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# Development of Structurally Modified Less Wrinkle and High Drape Soft Silk Fabric

**Abdul Kadhar KATPADI MOHAMMED\***, Hariraj GOPAL, Shivakumar P KUDLUR, Subhas V NAIK

Central Silk Technological Research Institute, Central Silk Board, Bangalore, India

\*kadharkma@gmail.com

## Article

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## ABSTRACT

*In India, high-quality silk fabrics are nearly exclusively made from the highly prized textile fibre of animal origin known as silk. Silk sarees and materials for clothing are sold in a variety of stores. The majority of these retail stores are constantly searching for various types of silk materials to advertise as unique goods. Central Silk Technological Research Institute (CSTRI) continuously works in this field to offer the silk industry a variety of silk yarns and fabrics produced under eco-friendly tags. In one such attempt, CSTRI developed a wet processing technique with a user-friendly chemical, calcium nitrate tetrahydrate, by optimizing the process parameter such as time, temperature and chemical concentration. The comfort properties of the fabric were improved by modifying the secondary structure of the fibroin protein that was ascertained by Raman and C-13 NMR spectroscopies. Significantly higher drape and crease recovery properties were measured by Kawabata and FTT analysis. The products were developed in commercial scales (beside their costing) and displayed for 21 days to assess the market potential in terms of subjective assessment, product preference and price. The survey findings were statistically analysed using Minitab and Python software. The project findings in unequivocal terms recommend the chemical process using the safe chemical to treat the grey-finished fabrics to have improved utility, durability and comfort properties.*

## KEYWORDS

*silk fabric, wrinkle, drape, FTT, Kawabata, spectroscopy*

## INTRODUCTION

In India, high-quality silk fabrics are nearly exclusively made from the highly prized textile fibre of animal origin known as silk. Silk sarees and materials for clothing are sold in a variety of stores around India. The majority of these retail stores are constantly searching for various types of silk materials to advertise as unique goods. Central Silk Technological Research Institute (CSTRI) continuously works in this field to offer the silk industry a variety of silk yarns and fabrics produced with user-friendly tags. The CSTRI has tested a variety of chemicals as part of its research investigations to increase the bulkiness of raw silk for the creation of silk sarees with higher comfort qualities. Initial investigations conducted by Japanese scientists in one such endeavour as pilot research did not produce the expected results [1]. The CSTRI has created a method that uses an eco-friendly chemical to increase the bulkiness of twisted silk by three times [2]. Similar research was done to add bulkiness to silk

garments [3]. Based on earlier research, the present study standardized the chemical concentration, treatment temperature, and treatment time to increase the drapeability and wrinkle resistance of soft silk materials.

In the silk handloom industry, mostly the acid class of dye is used in which acetic acid is used to attain the acid medium to avoid un-levelness in the dyed yarn or fabric. Also, mild acetic acid is used in the final finishing process to get the scooping effect (a rustling sound from friction between silk fibres) that is preferred by consumers. Thus, chemicals are used in the production of silk saree that is subsequently removed in the zero-discharge effluent treatment plant. The term “user friendly” is used to differentiate that this calcium nitrate tetrahydrate can be used by the dyer easily when compared to the acetic acid used in the dyeing and scooping process. The main objective of the research is to develop a silk product that would be very unique and have export demand because the Indian silk handloom industry consumes two-thirds of the raw silk produced (30000 t/year) domestically in India to cater to the needs of the buyers. There is no literature available on a product developed with better comfort properties using chemicals. Some literature is available on mechanical finishing that has improved the comfort properties and is being propagated by CSTRI in the field by providing the required gadgets at a subsidized rate so that the weaver can upgrade the machinery and equipment available with them to cater to the needs of the end consumers.

The research aims to treat the fabric with chemicals to achieve the desired results because 1) The development of wrinkle-resistant and high-drape silk sarees always has demand in the silk sector as it would resolve years of existing crease formation problem; 2) Silk sarees are not having wrinkle resistance despite adopting various methodologies like construction particulars modifications, chemical treatments using various chemicals etc.; 3) During the various interactions with consumers, they always requested CSTRI to resolve the problem of a wrinkle in silk sarees and high drape silk is also always warranted by the consumers. Accordingly, CSTRI under its research studies tried various chemicals for improving the bulkiness of raw silk for the production of silk sarees with better comfort properties. In one such attempt as a pilot study, CSTRI developed a technology for increasing the bulkiness of the twisted silk to the extent of three times using a chemical, which is an intervention worth mentioning. Also, as per the suggestions of experts from the Research Advisory Committee, CSTRI tried the same chemical with raw silk fabrics woven on power looms. The results have proved that the raw silk fabrics can be made wrinkle resistant and also improvement in drape characteristics can be achieved due to the chemical treatments.

Thus, the chemical concentration, treatment time and temperature that bear a direct effect on the economics of the treatment have been optimized using the Box-Behnken design of the experiment.

The optimized parameters were utilized to take commercial trials for the bulk production of treated fabric that was ultimately converted to garments to ascertain consumer preferences.

## MATERIALS AND METHODS

### Raw material

Raw silk produced from the bivoltine cocoons reeled on a multi-end reeling machine was used to produce the soft silk fabrics on power looms. The raw silk of 20 to 22 denier was utilized to produce the twisted silk for warp and weft. The silk thread, single twisted to the extent of 780-800 TPM, doubled and then compound twisted in opposite direction to the extent of 780-800 TPM, was used as warp, whereas the silk thread, doubled and twisted to the extent of 310-320 TPM, was used as weft. The bivoltine silk fabric of 50 to 60 g/m<sup>2</sup> was woven on a power loom and about 25 meters of the fabric have been developed for the purpose and used for experimentation. The fabric characteristics in the raw stage and after degumming are given in Table 1.

Table 1. Soft silk fabric characteristics before and after degumming

Particulars	Grey Fabric	Degummed Fabric
Fabric weight (g/m <sup>2</sup> )	51.0 (0.158)	45.0 (0.179)
Fabric thickness (mm)	20.0 (0.707)	14.9 (0.447)
Ends per Inch	104 (0.000)	105 (1.095)
Picks per Inch	78 (0.000)	90 (0.894)
Warp Denier	40.1 (0.493)	34.7 (1.506)
Weft Denier	121 (0.753)	65.5 (1.951)
Warp Crimp	4.80 (0.000)	4.80 (0.000)
Weft Crimp	1.60 (0.000)	4.80 (0.000)
Warp Twist	941 (13.13)	1023(14.25)
Weft Twist	132 (3.09)	124 (4.96)

Values in the parenthesis are the Standard deviation of the test results (Average of 5 readings)

### Design of Experiment

Preliminary experiments were carried out to study the influence of chemical (calcium nitrate tetrahydrate) concentration, the temperature of chemical treatment and the duration of chemical treatment to ascertain the range of the independent variables [4]. Accordingly, the response surface design of the experiment [5-6] was envisaged by adopting the Box-Behnken technique[7] using the said three variables like temperature, duration and concentration. The required soft silk fabrics were manufactured through M/s. BNS Fabrics, Bengaluru with 50 g/m<sup>2</sup> and 60 g/m<sup>2</sup>. The fabrics were tested initially in grey condition for all the utility, durability and comfort characteristics.

The fabric samples were sent to the industrial partner, for degumming [8-9] the same using soap and soda method. The fabrics were degummed to the extent of 24%. 21 samples of one meter each were prepared for the experiment. 20 samples for chemical treatment and the remaining one as a control sample. The chemical treatment of all 20 samples was carried out as per the run order by varying the dependent variables as per the design of the experiment (DoE). The treated samples were conditioned for 24 hours and weighed to study the mass loss on the initial mass taken and found that there is no mass gain in any of the chemical treatments carried out. The treated fabric samples were tested for various fabric utility and durability characteristics like crease recovery, drape coefficient, breaking load and elongation and bending length, to calculate the flexural rigidity.

In the optimization process, total crease recovery, drape co-efficient and tenacity and elongation properties were considered as dependent variables. The time, temperature and chemical concentration, which have a direct bearing on product cost, were considered independent variables. While in the optimized treatment, total crease recovery, drape co-efficient, bending length and flexural rigidity were studied that influence the wrinkle resistance and drapability of the fabric. The influence of pH as well as the effect after dyeing the chemically treated fabrics were also studied in the same line. Kawabata and FTT tests were carried out to ascertain the hand value of the treated fabric over the untreated fabric.

### **Standard test methods to study the properties of fabrics**

The chemically treated fabrics were tested using the standard test methods for the fabric utility, durability and comfort characteristics like tensile strength [10], crease recovery [11], flexural rigidity [12] and drape coefficient percentage [13]. The data were analysed using Minitab 19 version software statistical package.

### **Raman Spectroscopy**

To evaluate the effect of chemical treatment on the silk fibre structure, both the chemically treated silk fabrics at different temperatures along with control degummed fabric, in small swatches as a specimen, were subjected to a Raman spectroscopy test at the Indian Institute of Science, Bengaluru. The interpretation of the Raman Spectroscopy results was made in due consultation with the expert available at the Institute.

### **NMR (C-13) Spectroscopy**

To evaluate the effect of chemical treatment on the silk fibre structure, both the chemically treated silk fabrics at different temperatures along with control degummed fabric, cut into small pieces as a specimen, were subjected to an NMR spectroscopy test at the Indian Institute of Science, Bengaluru.

The interpretation of the NMR results was made in due consultation with the expert available at the Institute.

### **Low-stress mechanical properties**

The low-stress mechanical properties have been ascertained through Kawabata analysis as well as Fabric Touch Tester (FTT) technique.

### **Kawabata**

The Kawabata Evaluation System (KES) [14-15] is a series of instruments used to measure those textile material properties that enable predictions of the aesthetic qualities perceived by human touch. KES instruments quantify garment material tactile qualities through objective measurement of the mechanical properties related to comfort perception. With low forces applied, as in manipulating/touching fabrics, the Kawabata instruments define the role played by tensile (stretch), shear stiffness (drape), bending rigidity (flexing), compression (thickness, softness), and surface friction and roughness (next to the skin) on tactile sensations. This analytical power, combined with the capability to characterize energy loss in mechanical deformation and recovery processes, provides an unparalleled tool for use in fabric hand analysis. KES provides a unique capability, not only to predict human response but also to provide an understanding of how the variables of fibre, yarn, fabric construction and finish contribute to perceptions of comfort. A standard specimen size of 20 x 20 cm is used in three replications. All measurements are directional, except for compression, and are made in both the lengthwise direction and the crosswise direction of the sample.

### **FTT Technique**

The fabric touch tester can measure multiple physical properties of fabric specimens including thermal transmission, bending, compression and surface friction aspects in one single trial. Software with integrated prediction models is also designed to calculate touch-feeling scores for fabrics. This device is used to measure the fabric handle in less than five minutes per sample. The measurements are comprehensive and cover both sides (inside and outside of the fabric) and both directions (warp/wale and weft/coarse). Based on obtained values of the 13 fabric indices, the FTT software will also subsequently compute three primary comfort indices i.e., smoothness, softness and warmth, as well as two global comfort indices i.e. total hand and total touch values.

### **Commercial production**

The commercial production was carried out by purchasing the required ARM silk of 20/22 denier both warp and weft to produce 100 meters of the soft silk fabric. Also, the required soft silk fabric as well

as Crape silk fabric, Ready for Dyeing (RFD) and Ready for Stitching (RFS) were sourced from the market to produce garments.

### **Product Development**

The industry partner at Chennai, which is having multiple stores in the southern parts of India, was entrusted with the product development work that was carried out by the professional designers. Four models for women's wear and two models for children's wear were finalized in consultation with the market team of the industry partner. The services of a freelance designer were utilized to develop eight stoles and four scarves using bhandni techniques with natural dyes. Also, the services of garment manufacturer were utilized to develop three types of shirts with control and treated fabric.

### **Costing of the Products**

The costing of the product was carried out for all the products based on the actual expenditure incurred in developing the products that are on the higher side due to pilot-scale production. It is expected to reduce drastically in the event of commercial production which shall absorb the design cost to a greater extent.

### **Market survey and Analysis**

The market team of the industry partner were involved in taking the survey with knowledge of the technicalities of the products that were displayed for 21 days to assess the market potential in terms of subjective assessment, product preference and price. The participants rated the product and gave feedback on purchase preference and product price. Data were statistically analysed using Minitab and Python software.

## **RESULTS AND DISCUSSION**

### **Box-Behnken Design of experiments**

The finding of the experiments in terms of important fabric properties like crease, drape co-efficient, flexural rigidity and tenacity properties were assessed for all the 20 treated samples as per the run order. The data were analysed using Minitab software. The results are given in Table 2.

The statistical analysis of Box-Behnken experiment data of objective tests revealed that the chemical treatment and concentration of chemicals have a significant influence on the crease recovery, drape and elongation properties. The response surface equation along with the R squared value, a statistical measure that represents the proportion of the variance for a dependent variable that is explained by an independent variable are shown in Table 3.

Table 2. Influence of factors of chemical treatments on fabric characteristics

Run Order	S. No.	Duration (min)	Temperature (°C)	Concentration (%)	Total Crease Angle (°)	Drape Co- efficient (%)	Flexural Rigidity (mg. cm)		Breaking Load (kg)		Elongation (%)	
							Warp	Weft	Warp	Weft	Warp	Weft
9	1	3	75	6	255.00	25.69	0.01	0.02	22.57	39.93	20.50	15.03
1	2	5.5	66	3.6	247.80	27.01	0.00	0.01	24.00	43.67	20.20	15.00
5	3	5.5	66	8.4	254.60	25.19	0.00	0.02	24.30	43.10	19.80	12.93
3	4	5.5	84	3.6	249.60	26.68	0.00	0.02	24.30	44.60	17.60	14.53
7	5	5.5	84	8.4	260.20	25.85	0.00	0.01	23.37	41.27	18.53	12.43
11	6	9	60	6	248.60	25.52	0.01	0.02	24.70	42.33	18.53	13.67
13	7	9	75	2	248.80	24.86	0.00	0.01	25.20	39.97	20.00	15.13
16	8	9	75	6	255.10	23.20	0.00	0.01	24.79	40.60	18.47	11.35
19	9	9	75	6	254.60	23.45	0.00	0.01	25.00	40.57	18.33	11.33
18	10	9	75	6	255.70	23.48	0.00	0.01	24.77	40.90	18.17	11.47
15	11	9	75	6	254.00	23.70	0.00	0.01	25.12	41.38	18.47	11.65
20	12	9	75	6	254.00	23.56	0.00	0.01	24.53	41.10	18.60	11.63
17	13	9	75	6	254.20	24.05	0.00	0.01	24.70	40.63	18.07	11.27
14	14	9	75	10	258.90	21.72	0.00	0.02	26.00	42.27	21.13	14.60
12	15	9	90	6	259.30	25.00	0.00	0.02	24.20	40.70	17.40	13.63
2	16	12.5	66	3.6	245.90	27.51	0.01	0.02	24.23	40.57	17.80	13.70
6	17	12.5	66	8.4	257.80	26.56	0.01	0.02	23.33	41.20	18.20	14.83
4	18	12.5	84	3.6	246.60	27.51	0.00	0.01	23.40	35.07	17.50	12.33
8	19	12.5	84	8.4	258.00	23.71	0.00	0.01	23.57	42.27	19.33	14.80
10	20	15	75	6	250.60	27.84	0.00	0.02	25.50	40.57	20.87	15.40

Table 3. Response surface equations of fabric properties of chemically treated soft silk fabrics

Dependent Variable	Regression Equation	R-Square	p Value
Crease Recovery Angle - Total (°)	$Y1 = 187.6 + 2.33 X1 + 1.14 X2 + 0.51 X3 - 0.0648 X1^2 - 0.00539 X2^2 - 0.08815 X3^2 - 0.0258 X1 * X2 + 0.0886 X1 * X3 + 0.0193 X2 * X3$	89.26	0.000**
Drape co-efficient (%)	$Y2 = 80.2 - 0.683 X1 - 1.415 X2 + 0.43 X3 + 0.1060 X1^2 + 0.01044 X2^2 + 0.0231 X3^2 - 0.01265 X1 * X2 - 0.0315 X1 * X3 - 0.0109 X2 * X3$	87.23	0.002**
Elongation – Warp (%)	$Y3 = 22.0 - 2.436 X1 + 0.425 X2 - 2.44 X3 + 0.0461 X1^2 - 0.00487 X2^2 + 0.0948 X3^2 + 0.01865 X1 * X2 + 0.0255 X1 * X3 + 0.0162 X2 * X3$	78.08	0.021*
Elongation – Weft (%)	$Y4 = 76.5 - 2.163 X1 - 1.148 X2 - 3.819 X3 + 0.0901 X1^2 + 0.00732 X2^2 + 0.1795 X3^2 - 0.00172 X1 * X2 + 0.1166 X1 * X3 + 0.00759 X2 * X3$	92.5	0.000**

X1 = Duration of treatment, X2 = Temperature of treatment, X3 = Concentration of chemical; \* - Significant at 95% ; \*\* - Significant at 99%

The plot diagram of the dependent variables concerning the independent control variables of the experiment is given in Figure 1. Thus, it was observed that chemical treatment of the soft silk fabrics significantly increases the crease recovery, reduces the drape co-efficient and improves the elongation characteristics. Based on the optimization analysis, it was found that a 6% concentration of chemically treated at 90 °C temperature for 5 minutes has shown significant improvement in the fabric characteristics compared to degummed soft silk fabrics.

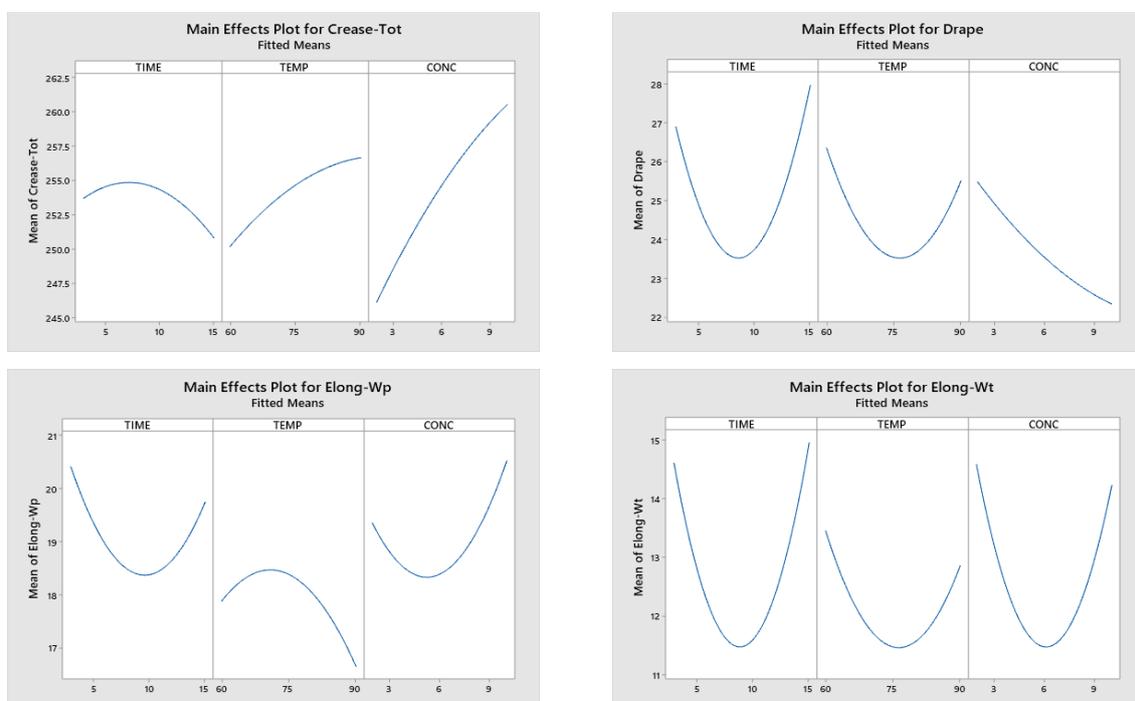


Figure 1. Plot Diagram of Box-Behnken design of experiment

The surface plot diagram in respect of three dependent variables like crease recovery angle, drape coefficient and elongation in warp direction for the optimized dependent variables (time: 5 minutes, concentration: 6% and temperature: 90 °C) are given in Figure 2.

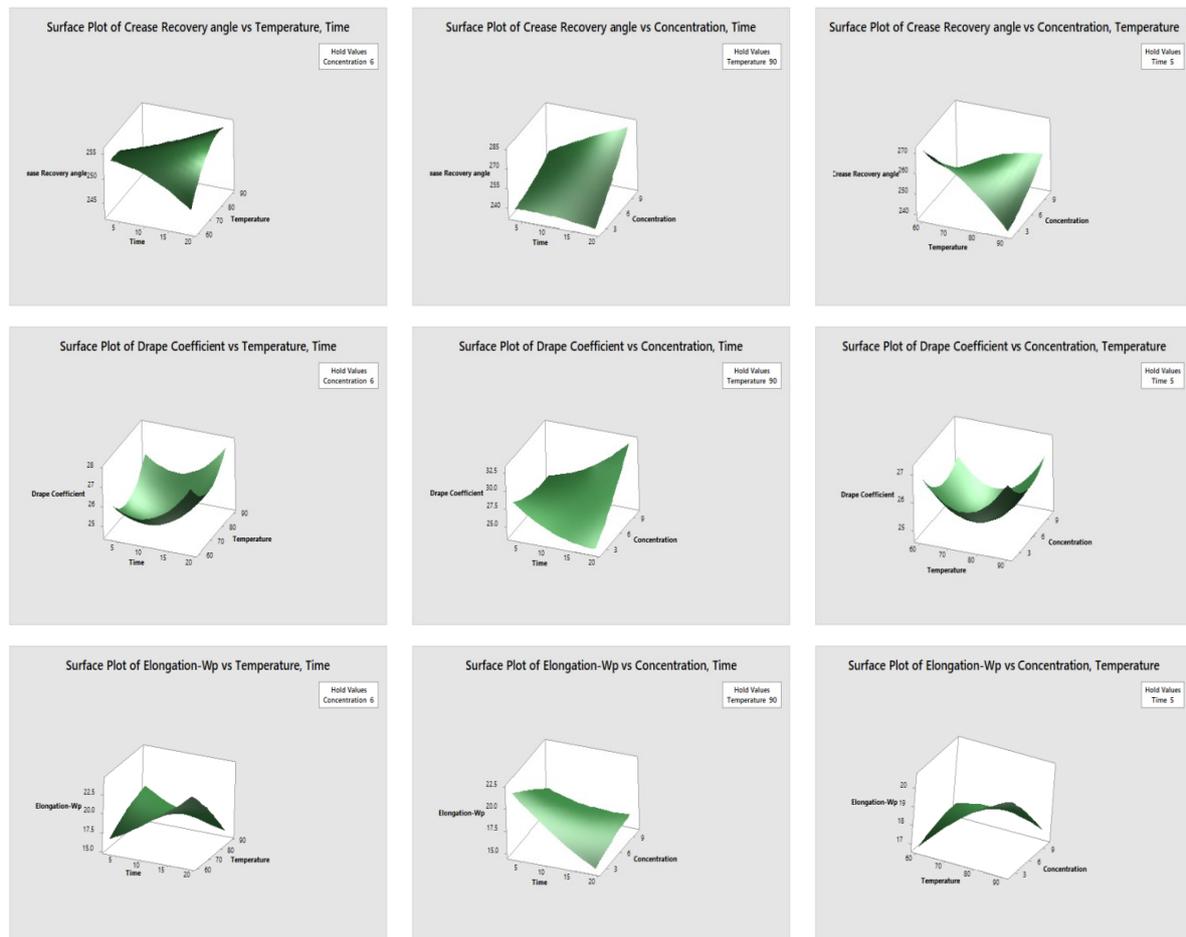


Figure 2. Surface Plot Diagram on the effect of the optimized independent variable on fabric quality

Moreover, the influence of all the independent variables on the dependent variable would be directly proportional. Higher time, temperature and chemical concentration individually would influence the dependent variable but higher temperature, which is not desirable as process parameters and higher concentration are not preferred due to the associated cost of treatment. Thus, based on the plot diagram, the independent variables were selected to get the optimum results as envisaged in the Box-Behnken design of the experiment.

### Process Optimization

To further optimize the treatment at low temperatures for better economics, three more trials with replications were taken by varying the temperature of treatment and keeping the other two variables constant as optimized earlier (temperature - 40, 60, 75 °C; time - 5 min; concentration - 6%).

It was found that the desired fabric characteristics deteriorate if the temperature is reduced and the details are given in Table 4 and Figure 3.

Table 4. Optimization of treatment temperature for better economics

Sample / Statistics	Crease Recovery Angle (°)		Drape co-efficient (%)		Bending Length (cm)		Flexural Rigidity (mg. cm)	
	Warp	Weft	Face	Back	Warp	Weft	Warp	Weft
<i><math>\bar{x} \pm s</math> (n=3)</i>								
Control – (90°C) - A	243 ±3.055	275±3.055	1.58 ±0.020	1.60 ±0.006	0.73 ±0.058	1.13 ±0.058	0.0027 ±0.0001	0.0085 ±0.0004
Treated (40°C) - B	235 ±1.155	265±2.309	1.66 ±0.020	1.67 ±0.012	0.83 ±0.058	1.37 ±0.058	0.0033 ±0.0001	0.0133 ±0.0029
Treated (60°C) - C	236 ±2.000	267±1.155	1.65 ±0.017	1.66 ±0.025	0.90 ±0.006	1.37 ±0.058	0.0033 ±0.0001	0.0133 ±0.0029
Treated (75°C) – D	237 ±1.155	267±3.055	1.63 ±0.006	1.64 ±0.021	0.83 ±0.006	1.33 ±0.058	0.0032 ±0.0001	0.0099 ±0.0001
<i>Student's t-Test (P values)</i>								
A Vs B	0.02	0.01	0.00	0.00	0.05	0.00	0.00	0.05
A Vs C	0.02	0.01	0.01	0.03	0.02	0.00	0.00	0.05
A Vs D	0.04	0.02	0.02	0.05	0.05	0.01	0.01	0.01

Plain silk degummed fabric of 53 GSM is used for the experiment.

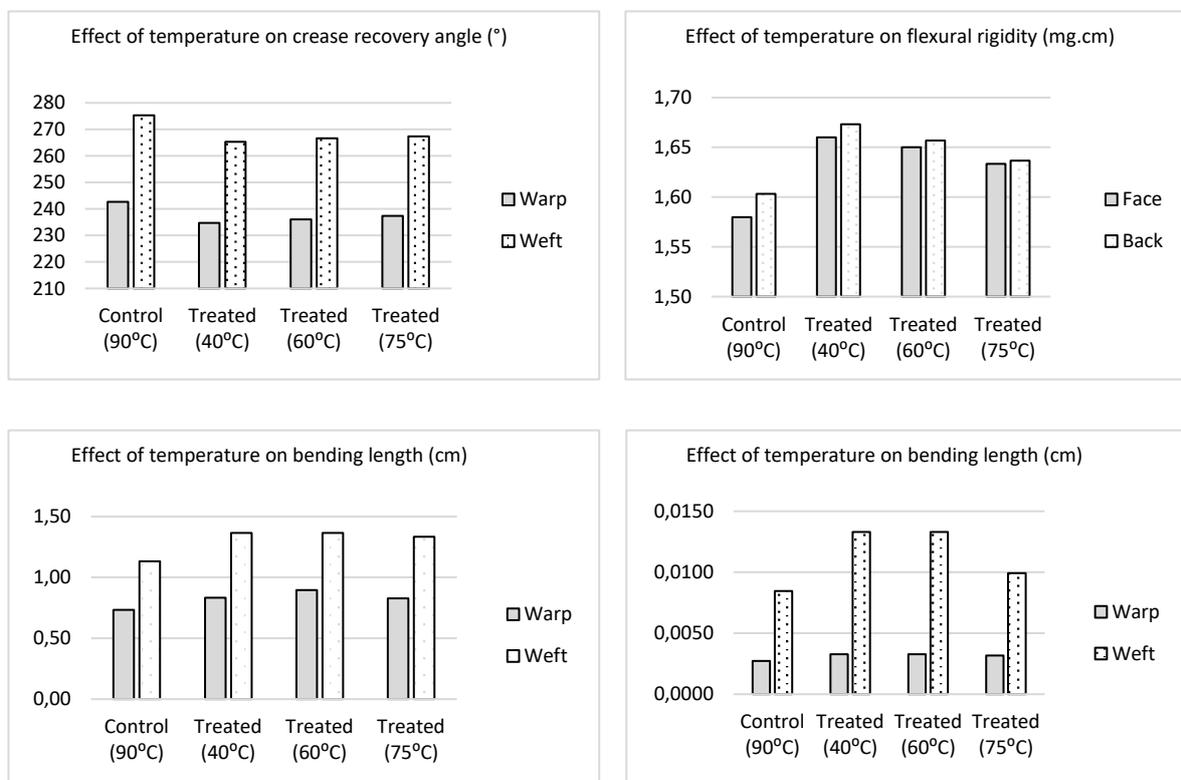


Figure 3. Effect of Temperature on treated fabric properties

### Raman Spectroscopy

To ascertain the effect of chemical reactions on the molecular structure of the fibroin protein, RAC suggested studying the structural modifications on the treated silk fabrics. Accordingly, the chemically treated silk fabrics at different temperatures along with control degummed fabric were subjected to a Raman spectroscopy test at the Indian institute of science, Bengaluru [16]. The results obtained are shown in Figure 4.

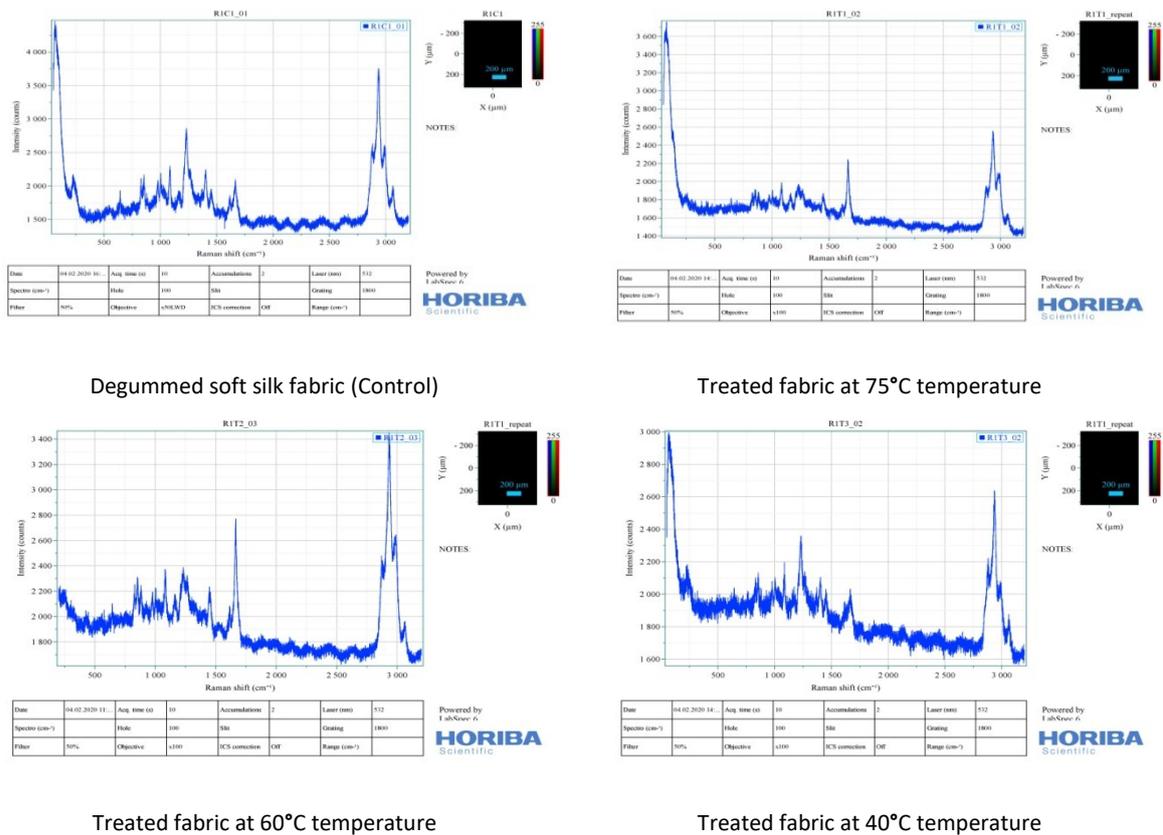


Figure 4. Raman spectroscopy of chemically treated fabrics with control fabric

The Raman spectroscopy data has been analysed and it could be observed that the chemical treatment at 60 °C and 75 °C has shown a shift at 1225 and 1650 wave numbers as shown in Figure 5, which may be attributed to tertiary level molecular structure change in the amide I and amide III level due to chemical treatment.

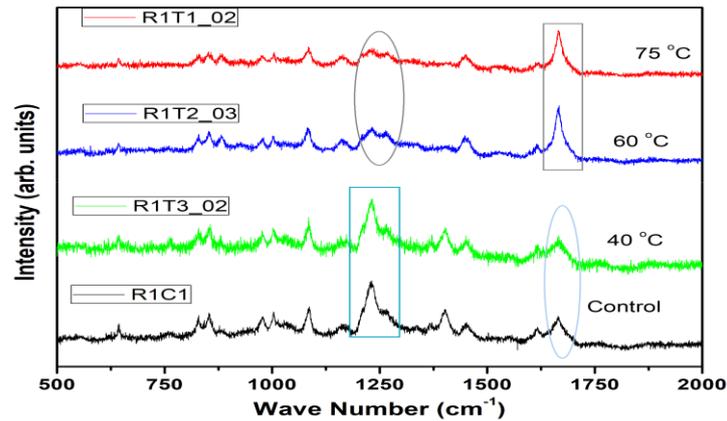


Figure 5. Analysis of Raman spectroscopy results of chemically treated fabric at 40 °C, 60 °C and 75 °C temperature compared with control fabric

### NMR (C-13) Spectroscopy

Further, to re-ascertain the structural modification, the chemically treated fabrics along with the control fabrics were subjected to NMR spectroscopy at the Indian Institute of Science, Bengaluru, wherein all the three treated fibroin proteins along with the control sample were analysed using C-13, NMR spectra in the solid state. In C-13, NMR spectra, each peak identifies a carbon atom in a different environment/ arrangement within a molecule. The results obtained are shown in Figure 6.

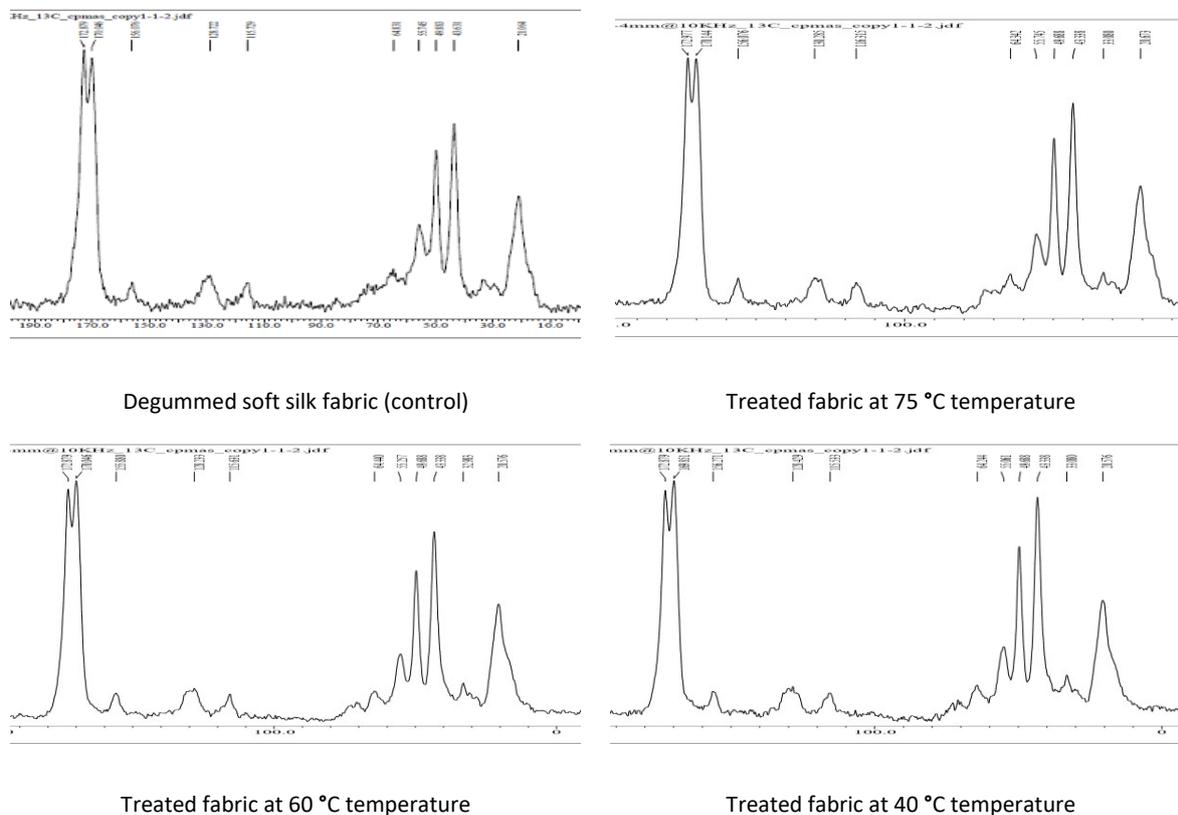


Figure 6. NMR spectroscopy (C-13) of chemically treated fabrics with control fabric

It could be observed from the figures that, the number of peaks in each spectrum along with chemical shifts at 0~50 ppm has increased. Thus it could be concluded that the terminal amino group has reacted with the chemical to modify the structure of silk fibre.

### Influence of pH of chemical treatment

To ascertain the influence of pH on chemical treatment, experiments were carried out in an Alkaline medium (pH 10-11 & 11-12) using buffer solutions, the results were encouraging and the alkaline pH greatly influence the fabric characteristics. The details are given in Table 5. The significant influence of the chemical treatment at alkaline pH on the fabric quality may be attributed to the presence of the amino group in Lysine and phenol group in Tyrosine that are prone to be active in alkaline pH.

Table 5. Influences of pH on chemical treatment

Sample / Statistics	Crease Recovery Angle		Drape co-efficient		Bending Length		Flexural Rigidity	
	(°)		(%)		(cm)		(mg. cm)	
	Warp	Weft	Face	Back	Warp	Weft	Warp	Weft
$\bar{x} \pm s$ (n=3)								
Alkaline pH 7.5 (Temperature - 75°C) - A	215 $\pm 5.033$	239 $\pm 5.774$	1.90 $\pm 0.015$	1.91 $\pm 0.021$	1.17 $\pm 0.058$	1.67 $\pm 0.058$	0.0049 $\pm 0.0001$	0.0157 $\pm 0.0001$
Alkaline pH 10-11 (Temperature - 75°C) - B	230 $\pm 2.000$	250 $\pm 2.000$	1.85 $\pm 0.006$	1.84 $\pm 0.006$	0.97 $\pm 0.058$	1.53 $\pm 0.058$	0.0045 $\pm 0.0001$	0.0153 $\pm 0.0001$
Alkaline pH 11-12 (Temperature - 75°C) - C	236 $\pm 2.000$	265 $\pm 2.309$	1.67 $\pm 0.012$	1.62 $\pm 0.020$	0.83 $\pm 0.058$	1.33 $\pm 0.058$	0.0029 $\pm 0.0006$	0.0069 $\pm 0.0051$
Alkaline pH 10-11 (Temperature - 90°C) - D	230 $\pm 2.000$	257 $\pm 1.155$	1.74 $\pm 0.017$	1.71 $\pm 0.026$	0.93 $\pm 0.058$	1.50 $\pm 0.100$	0.0037 $\pm 0.0007$	0.0152 $\pm 0.0002$
Alkaline pH 11-12 (Temperature - 90°C) - E	237 $\pm 1.1550$	264 $\pm 4.000$	1.65 $\pm 0.015$	1.64 $\pm 0.021$	0.780 $\pm 0.029$	1.33 $\pm 0.058$	0.0032 $\pm 0.0001$	0.0069 $\pm 0.0051$
Student's t-Test (P values)								
A Vs B	0.01	0.03	0.00	0.01	0.01	0.02	0.01	0.01
A Vs C	0.01	0.00	0.00	0.00	0.00	0.00	0.01	0.049
A Vs D	0.01	0.01	0.00	0.00	0.00	0.00	0.045	0.02
A Vs E	0.01	0.00	0.00	0.00	0.00	0.00	0.01	0.049

Plain silk degummed fabric of 45 GSM is used for the experiment

## Kawabata Analysis

Moreover, 4 Nos. of chemically treated silk fabric samples at different temperatures along with the control sample were sent to Central Institute for Research on Cotton Technology (CIRCOT), Mumbai for conducting the test under the Kawabata evaluation system and the results are given as Table 6. It is found that the bending stiffness, shear stiffness and coefficient of friction have significantly improved. More clearly, the bending stiffness of the treated fabrics decreased by 19%, 7% and 19% in respect of the chemical treatment carried out at 60 °C, 75 °C and 90 °C respectively for the untreated fabric (control). The shear stiffness decreased by 17%, 14% and 4% in respect of the chemical treatment carried out at 60 °C, 75 °C and 90 °C respectively. Also, the coefficient of friction decreased by 27% and 37% in respect of the chemical treatment carried out at 75 °C and 90 °C.

Table 6. KAWABATA Analysis

Parameter/ Statistics	RT - tensile resilience (%)	EMT - fabric extension at (500 g/cm) (%)	RC - compression resilience (%)	B - bending stiffness (g.f.cm <sup>2</sup> /cm)	G - shear stiffness (g. f/cm. deg)	MIU - coefficient of friction	SMD - geometrical roughness
$\bar{x} \pm s$ (n=3)							
Control (A)	31.12 ±5.51	8.62 ±0.64	52.64 ±3.08	0.016 ±0.0001	0.653 ±0.051	0.219 ±0.017	4.319±0.168
Treated - 40° C (B)	23.72 ±2.35	9.64 ±1.12	50.86 ±3.47	0.016 ±0.0006	0.647 ±0.133	0.177 ±0.011	4.318±0.213
Treated - 60° C (C)	20.31 ±4.62	9.92 ±0.61	51.04 ±3.58	0.013 ±0.0011	0.543 ±0.020	0.265 ±0.005	4.002±0.598
Treated - 75° C (D)	26.58 ±3.98	7.95 ±0.34	52.33 ±4.36	0.015 ±0.0002	0.560 ±0.040	0.160 ±0.004	3.805±0.572
Treated - 90° C (E)	28.90±2.87	10.88 ±1.39	54.03 ±2.74	0.013 ±0.0004	0.630 ±0.139	0.137 ±0.004	4.511±0.135
Student's t-Test (P values)							
A Vs B	0.12	0.21	0.34	0.20	0.47	0.05	0.50
A Vs C	0.08	0.09	0.34	0.02	0.03	0.03	0.26
A Vs D	0.22	0.11	0.46	0.01	0.05	0.01	0.12
A Vs E	0.27	0.09	0.31	0.00	0.35	0.00	0.17

Plain silk degummed fabric of 53 GSM is used for the experiment

The Data analysis for parameter optimization revealed that 90 °C is the best fit for all parameters like SMD, MIU, G, B, RC, EMT, RT (55% explained) and B, G & RC (73%). Also, 60 °C is the best fit for B and G only (85%). The details are shown in Table 7.

Table 7. Parameter optimization of Kawabata analysis

Response Optimization: SMD, MIU, G, B, RC, EMT, RT

**Parameters**

Response	Goal	Lower	Target	Upper	Weight	Importance
SMD	Minimum		3.1500	4.6620	1	1
MIU	Minimum		0.1340	0.2680	1	1
G	Minimum		0.5200	0.8000	1	1
B	Minimum		0.0115	0.0163	1	1
RC	Maximum	47.06	57.1400		1	1
EMT	Maximum	7.66	12.4300		1	1
RT	Maximum	15.36	35.8900		1	1

**Solution**

Solution	PARTICULARS	REP	SMD Fit	MIU Fit	G Fit	B Fit	RC Fit	EMT Fit	RT Fit	Composite Desirability
1	90C	S1	4.44067	0.133533	0.643333	0.0129133	53.2907	10.718	29.77	<u>0.554282</u>

Response Optimization: G, B, RC

**Parameters**

Response	Goal	Lower	Target	Upper	Weight	Importance
G	Minimum		0.5200	0.8000	1	1
B	Minimum		0.0115	0.0163	1	1
RC	Maximum	47.06	57.1400		1	1

**Solution**

Solution	PARTICULARS	REP	G Fit	B Fit	RC Fit	Composite Desirability
1	90C	S2	0.583333	0.0130733	54.5967	<u>0.729942</u>

Response Optimization: G, B

**Parameters**

Response	Goal	Lower	Target	Upper	Weight	Importance
G	Minimum		0.5200	0.8000	1	1
B	Minimum		0.0115	0.0163	1	1

**Solution**

Solution	PARTICULARS	REP	G Fit	B Fit	Composite Desirability
1	60C	S2	0.496667	0.0128067	<u>0.853099</u>

The Kawabata analysis also revealed that the Primary Hand Values (PHV) for women's thin dress materials like Koshi (Stiffness), Numeri (Smoothness) and Fukurami (Softness) are better than that of regular silk fabrics. The Total Hand Value (THV) of the treated fabric is better than that of the untreated silk fabric. The Q-max, CLO and TIV of the treated silk fabric are significantly better than that of untreated silk fabric.

### Fabric Touch Tester (FTT) analysis

To re-ascertain the low-stress mechanical properties of the treated fabrics, 2 Nos. fabric samples (Soft silk and crape silk) each measuring 1.5 meters, procured, treated and sent along with a control sample for testing using the FTT technique (Fabric Touch Tester) at The South India Textile Research Association (SITRA), Coimbatore. The Primary Sensory Index (PSI) of Smoothness, Softness, Warmness, Total Hand Value (THV) and Total Touch Value (TTV) of the treated fabric have greatly improved including its grade for both soft silks as well as crape silk. The details are given in Table 8 and Figure 7-9.

Table 8. Analysis of Fabric Touch Tester (FTT Technique)

Parameter	Values				Grade			
	Fabric face		Fabric back		Fabric face		Fabric back	
Control (C)/ Treated (T)	C	T	C	T	C	T	C	T
Soft Silk Fabric								
Primary Sensor Index								
PSI- Smoothness	0.59	1.11	0.66	1.45	3.5	5	4	5
PSI- Softness	0.67	0.88	0.6	1.04	4	5	3.5	5
PSI- Warmness	0.36	-0.14	0.31	0.01	2	1	2	1
Total Hand Value	0.56	0.73	-	-	3	5	-	-
Total Touch Value	-	-	0.58	1.08	-	-	3	5
Crape Silk Fabric								
Primary Sensor Index								
PSI- Smoothness	0.64	1.31	0.66	0.86	4	5	4	5
PSI- Softness	0.67	0.98	0.6	0.72	4	5	4	5
PSI- Warmness	0.34	-0.32	0.3	0.23	2	1	2	1
Total Hand Value	0.57	0.81	-	-	3	5	-	-
Total Touch Value	-	-	0.57	0.7	-	-	3	5

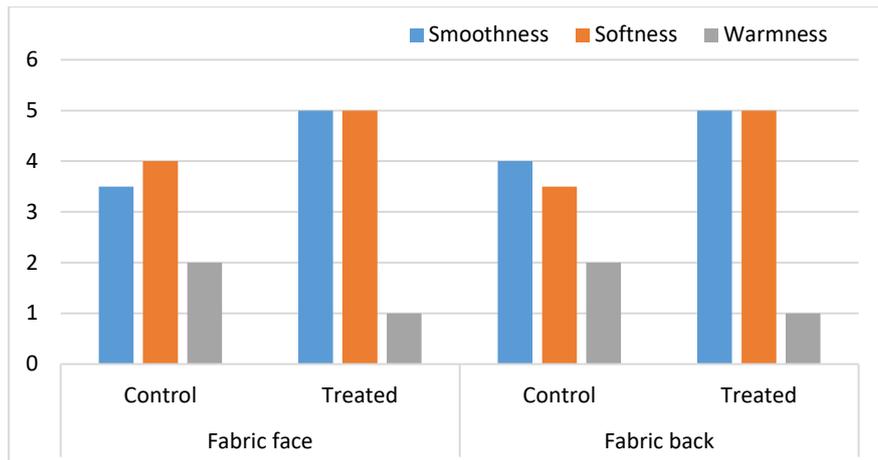


Figure 7. The grades of primary sensor index of soft silk fabric

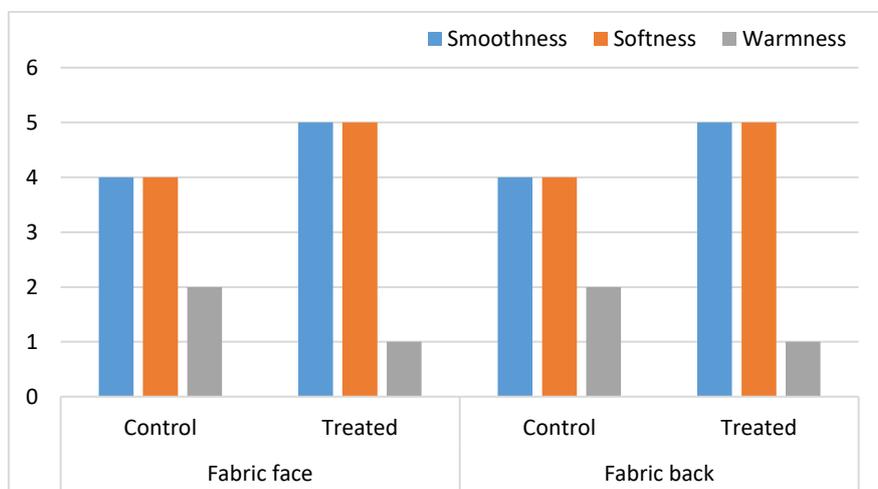


Figure 8. The grades of primary sensor index of crape silk fabric

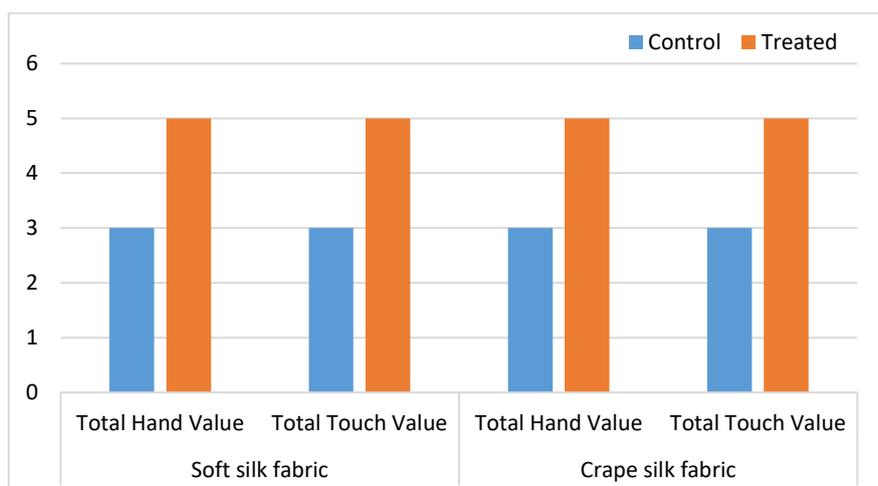


Figure 9. The grades of total hand value and touch values of the soft silk and crape silk fabric

### Fabric weight (gsm) and treatment temperature

To ascertain the effect of fabric weight in gsm, experiments were carried out using three different gsm fabrics that were treated at four different temperatures. ANOVA (two-way) analysis of the effect of chemical treatment of different fabric weights in gsm at different temperatures revealed that fabric weight and treatment temperature have a significant influence on the crease recovery, drape coefficient, bending length and Flexural rigidity of the fabrics as given in Table 9.

Table 9. Effect of fabric weight and treatment temperature

ANOVA - Two factors with replications - fabric weight in GSM (40, 50, 60) and treatment temperature (40, 60, 75, 90 °C)						
Source of Variation	SS	df	MS	F	P-value	F crit
<u>Crease Recovery – Warp</u>						
Fabric weight	1223.72	2	611.86	310.24	0.00	3.40
Temperature	5619.64	3	1873.21	949.80	0.00	3.01
Interaction	1148.94	6	191.49	97.09	0.00	2.51
Within	47.33	24	1.97			
Total	8039.64	35.00				
<u>Crease Recovery – Weft</u>						
Fabric weight	323.56	2	161.78	64.71	0.00	3.40
Temperature	6313.89	3	2104.63	841.85	0.00	3.01
Interaction	435.11	6	72.52	29.01	0.00	2.51
Within	60.00	24	2.50			
Total	7132.56	35				
<u>Drape – Face</u>						
Fabric weight	0.0055	2	0.0028	23.7381	0.0000	3.4028
Temperature	0.0945	3	0.0315	269.8730	0.0000	3.0088
Interaction	0.0107	6	0.0018	15.2302	0.0000	2.5082
Within	0.0028	24	0.0001			
Total	0.1135	35				
<u>Drape – Back</u>						
Fabric weight	0.0105	2	0.0053	14.9370	0.0001	3.4028
Temperature	0.1102	3	0.0367	104.1549	0.0000	3.0088
Interaction	0.0241	6	0.0040	11.3990	0.0000	2.5082
Within	0.0085	24	0.0004			
Total	0.1534	35				
<u>Bending length – Warp</u>						
Fabric weight	0.0706	2	0.0353	8.4667	0.0017	3.4028
Temperature	3.5475	3	1.1825	283.8000	0.0000	3.0088
Interaction	0.3583	6	0.0597	14.3333	0.0000	2.5082
Within	0.1000	24	0.0042			
Total	4.0764	35				

ANOVA - Two factors with replications - fabric weight in GSM (40, 50, 60) and treatment temperature (40, 60, 75, 90 °C)						
Source of Variation	SS	df	MS	F	P-value	F crit
<u>Bending length - Weft</u>						
Fabric weight	0.0939	2	0.0469	12.0714	0.0002	3.4028
Temperature	0.3978	3	0.1326	34.0952	0.0000	3.0088
Interaction	0.0439	6	0.0073	1.8810	0.1256	2.5082
Within	0.0933	24	0.0039			
Total	0.6289	35				
<u>Flexural Rigidity – Warp</u>						
Sample	1.35E-06	2	6.77E-07	48.74	3.54E-09	3.40
Columns	8.15E-06	3	2.72E-06	195.49	5.39E-17	3.01
Interaction	5.72E-08	6	9.54E-09	0.69	0.662228	2.51
Within	3.33E-07	24	1.39E-08			
Total	9.89E-06	35				
<u>Flexural Rigidity – Warp</u>						
Sample	0.00002	2	0.00001	1749.60	0.00	3.555
Columns	0.00008	2	0.00004	6933.80	0.00	3.555
Interaction	0.00004	4	0.00001	1749.80	0.00	2.928
Within	0.00000	18	0.00000			
Total	0.00014	26				

### Dyeability of treated fabrics

To ascertain the dyeing feasibility of the chemically treated fabrics before taking up the garmenting work, 3.75 meters of degummed with the treated fabric was sent to the industry partner in Chennai, where the dyeing trials for mass-scale production were taken before taking up the garmenting work on a commercial scale, for which 20 Nos hues were considered. Also, fabrics of different gsm were dyed using acid colour to ascertain the dyeing feasibility and the utility characteristics of the said fabric samples were assessed. The statistical analysis was carried out and the results are given in Table 10. It is evident from the results that crease recovery angle, drupe co-efficient, bending length and flexural rigidity have significantly improved.

Table 10. Statistical analysis of treated and dyed silk fabrics

Sample / Statistics	Crease Recovery		Drape co-efficient		Bending Length		Flexural Rigidity	
	Angle (°)		(%)		(cm)		(mg. cm)	
	Warp	Weft	Face	Back	Warp	Weft	Warp	Weft
$\bar{x} \pm s$ (n=3)								
Control - A	237	254	1.85	1.84	0.98	1.50	0.0045	0.0615
	$\pm 1.155$	$\pm 0.577$	$\pm 0.006$	$\pm 0.006$	$\pm 0.029$	$\pm 0.029$	$\pm 0.0001$	$\pm 0.0801$

Sample / Statistics	Crease Recovery		Drape co-efficient		Bending Length		Flexural Rigidity	
	Angle (°)		(%)		(cm)		(mg. cm)	
	Warp	Weft	Face	Back	Warp	Weft	Warp	Weft
Treated (75°C) - B	258 ±0.577	279 ±1.155	1.62 ±0.006	1.62 ±0.006	0.80 ±0.058	1.50 ±0.029	0.0029 ±0.0006	0.0099 ±0.0001
Treated & Dyed (45 g/m <sup>2</sup> ) - C	255 ±1.155	273 ±2.646	1.65 ±0.006	1.67 ±0.017	0.80 ±0.058	1.50 ±0.029	0.0032 ±0.0001	0.0099 ±0.0001
Treated & Dyed (65 g/m <sup>2</sup> ) - D	259 ±1.155	280 ±2.000	1.62 ±0.006	1.62 ±0.015	0.78 ±0.029	1.32 ±0.029	0.0032 ±0.0001	0.0099 ±0.0001
Student's t-Test (P values)								
A Vs B	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
A Vs C	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
A Vs D	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

### Commercial production of fabric

To develop varieties of garments, 8 kg of Automatic Reeling Machine (ARM) silk was procured and given for weaving to produce 100 meters (60 g/m<sup>2</sup>) of grey fabric on a power loom as per the specification given in Table 11. The performance of the ARM silk on the power loom was satisfactory and around 91.5 meters of fabric only could be woven on the power loom due to a weaving problem instead of 100 meters as envisaged earlier. The Degumming of 91.5 meters of soft silk fabric was carried out at a printing unit in Bangalore and found that the fabric had a slippage issue due to over-degumming to the tune of 28% instead of 24% meant for Bivoltine silk fabrics. The said fabric was stentered at a finishing unit in Bangalore to reduce the slippage. Thus, it was decided to use part of the fabric (24 m) to prepare Stole (Dupatta) and Scarf with natural colours using Bhandani (traditional tie-dye) technique to make exclusive items for the consumer survey. The remaining part of the fabric (67.5 m) was meant for preparing the fashion garments through the industry partner in Chennai, Tamil Nadu of Indian province.

Table 11. Quality parameter of soft silk fabric

Fabric particulars	Construction details		
Sort	100 % Silk Organza	REED	120
g/m <sup>2</sup> / Width	60 (Grey) / 45 Inches	EPI	182
Warp	20/22, 2 Ply Organzine	PPI	68
Weft	20/22, 3 Ply Tram		

## Experiment on Crape fabric

Trials were also taken with crape fabrics to ascertain the effect of chemical treatment and found that the fabric quality after treatment is at par with the soft silk fabrics. The details are given in Table 12. Also, trials were taken with a crape saree of 6 meters in length and found that the fabric quality of the saree after treatment is good. Thus, it was decided to include the crape variety of silk fabric for garment production to conduct a consumer survey.

Table 12. Crape silk fabric analysis

Sample / Statistics	Crease Recovery		Drape co-efficient		Bending Length		Flexural Rigidity	
	Angle (°)		(%)		(cm)		(mg. cm)	
	Warp	Weft	Face	Back	Warp	Weft	Warp	Weft
$\bar{x} \pm s$ (n=3)								
Control (A)	258	279	1.62	1.62	1.03	1.50	0.0045	0.0107
	$\pm 0.577$	$\pm 1.155$	$\pm 0.006$	$\pm 0.006$	$\pm 0.058$	$\pm 0.029$	$\pm 0.0001$	$\pm 0.006$
Treated (T)	283	295	1.53	1.53	0.80	1.13	0.0029	0.0099
	$\pm 1.155$	$\pm 2.309$	$\pm 0.058$	$\pm 0.046$	$\pm 0.058$	$\pm 0.058$	$\pm 0.0006$	$\pm 0.0001$
Student's t-Test (P values)								
A Vs B	0.00	0.03	0.02	0.01	0.00	0.00	0.00	0.00

## Product development and Costing

The required fabric of 45 m each of soft silk (58 g/m<sup>2</sup>) and crape silk fabric (70 g/m<sup>2</sup>) ready for dyeing (RFD) was procured for garment production. The said fabrics along with the soft silk fabric (67.5) were chemically treated as per the optimized process parameter at the industry partner's modern dyeing unit at Arni, Tamil Nadu and the treated fabrics were handed over to them for making garments like women's wear and children's wear with different styles using their expertise involving National Institute of Fashion Technology (NIFT). Moreover, 24 meters of soft silk fabric was treated at CSTR, Bangalore and given for making garments using the Bandhani technique with natural dyes to a freelance designer in Chennai to prepare stoles and scarves. Also, three different types of fabrics, ready to stitch (RTS), 5 m each, including the dupion silk fabric were procured to produce men's garment formal shirts with chemically treated fabric along with the control fabric for which the services of a master weaver at Bangalore, Karnataka were utilized. The costing sheet is given in Table 13.

Table 13. Costing sheet of various garments developed (Development and characterization of wrinkle resistant and high drape soft silk fabrics; in Rupees)

Particulars / Products	Women's wear Model (1&2)	Women's wear Model (3&4)	Kids' wear Model (1&2)	Stole	Scarf	Soft Silk Shirt (RFF-55 GSM)	Soft Silk Shirt (RFF-60 GSM)	Dupion Shirt (RFF-110 GSM)
Direct cost	[1]	[2]	[3]	[4]	[5]	[6]	[7]	[8]
Raw material (Degummed Fabric Cost)	22990	21704	21930	7190	2006	1081	1173	1863
Expenses - Chemical Treatment	770	627	602	241	241	35	35	35
Labour - Garment making	6900	6900	6900	17200	4800	225	225	225
Total Direct Cost	30660	29230	29432	24630	7047	1341	1433	2123
No. of Garment made (No.)	12	12	24	8	4	1	1	1
Add - Oveheads (25% for 1 to 3, 5% for 4-5, 10% for 6-8)	38325	36538	36790	25862	7400	1475	1576	2335
Cost per Garment	3194	3045	1533	3233	1805	1475	1576	2335
Add - Profit (25 %)	798	761	383	808	463	369	394	584
Selling Price per Piece- Rounded	4000	3800	1900	4000	2300	1800	2000	2900

### Consumer survey and market analysis

The products so developed using the chemically treated fabrics were displayed at one of the showrooms at Thyagaraya Nagar, Chennai, an important commercial hub for silk and textile marketing to ascertain the customer feedback on the subjective qualitative analysis for 21 days in which 121 volunteers including 12 men participated in the market survey analysis and provided their feedback.

#### *Subjective Assessment*

The results of the subjective assessment on the grade of the products for its Feel (F), Drape (D) and Less wrinkle (W) are given in Table 14. Most of the participants graded the products as good and above.

Table 14. Subjective assessment of grading the products based on feel (F), drape (D) and less wrinkle (W)

Products	Women's wear			Childrens' wear			Stoles			Scarves			Men's shirts		
	F	D	W	F	D	W	F	D	W	F	D	W	F	D	W
Not Participated	3	3	3	35	35	35	56	56	56	67	68	67	55	55	55
Participated	118	118	118	86	86	86	65	65	65	54	53	54	66	66	66
Total	121	121	121	121	121	121	121	121	121	121	121	121	121	121	121
Excellent	46	30	31	40	33	37	19	17	12	13	9	11	18	11	12
Very Good	50	59	55	35	41	37	22	25	30	16	20	19	26	29	23
Good	22	29	30	10	8	10	23	22	21	24	23	22	19	22	25
Average	0	0	2	1	4	2	1	1	2	1	1	2	3	4	6
Poor	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Total	118	118	118	86	86	86	65	65	65	54	53	54	66	66	66

### Statistical analysis

While comparing women's wear to children's wear and by considering the customers who have rated both women's wear and children's wear models, this can be treated as a dependent sample as the ratings were given by the same person. The two samples paired t-test was used.

1. The average ratings for the feel of women's wear and children's wear are the same.
2. The average ratings for the drape of women's wear and children's wear are the same.
3. The average ratings for fewer wrinkles in women's wear and children's wear are not the same.  
(soft vs crape)

Also, while comparing the stoles to scarves and considering the customers who have rated both stoles and scarves, this can be treated as a dependent sample as the ratings were given by the same person. The two samples paired t-test was used.

1. Average ratings for the feel of stoles and scarves are the same.
2. Average ratings for the drape of stoles and scarves are not the same. (garment length)
3. Average ratings for fewer wrinkles of stoles and scarves are the same.

### Buying Preference and Price

The results of the subjective assessment of the buying preference of the product and price offered for the products are given in Table 15 along with the statistical analysis of the cost of production in comparison with the price quoted by the participants. It is evident from the t-value that all the products developed have passed the test except stoles and scarves as the cost of production of the said items was high due to natural dyeing using the Bandhani technique.

Table 15. Subjective assessment on the buying preference, the price offered and statistical analysis of women's wear (WW), Kid's wear (KW) and other products

Product	WW Model 1		WW Model 2		WW Model 3		WW Model 4	
Buying Preference	P	Rs.	P	Rs.	P	Rs.	P	Rs.
Not preferred	34		40		38		49	
Preferred	87		81		83		72	
Total	121		121		121		121	
Price								
Not quoted		34		40		38		49
Quoted		87		81		83		72
Total		121		121		121		121
Minimum		600		400		600		600
Maximum		9000		10000		20000		7000
Average		1973.6		2043.2		2218.7		2125
Std Deviation		1397		1643		2312.9		1480.4
Statistical Analysis								
Production Cost		2200		2200		2300		2300
Student t value		-0.945		-0.604		-0.217		-0.827
Survey Result		PASS		PASS		PASS		PASS
Buying Preference	P	Rs.	P	Rs.	P	Rs.	P	Rs.
Not preferred	50		54		66		79	
Preferred	71		67		55		42	
Total	121		121		121		121	
Price								
Not quoted		50		54		66		79
Quoted		71		67		55		42
Total		121		121		121		121
Minimum		450		350		150		150
Maximum		6000		5000		3500		3000
Average		2006.3		1893.3		804.55		734.52
Std Deviation		1242.2		1145		617.59		587.83
Statistical Analysis								
Production Cost		1200		1200		3300		1700
Student t value		4.5899		4.4492		-32.83		-14.6
Survey Result		PASS		PASS		FAIL		FAIL

### *Consumer suggestions and remarks*

Some of the important remarks made by the participants are listed below that show the consumer preferred the product developed using the chemically treated fabric and is ready to purchase at a premium price.

- Good collection - Expecting sales
- Looks decent but bit simple for party wear
- Good and elegant - More varieties required
- Good display but more collections required
- So soft and good quality silk
- Some more Designs and Patterns are required
- Sarees with handwork would be good
- More colourful work on dress material required
- Colours are very dull - Stoles and Scarves
- The Dupion silk shirt looks wrinkled.

### *Association Rules Mining*

This is one of the important marketing techniques to ascertain the preference of the consumer who has already purchased one or two items and is keen to purchase one more item from the same shop. Python programming software [17-18] was used to ascertain the buying preference of the participants that are enumerated below.

- In 100% of the cases where WW models 4 and 3 were preferred, WW model 1 was also preferred
- In 98.5% of the cases where WW model 4 and 1 were preferred, WW model 3 was also preferred
- In 98.4% of the cases where WW models 4 and 3 were preferred, WW model 2 was also preferred
- Customers who preferred only WW model 4, a 90% chance that they also preferred WW model 1
- In 92.5% of cases, customers who preferred KW model 2, also preferred KW model 1
- In 87.3% of cases, customers who preferred KW model 1, also preferred KW model 2
- In 97.6% of cases, customers who preferred scarves also preferred stoles
- In 74.5% of cases, customers who preferred stoles, also preferred scarves.

## **CONCLUSION**

The process parameters of the chemical treatment of silk fabric have been optimized to get the desired results that have been commercially tested. The products were evaluated based on consumer preference. It is concluded that silk fabrics can be chemically treated to improve wrinkle resistance

and drapeability characteristics. The Raman spectroscopy as well as C-13 NMR spectroscopy confirmed that the chemical treatment has made a tertiary-level molecular structure change which facilitates improvement in fabric characteristics. The findings recommend the chemical process using the safe chemical to treat the grey/finished fabrics to have improved utility, durability and comfort properties that are permanent as evident from the spectroscopy analysis. Hence, the products can be popularized.

#### *Author Contributions*

Conceptualization –Gopal H; methodology – Gopal H and Katpadi Mohammed AK; formal analysis – Gopal H and Katpadi Mohammed AK; investigation – Gopal H and Katpadi Mohammed AK; resources – Gopal H, Katpadi Mohammed AK, Kudlur SP and Naik S; writing-original draft preparation – Katpadi Mohammed AK; writing-review and editing – Katpadi Mohammed AK; visualization – Katpadi Mohammed AK, Gopal H, Kudlur SP and Naik S; supervision – Naik S. All authors have read and agreed to the published version of the manuscript.

#### *Conflicts of Interest*

The authors declare no conflict of interest

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