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Research Article

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Characterization of Newly Developed Crease Resistant and High Drape Soft Silk Fabrics and Confirmation of Chemical Changes using Raman Spectroscopy

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Abstract

Soft silk fabrics are extensively used for the production of dress materials and soft sarees. The perennial problem in soft silk fabrics is the wrinkle and drape ability problems. CSTRI has developed new technology for the production of wrinkle resistant and high drape degummed soft silk fabrics using chemical treatment. In the earlier studies the suitability of the chemical has been confirmed for the improvement of softness of fabrics and standardized the processing conditions. In the present study influence of chemical concentration at different temperatures and duration of treatment were studied. It was found that higher the temperature of treatment the chemical reaction was effective and the fabrics have shown improved drapeability and wrinkle resistant. In order to study the chemical modification on the fabrics, the untreated and untreated at different temperatures were studied for Raman spectroscopy. The Raman spectroscopy data has been analyzed and it could be observed that the chemical treatment at 60 °C and 75 °C has shown shift at 1225 and 1650 wave number, which may be attributed to tertiary level molecular structure change in amide I and amide III level due to chemical treatment. Thus the study indicate that using the chemical treatment at high temperatures, the degummed soft silk fabrics could be improved to have better crease recovery, drape, Flexural rigidity and tensile characteristics.

1. Introduction

Silk industry is using raw silk for the production of variety of fabrics *viz*., Organza, soft silk, taffeta, crape and chiffon for its customers. Presently in India, 25384 MT of mulberry raw silk is being produced, out of which, 70% is being consumed for saree production. The silk sarees are not having wrinkle resistant in spite of adopting various methodologies *viz*., construction particulars modifications, chemical treatments using various chemicals *etc*. However, there is always demand for the sarees in the market due to its softness, lustre, durability, elegance, smooth texture and mechanical strength. The consumer interactions have brought out the fact that silk sarees with better drape and less wrinkles properties, would be of help them to wear for longer time. CSTRI under its research studies has tried various chemicals for improving the bulkiness of raw silk for production of silk sarees with better comfort properties.

The concept of chemically treating the silk yarns and fabrics have been tried and found to impart bulkiness in them to a great extent (Natsuki Ikegami *et al.,* 1998; Hariraj *et al.,* 2015, 2017). For silk industry, the wrinkle resistant and high drape fabrics are quite helpful for better comfort properties for the production of both sarees as well as dress materials. In the present study, the effect of chemical treatment, temperature of treatment and duration of treatment on improving the wrinkle resistant and high drape fabrics have been analysed. Further, the modifications in the structure of silk fibre due to chemical treatment have been studied using Raman spectroscopy.

2. Materials and Methods

2.1 Raw Material

The soft silk fabrics have been developed using 2 ply bivoltine raw silk as warp and weft and the number of picks is adjusted to have 50 grams per square meter (GSM) fabrics and used for the studies.

2.2 Degumming Conditions

The soft silk fabrics are degummed at 90 °C for about 45 minutes using soap 4 gpl and soda 1 gpl. The degummed silk fabrics are hot washed followed by cold wash with water and

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dried in the shade (Abdul Kadhar *et al.,* 2008, 2009). The initial weight and degummed weight of the fabrics after conditioning were determined and using the following formula degumming loss was calculated.

$$\text{Degumming loss (\%)} = \frac{\text{Initial weight of fabric - Degummed weight of fabric}}{\text{Degummed weight of fabric}} \times 100$$

The average degumming loss of soft silk fabrics was found to be 23%.

2.3 Design of Experiment

The 12 meters of degummed silk fabrics were divided into 4 batches and one batch kept as it is and the rest three batch fabrics were taken for experimentation. The chemical calcium nitrate was prepared with 6 gpl and used for treatment. Based on the weight of the fabric taken in the material liquor ratio of 1:30, the degummed fabrics are treated at different temperatures of 40 °C, 60 °C and 75 °C for 3 minutes duration, which is based on the standardization carried out earlier. The experiment was repeated three times using one meter cloth each time.

2.4 Standard Test Methods used to Study the Properties of Fabrics

The degummed fabrics treated at different temperatures using the chemical were tested for various characteristics following the standard testing procedures *viz*., Tensile strength IS 1969:1985, Crease recovery IS 4681:1981, Flexural rigidity ASTM D 1388-96, Option A, Drape coefficient percentage IS 8357:1977, (Bureau of Indian standards IS 6936, (1984)).

2.5 Data Collected

The various fabric characteristics *viz*., Tensile strength *viz*., breaking load and elongation warp/ weft, crease recovery, drape coefficient, bending length and flexural rigidity, of degummed silk fabrics and chemically treated silk fabrics were studied. The data were analyzed statistically using SPSS package.

2.6 Raman Spectroscopy

The Raman microscope system, made by HORIBA Scientific with a spatial resolution in the order of 0.5-1 µm as shown in Figure 1, measures the intensity of electromagnetic energy wavelength by wavelength from a sample that scatters laser light of specific wavelength. This type of scattering is called Raman scattering and is the basis of this type of spectroscopy. Raman Spectroscopy is a non-destructive chemical analysis technique which provides detailed information about chemical structure, phase and polymorphs, crystallinity and molecular interactions. It is based upon the interaction of light with the chemical bonds within a material. A Raman spectrum features a number of peaks, showing the intensity and wavelength position of the Raman scattered light. Each peak corresponds to a specific molecular bond vibration, including individual bonds such as C-C, C=C, N-O, C-H, *etc*. and groups of bonds such as benzene ring breathing mode, polymer chain vibrations, lattice modes, *etc*. In order to evaluate the effect of chemical treatment on the silk fibre structure, the both the chemically treated silk fabrics at different temperatures along with control degummed fabric were subjected to Raman spectroscopy test at Indian Institute of Science, Bengaluru.

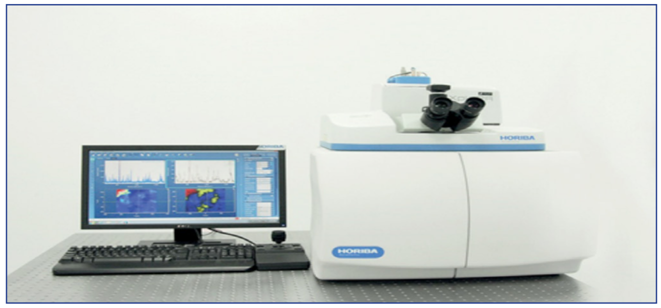


Figure 1: Modern Raman microscope system used for experimentation at IISC, Bengaluru

3. Results and Discussion

The fabric characteristics results of the degummed silk fabrics and chemically treated silk fabrics samples are given in Table 1 and Figure 2(a–f). The ANOVA results of all the fabric characteristics of degummed silk fabrics and chemically treated silk fabrics samples are given in Table 2.

3.1 Crease Recovery Angle

Crease recovery angle of the degummed soft silk fabric was significantly less compared to chemically treated soft silk fabrics to the extent of indicating that chemically treated soft silk fabrics are having better crease resistance as indicated in Table 1 and Figure 2a. The crease recovery angle is increased significantly, as the temperature of treatment is increased from 40-75 °C both in warp and weft way to the extent of 8.64% in warp way and 9.31% in weft way.

3.2 Drape Coefficient

Drape coefficients of the degummed soft silk fabric were found to be 1.85 on both face and back side of the fabrics. Higher the drape coefficient, stiffer the fabric. The chemically treated soft silk fabrics have shown less drape compared to untreated fabrics on both face and back sides. The soft silk fabrics treated with chemical at 75 °C have shown 12.4% better drapeability indicating that the fabrics have become flexible and easily drapeable, so that it is comfortable to wear as shown in (Table 1 and Figure 2b).

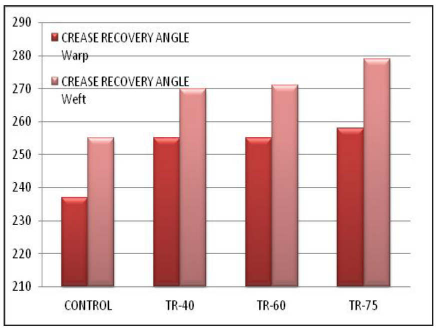
3.3 Tensile Properties

From the Table 1 and Figure 2c & 2d, it could be observed that the breaking load and elongation of the chemically treated fabric at 75 °C in warp direction has increased by 4.82% and 25% respectively, whereas breaking load and elongation of the chemically treated fabric at 75 °C in weft direction has decreased compared to untreated fabrics, which may be attributed to slight shrinkage due to chemical treatment.

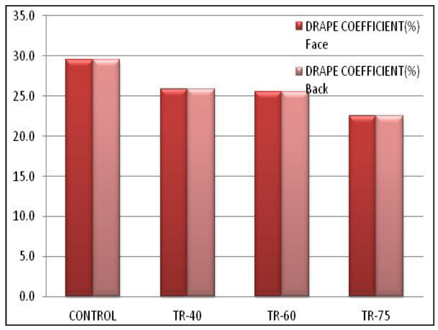
Table 1: Average fabric characteristics of degummed soft silk and chemically treated soft silk fabrics at different temperatures

Particulars	Samples	N	Mean	Std deviation	Minimum	Maximum
Crease Recovery Angle - Warp	Control	8	237.25	1.035	236	238
	Temp- 75 °C	8	257.75	1.282	256	260
	Temp- 60 °C	8	254.88	1.246	254	257
	Temp- 40 °C	8	255.25	1.035	254	256
Crease Recovery Angle - Weft	Control	8	255	1.069	254	256
	Temp- 75 °C	8	278.75	1.035	278	280
	Temp- 60 °C	8	271.13	1.246	270	273
	Temp- 40 °C	8	269.75	1.282	268	272
Drape Coefficient - Face	Control	8	1.8463	0.00518	1.84	1.85
	Temp- 75 °C	8	1.6425	0.01753	1.62	1.66
	Temp- 60 °C	8	1.7325	0.01669	1.71	1.76
	Temp- 40 °C	8	1.7413	0.01458	1.72	1.76
Drape Coefficient - Back	Control	8	1.8475	0.0707	1.84	1.86
	Temp- 75 °C	8	1.645	0.01927	1.62	1.67
	Temp- 60 °C	8	1.7325	0.01669	1.71	1.76
	Temp- 40 °C	8	1.73438	0.01408	1.72	1.76
Breaking Load - Warp	Control	8	22.363	0.2066	22.1	22.6
	Temp- 75 °C	8	23.213	0.3758	22.4	23.6
	Temp- 60 °C	8	21.6	2.0029	19	23.8
	Temp- 40 °C	8	22.238	0.5317	21.5	23
Breaking Load- Weft	Control	8	45.725	0.5523	45	46.6
	Temp- 75 °C	8	31.213	0.3834	31	32.1
	Temp- 60 °C	8	40.913	3.9961	37.4	46.3
	Temp- 40 °C	8	34.413	2.076	32.4	37.8
Elongation Percentage - Warp	Control	8	18.3	0.3546	17.9	18.8
	Temp- 75 °C	8	22.9	0.3817	22	23
	Temp- 60 °C	8	21.575	0.8345	20.3	23
	Temp- 40 °C	8	21.668	1.0218	20	23
Elongation Percentage - Weft	Control	8	12.488	0.3357	12	12.8
	Temp- 75 °C	8	11.7	0.769	11	13.3
	Temp- 60 °C	8	12.275	1.0375	11.1	14
	Temp- 40 °C	8	11.3	1.077	10	13
Bending Length - Warp	Control	8	1	0	1	1
	Temp- 75 °C	8	0.875	0.463	0.8	0.9
	Temp- 60 °C	8	0.888	0.0641	0.8	1
	Temp- 40 °C	8	0.938	0.0518	0.9	1
Bending Length - Weft	Control	8	1.513	0.0354	1.5	1.6
	Temp- 75 °C	8	1.325	0.0463	1.3	1.4
	Temp- 60 °C	8	1.425	0.0463	1.4	1.5
	Temp- 40 °C	8	1.438	0.058	1.4	1.5

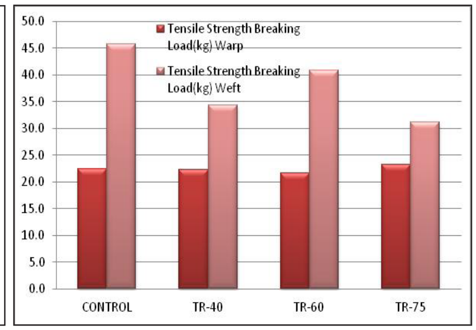
Particulars	Samples	N	Mean	Std deviation	Minimum	Maximum
Flexural Rigidity - Warp	Control	8	0.004488	0.0000354	0.0044	0.004
	Temp- 75 °C	8	0.003163	0.0003503	0.0023	0.0033
	Temp- 60 °C	8	0.00245	0.000233	0.0022	0.0028
	Temp- 40 °C	8	0.003438	0.000256	0.0032	0.0038
Flexural Rigidity - Weft	Control	8	0.015213	0.0000354	0.0152	0.0153
	Temp- 75 °C	8	0.009763	0.0003114	0.009	0.0099
	Temp- 60 °C	8	0.011813	0.0002031	0.0114	0.0121
	Temp- 40 °C	8	0.012313	0.0002167	0.012	0.0126



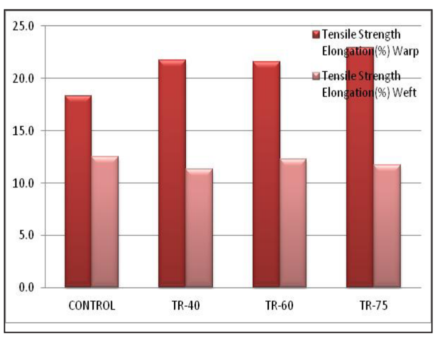
a) Crease recovery angle



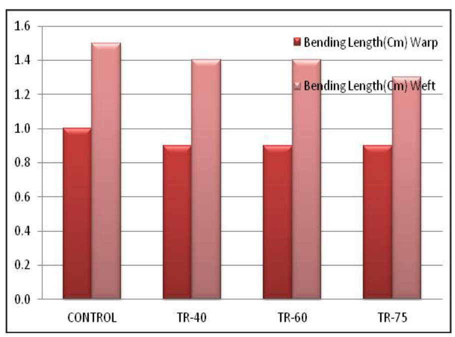
b) Drape coefficient



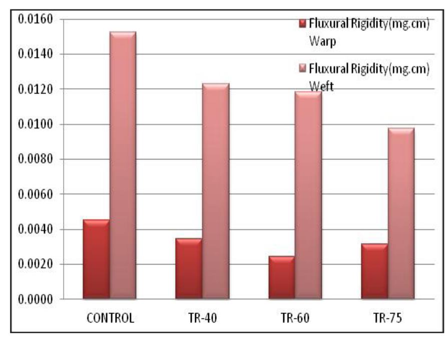
c) Tensile strength (Breaking Load)



d) Tensile strength (Elongation)



e) Bending length



f) Flexural rigidity

Figure 2: Effect of chemical treatment at different temperatures on fabric characteristics of soft silk fabric characteristics

3.4 Bending Length

The flexibility of fabric can be measured by determining by bending length. High bending length indicates the stiffer of fabric. From Table 1, it could be observed that bending length was decreased by 12.5% both in warp and weft direction when the soft silk fabrics are treated with chemicals (Figure 2e). Higher the temperature of treatment, the fabrics has more flexibility.

3.5 Flexural Rigidity

It is a measure of the resistance of a fabric to bending by external forces. From Table 1, it could be observed that flexural rigidity was decreased by 30% in warp direction and in weft direction by 36%, when the soft silk fabrics are treated with chemicals at high temperature (Figure 2f).

Based on the analysis, it was found that the chemically treated soft silk fabric characteristics at high temperature were found to be superior in terms of crease recovery, drape coefficient, tensile properties, bending length and flexural rigidity characteristics. It may be pointed out that the present observations were similar to studies conducted by Park suzin *et al.,* (2010).

3.6 Analysis of Variance Results

The untreated degummed soft silk fabrics and chemically treated degummed soft silk fabrics characteristics studied were analyzed for ANOVA. The results are given in Table 2. From the results in Table 2, it could be seen that most of the fabric characteristics of chemically treated soft silk fabrics were found be significantly different at 1% level, whereas the breaking load in warp and elongation percentage in weft are significant at 5% level. Thus, indicating that chemically treated degummed soft silk fabrics were found to be superior in terms wrinkle resistance and drapeability characteristics.

3.7 Raman Spectroscopy Results

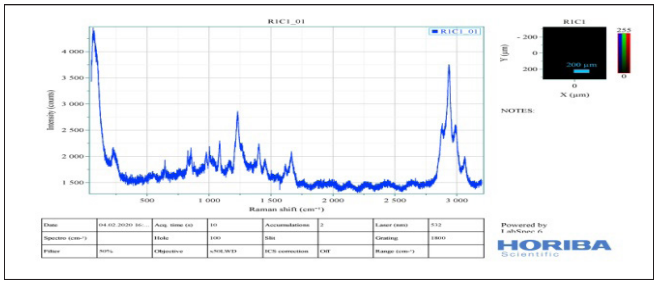
The degummed soft silk fabrics and the chemically treated soft silk fabrics at different temperatures were analysed for Raman spectroscopy. The graphs generated by the Raman spectrophotometer are given in Figure 3a to 3d. From the Figure 3a to 3d, it could be observed that chemical treatment of soft silk fabrics has shown shift at higher temperature. The Raman spectroscopy data has been analyzed and the results are combined in Figure 4. From the results it could be observed that the chemical treatment at 60 °C and 75 °C has shown shift at 1225 and 1650 wave number as shown in Figure 4, which may be attributed to tertiary level molecular structure change in amide I and amide III level due to chemical treatment.

Table 2: ANOVA results of degummed silk fabrics and chemically treated at different temperatures soft silk fabrics characteristics

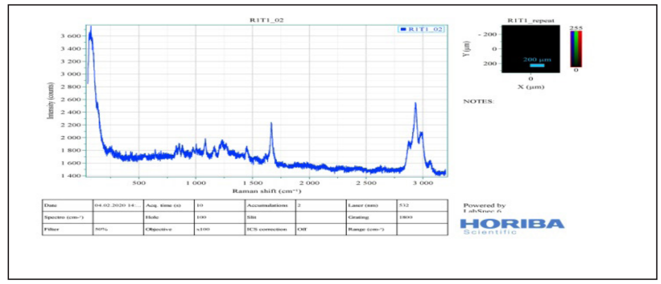
Particulars	Samples	Sum of square	Degree of freedom	Mean sum square	F-Value	Significance
Crease Recovery Warp	Between	2139.09	3	713.031	534.18	0.000**
	Within	37.375	28	1.335	-	-
	Total	2176.47	31	-	-	-
Crease Recovery Weft	Between	2365.34	3	788.448	582.88	0.000**
	Within	37.875	28	1.353	-	-
	Total	2403.22	31	-	-	-
Drape Coefficient Face	Between	0.167	3	0.056	269.6	0.000**
	Within	0.006	28	0.000	-	-
	Total	0.173	31	-	-	-
Drape Coefficient Back	Between	0.165	3	0.055	245.02	0.000**
	Within	0.006	28	0.000	-	-
	Total	0.171	31	-	-	-
Breaking Load Warp	Between	10.553	3	3.518	3.142	0.041*
	Within	31.346	28	1.120	-	-
	Total	41.90	31	-	-	-
Breaking Load Weft	Between	1016651	3	338.884	66.151	0.000**
	Within	143.441	28	5.123	-	-
	Total	1160.09	31	-	-	-
Elongation Percentage Warp	Between	93.198	3	31.066	61.763	0.000**
	Within	14.084	28	0.503	-	-
	Total	107.28	31	-	-	-
Elongation Percentage Weft	Between	7.033	3	2.344	3.189	0.039*
	Within	20.58	28	0.735	-	-
	Total	27.62	31	-	-	-
Bending Length Warp	Between	0.077	3	0.026	11.573	0.000**
	Within	0.063	28	0.002	-	-
	Total	0.14	31	-	-	-
Bending Length Weft	Between	0.142	3	0.047	23.13	0.000**
	Within	0.058	28	0.002	-	-
	Total	0.200	31	-	-	-
Flexural Rigidity Warp	Between	0.000	3	0.000	93.735	0.000**
	Within	0.000	28	0.000	-	-
	Total	0.000	31	-	-	-

Particulars	Samples	Sum of square	Degree of freedom	Mean sum square	F-Value	Significance
Flexural Rigidity Warp	Between	0.000	3	0.000	86.213	0.000**
	Within	0.000	28	0.000	-	-
	Total	0.000	31	-	-	-

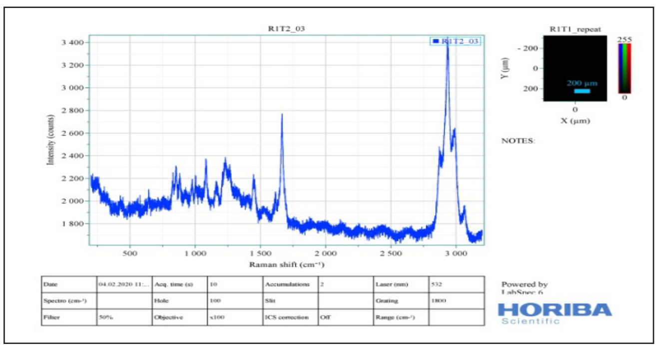
** - Significant at 1% level, * - Significant at 5% level



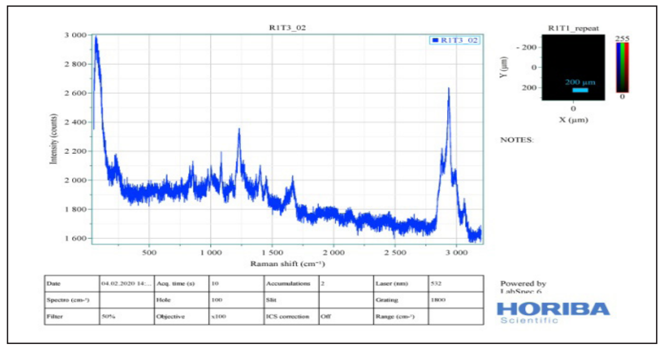
a) Control fabric



b) Fabric chemically treated at 75 °C



c) Fabric chemically treated at 60 °C



d) Fabric chemically treated at 40 °C

Figure 2: Effect of chemical treatment at different temperatures on fabric characteristics of soft silk fabric characteristics

4. Conclusion

Based on the studies it could be concluded that soft silk fabrics can be chemically treated to improve wrinkle resistance and drapeability characteristics. The study indicates that higher the temperature of treatment the chemical effect is effective on the fabric structure. The Raman spectroscopy confirmed that higher temperature of chemical treatment has shown a tertiary level molecular structure change which facilitates improvement in fabric characteristics. The study is being continued on large scale at industry level to produce wrinkle resistant high drape fabrics for marketing.

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