|---|---|---|---|---|
| | | | Warp | Weft | Warp | Weft | | | |
| | | Silk Fabric with No Treatment | | | | | | | |
| 1 | 00 | 00 | 55.12 | 47.88 | 16.7 | 15.8 | 0.677 | 98.87 | 0.0014 |
| S. No. | PAA (%w/v) | Silk Fabric with PAA Treatment | | | | | | | |
| 1 | 0.5 | 30 | 58.15 | 50.96 | 16.6 | 15.7 | 0.676 | 98.86 | 0.0015 |
| 2 | 1.0 | 30 | 58.24 | 51.05 | 16.6 | 15.6 | 0.676 | 98.81 | 0.0015 |
| 3 | 2.0 | 30 | 59.17 | 51.28 | 16.2 | 15.3 | 0.675 | 98.70 | 0.0016 |
| 4 | 3.0 | 30 | 59.56 | 51.94 | 15.8 | 14.9 | 0.674 | 98.48 | 0.0016 |
| 5 | 4.0 | 30 | 61.15 | 53.86 | 15.0 | 14.0 | 0.670 | 97.86 | 0.0020 |
| 6 | 5.0 | 30 | 61.26 | 53.92 | 14.9 | 13.9 | 0.670 | 97.81 | 0.0022 |
| 7 | 6.0 | 30 | 61.39 | 53.99 | 14.8 | 13.8 | 0.669 | 97.79 | 0.0023 |
| S. No. | Chitosan (%w/v) | Silk Fabric with Chitosan Treatment | | | | | | | |
| 1 | 0.5 | 30 | 60.65 | 53.08 | 16.0 | 15.1 | 0.673 | 98.84 | 0.0016 |
| 2 | 1.0 | 30 | 60.84 | 53.25 | 15.6 | 15.0 | 0.672 | 98.79 | 0.0017 |
| 3 | 2.0 | 30 | 61.57 | 53.58 | 15.6 | 14.7 | 0.670 | 98.68 | 0.0017 |
| 4 | 3.0 | 30 | 61.96 | 54.24 | 15.2 | 14.3 | 0.667 | 98.45 | 0.0018 |
| 5 | 4.0 | 30 | 63.75 | 56.16 | 14.4 | 13.4 | 0.658 | 97.82 | 0.0022 |
| 6 | 5.0 | 30 | 63.87 | 56.25 | 14.3 | 13.3 | 0.657 | 97.79 | 0.0023 |
| 7 | 6.0 | 30 | 63.98 | 56.31 | 14.2 | 13.2 | 0.657 | 97.78 | 0.0023 |



**Table 2a.** Physical Properties of the Silk Fabrics

| S. No. | Dyes | Tensile Strength (Kgf/mm2) | | Elongation (%) | | Drape Co-efficient | Thermal Resistance ($m^2$.mk/w) | Stiffness / Bending Modulus Value |
|---|---|---|---|---|---|---|---|---|
| | | Warp | Weft | Warp | Weft | | | |
| | | PAA Treated Silk Fabric | | | | | | |
| 1 | Kum kum | 63.25 | 55.67 | 14.0 | 13.0 | 0.668 | 97.83 | 0.0021 |
| 2 | Bar berry | 63.32 | 55.77 | 14.3 | 13.3 | 0.666 | 97.82 | 0.0023 |
| 3 | Annatto | 63.48 | 55.82 | 14.2 | 13.2 | 0.667 | 97.82 | 0.0022 |
| 4 | Onion | 63.29 | 55.74 | 14.3 | 13.4 | 0.668 | 97.81 | 0.0021 |
| 5 | Red Sandalwood | 63.37 | 55.86 | 14.6 | 13.6 | 0.667 | 97.80 | 0.0021 |
| 6 | Grape | 63.42 | 55.84 | 14.3 | 13.3 | 0.666 | 97.83 | 0.0022 |
| 7 | Madder | 63.27 | 55.79 | 14.2 | 13.4 | 0.667 | 97.81 | 0.0021 |
| 8 | Reactive Dye | 63.53 | 55.93 | 14.8 | 13.9 | 0.669 | 97.84 | 0.0023 |
| | | Chitosan Treated Silk Fabric | | | | | | |
| 1 | Kum kum | 65.85 | 57.86 | 13.7 | 12.7 | 0.655 | 97.78 | 0.0024 |
| 2 | Bar berry | 65.87 | 57.22 | 13.9 | 13.0 | 0.654 | 97.77 | 0.0024 |
| 3 | Annatto | 65.86 | 57.98 | 14.1 | 12.9 | 0.654 | 97.78 | 0.0025 |
| 4 | Onion | 65.85 | 57.37 | 13.9 | 13.1 | 0.656 | 97.79 | 0.0025 |
| 5 | Red Sandalwood | 66.17 | 57.44 | 14.0 | 12.9 | 0.655 | 97.78 | 0.0024 |
| 6 | Grape | 65.88 | 57.97 | 14.1 | 13.1 | 0.654 | 97.77 | 0.0025 |
| 7 | Madder | 65.86 | 57.79 | 13.9 | 13.0 | 0.655 | 97.78 | 0.0024 |
| 8 | Reactive Dye | 66.47 | 58.12 | 14.2 | 13.2 | 0.657 | 97.81 | 0.0027 |
| | | Silk Fabric with No Treatment | | | | | | |
| 0 | No Dye | 55.12 | 47.88 | 16.7 | 15.8 | 0.677 | 98.87 | 0.0014 |
| 1 | Kum kum | 58.35 | 51.09 | 15.9 | 14.3 | 0.676 | 98.11 | 0.0014 |
| 2 | Bar berry | 58.76 | 51.37 | 15.2 | 14.4 | 0.675 | 98.20 | 0.0014 |
| 3 | Annatto | 58.54 | 51.15 | 15.3 | 14.2 | 0.673 | 98.15 | 0.0015 |
| 4 | Onion | 58.47 | 51.14 | 15.1 | 14.3 | 0.674 | 98.18 | 0.0015 |
| 5 | Red Sandalwood | 58.68 | 51.18 | 15.2 | 14.4 | 0.676 | 98.16 | 0.0014 |
| 6 | Grape | 58.59 | 51.15 | 15.4 | 14.2 | 0.674 | 98.21 | 0.0014 |
| 7 | Madder | 58.62 | 51.17 | 15.3 | 14.3 | 0.674 | 98.19 | 0.0014 |
| 8 | Reactive Dye | 58.78 | 51.39 | 15.6 | 14.8 | 0.676 | 98.22 | 0.0015 |

### 3.2 Colorimetric Data of Dyed Silk Fabrics

Table 3 shows the colorimetric data of silk fabric with no treatment, PAA and CN treatments followed by dyeing with natural dyes and synthetic reactive dye. It is observed that the K/S values of PAA and CN treated dyed fabrics are significantly high compared with that of no treated and dyed silk fabrics. The increase of K/S value for the respective PAA and CN treated dyed fabrics over that of untreated dyed one is an average of 13% and 15% respectively. Between the PAA and CN treated dyed silk fabrics, the latter one has the edge with the increase of nearly 1.5%. There is an enhancement of reactive groups, based on the application of PAA and CN on the silk fabrics that are responsible for the increase of dye uptake compared with the untreated one [39,41,42,43].

**Table 3.** Colorimetric Data of dyed Silk Fabrics

| S. No. | Dyes | Colorimetric Data of Dyed Silk Fabrics | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | PAA Treatment | | | | | | Chitosan Treatment | | | | | |
| | | L* | a* | b* | C | h° | K/S | L* | a* | b* | C | h° | K/S |
| 1 | Kum kum | 34.3 | -4.9 | -16.6 | 21.2 | 257 | 14.6 | 34.6 | -5.6 | -15.9 | 21.6 | 259 | 14.7 |
| 2 | Bar berry | 35.5 | -5.8 | -14.7 | 22.6 | 258 | 14.5 | 33.9 | -6.9 | -15.3 | 23.6 | 258 | 14.5 |
| 3 | Annatto | 35.2 | -5.9 | -16.2 | 23.2 | 246 | 14.6 | 35.3 | -5.7 | -15.1 | 22.7 | 253 | 14.7 |
| 4 | Onion | 29.2 | -5.9 | -14.5 | 22.2 | 249 | 14.9 | 34.9 | -5.1 | -17.9 | 22.1 | 259 | 14.9 |
| 5 | Red Sandalwood | 33.1 | -4.0 | -15.3 | 21.7 | 254 | 14.3 | 36.9 | -5.4 | -16.4 | 23.3 | 256 | 14.5 |
| 6 | Grape | 34.9 | -4.5 | -15.9 | 21.4 | 258 | 14.0 | 35.7 | -5.8 | -15.3 | 22.3 | 256 | 14.3 |
| 7 | Madder | 35.6 | -5.8 | -14.6 | 22.9 | 245 | 14.6 | 35.6 | -6.8 | -17.6 | 23.7 | 248 | 14.7 |
| 8 | Reactive Dye | 35.3 | -4.3 | -15.2 | 23.5 | 241 | 14.9 | 32.2 | -6.3 | -16.3 | 21.6 | 253 | 14.9 |

| S. No. | Dyes | Colorimetric Data of Dyed Silk Fabrics (No Treatment) | | | | | |
|---|---|---|---|---|---|---|---|
| | | L* | a* | b* | C | h° | K/S |
| 1 | Kum kum | 33.3 | -6.7 | -15.6 | 23.2 | 253 | 12.6 |
| 2 | Bar berry | 33.6 | -6.4 | -17.7 | 22.3 | 245.4 | 12.5 |
| 3 | Annatto | 34.7 | -5.5 | -14.9 | 22.5 | 246.0 | 12.9 |
| 4 | Onion | 35.9 | -5.0 | -15.6 | 22.7 | 267 | 13.2 |
| 5 | Red Sandalwood | 32.8 | -5.6 | -17.6 | 21.3 | 249.4 | 12.2 |
| 6 | Grape | 25.5 | -5.6 | -15.9 | 22.3 | 252.2 | 11.7 |
| 7 | Madder | 32.5 | -4.6 | -17.9 | 22.3 | 250.8 | 12.8 |
| 8 | Reactive Dye | 34.8 | -6.4 | -15.6 | 21.1 | 253 | 13.5 |



### 3.3 Fastness Properties of Dyed Silk Fabrics

The data of the fastness properties (wash, light, stain and rub) obtained from the silk fabric subjected with PAA and CN treatments followed by dyeing with natural dyes (kum kum, bar berry, annatto, onion, red sandalwood, grape, and madder) and synthetic reactive dye are given in Table 4. The PAA and CN treated dyed silk fabrics show very good overall fastness properties over that of the corresponding untreated silk fabrics. The CN treated dyed silk fabrics show the maximum over all fastness properties. The treated (PAA and CN) dyed silk fabrics show good to very good (3 – 4) wash fastness properties; very good (4 - 5) light fastness properties; very good (3 – 4) stain fastness properties; and poor to moderate (2 – 3) rubbing fastness properties. The good over all fastness properties in general on the PAA and CN treated silk fabrics is revealed due to the good application and presence of the reactive groups involved with the material [39,44,45].

**Table 4.** Fastness Properties of Dyed Silk Fabrics

| S. No. | Dyes | Fastness Properties of Dyed Silk Fabrics | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | PAA Treatment | | | | | Chitosan Treatment | | | | |
| | | Wash | Light | Stain | Rub | | Wash | Light | Stain | Rub | |
| | | | | | Wet | Dry | | | | Wet | Dry |
| 1 | Kum kum | 3 | 4 | 2-3 | 2-3 | 3 | 3 | 4-5 | 3 | 2-3 | 3-4 |
| 2 | Bar berry | 3 | 4 | 3 | 2 | 3 | 3 | 4 | 3-4 | 2-3 | 3-4 |
| 3 | Annatto | 3 | 4 | 2-3 | 2-3 | 3-4 | 3-4 | 4-5 | 3 | 2-3 | 3-4 |
| 4 | Onion | 3-4 | 4-5 | 3-4 | 2-3 | 3 | 3-4 | 5 | 4 | 2-3 | 3-4 |
| 5 | Red Sandalwood | 3 | 4 | 3 | 2 | 3 | 3 | 4 | 3-4 | 2-3 | 3-4 |
| 6 | Grape | 3 | 3-4 | 2-3 | 2-3 | 3-4 | 3 | 4 | 3 | 2-3 | 3-4 |
| 7 | Madder | 3 | 4 | 3 | 3 | 3-4 | 3-4 | 4 | 3-4 | 3 | 3-4 |
| 8 | Reactive Dye | 3-4 | 4-5 | 4 | 2 | 3 | 4 | 5-6 | 4-5 | 2-3 | 3 |

| S. No. | Dyes | Fastness Properties of Dyed Silk Fabrics (No Treatment) | | | | |
|---|---|---|---|---|---|---|
| | | Wash | Light | Stain | Rub | |
| | | | | | Wet | Dry |
| 1 | Kum kum | 3 | 4 | 2-3 | 2 | 2 |
| 2 | Bar berry | 2-3 | 3-4 | 3 | 2 | 2-3 |
| 3 | Annatto | 3 | 4 | 2-3 | 2 | 2-3 |
| 4 | Onion | 3 | 4 | 3-4 | 2 | 3 |
| 5 | Red Sandalwood | 3 | 3-4 | 3 | 2 | 2 |
| 6 | Grape | 2-3 | 3 | 2-3 | 2 | 2-3 |
| 7 | Madder | 3 | 4 | 3 | 2 | 3 |
| 8 | Reactive Dye | 3-4 | 4-5 | 3-4 | 2 | 2-3 |



### 3.4 Primary Hand Value (PHV) of the Silk Fabrics

The primary hand values (1. Smoothness, 2. Stiffness, and 3. Fullness) of the silk fabric treated with PAA and CN followed by dyeing with natural dyes and synthetic reactive dye are given in Table 5. From this table it is observed that the smoothness of the CN treated dyed silk fabric is maximum followed by the PAA treated and no treated silk fabrics. "The CN treated dyed silk fabrics show a significant increase of smoothness value nearly 25% and 7% over that of untreated and PAA treated ones respectively. The PAA and CN treated dyed silk fabrics show reasonably decrease in stiffness values (av. 7% and 10%) over that of untreated one. The fullness of the PAA and CN treated dyed silk fabrics (av. 11% and 15%) is considerably more compared to that of untreated one. Between the CN and PAA treated dyed silk fabrics the former one is reasonably more of around 5%" [39,43,46,47].

**Table 5.** Primary Hand Value (PHV) of the Silk Fabrics

| S. No. | Dye | Primary Hand Value (PHV) of the Silk Fabrics | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | PAA Treatment | | | Chitosan Treatment | | | No Treatment | | |
| | | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 |
| 0 | No Dye | 7.2 | 7.0 | 7.3 | 8.0 | 6.7 | 7.7 | 5.9 | 7.4 | 6.5 |
| 1 | Kum kum | 7.6 | 6.8 | 7.5 | 8.2 | 6.6 | 7.8 | 6.1 | 7.3 | 6.6 |
| 2 | Bar berry | 7.6 | 6.9 | 7.6 | 8.3 | 6.6 | 7.9 | 6.2 | 7.3 | 6.7 |
| 3 | Annatto | 7.5 | 6.8 | 7.6 | 8.3 | 6.5 | 7.9 | 6.2 | 7.2 | 6.6 |
| 4 | Onion | 7.7 | 6.7 | 7.8 | 8.5 | 6.4 | 8.2 | 6.3 | 7.2 | 6.8 |
| 5 | Red Sandal wood | 7.5 | 6.8 | 7.7 | 8.3 | 6.6 | 8.1 | 6.2 | 7.3 | 6.6 |
| 6 | Grape | 7.5 | 6.9 | 7.5 | 8.4 | 6.6 | 8.0 | 6.1 | 7.3 | 6.7 |
| 7 | Madder | 7.6 | 6.8 | 7.6 | 8.4 | 6.5 | 7.9 | 6.2 | 7.3 | 6.7 |
| 8 | Reactive Dye | 7.8 | 6.6 | 7.9 | 8.6 | 6.4 | 8.2 | 6.3 | 7.2 | 6.9 |

1. Smoothness; 2. Stiffness; 3. Fullness

### 3.5 Bending Length and Crease Recovery of the Silk Fabrics

The data of bending length and crease recovery of PAA and CN treated dyed silk fabrics are given in Table 6. The bending length is comparatively reduced both in warp and weft way respectively when the silk fabrics are treated with PAA and CN and dyed. It is known that the bending length is also referred as the flexibility of the fabric, and that is considered as the very important aspects of the fabric responsible for many applications. The textile fabric which comprises more bending length are stiffer, lack good drape and flexibility; and therefore when a fabric falls under its own weight to a specific length and specific angle, then the bending length can also be considered as the falling length. Crease recovery is observed as one of the important properties of a textile material by which it can return to its original shape after being creased. Accordingly, crease recovery angle is the specified quantitative measure of the crease resistance. Subsequently, the CN treated dyed silk fabrics show the maximum crease recovery followed by PAA treated and untreated one [43,45,48,49].

**Table 6.** Bending Length and Crease Recovery of the Silk Fabrics

| S. No. | Dyes | Bending Length and Crease Recovery of the Silk Fabrics | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Bending Length (mm) of Dyed Silk Fabrics | | | | | | Crease Recovery ($^o$) of Dyed Silk Fabrics | | | | | |
| | | PAA | | CN | | NO | | PAA | | CN | | NO | |
| | | Warp (Cw) | Weft (Cf) | Warp (Cw) | Weft (Cf) | Warp (Cw) | Weft (Cf) | Warp (Cw) | Weft (Cf) | Warp (Cw) | Weft (Cf) | Warp (Cw) | Weft (Cf) |
| 0 | No Dye | 10.0 | 09.7 | 09.6 | 09.3 | 10.4 | 10.2 | 114 | 112 | 117 | 113 | 111 | 109 |
| 1 | Kum kum | 09.9 | 09.6 | 09.4 | 09.2 | 10.3 | 10.1 | 112 | 111 | 115 | 110 | 108 | 105 |
| 2 | Bar berry | 09.8 | 09.5 | 09.4 | 09.2 | 10.3 | 10.1 | 112 | 111 | 114 | 110 | 109 | 106 |
| 3 | Annatto | 09.9 | 09.6 | 09.5 | 09.3 | 10.2 | 10.0 | 112 | 110 | 115 | 111 | 108 | 105 |
| 4 | Onion | 09.7 | 09.4 | 09.2 | 08.9 | 10.1 | 09.9 | 110 | 108 | 113 | 109 | 107 | 103 |
| 5 | Red Sandal wood | 09.9 | 09.6 | 09.4 | 09.2 | 10.2 | 10.1 | 111 | 110 | 114 | 110 | 108 | 105 |
| 6 | Grape | 09.8 | 09.5 | 09.3 | 09.1 | 10.2 | 10.1 | 112 | 110 | 115 | 111 | 109 | 106 |
| 7 | Madder | 09.9 | 09.6 | 09.5 | 09.2 | 10.2 | 10.1 | 111 | 109 | 114 | 110 | 109 | 106 |
| 8 | React ive Dye | 09.6 | 09.3 | 09.1 | 08.8 | 10.0 | 09.8 | 109 | 107 | 111 | 108 | 106 | 102 |

PAA – Polyacrylic treated ; CN – Chitosan Treated ; NO – No Treatment Silk Fabrics

### 3.6 Antibacterial Assessment of the Silk Fabrics

The antibacterial assessment vales of PAA and CN treated dyed silk fabrics for Staphylococcus aureus and Escherichia coli are provided in the Table 7. By qualitative test method, the antimicrobial activity of the silk fabrics was assessed. It is observed from the Table 7 that the silk fabrics when compared to Escherichia coli, show a higher zone of inhibition against Staphylococcus aureus. Over the untreated one, the CN and PAA treated dyed silk fabrics show a significant increase of antibacterial activity of around 30% and 25% respectively; moreover between the CN and PAA treated dyed silk fabrics the former shows around 8% increase over the latter. Towards the consumers' attitude, the hygiene and active lifestyle has created a rapidly increasing market over a wide range of textile products possessed with antimicrobial properties, which subsequently has stimulated intensive research and development [40,45,50,51,52].

**Table 7.** Antibacterial Assessment of the Silk Fabrics

| S. No. | Dyes | Antibacterial Assessment of the Silk Fabrics | | | | | |
|---|---|---|---|---|---|---|---|
| | | PAA Treatment | | Chitosan Treatment | | No Treatment | |
| | | SA | EC | SA | EC | SA | EC |
| 1 | Kum kum | 36 | 34 | 39 | 36 | 27 | 25 |
| 2 | Bar berry | 37 | 35 | 40 | 37 | 27 | 26 |
| 3 | Annatto | 36 | 33 | 39 | 37 | 28 | 25 |
| 4 | Onion | 38 | 36 | 41 | 39 | 29 | 26 |
| 5 | Red Sandal wood | 36 | 34 | 39 | 36 | 28 | 25 |
| 6 | Grape | 36 | 34 | 38 | 35 | 27 | 24 |
| 7 | Madder | 37 | 35 | 39 | 36 | 28 | 25 |
| 8 | Reactive Dye | 40 | 37 | 43 | 41 | 31 | 29 |



## 3.7 UPF Assessment of the Silk Fabrics

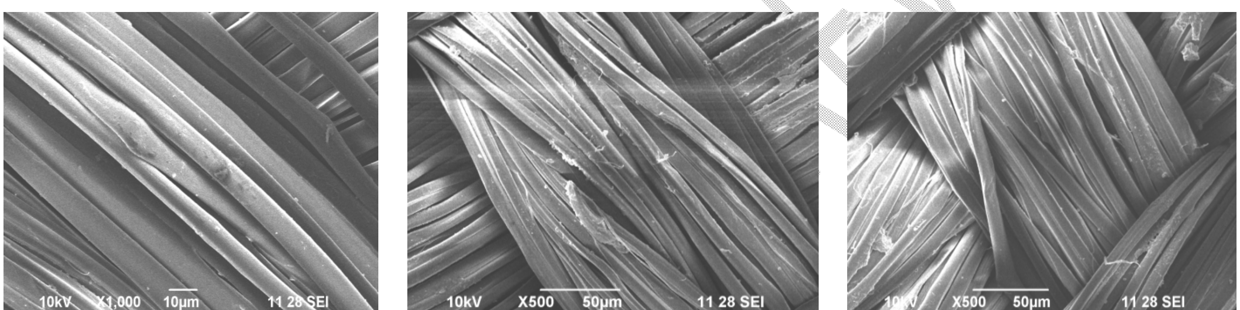
The results of UPF (uv protection factor) for PAA and CN treated dyed silk fabrics are presented in the Table 8. It is clearly seen from this table that there is a considerable improvement in the uv protection factor on the PAA and CN treated dyed silk fabrics. The increase of UPF on CN and PAA treated dyed fabric is around 52% and 42% respectively over that of the untreated one. The CN treated silk fabric shows more than 15% increase of UPF over that of PAA treated one. This data reveals that the CN treated dyed silk fabrics are in the excellent uv protection category compared to that of PAA treated (very good) and untreated ones. This gives the indication that if the silk fabric is to be protected from the uv rays chitosan application is considered to be the excellent choice. The scientific term, ultraviolet protection factor (UPF) is used to indicate the quantum of protection provided to the human skin by the textile fabric from the ultraviolet rays. "The distinction between SPF (Sun Protection Factor) and UPF values are that the SPF values for sunscreens are determined through human testing whereas UPF values are based on instrumental measurements. Ultraviolet protection factor is known as the ratio of the average effective UV irradiance calculated for unprotected skin to the average UV irradiance calculated for skin protected by the test textile fabric. The amount of UV radiation that penetrates a textile fabric and reaches the skin is considered as the measure of UPF. Both UV-A & UV-B radiation blocked are measured by UPF. UPF rating refers towards its material and does not to the design of the textile products" [53,54,55].

**Table 8.** UV Protection Factor Assessment of the Silk Fabrics

| S. No. | Dyes | UPF Rating of Silk Fabrics | | | Standard Chart for UPF Rating for the Textile Fabrics | | |
|---|---|---|---|---|---|---|---|
| | | PAA Treatment | Chitosan Treatment | No Treatment | UPF Rating | Protection Category | UV Radiation Blocked (%) |
| 1 | Kum kum | 35 | 42 | 20 | 15 to 24 | Good | 93.3 - 95.9 |
| 2 | Bar berry | 36 | 41 | 21 | | | |
| 3 | Annatto | 36 | 41 | 20 | | | |
| 4 | Onion | 38 | 44 | 22 | 25 to 39 | Very Good | 96 - 97.4 |
| 5 | Red sandal wood | 36 | 42 | 20 | | | |
| 6 | Grape | 35 | 41 | 20 | 40 to 50 | Excellent | 97.5 or more |
| 7 | Madder | 36 | 42 | 21 | | | |
| 8 | Reactive Dye | 39 | 45 | 22 | | | |

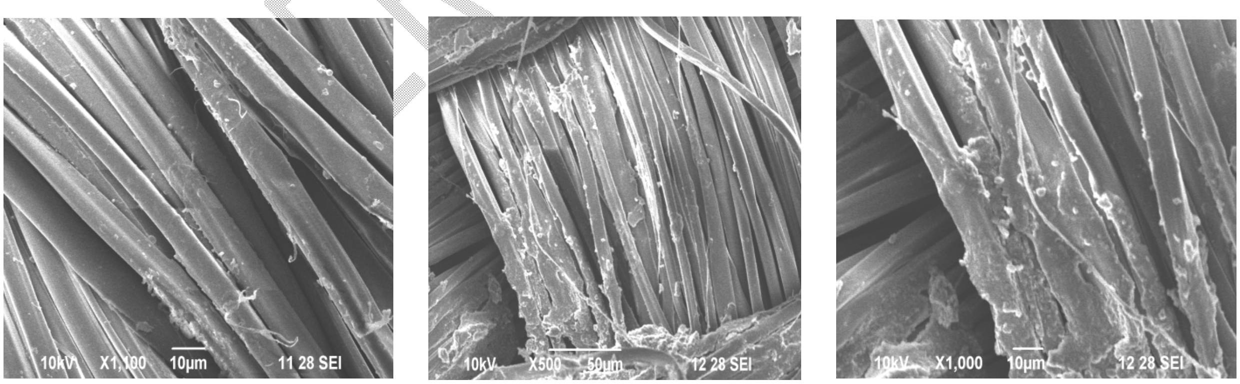
### 3.8 SEM Images of the Silk Fabrics

The SEM images of the silk fabric with no treatment, PAA and CN treatments followed by dyeing with natural dyes and synthetic reactive is shown in Figures 1a, 1b and 1c respectively. These figures give the clear picture of the differences in the effect of applications on the silk fabrics. The Figure 1a show the SEM images of untreated silk; and natural and synthetic reactive dyed silk fabrics. This figure does not give any appreciable difference in the appearance. The Figures 1b and 1c give a significant difference in the appearance compared to that seen in the Figure 1a. This gives a clear revelation that the effect of application of both PAA and CN is considerably good in the silk fabrics. A focused beam of high-energy electrons is used in the scanning electron microscope to generate a variety of signals at the surface of solid (textile) specimens. The external morphology (texture), crystalline structure and orientation of materials making up the sample is revealed from the information of the signals that derive from electron-sample interactions. The analyses of selected point locations on the textile sample is also capable by SEM performance [40,43,56,57,58].



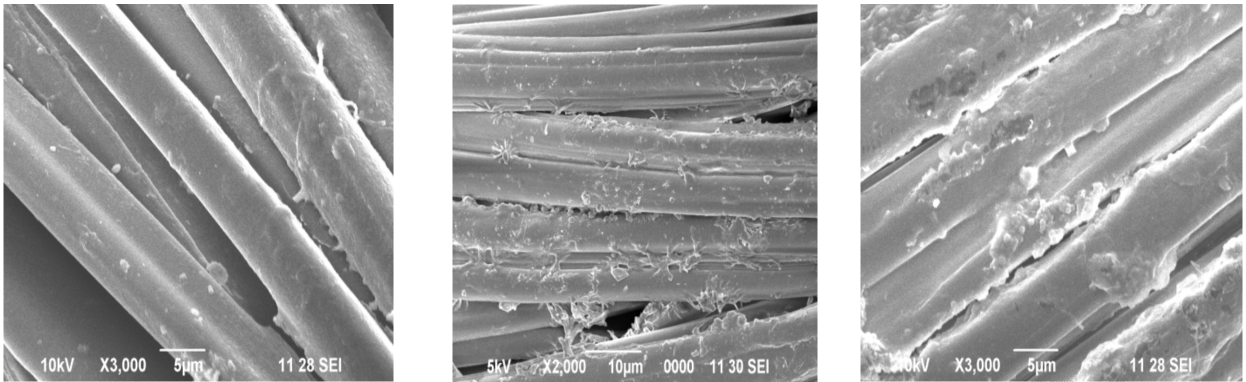

(i) Untreated (ii) Untreated & Natural Dyed (iii) Untreated & Reactive Dyed

**Figure 1a.** SEM Images of the Untreated and Dyed Silk Fabrics




(i) PAA Treatment only (ii) PAA Treated & Natural Dyed (iii) PAA Treated & Reactive Dyed

**Figure 1b.** SEM Images of the PAA Treated and Dyed Silk Fabrics

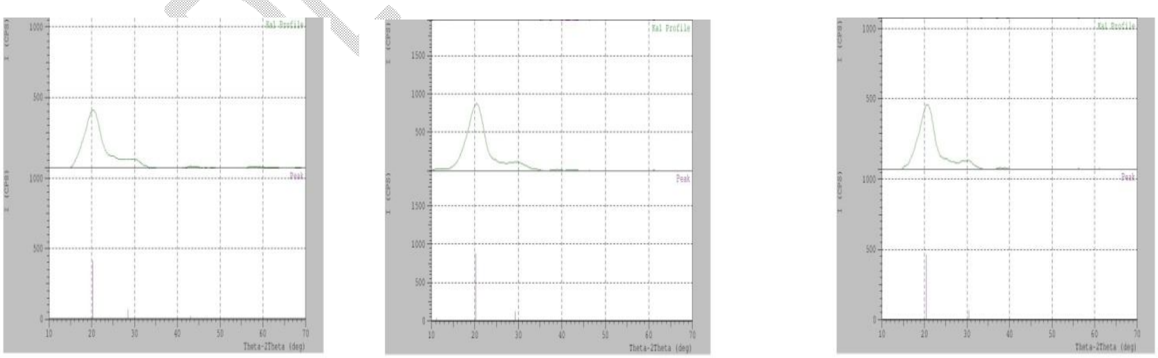



(i) CN Treatment only (ii) CN Treated & Natural Dyed (iii) CN Treated & Reactive Dyed

**Figure 1c.** SEM Images of the CN Treated and Dyed Silk Fabrics

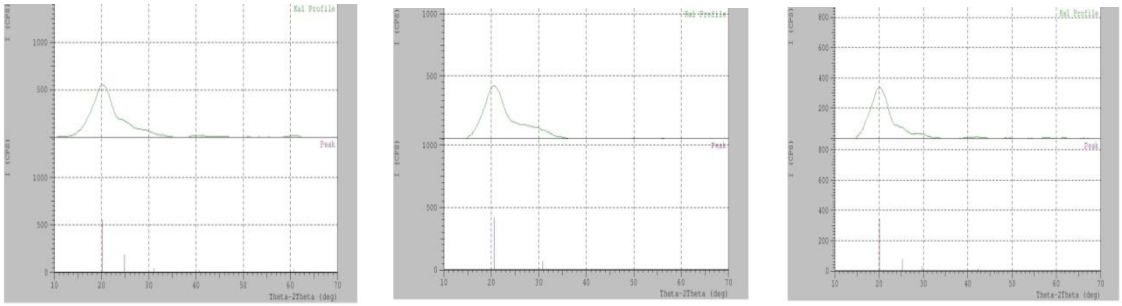
### 3.9 XRD Analysis of the Silk Fabrics

The silk fabrics with no treatment, PAA and CN treatments followed by dyeing with natural dyes and synthetic reactive were subjected for XRD study and the graphs are shown in Figures 2a, 2b and 2c respectively. These graphs give the indication about the effect of PAA and CN treatment on silk fabrics. The Figure 2a gives the XRD graphs of untreated silk; and natural and synthetic reactive dyed silk fabrics. There is no appreciable difference in the graphs due to the dyeing on the untreated silk fabric. From the Figures 2a and 2c it is revealed that as the silk fabrics are applied with PAA and CN followed by dyeing the bandwidth of the XRD graph is differed which gives the indication for the effect of application of PAA and CN on the silk fabric. X-ray diffraction technique is a common materials characterization which allows for the identification of crystal orientations and the inter atomic spacing. X-rays are used for this purpose based on the fact that the wavelength is on the same length scale as inter atomic spacing and the lattice parameter values [59,60,61].



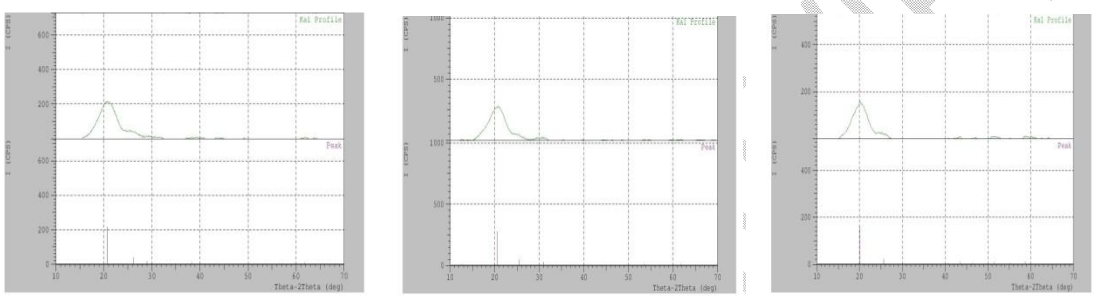
(i) Untreated (ii) Untreated & Natural Dyed (iii) Untreated & Reactive Dyed

**Figure 2a.** XRD Images of the Untreated and Dyed Silk Fabrics



(i) PAA Treatment only (ii) PAA Treated & Natural Dyed (iii) PAA Treated & Reactive Dyed

**Figure 2b.** XRD Images of the PAA Treated and Dyed Silk Fabrics



(i) CN Treatment only (ii) CN Treated & Natural Dyed (iii) CN Treated & Reactive Dyed

**Figure 2c.** XRD Images of the CN Treated and Dyed Silk Fabrics

## 4. CONCLUSION

From this research work it is concluded that the basic physical properties like tensile strength, elongation, drape co-efficient, thermal resistance, and stiffness / bending modulus value of the PAA treated and CN treated silk fabrics are in good trend possessed for the protection behavior. The overall K/S values and the fastness properties of the PAA and CN treated dyed silk fabrics are significantly good. The primary hand values such as smoothness, stiffness, fullness of the PAA and CN treated dyed silk fabrics are considerably good. The bending length and crease recovery of PAA and CN treated dyed silk fabrics both in warp and weft directions are present as required for the fabric and garments. The CN and PAA treated dyed silk fabrics show a significant increase of antibacterial activity for Staphylococcus aureus and Escherichia coli and indicated the influence of the protection behavior against the bacterial activity in the silk fabric and garments. The uv protection factors of the CN treated and PAA treated dyed silk fabrics are convincingly in the higher trend suitable for the protection of silk fabrics and garments. The SEM images and XRD graphs reveal and confirm the effect of the PAA and CN treatments followed by dyeing with natural dyes and synthetic reactive dye in the silk fabrics.

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