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A Troubleshooting Approach towards the Generation of White Patches on Silk Fabric

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ABSTRACT: *The phenomenon of occurrence of white patches on dyed silk fabric stored for few months, after production, is an intricate matter. There may be many reasons behind this occurrence. The possible sources may be deposition of fatty acid salts or growth of certain fungus or micro-organisms. This study focuses a detailed observation and analysis of white patches occurring in dyed silk fabric that has been stored for some months. An attempt has been made in this study to identify the possible reason behind the occurrence of these patches on the fabric and analyze the surface morphology of the affected areas of the fabric by adopting Scanning Electron Microscope (SEM) study, Fourier Transform Infrared Spectroscopy (FT-IR) and fungal growth study. The studies have identified the presence of some chemical compounds in this white deposition. Furthermore, the article also discusses the mechanism of deposition of these chemical compounds.*

Keywords *-silk, fungus, SEM, FTIR, microbiological testing, fatty acid, salt deposition*

I. INTRODUCTION

Silk is a naturally occurring protein fibre produced by the worms and has been potentially used as a textile material for over five thousand years. Natural raw silk is composed mainly of sericin about (22-25 %), fibroin about (62.5-67.0 %) and the rest is of water and mineral salts [1]. Fibroin is a single protein which is insoluble in hot water. On the other hand, sericin is primarily amorphous and acts as a gum binder to maintain the structural integrity of the cocoon, which makes it more water-soluble than fibroin [2]. This delicate filamentous fibre is well known for its sheen texture, water absorbency, dyeing affinity, thermal tolerances along with insulation properties [3]. Silk is basically a strong fibre having strength 3.6-4.0 cN/tex which can be attributed for its linear and beta configuration and considerable crystalline polymer system [4]. Amongst the different species of moths producing raw silk the best known variety is the Lepidopter Bombyx mori. Customarily, prior to weaving, raw silk is degummed for removal of sericin to obtain its soft and lustrous feel. Chemical substances like lubricants, antistatic agents, natural-based auxiliaries which are added during degumming of the silk fibres provide food source for microorganisms. Synthetic fibres are not totally immune to micro-organisms but it has been found that certain synthetic fibres like polyurethane fibres can get damaged due to the growth of micro-organisms. On the other hand, natural fibres because of evolution are more easily attacked by micro-organisms as for example wool is more susceptible to bacterial attack while cotton is more likely attacked by fungi [5]. Once a fabric is invaded by fungal growth, various cleaning methods may not return the garment to its previous condition. Natural fabrics (i.e. wool, cotton and silk) appear to support more fungal growth than manmade fabrics (polyester) [6].

Heald, Commoner and Ballard have described in the article entitled 'A Study of deposits on glass in direct contact with mounted textiles' their observation about different appearances according to the material stored under glass using a whole range of analysis methods to identify the substances. These were generally identified as saturated fatty acids and a decomposition product of unsaturated fatty acids.

Migrated soaps present in exhibition fabrics and textile objects due to earlier processing and washing procedures were interpreted as the origin of these fatty acids [7]. In a silk processing unit it has been observed that the occurrence of white patches in the finished fabric cause a serious problem. Those industries which export expensive silk fabrics, suffer heavy loss due to such severe problems. These obstinate white patches are found to be visible all over the fabric in local colonies and may appear in any fabric lot. Sometimes there are some large spots observed near the selvedge ends of the fabrics. They visually appear like fungi. The fabric samples have been subjected to Scanning Electron Microscope (SEM) study, Fourier Transform Infrared Spectroscopy (FT-IR) and fungal growth study to identify the possible reason behind the occurrence of these patches on the fabric surface and analyze the surface morphology of the affected areas of the fabric. The studies have identified the presence of some chemical compounds in this white deposition.

II. MATERIAL AND METHODS

The fabric sample had been made from 100% filature and duppion silk yarn having the specifications as furnished in Table 1. The parameters of the produced fabric have been defined in Table 2. The conditions adopted and the chemicals applied here during the degumming followed by dyeing of the silk yarn have been provided in Table 3.

Table 1 Yarn specifications

Sl. No.	Yarn Particulars	Warp Yarn	Weft Yarn
1.	Variety	Mulberry filament filature silk	Duppion silk
2.	Count before degumming	2 ply 42 denier	2 ply 220 denier
3.	Count after degumming	2 ply 34 denier	2 ply 175 denier
4.	Twist per metre	700	180
5.	Twist direction	s-twist	z-twist

Table 2 Fabric Specifications

Sl. No.	Fabric property parameters	Values
1	Weave construction	Plain weave
2	Thread density (ends/cm × picks /cm)	48 × 33
3	Areal density (gsm)	91.00

Table: 3 Yarn dyeing process and recipe

	Recipe	Quantity in g/lit. (weft)	Quantity in g/lit. (warp)
Degumming Material: Liquor=1:20 30 min at 90° C, Ph 10.5-11	(1) Wetting & stain removal agent	2	2
	(2) Lubricating agent	2	2
	(3) Wetting agent	1	1
	(4) Enzyme, degumming agent	3.5	-------
	(5) Soap	------	5gpl
Bleaching Material: Liquor=1:20 30 min at 90° C, Ph 8-9	Hot wash and cold wash	10 min each	10min each
	(1) Hydrogen peroxide	3	3
	(2) Stabilizer AWNI	1	1
Dyeing Material: Liquor=1:20 30 min at 86°C	(1) Leveling agent (2) Acetic acid (for Ph 3-4) (3) Metal complex dye Navy blue (shade)	2 2 As per shade depth	2 2 As per shade depth
	Hot wash and cold wash	10 min each	10 min each
Dye fixation at 50° C for 10 min	Dye fixing agent (Optifix)	4	4
Scrooping at 25^0C	(1) Acetic acid (2) Anti-fungal agent (3) lubricating agent (4) Antistatic agent	2 4 2 ------	2 4 2 3

The defective silk fabric sample had been examined visually. The defect appeared like fungal growth as shown in Fig.1 below.

Fig.1 occurrence of white patches on the surface of the dyed silk fabric

III. MICROBIOLOGICAL TEST

The microbiological testing of the dyed silk fabric samples had been carried out as per Parallel Streak Method of AATCC Test Method 147-2004 which is useful for evaluating bacterial growth on treated textile materials. The dyed silk fabric samples had been placed in intimate contact with nutrient agar which had been previously streaked with an inoculum of a test bacterium. A standard strain of bacteria is used which is specific to the requirements of the material under test. Staphylococcus aureus was used as a representative gram positive organism. No bacterial growth had been found to be occurring on the surface of the textile material.

IV. SCANNING ELECTRON MICROSCOPE (SEM) STUDY

The scanning electron microscope (SEM) is one of the most versatile instruments available for the examination and analysis of the micro structure surface morphology and chemical composition characterizations. The electron microscope produces images of the dyed fabric samples by scanning it with a focused beam of electrons. The electrons interacted with atoms in the sample, producing various signals that can be detected and that contain information about the sample's surface topography and composition. This study had been conducted at several national and international research organizations. The defective places of the dyed fabric had been observed under SEM at magnification level of 500 X, 1.00 K X, 10.00 K X where the defects were seen as extraneous fine flakes and agglomeration of deposits as supported by the SEM photographs provided below [Figs. 2 (a), 2 (b) and 2 (c) respectively]. The SEM analysis and observations of the other national and international research organizations have been provided in Figs. 3, 4 respectively.

Fig. 2(a) SEM at Mag 500 X

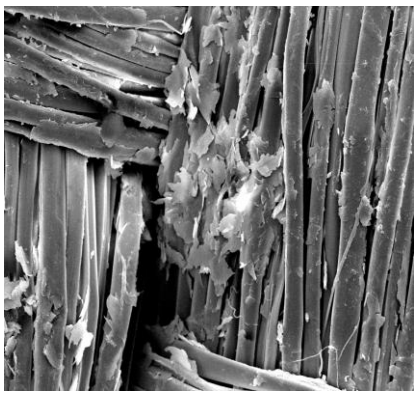

Fig. 2(b) SEM at Mag1.00 K X

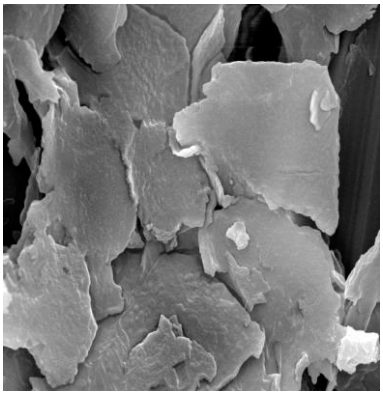

Fig.2(c) SEM at Mag 10.00 K X

Courtesy: *Razabazar Science College, University of Calcutta, India*

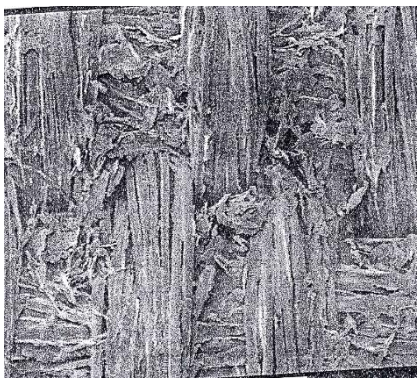
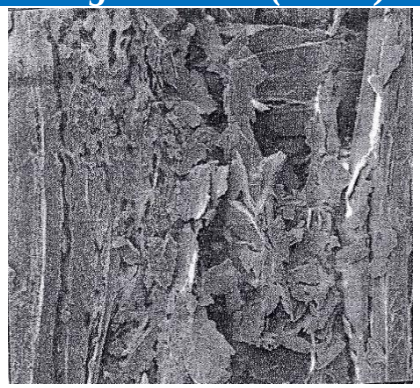
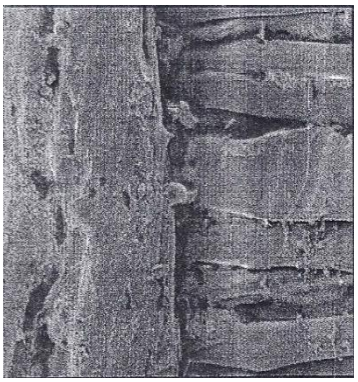

Fig. 3(a) SEM at Mag 200 X **Fig. 3(b) SEM at Mag 500 X** **Fig. 3(c) SEM at Mag 700 X**

Courtesy: *Bombay Textile Research Association, India*

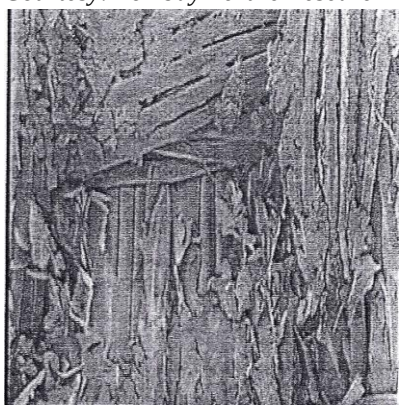
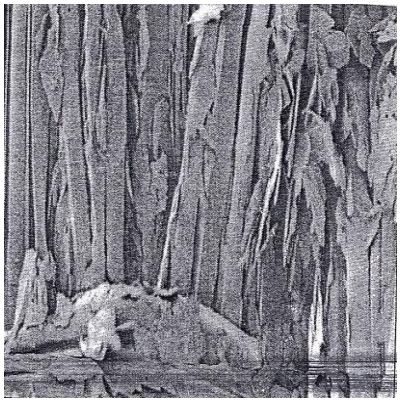
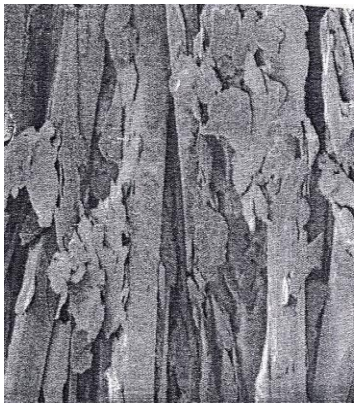

Fig. 4(a) SEM at Mag 350 X **Fig. 4(b) SEM at Mag 500 X** **Fig. 4(c) SEM at Mag 1.00 KX**

Courtesy: *Intertek Testing Services India Pvt. Ltd., Mumbai, India*

V. FTIR SPECTROSCOPY ANALYSIS

Fourier Transform Infrared spectroscopy (FT-IR) has wide applicability in structure elucidation analyzing both qualitatively and quantitatively, of materials which are either synthesized chemically or of natural origin [7].

In this method the dyed silk fabric samples had been placed on the FTIR spectrometer and the infrared radiation had been allowed to pass through the fabric samples. Some of the infrared radiation have been absorbed by the sample while the rest amount gets transmitted. The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. It had been observed that no two unique molecular structures produce the same infrared spectrum. The FTIR spectroscopy analysis of the dyed silk fabric samples both in the affected and unaffected areas have been carried out in National Institute of Research on Jute and Allied Fibre Technology, India and the results of the spectral analysis have been shown in Fig. 6 below.

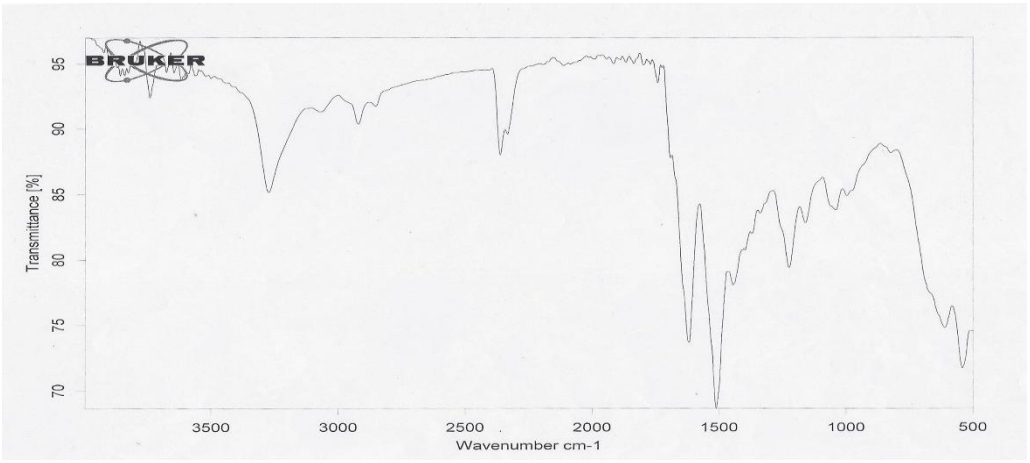

Fig. 6(a) FTIR spectroscopy analysis of the of the dyed silk fabric sample (without affected area)

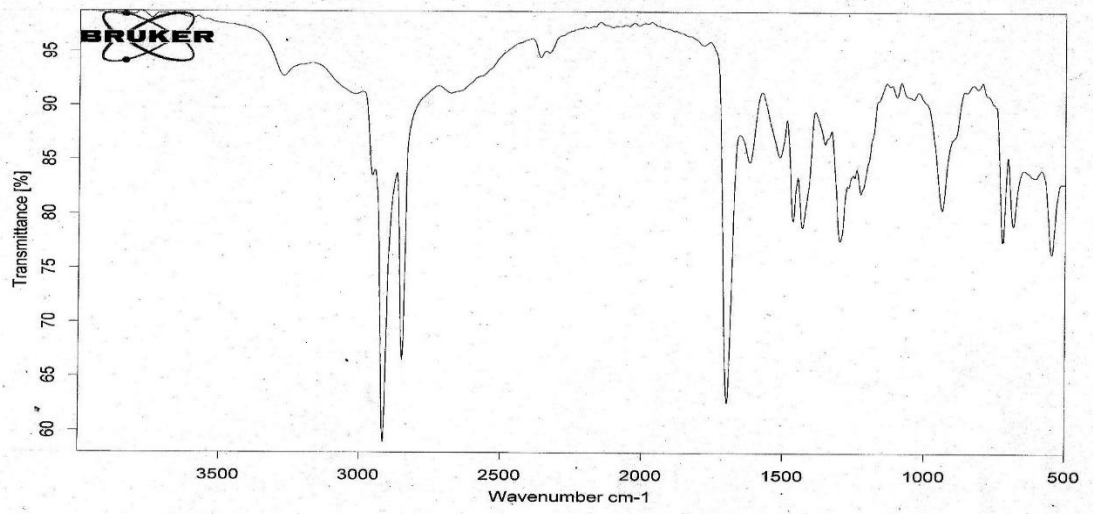

Fig. 6(b) FTIR spectroscopy analysis of the affected area (white patches) of the dyed silk fabric sample

Courtesy: *National Institute of Research on Jute and Allied Fibre Technology (NIRJAFT), India*

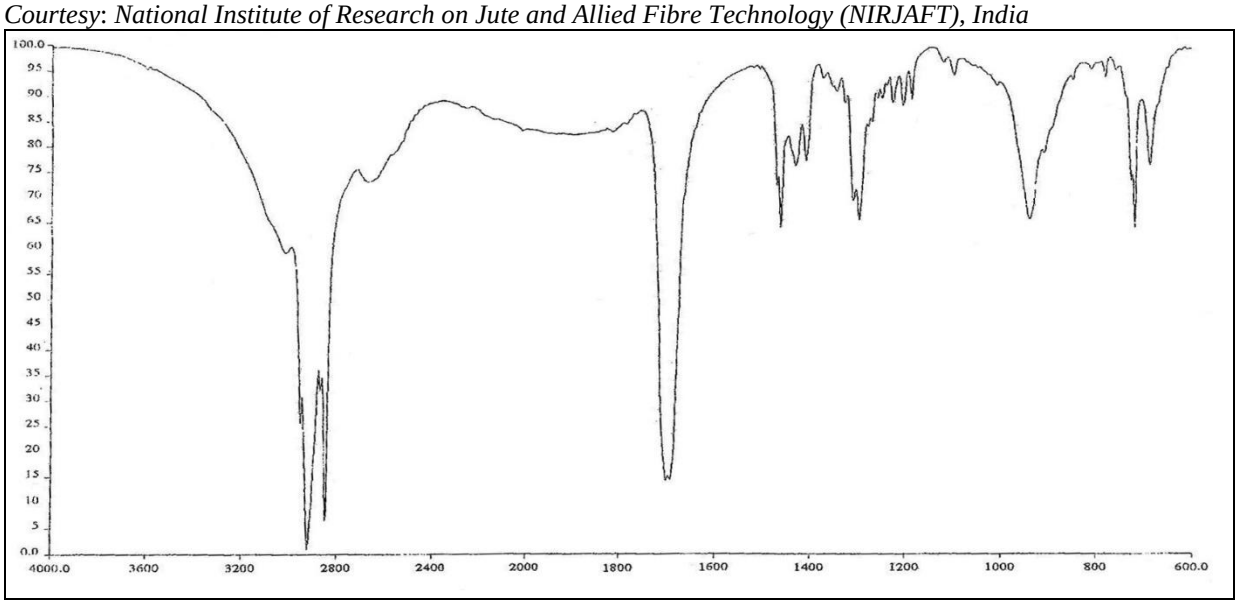

Fig. 7 FTIR spectroscopy analysis of the affected area (white patches) of the dyed silk fabric sample

Courtesy: *Stazione Sperimentale Seta, Italy*

VI. RESULTS AND DISCUSSIONS

The different testing methods carried out at the different national and international research organizations, their test reports and analysis indicate the presence of non-fibrous deposits on the fabric surface at the defective areas which is not seen in the normal areas of the fabric. All the national and international research organizations after performing the method of microbiological testing of the dyed silk fabric samples have unanimously confirmed that there was no microbial or fungal growth on the surface of the dyed silk fabric. The international research organization Stazione Sperimentale Seta, Italy have given hints about the agglomeration of fatty acid deposits which have appeared as white flakes under high magnification. Again, Davis and Mauer have achieved in their work the specific identification of fatty acid compounds from spectral analysis by focusing on specific absorbance regions [9] and their FTIR spectral findings show that the wavenumber of the fatty acid compounds belong in the spectral region of 2955 cm^{-1} to 2850 cm^{-1} which fairly correlates with the FTIR spectral analysis of the affected area (white patches) of this dyed silk fabric sample, Fig.7. According to Heald, Commoner, Ballard and Mary, a probable source of fatty acid deposits is the residual soap left in the textile from wet processing of textiles. Wulfert stated that fatty acids and fatty acid soaps diffusion had noticed in non-textile objects like panel paintings too, but these kinds of residues might have enhanced mobility due to the use of detergents in washing treatments but would not be rinsed off completely.

VII. CONCLUSION

The results obtained in the present study indicate that white patches on silk fabric are not due to the growth of fungus or microorganisms but indicates the deposition of some chemicals which have accumulated during the process. This observation and its reason behind occurrence is being proliferated by FT-IR study conducted by the several research organizations. According to the FT-IR report, deposition of fatty acid or its residual salts have originated due to use of soap applied as a degumming agent and insufficient rinsing may not have removed this completely. The authors are of the opinion that as the fabric production is a lengthy process and is handled by different workers during different stages of production like chemical measurement, machine running, checking of parameters, dyeing, weaving and storing therefore there may not be sufficient supervision and follow-up of the standard machine and process parameters. Moreover, proper washing maintaining the adequate time and rate of flow of water after each stage of process is very important in mitigating these type of defects.

VIII. ACKNOWLEDGEMENT

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