Applied Biological Research 24(4): 455-462; (2022)
DOI: 10.5958/0974-4517.2022.00054.4

TRADITIONAL WASHING FORMULATIONS OUTSTAND COMMERCIAL ONES FOR WASHING *Antheraea assamensis* (MUGA) SILK CLOTH

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(Received 7 June, 2022; accepted 26 October, 2022)

ABSTRACT

Safe silk washing is an issue faced by its users for long because silk is prone to get damaged during conventional washing. The current study focused on the possible *Antheraea assamensis* (Muga) silk washing formulation which was a rare event until people realized the importance of cleaning and hygiene with the onset of covid-19 pandemic. *Kolakhar*, lemon (citrus) juice, and some commercially available washing products were studied for bond changes by FTIR, surface changes by SEM and colour deviations from control samples. The study showed that there was formation of new bands in all treated samples (except water wash) between 1513-1518 cm^{-1} that is attributed to β-sheets; and 1698-1703 cm^{-1} linked with β-turn conformation in all the treatments. The morphological observation illustrated fibrillation and peeling off fibers in the commercial washed fabrics. Colour change (δE) was least in fabric conditioner (0.85) <citrus wash (1.06) <*Kolakhar* wash (1.13) and maximum for fabric whitener (5.46). Overall, the traditional agents proved excellent washing factor and has the potential in opening a window for optimized formulation that can be employed for Muga washing.

Keywords: Commercial washing agents, Muga, silk cloth, traditional washing

INTRODUCTION

Silk fabrics are tagged as “the queen of fibers" because of their softness, comfort, good hygroscopicity, mechanical strength, environmental stability, etc. (He *et al.,* 2017). Muga silk produced by *Antheraea assamensis* is exclusive to Assam, India which has got the Geographical tag (Devi *et al.,* 2011). The prime identity of Muga silk is its golden luster appearance that makes it one of the costliest silks (₹. 16000-18000 kg^{-1} yarn) (Goswami *et al.,* 2016; Gogoi *et al.,* 2017). One major characteristic of Muga silk is its longevity which gets threatened due to washing (Devi *et al.,* 2011). Muga silk washing is an infamous activity due to the non-availability of silk-specific commercial washing products; and dry cleaning is uneconomical. With the on-set of covid-19 pandemic, washing of clothes has become a mandatory task that is also extended to Muga silks which are more commonly worn in occasions that involve mass gatherings.

Assessing and analyzing the washing parameter of silk fabrics has been a matter of concern for researchers for quite a long (Talukdar *et al.,* 2011). It has been reported that chemical treatments causes modifications in the polypeptide chains and side chains of amino acids which influences the chemical, physical and mechanical properties of fiber (Talukdar *et al.,* 2011). The degumming of Muga silk is supposed to result in hydrolytic degradation of protein polypeptide that leads to functional and physical changes, and also esthetic aspect, such as dull appearance, fibrillations etc.(Freddi *et al.,* 2003; Choudhury *et al.,* 2016). Similar activities can be attributed to washing with

ordinary cloth detergent which may decrease the shine and lifespan of costly Muga silk. Very scanty information is available on the washing of Muga silk (Talukdar *et al.*, 2011). There are different non-Muga specific washing agents for normal Muga clothing that include fabric conditioner, detergent for wool, detergents, washing soap, fatty soap, fabric stiffener, etc. that makes it vulnerable to damage. With the influence of fashion and re-designing of traditional dresses, there is rising demand for suitable washing agents for Muga silk clothing. In view of above, the present work was aimed to assess the effect of non-specific commercially available washing agents and traditional washing formulation on Muga cloth.

MATERIALS AND METHODS

Muga fabric was collected from Dhakuakhana, Assam, India in 2020. The commercial washing agents and lemon were bought from the local market. *Kolakhar* was prepared in the laboratory.

Preparation of Kolakhar and lemon juice

Kolakhar was prepared as per Deka and Talukdar (2007) with some refinements. The peels of wild banana (*Musa balbisiana*) fruit were sun-dried; and 500 g dried peel was burned to charcoal and ashes. From the burned peels 6 g were suspended in 120 mL sterilized distilled water and thoroughly mixed by stirring it in a magnetic stirrer for 45 min at 500 rpm (Motorless magnetic stirrer, 4050, Tarson, India). After complete mixing, the solution was filtered using a muslin cloth that yielded a light black coloured filtrate. For lemon juice preparation, citrus limon were squeezed to extract juice which was then filtered to remove the pulp and seeds. *Kolakhar* washing (KWM) agent, lemon juice (LW) wash, water wash Muga (WWM) and untreated control i.e. raw Muga (RM), were evaluated.

Preparation of commercial washing agents

Washing agents claiming as detergent 'SEW' (Surf excel wash), laundry soap 'RS' (Rin supreme bar), commercial indigenous laundry soap 'DS' (White Amrit Dhela soap), fabric stiffener 'RFS' (Revive fabric stiffener), fabric conditioner 'CFC' (Comfort fabric conditioner), liquid detergent for woolen clothes 'EW' (Godrej Ezee), and fabric whitener 'VOX' (Vanish oxy action) were used as commercial washing agents. For all the washing agents, 1 g washing agent was dissolved in 30 mL sterile distilled water using a vortex (adjustable speed vortex, E11190, Abdos, India). These were selected in relation with traditional methods.

Washing process

Muga silk was cut into pieces weighing 60 mg each. These pieces were submerged in 30 mL of test solutions with material-to-liquid ratio (MLR) 0.002 mL^{-1} at 25°C. The solutions containing the fabric were shaken gently and kept suspended for 30 min (Deka and Talukdar, 2007). The pieces were then rinsed thoroughly with distilled water until washing agents were complete removed. The washed pieces were subjected to air drying before preparation of analysis.

Micrographic images

To observe post-treatment morphological changes in the fiber, the fabrics were cut into 5 x 5 mm size and dried in oven to ensure the complete removal of moisture. The samples were coated with platinum using auto-fine coater and examined under a scanning electron microscope (6390lv, Jeol, Japan) at an accelerating voltage of 20 kV. The image mode was secondary electron image with working distance of 10 mm (Rout *et al.*, 2001).

Muga colour analysis

Determination of the Red, Green and Blue values were carried out by using the method of Pan *et al.* (2009) and Saravanan *et al.* (2016) with some modifications. Instead of considering every one pixel of the segment, the RGB taken was the mean of the entire fabric piece that was considered for RGB

to HSL (Hue, saturation, and luminosity) colour space conversion. All the samples were scanned in Canon scanner (Canoscan Lide, 220, Canon, India) at 600 dpi to maintain the constant conditions. From the scanned image, three locations of different orientations were selected for RGB analysis using software ImageJ to get the complete colour information. The software determined the mean RGB values from which Hue angle (H), saturation (S) and luminosity (L) were derived. The obtained RGB values were in 0-255 range which was converted to 0-1 range by dividing each value by 255 (R',G' and B') for calculations (Saravanan *et al.,* 2016; Gowda and Yuan, 2018). L, S and H were determined using the following formula:

$$L = (C_{max} + C_{min})/2 \quad \text{(i)}$$

$$S = \Delta/(1-|2L-1|), \Delta <> 0 \quad \text{(ii)}$$

$$H = 60^{o} \times [\{(G'-B')/\Delta\} \text{mod} 6], C_{max} = R' \quad \text{(iii)}$$

Where C_{max} is maximum (RGB) and C_{min} is minimum (RGB) and Δ is the difference between C_{max} and C_{min}.

Colour difference, δE, indicates the perceived total difference in colour (visual) coordinates between two colours. The extracted RGB data from ImageJ was used to calculate δE values using δE calculator (*http://colormine.org/delta-e-calculator/cie2000*) (Gallagher, 2014; Kocaoğlu and Olguntürk, 2019). The hexadecimal (HEX) colour code and the associated colour were determined as per Plante and Cushman (2020).

Fourier transforms infrared spectra (FTIR) studies

FTIR spectrophotometer (Impact 410, Nicolet, USA) was used for the analysis of treated and untreated Muga silk samples. Approximately 30 mg samples were cut finely into small parts using clean scissors. The samples were made into pellet with potassium bromide. The measurement was seen with step size 1.0 cm^{-1} from 4000 to 400 cm^{-1}. OriginPro 8.5 was used for graph generation and peak analysis.

RESULTS AND DISCUSSION

Micrographic images

The images produced by SEM of the untreated and treated samples are shown in Fig. 1. The images clearly showed the changes in fiber surface due to the treatments as compared to the untreated control. The bending deformation of silk without breakage was high in silk that justified the intactness of the Muga fibers in all treated and untreated samples [Fig. 1] (Shah *et al.,* 2014). The fine external structure with longitudinal stripe-like structures of untreated (control) Muga in Fig. 1a indicated refined natural features like luster and colour (Dutta *et al.,* 2013) which was also observed in WW (Fig. 1b), KW (Fig. 1c) and LW (Fig. 1d). In case of SEW (Fig. 1e), RS (Fig. 1f), DS (Fig. 1g), RFS (Fig. 1h), CFC (Fig. 1i), EW (Fig. 1j) and VOX (Fig. 1k). Distinct damages in the form of fibrillation, peeling, scarring, and crumpling of fibers were observed. The micro-cracks observed in commercial washing product led to the roughness of Muga fabric that decreased the tensile property eventually. The core of silk fibers was highly crystalline which were insoluble in water but served as an excellent absorber that takes in the water leading to wet abrasion and swelling of the fiber (Mishra, 2000). Due to this the inter-microfibril interactions weakened that rendered the fiber susceptible to fibrillations (Ma *et al.,* 2014). The solutions made from commercial washing agents were more prone to get absorbed by fiber and the minute components found in them were assumed to have a friction effect and were damages which can be seen in SEM images.

Muga colour analysis

The Red Green Blue (RGB) colour model is employed to detect and portray the images in electronic systems as well as in conventional photographs (Villafuerte and Negro, 1998). The calculated Hue,

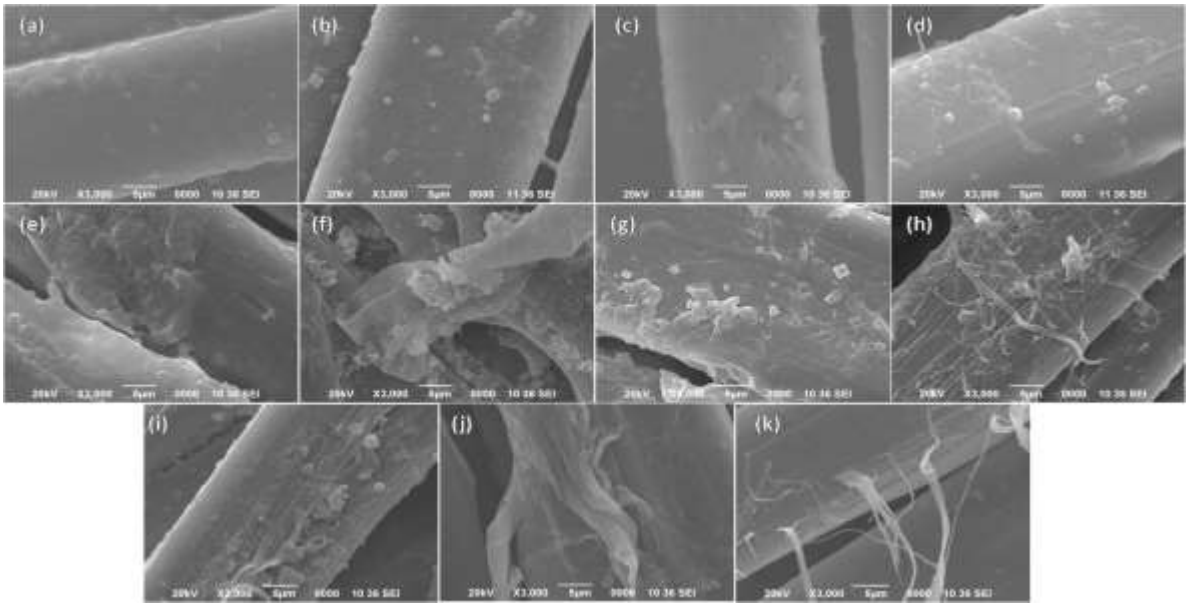

Fig. 1: SEM images of Muga silk at 3000X magnification of a) Raw (control), b) Water washed, c) *Kolakhar* washed, d) *Citrus* lemon washed, e) Fabric conditioner washed, f) Indigenous laundry soap washed, g) liquid detergent for woolen clothes washed, h) Fabric stiffener washed, i) Laundry soap washed, j) Detergent washed, and k) Fabric whitener washed

saturation and luminosity (HSL) values of scanned images are given in Table 1 and Fig. 2. The matching of deduced patches from the calculated values confirmed the correctness of equations. The maximum deviation for luminosity and saturation were observed for VOX (74.66%) whereas minimum deviations were seen for luminosity in *Citrus* (64.53%) treated followed by *Kolakhar* treated (66.36%). Colour code generated from HEX angle showed the closest color being *Citrus*-treated (#c79f82) silk with raw Muga (#c59d84). δE values revealed that the colour change was least in commercial fabric conditioners (0.85), followed by citrus-treated (1.06) and *Kolakhar* treated (1.13) Muga. The highest changes were noticed can in VOX (5.46) treated Muga (Fig. 2). The overall colour analysis suggested that CFC protects colour loss of Muga fabric followed by LW and KW.

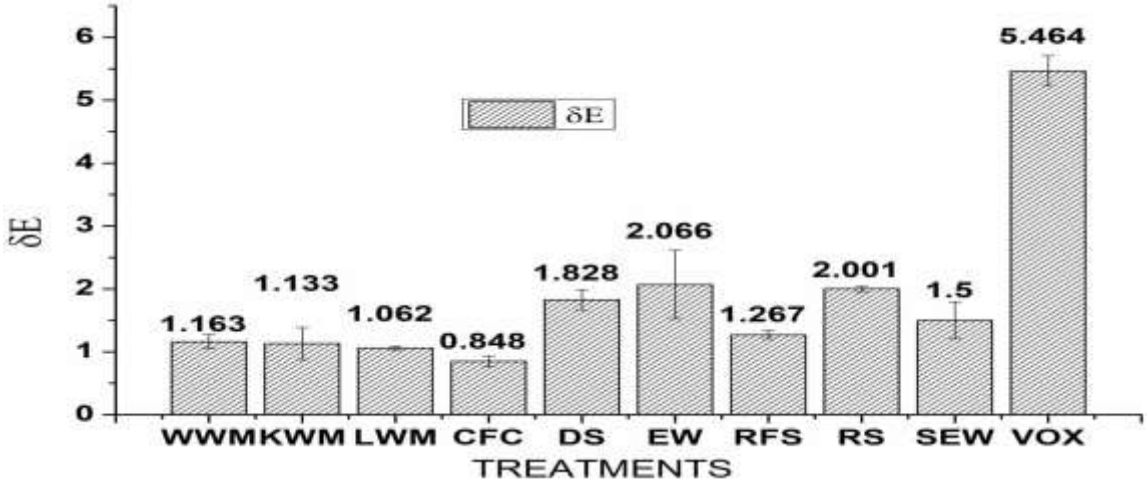

Fig. 2: Colour difference of treated Muga silk against raw Muga silk. WWM = Water washed, KWM = *Kolakhar* washed, LW = *Citrus* washed, CFC = Fabric conditioner washed, DS = Indigenous laundry soap washed, EW = Liquid detergent woolen clothes washed, RFS = Fabric stiffener washed, RS = Laundry soap washed, SEW = Detergent washed and VOX = Fabric Whitener washed, and δE = colour difference

Table 1: Luminosity, saturation, hue angle, fabric colour, generated colour code of the fabric colour and associated colour with the code of control (raw) and treated Muga silk

Treatments*	Luminosity (%)	Saturation (%)	Hue angle (°)	Fabric colour	Colour code	Associated colour (Hex)
RAW (control)	64.65	36.16	23.21		#c59d84	
WWM	66.68	37.69	25.23		#caa58a	
KWM	66.36	35.06	25.50		#c7a58b	
LW	64.53	37.77	25.02		#c79f82	
CFC	67.07	40.80	24.75		#cda589	
DS	70.00	37.93	24.01		#cfad96	
EW	71.14	41.50	24.93		#d4b097	
RFS	69.84	41.10	24.59		#d2ac93	
RS	66.41	34.71	26.36		#c7a68c	
SEW	68.35	41.49	26.28		#d0aa8c	
VOX	74.66	47.69	30.57		#ddbfa0	

*WWM = Water washed, KWM = *Kolakhar* washed, LW = *Citrus* washed, CFC = Fabric conditioner washed, DS = Indigenous laundry soap washed, EW = Liquid detergent woolen clothes washed, RFS = Fabric stiffener washed, RS = Laundry soap washed, SEW = Detergent washed, and VOX = Fabric whitener washed.

FTIR analysis

The FTIR results of treated- and untreated-Muga silk are depicted in Fig. 3. The changes in FTIR data were noted in treated Muga silk as compared to untreated Muga silk. The characteristics of amide absorption bands of protein amide I (1600-1800 cm^{-1}), amide II (1470-1570 cm^{-1}), and amide III (1259-1350 cm^{-1}) were found in FTIR results (Barth, 2007). Heterocyclic amine, NH stretch (3490-3430 cm^{-1}) that was present in RM (3435 cm^{-1}) and WWM (3433 cm^{-1}) samples got shifted to 3412-3427 cm^{-1} in all the rest treatments that is linked only with hydroxy-group, H bonded OH stretch (3570-3200 cm^{-1}) (Coates, 2006). SEW (2980 cm^{-1}) and VOX (2981cm^{-1}) had peak shifts not attributed to any bond in particular, which was present in RM (2956 cm^{-1}) and rest treated samples (2965-2970 cm^{-1}) linked to methyl C-H asymmetric stretch (2970-2950 cm^{-1}). Aromatic ring stretch (1510-1497 cm^{-1}) found in untreated raw Muga (1498 cm^{-1}) and water washed Muga (1504 cm^{-1}) was missing in all other treatments. Formation of new peak, except for water wash Muga, was seen in all the treatments at KWM (1515 cm^{-1}), CWM (1516 cm^{-1}), CFC (1514 cm^{-1}), DS (1513 cm^{-1}), EW (1513 cm^{-1}), RFS (1514 cm^{-1}), RS (1518 cm^{-1}), SEW (1514 cm^{-1}) and VOX (1516 cm^{-1}) which may be attributed to the stretching vibration of C-N group and deformation vibration of N-H groups as well as to the formation of β–sheets in case of secondary structures (Hay and Myneni, 2007; Masaki *et al.*, 2017). There was formation of a small peak linked to β-turn conformation of hairpin-folded antiparallel

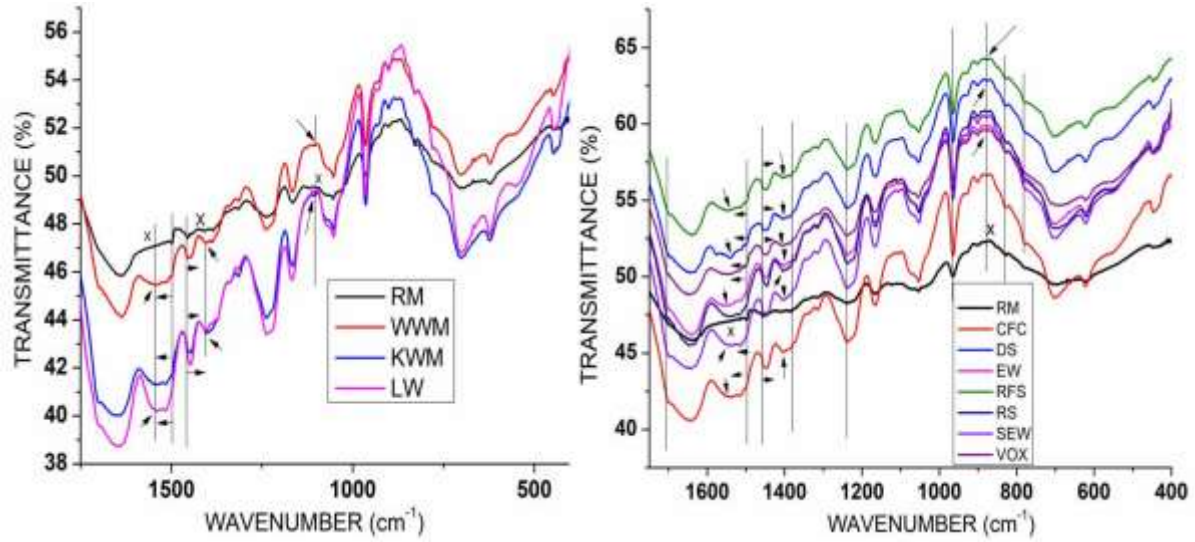

Fig. 3: FTIR graph of a) raw and traditional treated Muga silk; and b) raw and commercial agent treated Muga silk. RM = Raw (control), WWM = Water washed, KWM = *Kolakhar* washed, LW = *Citrus* washed, CFC = Fabric conditioner washed, DS = Indigenous laundry soap washed, EW = Liquid detergent woolen clothes washed, RFS = Fabric stiffener washed, RS = Laundry soap washed, SEW = Detergent washed, and VOX = Fabric whitener washed.

β-sheet structure in all the test samples (1698-1703 cm^{-1}), except the untreated raw sample (Asakura, 2021). The peaks from 1638-1646 cm^{-1} are associated with random coils and extended chains which can be seen in case of RM (1638 cm^{-1}), WW(1637 cm^{-1}), KWM (1642 cm^{-1}), LWM (1642 cm^{-1}), CFC (1641 cm^{-1}), DS (1641 cm^{-1}), EW (1640 cm^{-1}), RFS (1641 cm^{-1}), and RS (1643 cm^{-1}), and SEW (1643 cm^{-1}); however, VOX (1648 cm^{-1}), was linked only with random coils (1647-1655 cm^{-1}) (Tretinnikov and Tamada, 2001; Taddei and Monti, 2005; Teramoto and Miyazawa, 2005). No non-hydrogen bonded water was observed as the peaks associated with its symmetric and anti-symmetric stretching vibration were found between 3630-3700 cm^{-1}. The peaks between 3600-2900 cm^{-1} are linked with a bonded OH group which was observed in all the samples (Gellman *et al.*, 1991).

Conclusion: The high magnification images of SEM confirm the damages caused by commercial washing agents. The commercial fabric conditioner showed excellent resistance to colour change, followed by lemon juice wash and *Kolakhar* wash Muga. The FTIR results suggested that all the treatments have equal effect on the chemical nature of fabric. It may be concluded that traditional washing agents have a fairly good effect on Muga fabric in retaining the original colour without any damage to its fibers. Formation of washing agents with proper optimization of the traditional washing formulation can be a good approach as a Muga specific washing agent.

Conflict of interest: The authors declare that there is no conflict of interest regarding this publication.

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