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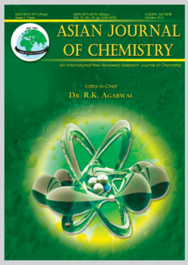

Effect of Degumming Performed with Different Type Natural Soaps and Through Microwave Energy Method on The Properties of Silk Fiber

METIN YUKSEK

Department of Textile, Faculty of Technical Education, Marmara University, Kadikoy, Istanbul, Turkey

Corresponding author: E-mail: myuksek@marmara.edu.tr

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Removal of sericin on the silk fibers is a surface modification process. Various chemical agents are used in the sericin removal process. In this article, effect of the sericin removal process performed in order to clear of the fiber surface from sericin through conventional and ultrasonic methods by using natural materials such as olive oil, daphne, turpentine soaps. The results obtained were examined with percentage weight loss values; bleaching properties, mechanical properties, morphological characteristics (SEM) followed by FT-IR. The most weight loss was observed in the sericin removal process performed with daphne and turpentine soaps through the microwave method. In all samples, microwave had a role in the highness of both the tensile strength and elongation values. Bleaching level was higher in the methods where microwave was used.

Key Words: Olive oil soap, Daphne soap, Turpentine soap, Degumming, Conventional method, Microwave irradiation.

INTRODUCTION

Silk fibers are composed of two proteins, one of them is fibroin in the form of filament and the other one is sericin (gum) which is not in the form of filament. Besides, silk contains naturally coloured substances, wax, carbohydrate and inorganic salts.

Silky material is cleared of sericin in order to prepare it for the processes of dyeing, printing and finish. Then it is generally bleached with hydrogen peroxide. Removed of sericin from silky material is called as degumming. Water, gelatine, soap, enzymes, alkalies and acids are some of the methods that are applied. The most widely used ones among these are soaps and enzymes[1].

Silk fiber is an all-purpose biopolymer as it possesses most of the properties required for a textile fiber such as slimness, stiffness, softness, smooth feeling, brightness, elegance and dyeability[2]. It is the only natural filament produced by the silkworm moth. Degumming is the process to extract the silk sericin from material as soap, alkali-soap or enzymatic. After the removal of sericin, silk fibroin fibers acquire pearlescence and flexibility of the silk fiber increases. Touch of silky fabric depends on the amount of sericin remaining in the silk fibroin. Silk fiber properties such as usage, brightness and friction behaviour are also affected by the amount of sericin remaining on the material. Thus, degumming is a rather important process[3].

In recent years, detergents are used instead of soaps in the continuous sericin removal. Its reason is that as the acidic hydrolysis products of sericin accumulating in the degumming baths can not be balanced, the use of degumming baths for weekly sericin removal phases is restricted.

It is stated that in enzymatic degumming of the silk fabric, a higher level of bleaching is obtained when compared to the fabric whose sericin is removed with soap-alkali but it has a higher level of creep and flexural rigidity, a lower weighting and soft touching in this method[4]. It is asserted that better resistance and elongation values are obtained in the degumming performed with alcalase, savinase and the combination of these when compared to the degumming process with soap[5]. In another research where the papain enzyme was compared with the soap-alkali method, properties such as weight loss, resistance and colour yield were examined[6].

Some degumming methods were analyzed in order to reduce the environmental and pollution burden. It is indicated that organic acids can be alternatives so as to deal with the problems to occur in the conventional soap/enzyme degumming process. It is also indicated that the yield and the intrinsic physico-mechanical properties of the silk fibers are considerably good in the degumming with tartaric acid and almost all of the sericin on the fiber can be removed in the degumming process performed with citric acid[6-8].

Microwaves are high frequency radio waves. Microwaves having a rather wide frequency spectrum are especially used in the field of telecommunication. Microwave range is 910-2850 MHz[9]. While 2450 MHz radiation is mainly used in applications such as household ovens and other low-power heating systems, high-power heating systems can operate more smoothly at 900 MHz frequency. Specific frequency bands are allowed in every country in order not to cause any confusion in communication[10].

The basis of the high frequency drying is that product to be dried is made to pass through two condenser leaves connected to high frequency alternative current. As water molecules form dipole, they want to take a specific layout shape. However, as high frequency alternative current is used, charge of the condenser leaves constantly changes and thus, layout shape of the water molecules also constantly change. Heat generated as a result of the friction of the molecules during this change repeated millions of times in a second enables the evaporation of the water[11].

Microwave technology includes such fields as heating, drying, condensation, dyeing and fixation in press and disinfection of the woollen fabrics. In 1966, patent was issued to Ciba Geigy Company, which introduced the use of microwaves to the dyeing process where the reactive dyestuffs are used[12].

There are many researches as regards to the use of the microwave energy in the textile industry. Issues discussed and nalyzed in these researches include alkalisation of polyester fabrics[13], fixation of polyester fabric[14], dyeing of polyester[15,16], development of the dyeability of the flax fibers[17], dyestuff fixing of the wool in the pad dyeing's[18], fixing of the reactive printingwoollen fabrics[19], degumming of the silk[20].

In this research, sericin removal process was performed through the conventional and the environment-friendly microwave energy methods by using the olive oil, turpentine and daphne soaps.

EXPERIMENTAL

Silk fibers used in this research were specially produced in the province of Bursa of Turkey. They were conditioned 48 h prior to testing under 20 ± 2 ºC and 65 ± 2 relative humidity % condition. Properties of the fibers are indicated in the Table-1.

TABLE-1
PROPERTIES OF THE SILK FIBERS

Stiffness (dtex)	1-4
Tensile strength (cN/tex)	7.5
Elongation (%)	17.4

Chemical treatments of silk fibers: Silk fibers were exposed to two different methods. Experimental details of the conventional and the microwave energy methods of the degumming process are given below.

Conventional method: In the study, polimat HT sample dyeing machine (Type A11612N-Emsey) was used. A treatment of 1 h was conducted at 95 ºC at a 1/30 flotte ratio with olive oil soap, daphne and turpentine soap solutions prepared at 0.7 % ratio with distillate water. At the end of the process, test samples were washed for 5 min in 1.5 L hot water and then rinsed for 5 min in 1.5 L cold water.

Microwave energy method: Microwave method with a Galanz/WP800T was carried out at a frequency of 2.45 GHz. Microwave oven had a maximal power f 800 W with six discrete settings. The mixtures were placed in a sealed glass vessel and treated by the microwave according to the experimental design. Degumming flotte which was brought to 50 ºC at the 1/30 flotte ratio with a 0.7 % soap solution was treated under high-level microwaves for 2 min. After 2 min, flotte outlet temperature from the microwave reached to 75 ºC. At the end of the process, test samples were firstly washed for 5 min in 1.5 L hot water and then rinsed for 5 min in 1.5 L cold water.

Determination of the weight loss: Raw and degummed fibers used in the experiments were conditioned under laboratory conditions and then, average weight was determined by making 5 measurements for each test sample according to ASTM D5848-10E[21]. Weight loss was found by measuring the weight difference between the untreated samples and the samples treated with soaps of different origins. After the treatment, samples were dried in the drying oven at 95 ºC until they reached to the fixed weighting. Weight loss was calculated and the initial weight was indicated as %. Amount of the weight loss was calculated with the following formula:

$$\text{WL}\ (\%) = \frac{W_1 - W_2}{W_1} \times 100 \qquad (1)$$

where WL: weight loss, W_1: raw silk weight, W_2: degumming silk weight.

Testing and characterization of silk fibers after chemical treatments: Applying the conventional and the microwave energy methods on silk fibers (single fiber length is 250 mm), mechanical characteristic values were determined based on ASTM D 3822 with Instron 4411 (50 N load, speed of 10 mm/min) resistance device[21]. Besides, morphological properties were analyzed by JEOL JSM-5410 LV operated at 20 kV.

RESULTS AND DISCUSSION

Weight loss: Weight losses occurring the surface processes applied to silk fiber through 2 different methods and with 3 different natural soaps are indicated by percentage (Fig. 1).

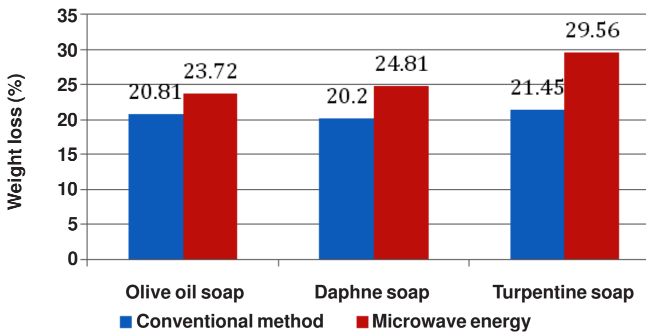

Fig. 1. Weight loss properties obtained as a result of the degumming process performed with conventional and microwave energy methods

When the weight loss ratios of the process performed with conventional method are evaluated in terms of methodology, they are seen within the limits specified in the literature[18].

However, degumming processes performed with all of three natural soaps yielded good results in both the conventional and the microwave energy methods and a great amount of sericin was removed. In the degumming process conducted through the microwave energy method, time was shortened, weight loss increased, a contribution was made to the amount of the water that was used. Positive effect and power of the environment-friendly microwave energy method has become clear[19-22].

Mechanical properties: When the changes in the resistance properties at the end of the degumming process performed through the conventional and the microwave energy methods by using the daphne soap, turpentine soap and olive oil soap are evaluated according to Fig. 2, resistance properties of the treated silk fibers are higher than the untreated fibers. An overall decrease is observed in the resistance values at the end of the degumming processes conducted through the conventional and the microwave energy methods. The reason of this decrease is the removal of the sericin and thus the emerging weight loss. This decrease observed in the resistance values is less in the degumming process performed with the microwave energy method. At this point, it is observed that microwave energy carries all of the three soaps to the fibers in the shortest time possible in the form of waves with the help of rapid mass transfer and it reduces the negative mechanical effect at the end of the process to the minimum when compared to the conventional treatment. Moreover, order of hydrogen bonds in the polymer chain structure of the silk fibers is not distorted due to sonication effect due to the microwave energy and even the order in the β-chain structure of the silk fiber is not distorted. This effect is more obvious to observe in the turpentine soap.

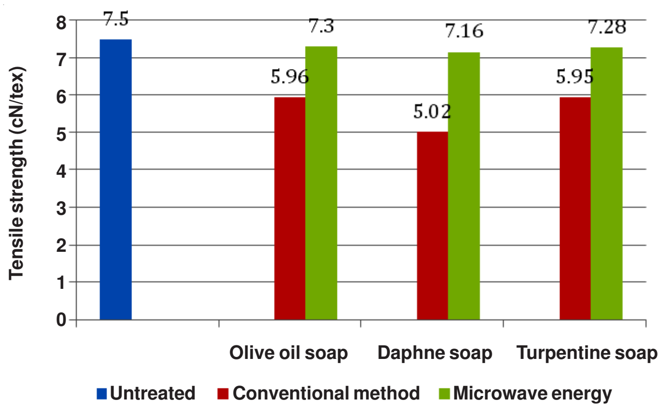

Fig. 2. Resistance properties obtained at the end of the degumming process performed through the conventional and the microwave energy methods

An overall increase is observed when the Fig. 3 is analyzed. This increase results from the fact that the fibers possess a more amorphous structure after the removal of the sericin. This effect can also be attributed to the amino acid groups of the polymer chain. This is because of the positive effect of the turpentine soap on the resistance properties and the fact that it forms the chemical bonding that microwave energy creates in the water without distorting the fibrillary structure of the fibers in a short time and even increases it.

Degree of whiteness: Percentage remission values measurement of the dyed samples was performed through Datacolour Spectra Flash 600 plus reflectance spectrophotometry according to CMC 2:1 CIELab and CIELch system. Colourmeasurements were conducted by using D65 light source with 10° observer. Degrees of whiteness were calculated

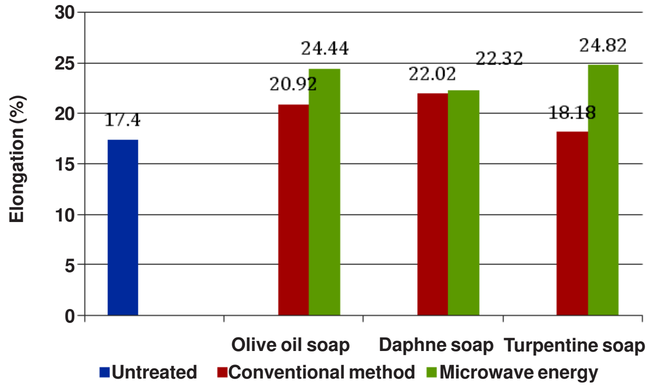

Fig. 3. Elongation properties obtained at the end of the degumming process performed through the conventional and the microwave energy methods

with Berger Whiteness formula[23] with the use of remission values. Measurements of the raw silk fibers and those undergoing a degumming process through the conventional and microwave energy methods with different soaps were performed in the spectrophotometry at 10 nm range for 400-700 nm wavelengths and % remission values of the samples are given in the Table-2. Degrees of whiteness determined according to Berger Whiteness index formula with the use of % remission values are also given in Fig. 4.

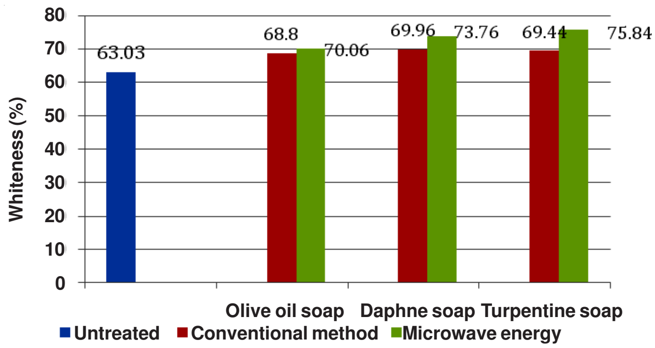

Fig. 4. Degrees of whiteness obtained at the end of the degumming process performed through the conventional and the microwave energy methods

It is already known that an overall increase is observed in the remission percentages obtained at the end of the removal of the sericin through the conventional and the microwave energy methods when compared to the untreated materials (Table-2 and Fig. 4). Perfect increases are observed in the remission values when the treatment is performed through the microwave energy rather than the conventional method. Beside to the increase in the remission values obtained with each of the three soaping, an additional positive increase is observed in the remission values obtained at the end of the degumming performed through the microwave energy method with the turpentine soap.

SEM of silk fibers: SEM photographs of the untreated silk fibers are observed in Figs. 5 and 6 In the Figs. 7 and 8, removal of the sericin through different methods by using the daphne soap is indicated, in Figs. 9 and 10, removal of the sericin through different methods by using the turpentine soap and in the Figs. 11 and 12, removal of the sericin through different methods by using the olive oil soap are given.

TABLE-2
PERCENTAGE REMISSION VALUES OBTAINED AT THE END OF THE DEGUMMING PROCESS PERFORMED THROUGH THE CONVENTIONAL AND THE MICROWAVE ENERGY METHODS

nm	Raw	Olive oil soap		Daphne soap		Turpentile soap	
		Conventional	Microwave	Conventional	Microwave	Conventional	Microwave
400	73.35	76.56	76.61	76.69	78.17	76.6	79.11
420	76.59	79.42	79.58	79.33	80.98	79.56	81.79
440	79.38	81.70	81.93	81.54	83.23	81.95	83.91
460	81.83	83.56	83.85	83.38	85.05	83.93	85.60
480	83.82	85.03	85.30	84.82	86.42	85.48	86.82
500	85.42	86.28	86.48	85.95	87.44	86.75	87.76
520	86.61	87.27	87.33	86.78	88.14	87.65	88.43
540	87.49	88.02	87.95	87.37	88.55	88.30	88.96
560	88.23	88.64	88.47	87.91	88.83	88.84	89.42
580	88.86	89.12	88.91	88.32	89.06	89.29	89.82
600	89.39	89.49	89.26	88.70	89.25	89.67	90.17
620	89.81	89.77	89.53	88.98	89.44	89.95	90.41
640	90.23	90.08	89.83	89.32	89.81	90.27	90.68
660	90.37	90.13	89.88	89.42	90.08	90.29	90.66
680	90.74	90.42	90.18	89.73	90.61	90.58	90.90
700	91.28	90.89	90.65	90.24	91.25	91.12	91.37

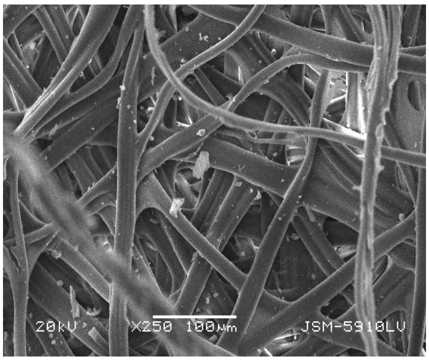

Fig. 5. Untreated silk fibre

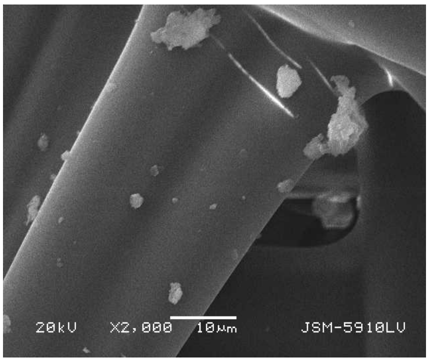

Fig. 6. Untreated silk fibre

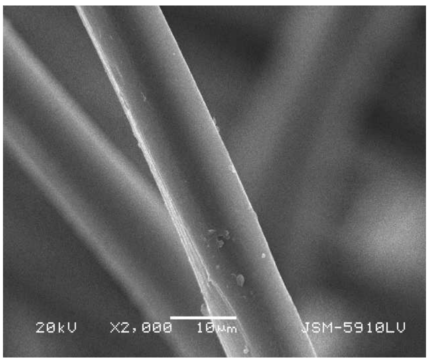

Fig. 7. Treated silk fibre with laurel soap by conventional process

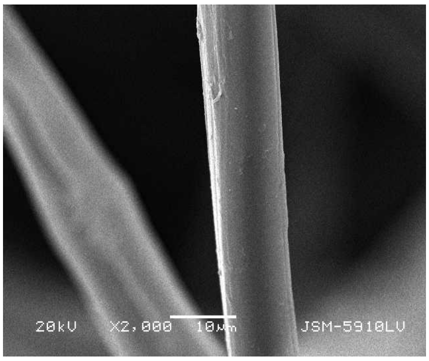

Fig. 8. Treated silk fibre with laurel soap by microwave energy process

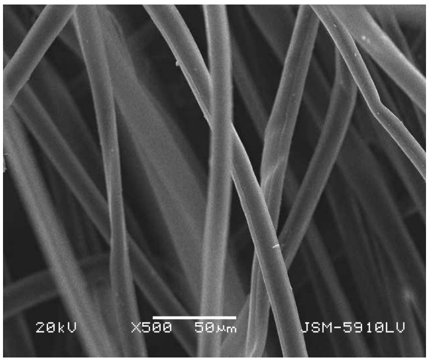

Fig. 9. Treated silk fibre with turpentine soap by conventional process

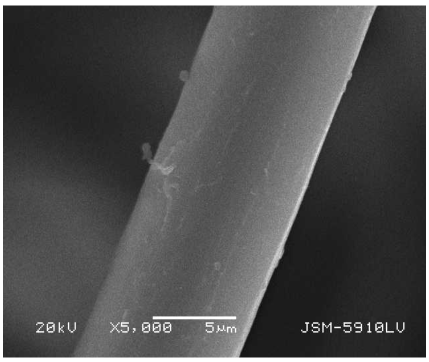

Fig. 10. Treated silk fibre with turpentine soap by microwave process

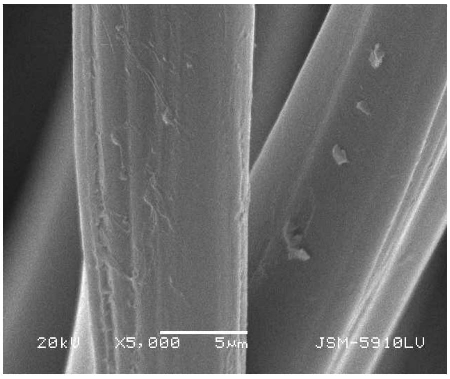

Fig. 11. Treated silk fibre with olive oil soap by conventional process

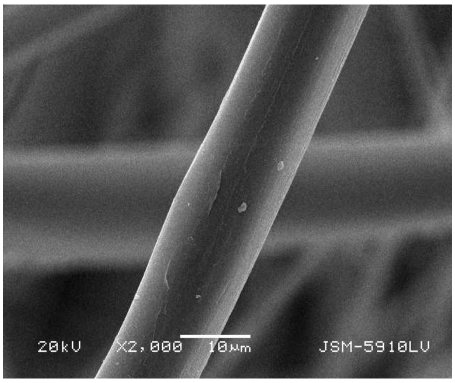

Fig. 12. Treated silk fibre with olive oil soap by microwave process

If a degumming process is insufficient and incompatible, residual sericin should appear as, deposits on the surface of the filaments and nonuniformity over the surface of the yarn must be observed[5]. When an overall assessment of the SEM photographs is made, it is observed that the degumming processes performed with the use of microwave energy are more successful than those performed through the conventional method. Power of the ultrasonic energy became clear once more in the surface morphology. Sericin of the exterior surface was removed completely. In the light of the other results, it was found out that sericin layer available on the exterior surface was removed completely in the microwave energy method where the turpentine soap was used and the fibrous structure of the silk fiber became more apparent.

Conclusion

In the degumming processes conducted through both the conventional and microwave energy methods with three different natural soaps, degumming process performed with the turpentine soap yielded positive results in terms of weight loss, whiteness degree and mechanical properties. The power of the microwave energy produced the most positive results in the degumming processes performed with the turpentine soap through the microwave energy method. Mass transfer of the turpentine soap, which is a natural soap within the water, did not harm the chemical bond structures as it took place quickly. The reason of the success of the microwave energy process is the strength that is achieved. In this study, % weight loss increase is outstanding with the success of removing the impurities. Weight loss results can be correlated with mechanical test results, which can be regarded as a proof for more enhanced interfacial interactions with microwave energy than the conventional method. Moreover, beyond all results, it is rather evident that chemical substance, energy, water and time savings are provided using the environment-friendly microwave energy method.

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