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RESEARCH ARTICLE

SURFACE CHARACTERIZATION AND PROPERTIES OF RAW AND DEGUMMED (*BOMBYX MORI*) SILK FIBROIN FIBER TOWARD HIGH PERFORMANCE APPLICATIONS OF "KISSWA AL-KABBA"

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ABSTRACT

The purpose of this paper is the early evaluation of pre-treatment processes of *Bombyx mori* silk fibroin used to produce "Kisswa Al-Kabba" in order to determine the degree of efficiency and the differences among the most commonly-used degumming processes. Silk fibroin from the *Bombyx mori* silkworm was degummed through reproducibility processes in a customized method including natural soap degumming (tap water and deionized water) along with laboratory scale and factory scale machines. The results revealed that the maximum average value of sericin content was found to be 28.1669% (for tap water) and 28.5715% (for deionized water), indicating removal of all or most of the sericin. After neutral soap degumming, the initial modulus decreased from 108.7 gf/den to 55.1 gf/den, while the breaking tenacity decreased from 4.0270 gf/den to 2.7185 gf/den, indicating partial harmful damages of silk fibers molecules after neutral soap degumming method. These values decreased after degumming treatment, suggesting that the silk fibers become soft and stretchable. There was not much influence on elongation after degumming for different scale conditions. The tensile properties of the silk fibers after degumming laboratory scale machine (tap water) treatment was the worst. The degumming process or boiling process of silk fibers on a laboratory scale with tap water results in a serious decrease in the tensile properties. The surface morphology of the silk fiber shows that there was no important difference among the degumming approaches used in this study. The molecular conformation estimated by FT-IR stayed unchanged regardless of the degumming processes.

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INTRODUCTION

The "Al-Kaaba" is a huge stone structure at the core of the Holy mosque, the City of Makkah, KSA. It constitutes a single large room with a marble floor that lies at the core of the Holy Mosque. It stands about 60 feet (18.288 m) in high and each side is about 60 feet (18.288 m) in length. The door of the "Al-Kaaba" is approximately 7 feet (2.1336 m) from about the ground and is located at the South-East wall. Its four outer walls are covered with a black curtain made of silk fabric, called the "Kisswa" that wrapped around the "Al-Kaaba", reaching the ground and fixed to the base with gold rings. The cover is made of 670 kg of pure silk fiber and is 658 m² long. The embroidery band is made up of 150 kg of gold threads and 47 pieces of cloth, and each part is 14 m long and 1.01 m wide. The silk fabric is used as material for making up the covering "Kisswa Al-Kabba" as it is universally acclaimed for

possessing the most desirable properties of textile silk fiber, such as, fineness, elasticity, strength, softness, dyeability, smooth feeling, luster, grace, elegance, flexibility and high rating (Khan et al. 2010, Sealy 2015, T-T et al. 2013). *Bombyx mori* (*B. mori*) silk has been used and regarded as a high-status luxury textile fibre for over 5000 years. Silk fibroin (SF), known as the "queen of all fabrics", was first revealed in China (Encyclopedia 2000, Li et al. 2012, Naskar et al. 2014). It is used as a natural bio-polymer of the protein fibres produced by the silkworm (*B. mori*), see Fig. 1(a) and Fig. 1(b). The SF is usually stable at temperatures up to 140°C and the thermal decomposition temperature is over 1500°C. The densities of silk fibres range between 1320 and 1400 kg/m³ with sericin and range between 1300 and 1380 kg/m³ without sericin. It is still considered as a premier textile material in the world today due to its high tensile strength of about 0.5 GPa, breaking elongation of 15%, breaking energy (toughness) of 62.104 J kg⁻¹, lustre and ability to bind chemical dyes.

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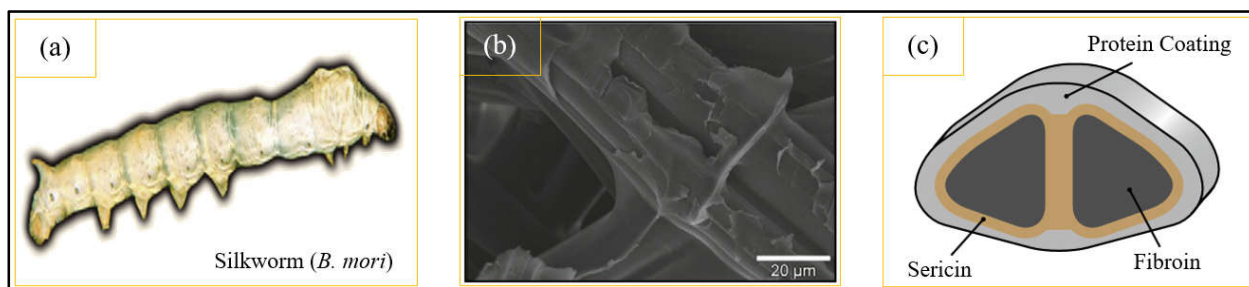


Fig. 1. (a) photograph of a *Bombyx mori* silkworm, (b) SEM image of *B. mori* silkworm cocoon fibers with two brins of fibroin and sericin and (c) schematic of structure of silkworm silk fiber with two brins surrounded by “sericin” and “protein” layers to protect against predators and microbes, adapted from (Sealy 2015)



Fig. 2. from “mori Eggs” to “Kisswa Al-Kabba”

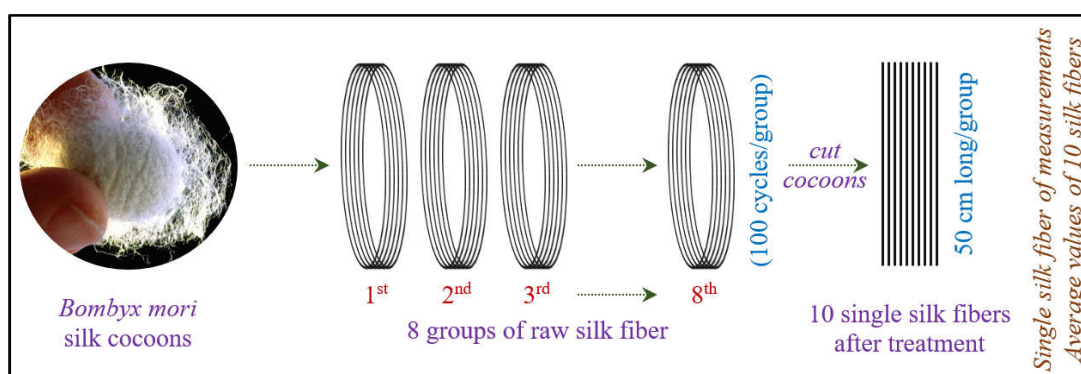


Fig. 3. sampling method for the measurement of single silk fiber

Silk fibres are remarkable materials displaying unusual mechanical properties, such as being strong, extensible and mechanically compressible (Babu 2015). In addition to their remarkable mechanical properties, silk fibers offer a wide range of valuable features, including, controllable biodegradability, bio-compatibility, morphological flexibility and environmental stability (Hu and Kaplan 2011). Generally, the chemical composition of native *B. mori* silk fibres mainly comprises protein polymer that consists of two totally different families, namely fibroin protein (~65-75%)

(Wang et al. 2015), coated with (~20-30%) sericin protein (Zhang 2002) and the remainder (~5%) is composed of pigments, sugars, wax and other associated impurities in the silk fibers (Liu et al. 2015, T-T et al. 2013, Wang et al. 2015). The silkworm *B. mori* cocoon is composed of fibroin, a typical crystalline protein fiber, surrounded by sericin, which is a family of glue-like proteins, which helps in maintaining and reinforcing the cocoon structure, see Fig. 1(c) (Sealy 2015), and also helps in cementing one fibroin to the next fibroin fiber, resulting in a single thread with a diameter of 10-25 μm

(Li et al. 2012). Sericin is a glue-like protein that contained 18 different kinds of hydrophilic polar amino acids; most are large molecular side chain, polar and hydrophilic amino acid residues and, thus, sericin has the characteristics of glycerol-like UV irradiation protection and absorption or release of moisture. The glue-like protein sericin is amorphous produced and excreted in the middle silk gland cells and can have noticeably different solubility degrees. The sericin layer directly surrounding the fibroin fiber is poorly soluble in water and is excreted in the posterior of the middle gland of the silkworm. The sericin surrounding this inner layer is water-soluble and is excreted in the anterior of the middle gland (T-T et al. 2013, Y-J and Y-Q 2011).

Sericin is a hydrophilic protein that can be swelled and dissolved in hot water with less hydrolysis, particularly in alkaline hot water. The Ancient Chinese used hot water to boil the *B. mori* silkworm cocoons and then wound the fiber silk onto a reel and dried it, which is like spinning on a large scale (Sun et al. 2012). The use of alkalis as degumming agents is still vital for current silk production processes (Fabiani et al. 1996, Sun et al. 2012, Vepari and Kaplan 2007). The general production process of silk, from raw cocoon to finished textile articles, involves cooking, reeling, degumming, dyeing, weaving, and finishing processes. Fig. 2 illustrates the story of production line from “*mori Eggs*” to “*Kisswa Al-kabba*”. Besides, the growing of mulberry trees (mulberry culture), the production of silk can be viewed as a culmination of several separate stages: (1) *time to hatch*: caterpillars, the larval stage in the life cycle of the silk moth, hatch from their *mori* eggs. (2) *mulberry meal*: workers, who work in sericulture, feed mulberry leaves to silk moth caterpillars. (3) *spin cycle*: silk moth caterpillars spin cocoons between the woven needs of flat frames.

The insects spend days wrapping themselves in the protective filament. (4) *harvest time*: millions of cocoons are sent to workers who will turn the filaments into silk strands. (5) *final product*: filaments from roughly five to eight cocoons are wound together to create a single raw silk filament thread. From the raw material of cocoons, the production of silk fibers involves a simple thermochemical process to remove or separate the sericin protein from the natural fibroin. This process is known as degumming which is a very important process in ensuring the proper dyeing and finishing of silk goods (Sargunamani and Selvakumar 2006). The procedures used for silk degumming in the laboratory are based entirely upon its solubility in hot water.

It is demonstrated that the removal of sericin involves treatment with a diluted solution of sodium carbonate (Na_2CO_3) (Vinson Joe A. 2000b), or highly concentrated urea (Rietveld and Wiseman 2003), or pH neutral soap (Marseilles soap) (Pastore and Fratellone). Lately, detergents have replaced soap because of its incapability to compensate for the acidity of sericin hydrolysis products, which tend to accumulate in the bath and limit the use of degumming baths for weekly cycles. The sericin removal mechanism can be described as a combination of hydrolysis and dispersion/solubilization, which is a typical process caused by the strong alkaline compounds used during the degumming process. Therefore, degumming process parameters, such as time, pH, temperature and alkalinity must be addressed carefully, ensuing only in sericin elimination without damaging fibroin by setting off hydrolytic degradation.

EXPERIMENTAL PROCEDURE

MATERIALS

More than 300 samples (raw silk fibers, from “*Kisswa Al-Kabba Factory*”, Makkah, KSA, 2016) were used in this study with identical dimensions of 100 m length \times 0.1 mm diameter. All raw silk fibers were collected together after reeling of cocoon filaments of a commercial silkworm variety of *B. mori* silk cocoons, see Fig. 3. To remove sericin, raw silk fiber was degummed through a customized method for “*Kisswa Al-Kabba Factory*” neutral soap degumming (NSD). The chemical used, which was a commercial grade agent, was of laboratory reagent grade and supplied by DyStar Singapore Pte Ltd.

Degumming of Raw Silk Fiber

Silk fibers processing from *B. mori* silk cocoons to finish clothing articles (as a final product) comprises a series of stages which include (1) reeling, (2) weaving, (3) degumming, (4) dyeing and (5) finishing. Usually, degumming is a procedure involving removing sericin coating, which gives a dull appearance, harsh and stiff feeling to the silk fibers' surface, and hides the typical soft handling, whiteness and shiny aspect of a silk fiber's surface. Also, it is the main cause of adverse selection problems with hyper-sensitivity and biocompatibility to silk fibers. In general, traditional industrial degumming methods of raw silk fibers, including alkali degumming (Rajkhowa et al. 2011), use of soap-alkaline degumming solution (Kato 1968), synthetic detergents (Bianchi and Colonna 1992), enzymatic degumming (Mokhtar Arami et al. 2007), organic acid organic acids such as fiber silk degumming solvents (Fabiani et al. 1996) and the low-temperature plasma method (Long et al. 2008). All degumming of raw silk fibers was carried out using customized neutral soap degumming as used in the “*Kisswa Al-kabba*” factory. Three different scale machine environments were used during the investigation namely (1) laboratory scale machine, (2) factory scale small machine and (3) factory scale large machine. Testing took place in an air-conditioned room, with an ambient temperature of $20 \pm 2^\circ\text{C}$ and a relative humidity of greater than $40 \pm 5\%$ RH. To achieve this, all raw silk fibers were degummed through the following procedure. All degumming processes were carried out on the laboratory scale and factory scale machines using an NSD customized method. Silk fiber hanks were prepared and weighted “*before*” degumming in grams. They were degummed in a controlled temperature according to the following stages (see, Fig. 4).

After measuring the initial pH of the degumming bath, silk fibers were treated at 90°C for one and a half hours with a solution consisting of tap water (0.320 L) used in each of the eight tanks and the pH of the tap water was measured, soda ash, Na_2CO_3 , which is the water-soluble sodium salt of carbonic acid (0.64 g), sera wash (17 drops, 0.48 g), Calonate T (0.64 g), olive soap oil (2.24 g). The higher temperature (90°C) and pH around 10.5 in the presence of harsh chemical materials in the treatment bath imposed a noticeably unnatural environment on the silk fiber and therefore caused partial degradation of fibroin. This degradation caused a loss of aesthetic quality in terms of whiteness and physical properties. All silk fibers were cooled down for 10 minutes and the pH of the degumming bath after the process was measured. The solution was then emptied off and the remaining fibroin fibres

were washed with normal temperature tap water for 15 minutes twice to remove soap and sericin. Then, all silk fibers immersed in 0.25 g of acetic acid and 440 L of tap water for 15 minutes at room temperature in order to reach complete neutralization, see Fig. 5. Finally, after the degumming procedure, all silk fibers were dried immediately at 80°C for 1 hour and then kept for another 48 hours in the same environment and for the same length of time that would be used for the testing to allow the single silk fiber surface to equilibrate with the environmental conditions. Note that the same procedure was repeated with the deionized water instead of tap water. The procedure that is described above was judged to be adequate at this stage as they used this method in "Kiswa Al-kabba Factory". After that, all silk fibers were weighted in order to calculate the quantity of removed sericin as stated in Equation (1) and the procedure for calculating the degumming rate was done in triplicate.

$$S(\%) = \frac{B-A}{B} \times 100 \quad (1)$$

where, S , is the amount of removed sericin, B , is the mass of dried silk fibre "before" the degumming and, A , is the mass of dried silk fibre "after" the degumming. After the ventilation drying process, the outer surface and cross-section of degraded silk fibers were examined and observed by scanning electronic microscopy (SEM).

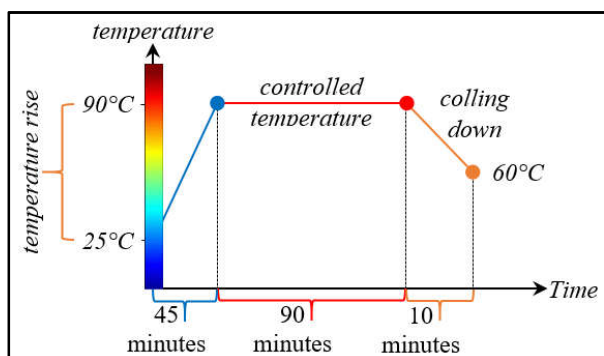


Fig. 4. Stages of temperature during degumming

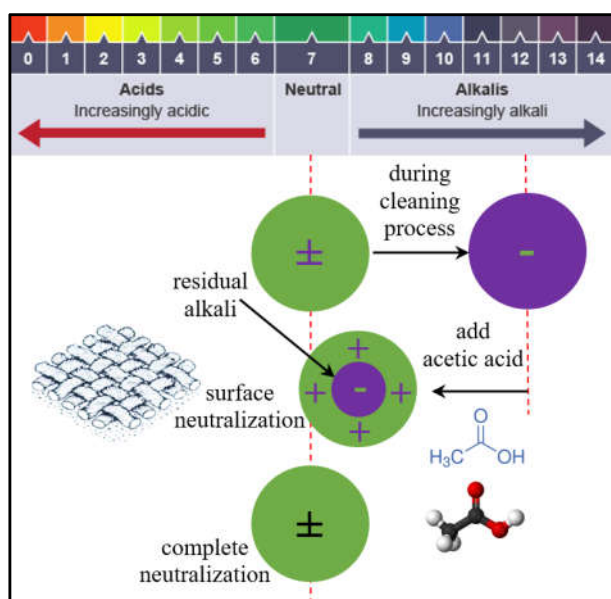


Fig.5. Schematic diagram of pH scale and chemical process of the silk fiber surface

Scanning Electron Microscopy (SEM)

The surface texture of silk fibres before and after the degumming procedure was examined by SEM of at least ten arbitrarily located scan areas on the surface of the silk fiber (scan size: 10 $\mu\text{m} \times 10 \mu\text{m}$). The SEM machine was performed with an LEO 440i (LEO Elektronen- mikroskopie GmbH, Oberkochen, Germany) also with the accelerating voltage of 20 kV. Observation was carried out with high magnifications of 1000x and 5000x and images were registered by digital image recording system Semafore. More details of the SEM machine procedure have been reported elsewhere (Alsoufi 2016, Alsoufi et al. 2016, Bawazeer et al. 2016).

Fourier Transform InfraRed (FTIR)

The FTIR spectrum of the silk fiber was recorded using a PerkinElmer Spectrum 100 FTIR spectrometer (PerkinElmer, Norwalk, CT, USA) equipped with a MIR TGS detector system. The Fourier transform infra-red spectra were recorded in the between 4000 to 400 cm^{-1} regions at an ambient temperature of 20 \pm 1°C and a relative humidity of 40 \pm 5% RH. All the data analysis was then collected automatically using appropriate software (Spectrum 100 software). Each silk fiber before and after the degumming process was scanned as three different replicates under the similar setting condition, all of which gave identical spectra. The mean IR spectra of these three replicates' data were then used in statistical analysis. More details of the FTIR spectrum procedure have been reported elsewhere (Bawazeer et al. 2016).

RESULTS AND DISCUSSION

Silk Degumming Rates

All samples were weighing before and after degumming. The quantity of removed sericin, expressed in % was obtained using the following equation (1) and all the results were stated in Fig. 6 for both tap water and deionized water. The maximum average value of sericin content was found to be 28.1669% (for tap water) and 28.5715% (for deionized water), indicating removal of all or most of the sericin. The pH values of baths measured before and after degumming processes, regardless of process type or stage did not exhibit significant change. The pH value of tap water and deionized water before the process was around 6.5, the pH value of initial degumming bath was around 10.5 and finally the pH value of degumming bath after process ended was around 10.3 with a 95% level of confidence.

The surface properties of silk fiber obtained by different methods were observed in a scanning electronic microscope at magnification of 150x, 750x and 1000x. Fig. 7 shows the SEM image of raw silk fiber with this magnification factor. The SEM observations show that the surface of control silk fiber image is rough due to its fully-coated with sericin appearance as partially non-uniform coating on the silk fibers and many granular deposits are still visible in the interstices between cocoon filaments. These single filaments of silk fibroin are around 8 - 10 μm in diameter but they are not a standard cylindrical shape and their morphology is irregular. On the other hand, Fig. 8 shows the SEM image of silk fiber after being degummed with tap water and deionized water methods. The degummed silk surfaces were both smooth with fine longitudinal striation attributable to the degumming process.

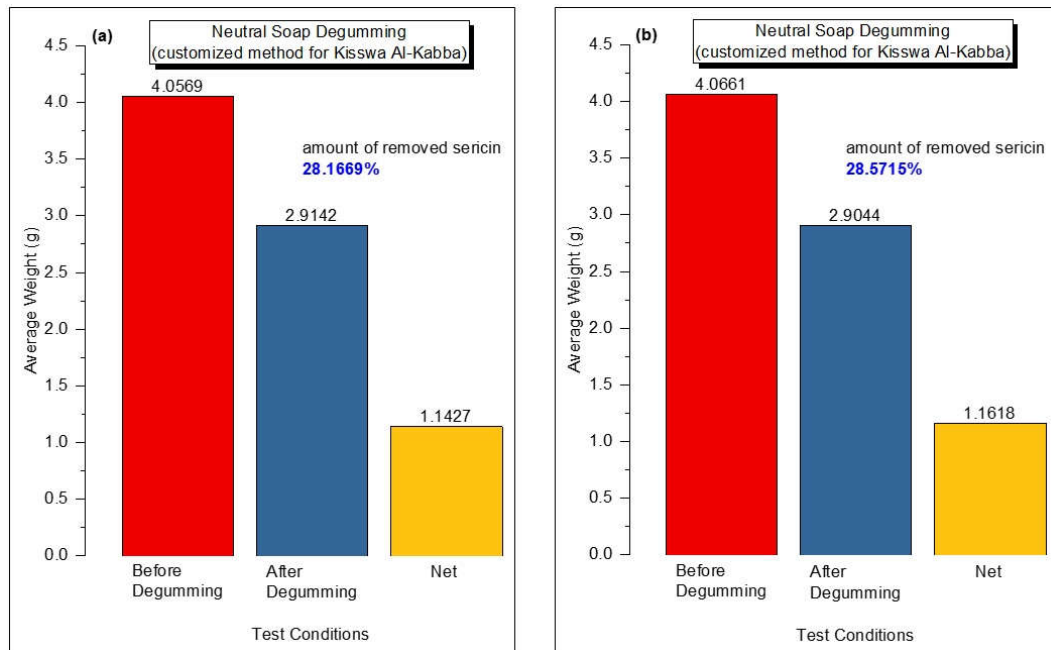


Fig. 6. Average weight before and after NSD and sericin content using (a) tap water and (b) deionized water

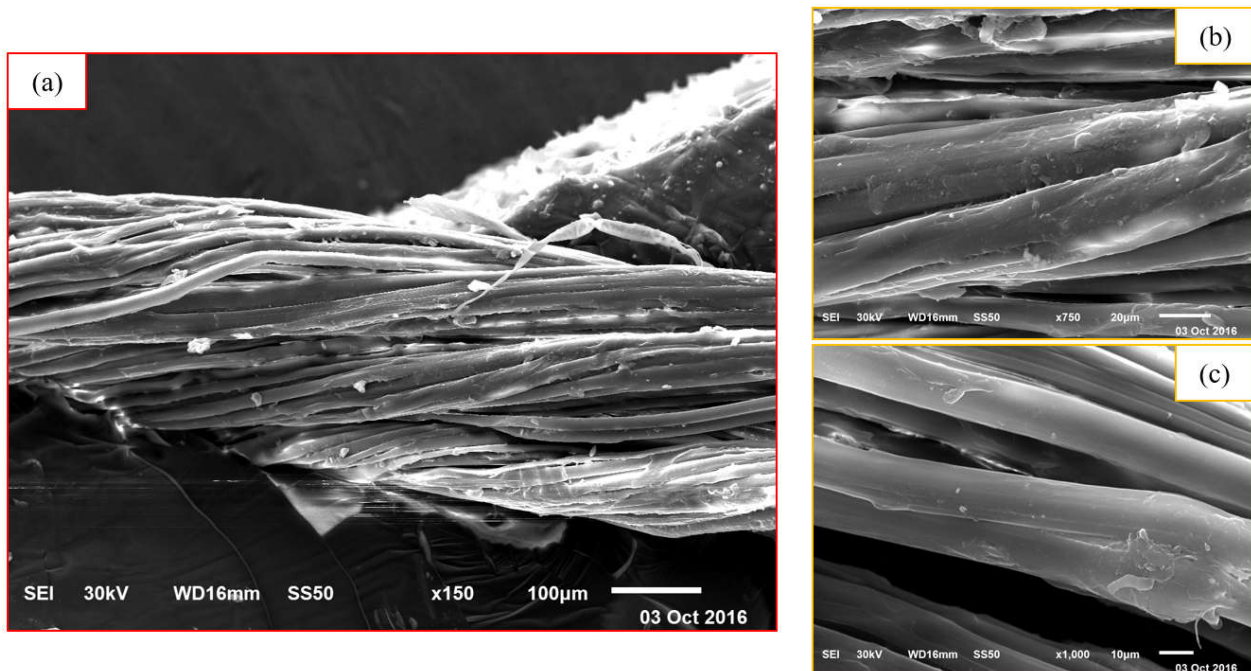


Fig. 7. SEM image of raw silk with magnification factor of (a) 150x (b) 750x and (c) 1000x

The SEM observations also showed that there is not enough evident difference among the products of the methods used in this study.

Mechanical Properties of Silk Fibers

Initial modulus, breaking force, breaking tenacity and elongation properties are the most vital parameters for assessing the reparability and producibility performance of silk fibers for suitable industrial applications. The mechanical properties were measured with a universal tensile testing machine (Tinius Olsen, Model H5KT), Japan using a standard technique at a gauge extension range of 50 mm, strain rate of 100% min⁻¹, load range of 6.0 kgf and a speed of 100 mm/min. Silk fiber with uniform thickness throughout, was held between two mechanical clamps positioned at a distance of 50 mm between them.

Thick paper was attached on the surface of the mechanical clamp to avoid the silk fiber from being cut by the grooves of the mechanical clamp. In order to reduce possible error in the measurements of the mechanical properties of the silk fibers, each value reported is the average of 10 measurements. The measurements were carried out pulling the top clamp at a rate of 100 mm/min. The initial modulus, breaking force, breaking tenacity and elongation properties of silk fibers degummed with different scale conditions along with the raw silk fibers at break was recorded and presented in Fig. 9. After neutral soap degumming (NSD), the initial modulus decreased from 108.7 gf/den to 55.1 gf/den, while the breaking tenacity decreased from 4.0270 gf/den to 2.7185 gf/den, indicating partial harmful damage to silk fiber molecules after NSD. These values decreased after degumming treatment, suggesting that the silk fibers become soft and stretchable.

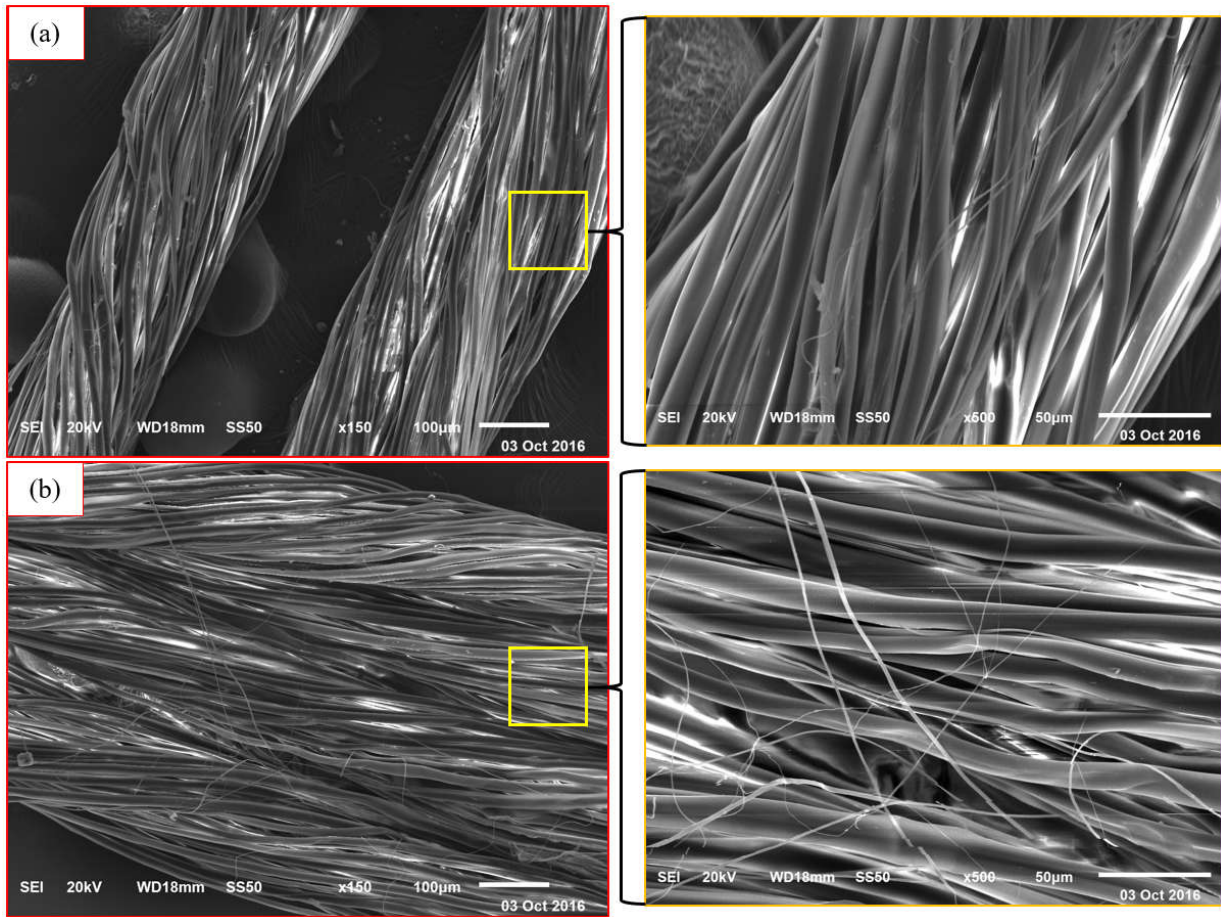


Fig. 8. SEM image of silk fiber after degummed with (a) tap water and (b) deionized water

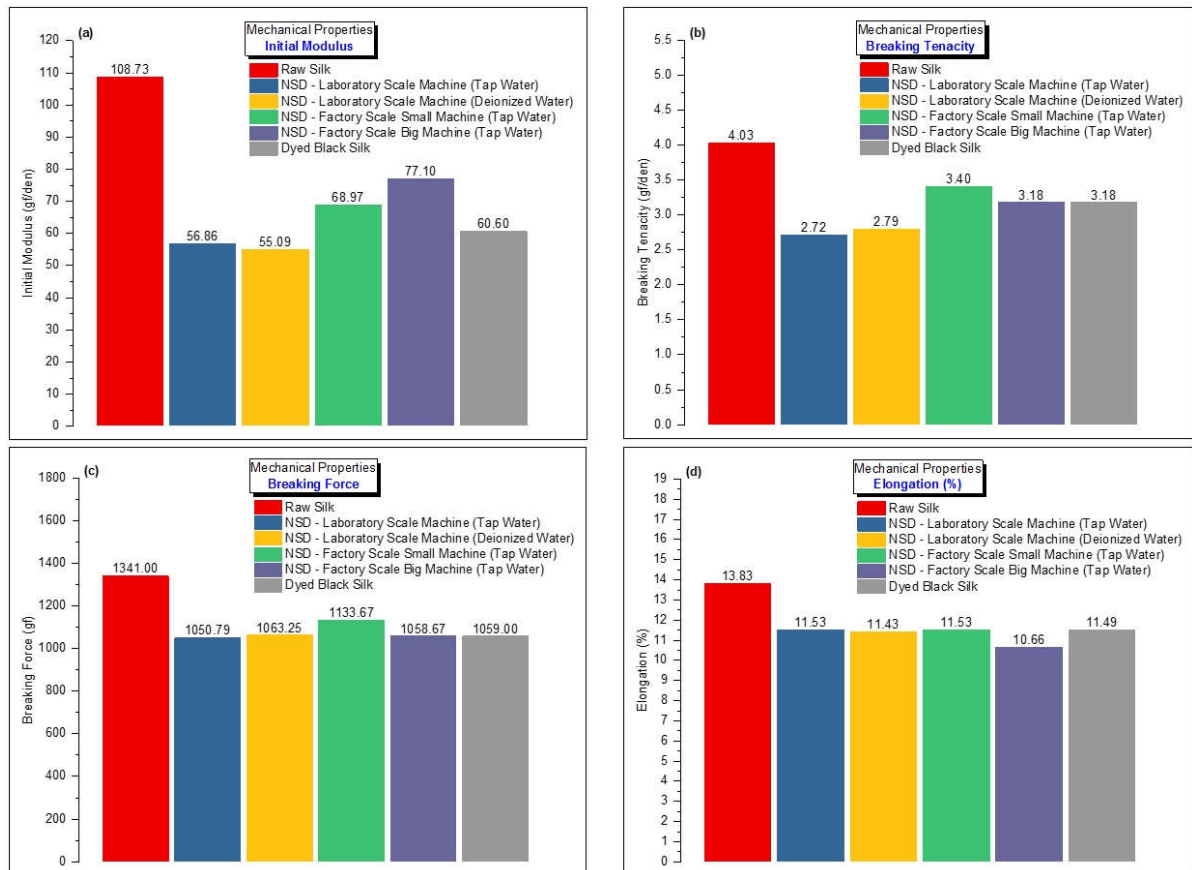


Fig. 9. Mechanical properties of silk fibers degummed with different scale conditions along with the raw silk fibers and dyed black silk

Table 3. Silk fibers categories and its mechanical properties, adapted from (Gupta and Kothari, 1997)

Categories	Tenacity (gf den ⁻¹)	Elongation (%)	Modulus of elasticity (gf den ⁻¹)
Silk fibers with <i>average</i> mechanical properties	3 - 5	35	30 - 60
Silk fibers with <i>above average</i> mechanical properties	7 - 8	8 - 15	50 - 80
Silk fibers with <i>superior</i> mechanical properties	8 - 20	5 - 15	80 - 250
Silk fibers with <i>outstanding</i> mechanical properties	15 - 50	0.5 - 5	250 - 4000

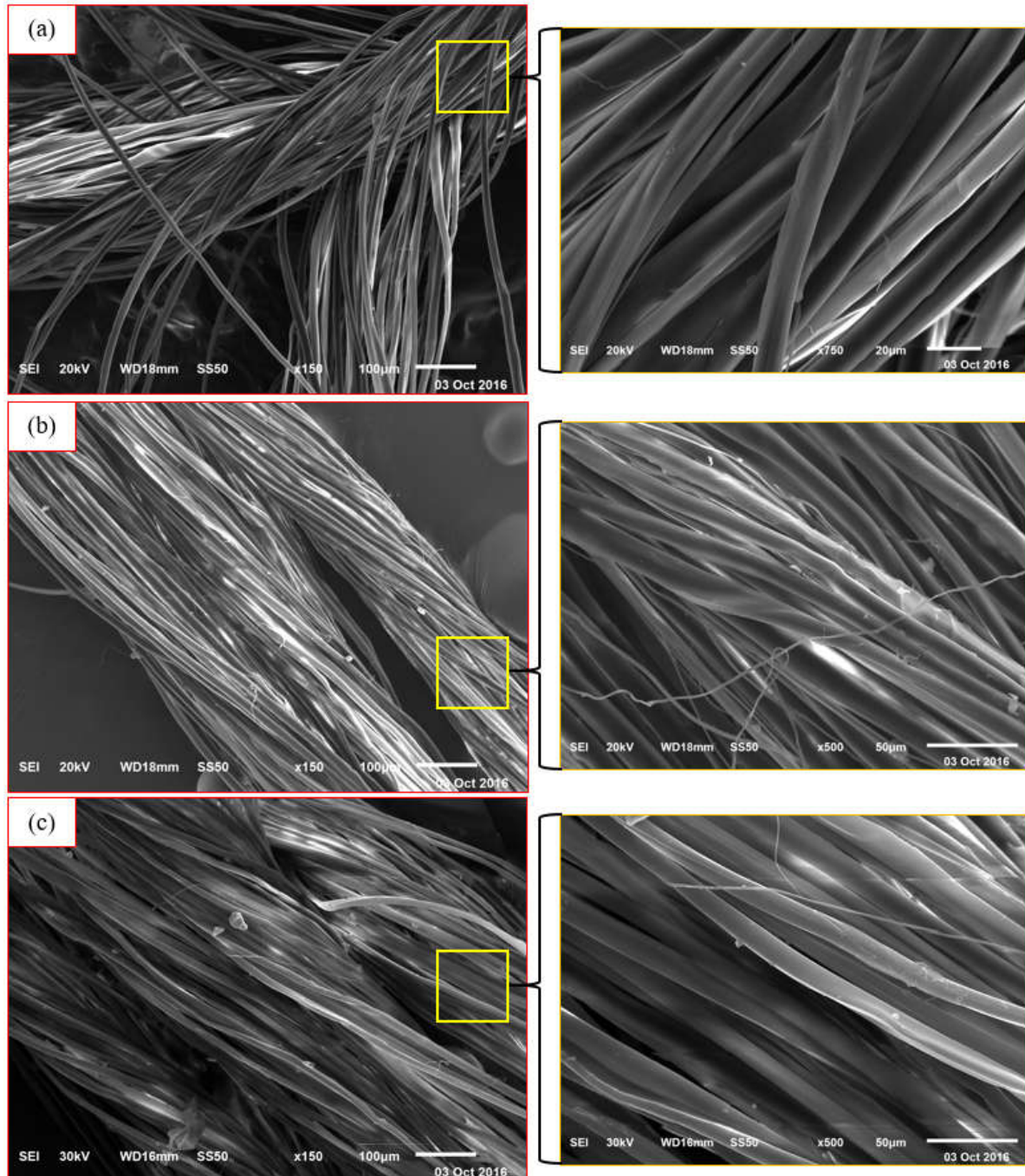


Fig. 10. SEM images of silk fibroin degummed from different degumming methods (a) factory scale small machine (tap water), (b) factory scale big machine (tap water) and (c) dyed black silk

There was not so much influence on elongation after NSD for different scale conditions. In general, the tensile properties of the silk fibers after NSD laboratory scale machine (tap water) treatment was the worst ($P < 0.01$). The degumming process or boiling process of silk fibers on a laboratory scale with tap water results in a serious decrease in the tensile properties. To facilitate this, the manufactured silk fibers need to be classified appropriately.

Table 3 illustrates the wide basis of mechanical properties' classifications which are in four categories of silk fibers. Based on the mechanical properties available in (Gupta and Kothari 1997), the results indicated that the tenacity of the silk fiber after treatment was in the region of average mechanical properties and possessed completely reversible elongation up to 5% strain. The elongation of the silk fiber after treatment in the region of above average mechanical properties and has a

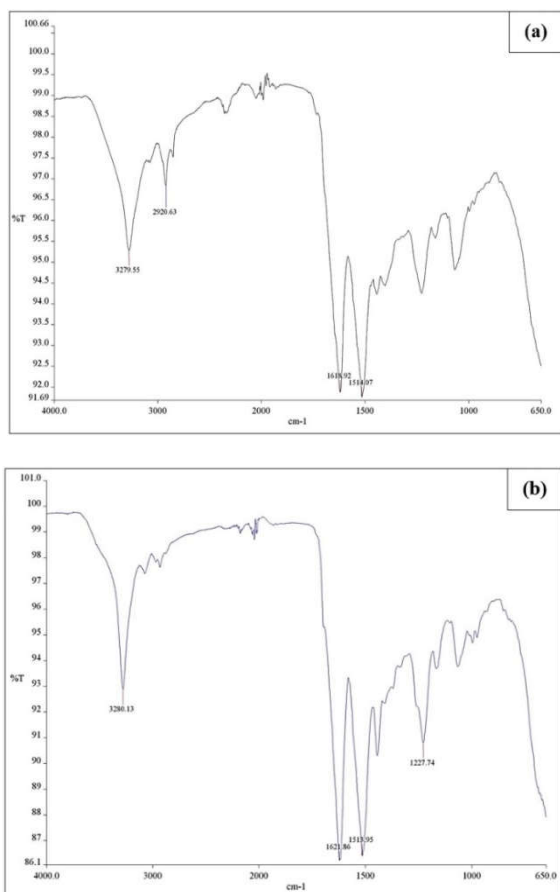


Fig. 11. IR spectrum of (a) raw and (b) degummed silk fibers

high degree of toughness. The modulus of elasticity of the silk fiber after degumming was in the region of superior mechanical properties. Bear in mind that the differences in the silk fiber properties were attributed to the dissimilarities in the amount of amino acids, environmental habitats of the insects, physical structure and the fact that different insect species have different properties of silk fibers.

Morphology of Silk Fibers

The silk fibers before and after NSD were observed by using a scanning electronic microscope (SEM). The SEM provides a wealth of direct information about morphology features of silk fiber with nano-meter resolution based on a significantly innovative principle that differs from conventional spectroscopic and microscopic methods. Fig. 10 shows the SEM images of the degummed silk fibroin from different degumming methods for a factory scale small machine (tap water), a factory scale big machine (tap water) and dyed black silk. The SEM observations show that, there was no important difference among the degumming approaches used in this study. However, the factory scale small machine degummed silk surface seemed to have a rough surface with remaining sericin residue. This indicates that the sericin in the silk fiber was not totally removed by the degumming method, which is equivalent to a lower degumming rate. What is more, the micrographs of samples degummed show the damage done to the degummed fibres such as uneven fibre surface, with thin visible fibrils on fibre surface. Submitting fibres to the relatively high degumming temperature of 90 °C for 90 minutes and the alkaline conditions under which the fibres were degummed caused visible fibrillation and surface rupture (Vinson J. A.), as seen in Fig. 10.

Damage of degummed fibres was enhanced in a subsequent dyeing fibroin as shown in Figure 10(c). Undesirable fibre degradation appears as a loss of aesthetic and physical properties, for instance, surface fibrillation, drop of tensile strength, dull appearance, poor handle, as well as, uneven dyestuff absorption during dyeing and printing processes (Whitehouse).

FT-IR Data Analysis

The effect of degumming processes on the molecular characteristics of silk fiber were investigated by FT-IR. The IR spectrum of raw and degummed silk fibers presented in Fig. 11(a) and Fig. 11(b) shows that the peaks from 1000 to 1800 are due to fingerprint regions for all silk fibers. The obvious absorption bands at 1618 cm^{-1} corresponds to amide I, which is characteristic of random coil conformation (amorphous), 1514 cm^{-1} corresponds to amide II, assigned to the β -sheet structure (crystalline) (Bhat and Ahirrao 1983). Also, the data show similar FT-IR spectra which implies that the molecular conformation of silk fibers does not change even after different treatment conditions of degumming and they assume a β -sheet structure and random coil conformation. The results also suggested that the sharpness of intensity of the β -sheet structure was increased in the IR spectrum of degummed silk as shown in Fig. 11(b). The appearance of β -sheet conformation may be associated with the packing of molecular chains in degummed silk fiber. Interestingly, the raw silk which exhibited prominent peaks in the region of 2921 cm^{-1} belonged to the stretching vibration of CH_2 ($\delta^2\text{CH}_2$ and $\nu^2\text{CH}_2$). This band was only observed sharply in raw silk samples. After being degummed at 90 °C for 90 minutes, the peak at 2921 cm^{-1} decreased, indicating that the sericin layer had being removed only leaving remaining sericin residual

Conclusion

In this paper, the method of degumming should be considered. This work has focused on examining the differences in both structure and properties of degummed silk fiber using a customized method including natural soap degumming (tap water and deionized water) along with laboratory scale and factory scale machines. The present study clearly indicates that degumming using an NSD method influences the properties of degummed mulberry silk fabrics. The profound differences in mechanical properties of laboratory scale machine compared with factory scale small machine, manifested by laboratory scale machine silk having much weaker mechanical properties, are attributed to the silk fibers becoming soft and stretchable after degumming processes. The silk degumming rate using deionized water (28.5715%) is higher than or equal to that of the method using tap water (28.1669%). The weight loss occurring in the treatment is due to the removal of sericin. The silk sericin removal percentage was almost 100% after degumming which resulted in a total weight loss of around ~28% in the silk fibers. Although the surface profile of degummed silk was clean and smooth, the treatment produced fibrillation in the material, which is evident from scanning electron micrographs. The flexural rigidity of the materials reduces due to the treatment using NSD. The boiling and scouring processing in the NSD method results in a decrease of tensile properties of the silk fibroin fiber. The tensile strength and elongation of degummed silk fiber decreased from 108.73 gf/den to 56.86 - 77.1 gf/den (for tensile strength) and from 13.83% to 10.66 - 11.53% (for elongation), indicating that the weakening molecular orientation of silk fibers by the action of

the soap-alkali solution towards silk fibroin fiber accompanied with the partial hydrolysis of silk fibroin molecules by the alkali action of NSD. The SEM observations show that the factory scale small machine degummed silk surface seemed to have a rough surface texture profile with remaining sericin residue. What is more, the molecular conformation estimated by IR spectrum stayed unchanged regardless of the degumming procedures.

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Declaration of Conflicting Interests

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