

Effect of Microwave Irradiation on the Physical Properties and Structure of Silk Fibre

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Shaoxing University,
College of Textile and Garment,
Shaoxing 312000

Clean Dyeing and Finishing Technology Research
Laboratory of Zhejiang Province,
Shaoxing 312000
E-mail: zhaoxue44455709@sina.com

Abstract

Microwaves are high frequency radio waves which are capable of penetrating many materials and causing heat to be generated in the process. To investigate the effect of microwave irradiation on the physical properties as well as the chemical, surface morphological and fine structure of silk fabric, silk fabric was treated with microwave irradiation under a variety of conditions in terms of the power and time of microwave treatment. The breaking strength, elongation at break, and whiteness of the treated silk fabric in a wet state were investigated. The structures of the untreated and treated silk were investigated with Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), differential scanning calorimetry (DSC) and X-ray diffraction (XRD). The results show that the physical properties of the treated silk fabrics were changed with the microwave irradiation time. The chemical and surface morphological structure as well as the decomposition temperature and crystallinity of the treated silk were changed.

Key words: silk fabric, microwave, structure, physical property.

Introduction

Silk has been used as textile fibres for more than 5000 years, as well as being widely used in commodities around the world and having a wide range of applications in apparel, furnishings, and industrial products. Silk-based reactions are very important for all possible chemical modifications of natural products. Much attention has been paid to the modification and utilisation of silk due to its good biodegradability and biocompatibility [1-3]. In recent years, the modifications and drying of some materials have been conducted under microwave irradiation conditions [4-6]. The dielectric constant (ϵ) of various natural fibres or synthetic polymers is as follows: The dielectric constant (ϵ) of silk is 4.2, cotton yarn – 6.0, polyethylene fibre – about 4.0~8.0, polyester fibre – 3.02, nylon 6 – about 3.5~3.7, nylon 66 – 3.33, and polyvinyl chloride – 3.3. Compared with other natural fibres or synthetic polymers, silk has a relatively higher dielectric constant (ϵ) [7]. Microwave irradiation is one of the powerful techniques of non-contact heating, because dielectric substances with a large dielectric constant are heated intensely due to the vibration and rotation of permanent dipoles in the microwave field. Silk has a higher polarisation ability in the microwave field compared with other natural fibres or synthetic pol-

ymers. The microwave has been used in the heating, drying and dyeing processing of silk materials [8-10].

In the conventional processing of fabric, a large amount of energy is consumed. Microwave heating, as an alternative to the conventional heating technique, has been proved to be more rapid, uniform and efficient. Microwave energy can easily penetrate to the particle inside, and all particles can be heated simultaneously, thus reducing heat transfer problems. However, microwave irradiation could affect the chemical, fine s and surface morphological structure of silk fabric, including some physical properties. Report of the effect of microwave irradiation on the physical properties and structure of wet silk has not yet been undertaken systematically [11-13].

In this paper, silk fabric was treated with microwave irradiation. The effect of microwave irradiation on the structure of silk was investigated with Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), differential scanning calorimetry (DSC) and X-ray diffraction (XRD). Physical properties of the treated silk were also discussed.

Experimental

Materials

100% silk weave fabric was obtained from the Rongrui Textile Company (Huzhou, China). Specifications of the silk fabric used: 16S \times 16 S 96 \times 82 inch.

Microwave irradiation treatment of silk fabric

A microwave oven, Yk-01, used in this study, with continuous adjustable power of 100~1000 W. A microwave frequency of 2450 MHz was chosen because this has been widely used as an ISM band (industrial, scientific, and medical use) that know-how in microwave industry were available for manufacturing of the apparatus used in this study.

Silk fabrics in a wet state were enclosed in polythene film and then treated with microwave irradiation at various power settings (119, 280, 462, 595, 700 W) for various lengths of time (10, 20, 30, 60, 90, 120, 140, 160, 180, 200, 220, 240 and 300 s, respectively), the fabrics were then removed from the microwave oven and slowly cooled under a vacuum for 24 h. The silk fabrics were immersed in a water bath for 30 min and then padded at room temperature; the liquor pickup can be 80% of the weight of dry fibre.

Fabric performance evaluation

The breaking strength and elongation at break of the fabric was measured according to the ASTM D 5034 test method (Measurement was made after conditioning samples at 25~30 °C, 60~70% R.H. for 24 h prior to testing), with a measurement error of $\pm 0.5\%$. Whiteness was measured according to Brightness ISO R457, with a measurement error of $\pm 3\%$. Three samples were examined for each measurement. Each point needed to be tested three times, and the final result

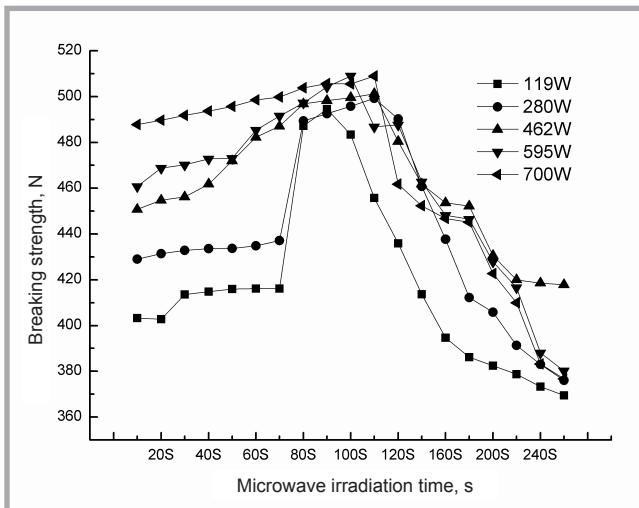


Figure 1. Breaking strength of silk fabric in a wet state treated with microwave irradiation. **Note:** Breaking strength of the untreated silk fabric is 405.8N.

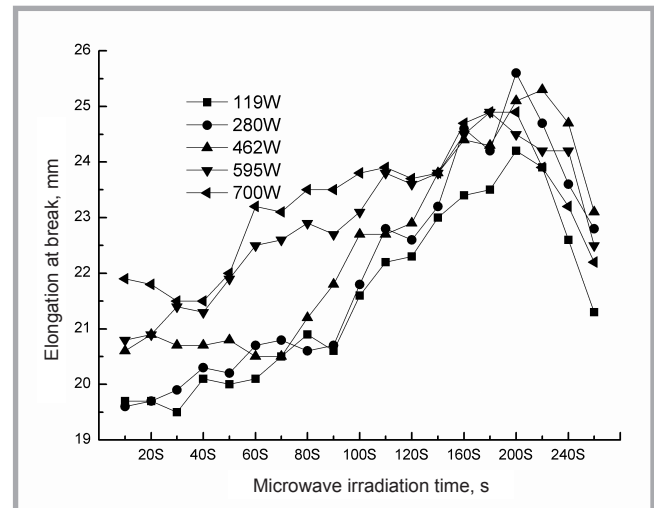


Figure 2. Elongation at break of silk fabric in a wet state treated with microwave irradiation. **Note:** Elongation at break of the untreated silk fabric is 16.2%.

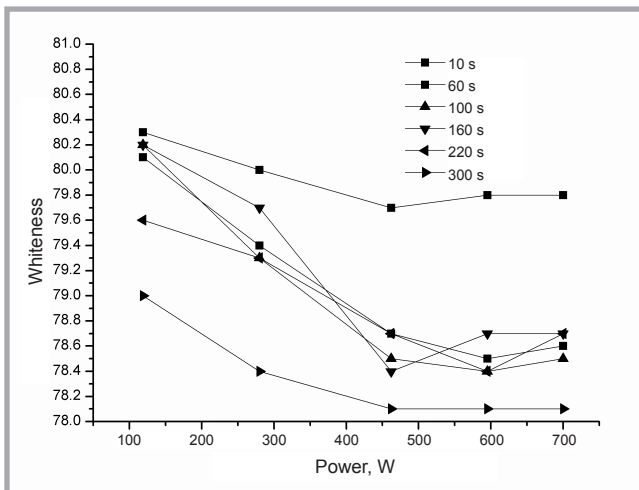


Figure 3. Whiteness of untreated and microwave-treated silk. **Note:** Whiteness of the untreated silk fabric is 80.4.

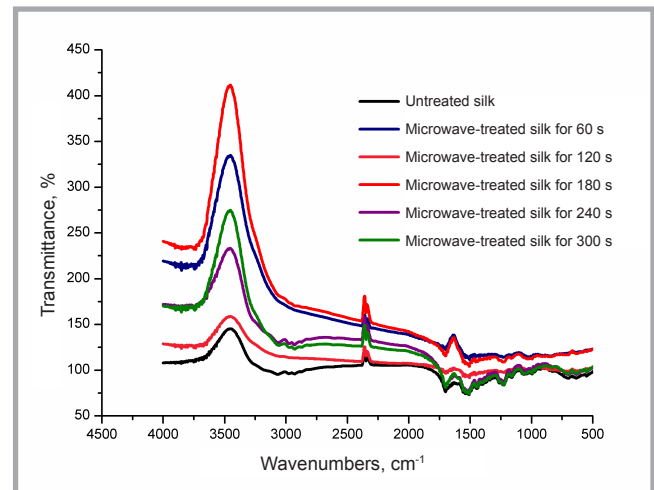


Figure 4. FTIR spectra of untreated and microwave-treated silk.

was the average value of the three numbers tested.

The surface morphological structure of untreated silk fabric and microwave-treated silk fabrics was measured by SNG-3000 scanning electron microscopy (SEC Ltd, Korea). The chemical structures of untreated and microwave-treated silk fabrics were measured by an IRPrestige-21 infrared spectrometer (SHIMADZU, Japan). Decomposition temperatures of the untreated and microwave-treated silk fabrics were measured by differential scanning calorimetry (Perkin-Elmer Ltd, USA). The crystallinity of the untreated and microwave-treated silk fabrics was measured by an Empyrean X-ray Diffractometer (PANalytical Ltd, Holland), which used the Cu-K target at 40 kV, 300 mA and $k = 1.54056$.

Results and discussion

Physical properties of the microwave-treated silk fabric

Breaking strength and elongation at break

The breaking strength and elongation at break of silk fabric in a wet state treated with microwave irradiation may change. The microwave power and treatment time under microwave irradiation conditions may also impact the breaking strength and elongation at break of silk fabric. The breaking strength and elongation at break of untreated and treated silk fabric in a wet state with microwaves at various power settings (119, 280, 462, 595 & 700 W) for various lengths of time (10, 20, 30, 60, 90, 120, 140, 160, 180, 200, 220, 240 and 300 s) are presented in **Figures 1** and **2**.

Figure 1 shows that the treated silk fabric has a higher breaking strength in a wet state as compared to the untreated silk fabric, which increases with the processing time at the beginning. This increase in breaking strength is thought to be due to the presence of water molecules in silk fibres, which promotes the adjustment of the fine structure of silk fibres through the absorption of microwave energy, and eliminates the residual stress existing in the fibre.

Compared with the untreated silk, the treated silk fabric decreased significantly with increasing treatment time in a wet state due to the more heat generated by free water molecules in a wet state. Higher temperature conditions leads to a decrease in breaking strength. Silk fabrics in a wet state contain more free water

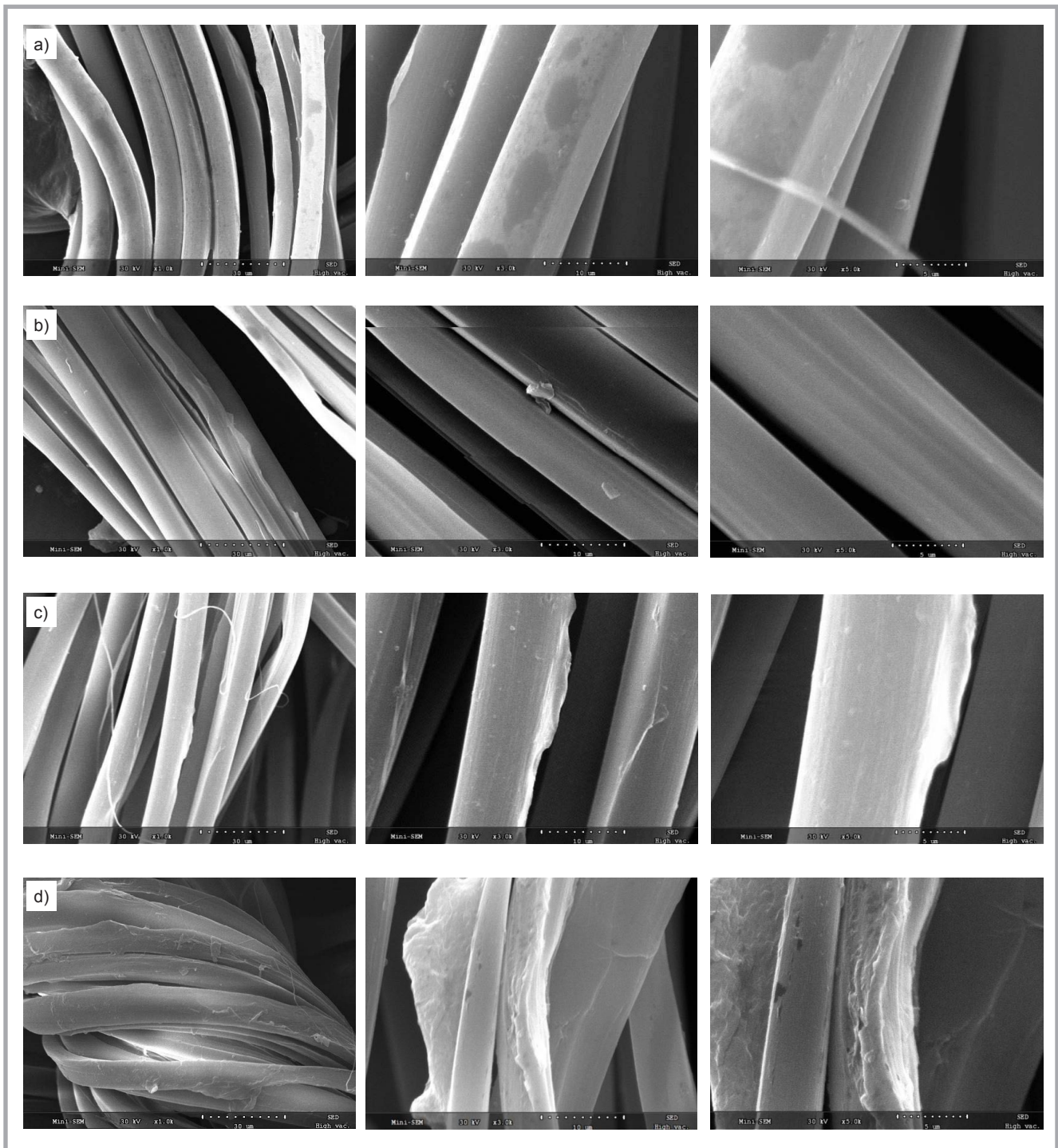


Figure 5. SEM micrographs of a) untreated silk and microwave-treated silk: b) 60 s, c) 180 s, d) 300 s.

molecules, hence the fibres themselves absorb less microwave energy. Microwaves are high frequency radio waves which are capable of penetrating water molecules in silk fibres and causing heat to be generated in the process. The electric field energy is converted into heat through the dielectric losses of water in the wet state. In the presence of a high frequency electromagnetic field, the water molecules oscillate synchronously with it [14.15].

It can be clearly seen from **Figure 2** that after a short period of microwave irradiation, the elongation at break of the silk fabric in a wet state is significantly increased, which may be attributed to the thermal shrinkage of the silk fibre in the wet state. After a long period of microwave irradiation, the elongation at break of the treated silk fabric in a wet state decreases. These changes in elongation at break are mainly due to the decreased breaking strength of microwave treated silk fabrics.

Whiteness

The treatment time and power under microwave irradiation conditions may also impact the whiteness of silk fabric. The whiteness of silk fabric treated in a wet state with microwaves at various power settings (119, 280, 462, 595 & 700 W) for various lengths of time (10, 60, 100, 160, 220 and 300 s) is presented in **Figure 3**.

It can be seen from **Figure 3** that compared with the untreated silk, the

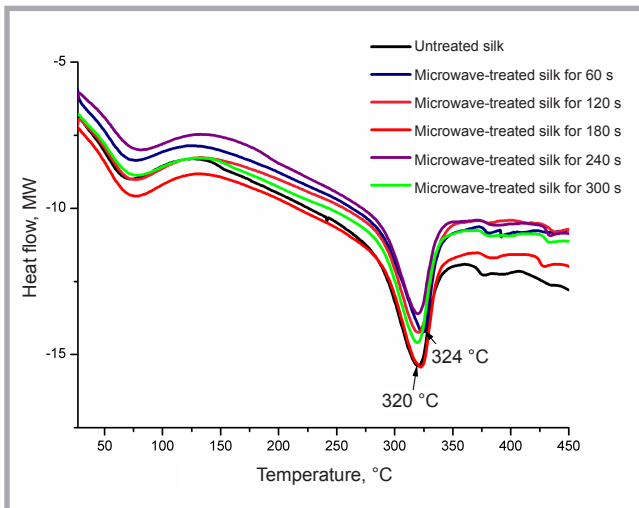


Figure 6. DSC Curve of untreated silk and microwave-treated silk.

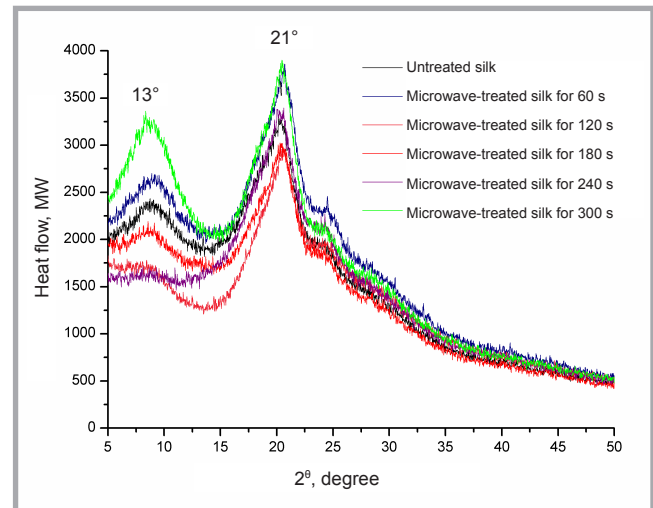


Figure 7. XRD Curve of untreated silk and microwave-treated silk.

whiteness of silk fabric subjected to microwave treatment was significantly decreased. Microwave irradiation had a significant effect on the whiteness of silk fabric.

FTIR analysis

To investigate the influence of the treatment with microwaves on the chemical structure of silk fibres, experiments with silk fabric in a wet state under microwave irradiation at 700W power for various lengths of time (60, 120, 180, 240 and 300 s) were carried out. Figure 4 shows the FTIR curve of untreated silk samples and those treated with microwaves.

As can be seen from Figure 4, the FTIR curve of the microwave-treated silk was changed compared to the untreated silk. The wavelength region has a difference of 3000, 1700⁻¹, 500, 1000 cm⁻¹, which is due to the more heat generated by free water molecules in a wet state. As can be seen from Figure 4, under high temperature conditions can cause changes in chemical structure. Microwave radiation has a significant effect on the chemical structure of silk fibres.

Table 1. CI value of untreated silk and microwave-treated silk.

Silk samples	I ₂₁	I ₁₃	CI
Untreated	3271	1750	0.46
Treated with 60 s	3859	1956	0.49
Treated with 120 s	2964	1482	0.50
Treated with 180 s	3020	1531	0.49
Treated with 240 s	2969	1562	0.47
Treated with 300 s	3904	2065	0.47

SEM analysis

To investigate the influence of the treatment with microwaves on the surface morphological structure of silk fibres, experiments with silk fabric in a wet state under microwave irradiation of 700 W power were carried out for various lengths of time (60, 120, 180, 240 and 300 s). Scanning electron microscopy photographs of the surface morphological structure of the untreated silk samples and those treated with microwaves are shown in Figure 5.

Figure 5 shows the microwave irradiation had an obvious damaging effect on the surface structure of the silk compared with the untreated silk fabric. For silk fabric in a wet state wet at 300 s microwave treatment time, some of the surface morphological structure of the silk fibres was slightly damaged.

DSC analysis

To investigate the influence of the treatment with microwaves on the silk fibre decomposition temperature, experiments with silk fabric in a wet state under microwave irradiation of 700 W power were carried out for various lengths of time (60, 120, 180, 240 and 300 s). Decomposition temperatures of the untreated silk samples and those treated with microwaves are shown in Figure 6.

It can be seen from Figure 6 that the decomposition temperature of untreated silk is 320 °C. Compared with the untreated silk, the decomposition temperature of the silk treated with microwave irradiation in a wet state increased to 324 °C,

which can be attributed to the adjustment of the fine structure of silk fibres in a wet state by absorption of microwave energy. For a treatment time of 120 s to 300 s, the decomposition temperature of the treated silk slightly decreased. The decrease in the decomposition temperature of the treated silk fabric changed the physical properties.

XRD analysis

To investigate the influence of the treatment with microwaves on silk fibre crystallinity, experiments with silk fabric in a wet state under microwave irradiation of 700 W power were carried out for various lengths of time (60, 120, 180, 240 and 300 s). The crystallinity of the untreated silk fibres and those treated with microwave are shown in Figure 7.

The crystallisation index (CI) of silk fibres is calculated using the following equation [16].

$$CI (\%) = (I_{21} - I_{13}) / I_{21} \quad (1)$$

Where I₂₁ is the intensity at 2θ = 21° and I₁₃ at 2θ = 13°

On the basis of Equation (1), CI results of the untreated and microwave-treated silk fibres are listed in Table 1.

It can be seen from Table 1 that compared with the untreated silk, the crystallinity of the treated silk in a wet state with microwave irradiation increased, which can be attributed to the adjustment of the fine structure of silk fibres in a wet by absorption of microwave energy. For a treatment time of 120 s to 300 s, the crystallinity of the treated silk slightly

decreased. The decrease in the crystallinity of the treated silk fabric changed the physical properties .

■ Conclusions

FPAE emulsion with a core-shell structure was synthesised by semi-continuous seed emulsion polymerisation. Then linen fabrics weh resistance was excellent and the wear-resistance of the finished linen fabric was better than for the unfinished linen fabric; however, it had a negative effect on bending rigidity as well as the air and moisture permeability.

It can be concluded from the investigation that microwave irradiation could impact the breaking strength and elongation at break of wet silk fabrics. The whiteness of silk fabrics subjected to microwave treatment was significantly decreased. It was also found that microwave irradiation could also affect the chemical, surface morphological and fine structure of silk. The chemical structure of microwave-treated silk had an obvious change. The microwave treatment caused a slight damaging effect on the surface morphological structure of the silk fibres at a microwave treatment time of 300 s. Compared with the untreated silk fabric, the decomposition temperature and crystallisation of the treated silk fabric in a wet state increased with an increase in the treatment time, and decreased slightly as the treatment time increased. Microwave heating is more efficient than conventional heating methods. During conventional heating, heat is generated outside the treated product and conveyed by conduction or convection. On the contrary, in microwave treatment, heat is generated in a distributed manner

inside the material, allowing more uniform and faster heating. The microwave irradiation technique has good potential for industrial application as microwave is a clean, environmentally friendly heating technology.



Disclosure statement

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