Effect of Microwave Irradiation on the Physical Properties and Structures of Wool Fabric

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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 property, chemical structure, surface morphological structure, and fine structure of wool fabric, wool fabric was treated with microwave irradiation under variety of conditions in terms of the power and the time of microwave treatment. The breaking strength, breaking elongation, and whiteness of the treated wool fabric in different humid state were investigated. The structures of the untreated and treated wool were investigated with Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and Laser Raman spectroscopy (LRS). The results show that the physical properties of the treated wool fabrics were changed with microwave irradiation time and power. The chemical structure had not significant change. The surface morphological structure, the concentration of cystine S—S bonds and crystallinity of the treated wool were changed. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 944–952, 2011

Key words: wool fabric; microwave; structure; physical property

INTRODUCTION

Wool is one of the most widespread natural polymers. For all the possible chemical modifications of natural products, the reactions based on wool are the most important. Much attention has been paid on modification and utilization of wool due to its good biodegradability and biocompatibility. Modifications of wool with graft copolymer or compounds expand the applications as functional hybrid materials. In recent years, modifications and drying of some materials have been conducted under microwave irradiation condition.[1–5] The dielectric constant (ε) of various natural fibers or synthetic polymers are as follows[6]: The dielectric constant (ε) of wool is about 6.0∼ 10. The dielectric constant (ε) of silk is 4.2. The dielectric constant (ε) of cotton yarn is 6.0. The dielectric constant (ε) of polyethylene fiber is about 4.0∼ 8.0.The dielectric constant (ε) polyester fiber is 3.02. The dielectric constant (ε) of nylon 6 is about 3.5∼ 3.7. The dielectric constant (ε) of nylon 66 is 3.33. The dielectric constant (ε) of polyvinyl chloride is 3.3. Compared with other natural fibers or synthetic polymers, wool have a relatively higher dielectric constant (ε). Microwave irradiation is one of powerful techniques of noncontact heating, because the dielectric substances with large dielectric constant vigorously fever by vibration and rotation of permanent dipoles in microwave field. Wool has higher polarization ability in microwave field compared with other natural fibers or synthetic polymers. Microwave has been used in heating, drying, and dyeing processing of wool materials.[7–9]

In the conventional processing of fabric, a large amount of energy is consumed. Some new techniques and methods for saving energy were investigated.[10–14] Microwave heating, as an alternative to conventional heating technique, has been proved to be more rapid, uniform, and efficient. The microwave energy can easily penetrate to particle inside and all particles can be heated simultaneously, thus reducing heat transfer problems. However, the microwave irradiation could affect the chemical structure, fine structure, and surface morphological structure of wool fabric, including some physical properties. The report of the effect of microwave irradiation on the physical properties and structure of wool was scarce.

In this article, wool fabric was treated with microwave irradiation. The effect of microwave irradiation on the structure of wool was investigated with Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), Laser Raman spectroscopy (LRS), and X-ray diffraction (XRD). The physical properties of the treated wool were also discussed.

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EXPERIMENTAL

Materials

100% wool weave fabric was obtained from Qingfeng Textile Company (Beijing, China). Specifications of the wool fabric used: 15S/2 × 15S/2 74 × 64/inch.

Microwave irradiation treatment of wool fabric

A microwave oven, Yk-01, used in this study had continuous adjustable power of 250∼ 1000 W. The microwave frequency of 2450 MHz was chosen because 2450 MHz had been widely used as ISM band (industrial, scientific, and medical use) that know-how in microwave industry were available for manufacturing of the apparatus used in this study.

Wool fabrics in different humid state were enclosed in polythene film and then treated with microwave irradiation at various power settings (300, 500, 700 W) for various lengths of time (1, 1.5, 2, 2.5, 3, 3.5, and 4 min, respectively), the fabrics were removed from microwave oven and slowly cooled under vacuum for 24 h. Wool fabrics in different humid state were explained below (1) Wool fabrics in dry state (moisture absorption with anhydrous calcium in dryer for one week), (2) Wool fabric in nature humid state (Lay wool fabric to condition at 25∼ 30°C, 60∼ 70% R.H. to achieve an equilibrium moisture content.), (3) Wool fabric in wet state (The wool fabric were immersed in the water bath for 30 min, and then were padded at room temperature, the liquor pickup can be 70% on weight of dry fiber).

Fabric performance evaluation

The breaking strength and elongation of the fabric was measured according to ASTMD 5034 test method (Measurement was made at after conditioning samples at 25∼ 30°C, 60∼ 70% R.H. for 24 h prior to testing). CIE Whiteness was evaluated using a Datacolor SF650 color measuring and matching instrument (Datacolor). The surface morphological structure of untreated wool fabric and microwave-treated wool fabrics were measured by a JSM-5600LV scanning electron microscopy (JEOL, Japan). Crystallinity of untreated and microwave-treated wool fabrics were measured by a D/Max-2550 PC X-ray Diffractometer (Rigaku, Japan), which used Cu-K target at 40 kV, 300 mA, and $k = 1.54056$. The chemical structures of untreated and microwave-treated wool fabrics were measured by a NEXUS-670 infrared spectrum (Nicolet). The concentration of cystine S—S bonds of untreated and microwave-treated wool fabrics were measured by a Laser Raman microscope (Renishaw, England).

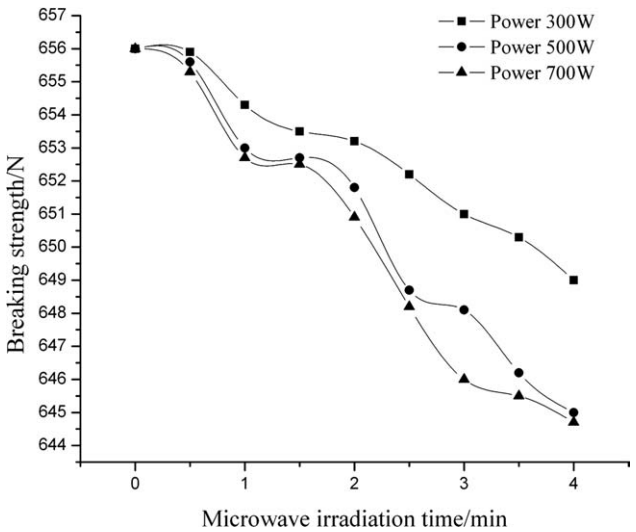

Figure 1 Breaking strength of the wool fabric in dry state treated with microwave irradiation.

RESULTS AND DISCUSSION

Physical properties of the microwave-treated wool fabric in different humid state

Breaking strength and elongation

Breaking strength and elongation of the wool fabric in different humid state treated with microwave irradiation may be changed. Microwave power and treating time under microwave irradiation condition may also impact the breaking strength and elongation of wool fabric. The breaking strength of the wool fabric untreated and treated in different humid state with microwave at various power settings (300, 500, 700 W) for various lengths of time (0.5, 1, 1.5, 2, 2.5, 3, 3.5, and 4 min) are presented in Figures 1–6.

Figure 1 shows that the breaking strength of the treated wool fabric in dry state was decreased with increasing treating time and power. Wool fabric in dry state doesn't contain water molecules. Microwave energy is mainly focused on wool fibers, which results in difficulty of intermolecular position adjustment. It is difficult for fibers to adjust the fine structure and eliminate the residual stress without the existence of water molecules in wool fiber under microwave irradiation. Obtaining of more heating and difficulty of fine structure adjustment under longer microwave treatment resulted in the breaking strength decreasing of wool fibers.

Figure 2 shows that compared with the untreated wool fabric, the breaking strength of the treated wool fabric in nature humid state was higher and increased with increasing treating time and power. There exist three different kinds of water adsorbed on wool, free water, freezing, and nonfreezing bound water. An amount of bound water on wool is about 33.5%. Free water adsorbs on wool until the

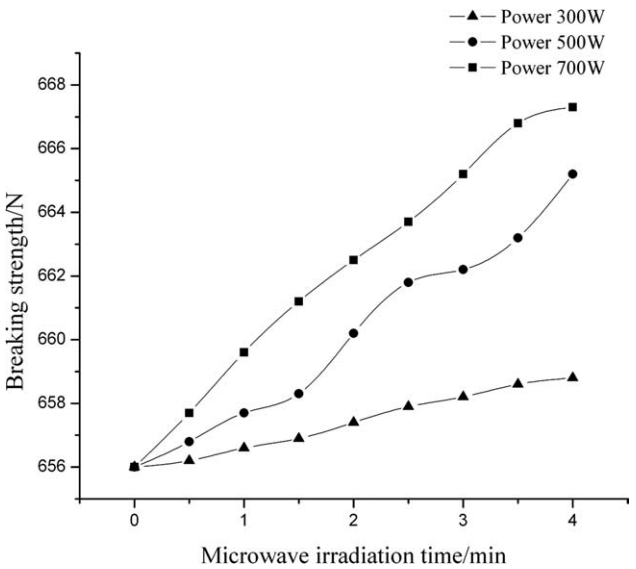

Figure 2 Breaking strength of the wool fabric in nature humid state treated with microwave irradiation.

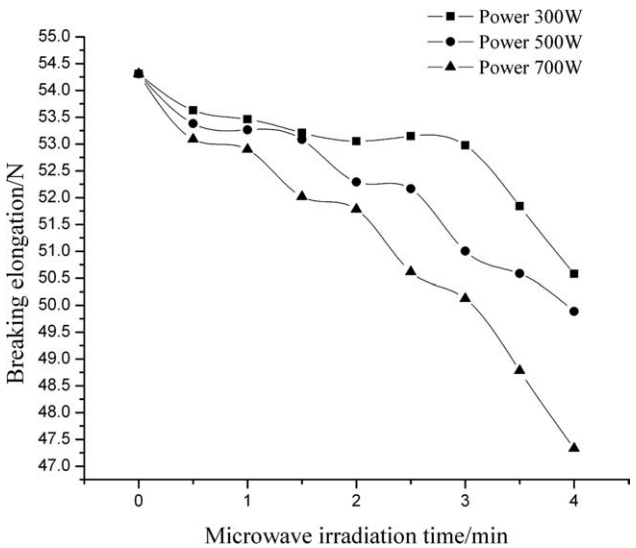

Figure 4 Breaking elongation of the wool fabric in dry state treated with microwave irradiation.

water content exceeds 30% by weight.[15,16] An amount of water on wool in nature humid state is about 12%. Wool fabric in nature humid state mainly contains bound water molecules, which express less thermal effect.[6] Fiber itself can also adsorb more microwave energy. The reason of such an increase in breaking strength was considered to be due to the existence of water molecules in wool fiber, which promoted adjustment of the fine structure of wool fibers resulted from absorption of microwave energy and eliminated the residual stress existed in wool fibers.

It can be seen from Figure 3 that compared with the untreated wool, the treated wool fabric in wet state was obviously decreased with increasing treating time and power, which is caused by more heat generated through free water molecules in wet state. Longer high temperature conditions results in decreases of breaking strength. Wool fabric in wet state contains more free water molecules so that fibers itself adsorbs few microwave energy. Microwaves are high frequency radio waves which are capable of penetrating water molecules in wool fibers and causing heat to be generated in the process. The electric field energy is converted into heat through the dielectric losses of the water in wet state. In the presence of the high frequency electromagnetic field the water molecules oscillate synchronously with it,

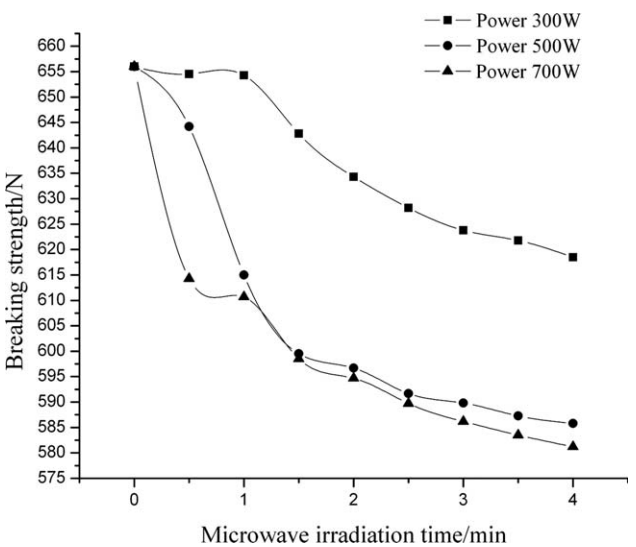

Figure 3 Breaking strength of the wool fabric in wet state treated with microwave irradiation.

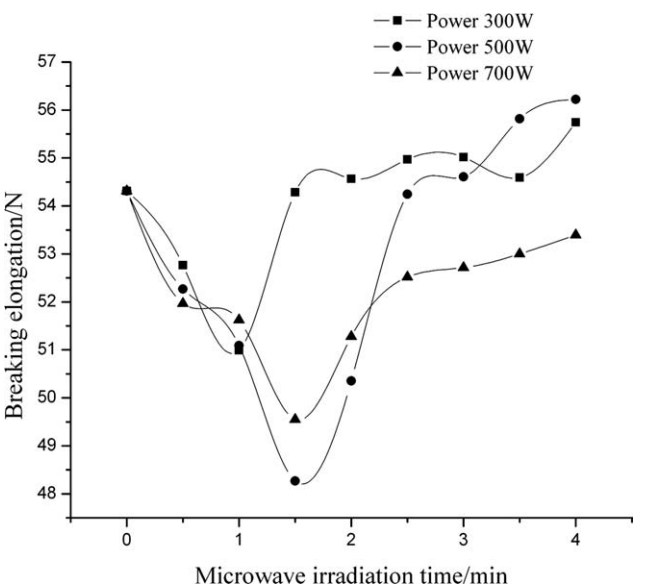

Figure 5 Breaking elongation of the wool fabric in nature humid state treated with microwave irradiation.

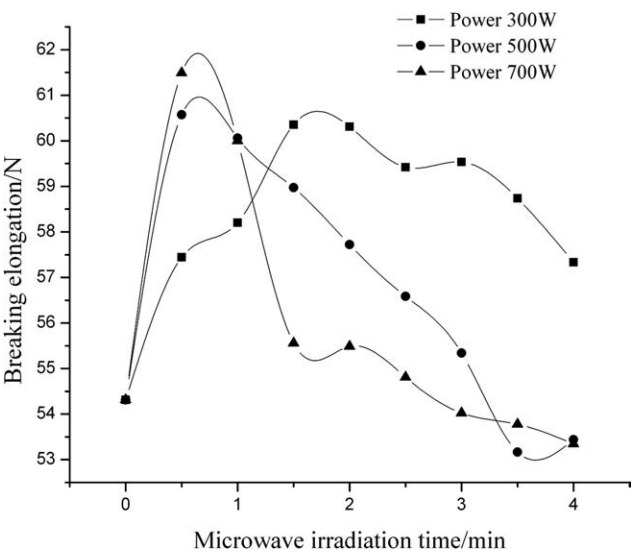

Figure 6 Breaking elongation of the wool fabric in wet state treated with microwave irradiation.

and this causes heat to be generated through intermolecular friction.

It was clear from Figures 4–6 that the elongation at break of treated wool fabric in dry state was decreased with increasing treating time and power. The elongation at break of treated wool fabric in nature humid state was increased after longer microwave irradiation. These changes of the elongation at break were mainly because the breaking strength of the wool fabric treated with microwave might be increased or decreased. The elongation at break for wool fabric in wet state has the obvious increase after shorter microwave irradiation, which can be attributed to the heat shrinkage of wool fibers in wet state.

Whiteness

Treating time and power under microwave irradiation condition may also impact the whiteness of wool fabric. The whiteness of the wool fabric treated in different humid state with microwave at various power settings (300, 500, 700 W) for various lengths of time (0.5, 1, 1.5, 2, 2.5, 3, 3.5, and 4 min) are presented in Table I.

It can be seen from Table I that compared with the untreated wool, the whiteness of wool fabric subjected to microwave treatment were slightly decreased. Microwave irradiation had no significant effect on the whiteness of wool fabric.

FTIR analysis

To investigate the influence of the treatment with microwave on the wool fibers chemical structure, experiments with wool fabric in different humid state under microwave irradiation 700W power for 4 min were carried out. Figure 7 shows the FTIR curve of the wool samples untreated and treated with microwave.

It can be seen from Figure 7 that compared with the untreated wool, the FTIR curve of the treated wool with microwave almost unchanged. Microwave irradiation had no significant influence on the chemical structure of wool fibers.

XRD analysis

To investigate the influence of the treatment with microwave on the crystallnity of the wool fibers, experiments with wool fabric in different humid state under microwave irradiation 4 min was carried out. The X-ray diffraction analysis of the crystallnity of the wool samples untreated and treated with microwave are showed in Figure 8.

The crystallization index (CI) of wool fibers is calculated by the following equation.[17]

$$\mathrm{CI}\ (\%) = (\mathrm{I}_9^\circ - \mathrm{I}_{14}^\circ)/\mathrm{I}_9^\circ, \quad (1)$$

where I_9° is the intensity at $2\theta = 9^\circ$ and I_{14}° is the intensity at $2\theta = 14^\circ$.

TABLE I
Whiteness of the Wool Fabric in Different Humid State Treated with Microwave Irradiation

		Whiteness in different microwave irradiation time (min)							
	Irradiation power (W)	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0
Dry state	300	−17.06	−17.55	−17.43	−17.44	−18.07	−18.61	−18.69	−18.18
	500	−17.19	−17.62	−17.59	−17.56	−17.58	−18.24	−18.49	−18.34
	700	−15.46	−15.22	−15.69	−15.94	−17.33	−17.60	−17.49	−17.88
Nature humid state	300	−15.45	−16.40	−16.10	−16.15	−16.19	−16.36	−16.78	−16.91
	500	−12.85	−13.23	−15.53	−16.06	−16.02	−16.43	−16.67	−16.58
	700	−16.19	−16.97	−17.56	−17.40	−17.82	−18.52	−18.78	−18.23
Wet state	300	−14.66	−15.62	−15.73	−15.25	−14.51	−14.85	−13.59	−13.46
	500	−13.30	−15.26	−16.13	−16.36	−16.75	−16.36	−16.95	−17.91
	700	−14.61	−16.96	−16.94	−17.91	−22.68	−22.31	−22.77	−22.78
Untreated sample					−7.09				

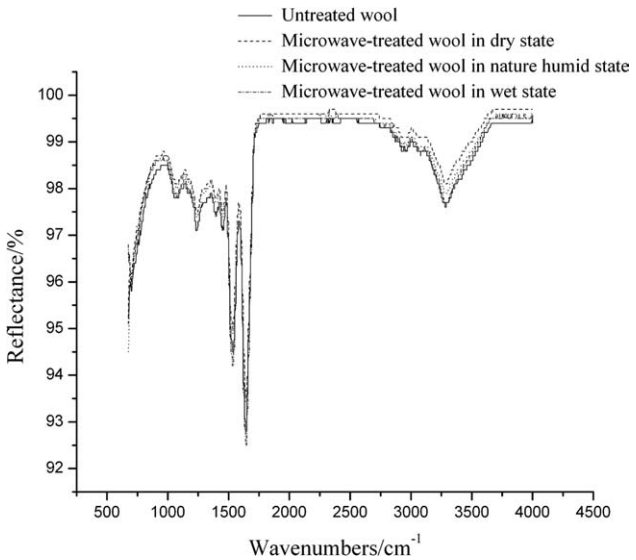

Figure 7 FTIR spectra of untreated wool and microwave-treated wool.

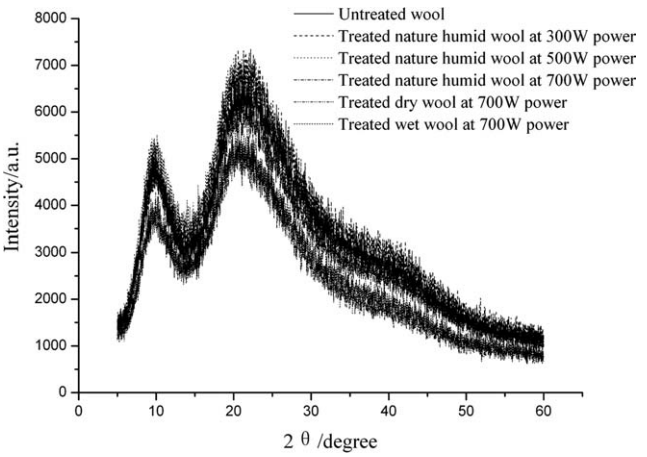

Figure 8 XRD plot of untreated wool and microwave-treated wool.

On the basis of eq. (1), the CI results of the untreated wool and microwave-treated wool fibers are listed in Table II.

It can be seen from Table II that compared with the untreated wool, the crystallnity of the treated wool in nature humid state with microwave irradiation increased, which can be attributed to the adjustment of the fine structure of wool fibers in nature humid state resulted from absorption of microwave energy. With treating power, from 300 to 700 W, the crystallnity of the wool treated slightly increased. The crystallnity of the treated wool in dry state and wet state with microwave irradiation decreased. The increase and decrease of the crystallnity of the treated wool fabric made the physical property changed.

SEM analysis

To investigate the influence of the treatment with microwave on the wool fibers surface morphological structure, experiments with wool fabric in different humid state under microwave irradiation 4 min was carried out. The scanning electron microscopy photographs of surface morphological structure of the wool samples untreated and treated with microwave are showed in Figure 9.

Figure 9 shows the microwave irradiation had an obvious damaging effect on the surface scale-like structure of wool compared with the untreated wool fabric. For wool fabric in nature humid state, after 300 W power microwave treatment, some of the escarpments on the surface of wool fibers were raised resulting in cleft lines along the scale edge; the scale edges were slightly eroded, after 500 W power microwave treatment, there are more fragments on the surface of wool fibers, which can be attributed to the etching effect caused by bombardment of the microwave. The cuticular escarpments were partially removed after 700 W power of microwave treatment for wool fabric in nature humid state and dry state, treated wool fibers in nature humid state and dry state showed much smoother surface than untreated wool. Microwave irradiation had a more serious damaging effect on the surface structure of wool in wet state than wool in nature humid state and dry state, the wool fiber in wet state lost its original scale-like structure after 700 W power microwave treatment. Although microwave irradiation had an obvious damaging effect on the

TABLE II
CI and Relative CI Values of Untreated Wool and Microwave-Treated Wool

Wool samples	I_9°	I_{14}°	CI	RCI (%)
Untreated wool	348	181	0.48	100
Treated nature humid wool at 300 W power	351	176	0.50	104
Treated nature humid wool at 500 W power	357	174	0.51	106
Treated nature humid wool at 700 W power	361	169	0.53	110
Treated dry wool at 700 W power	380	220	0.42	88
Treated wet wool at 700 W power	362	264	0.27	56

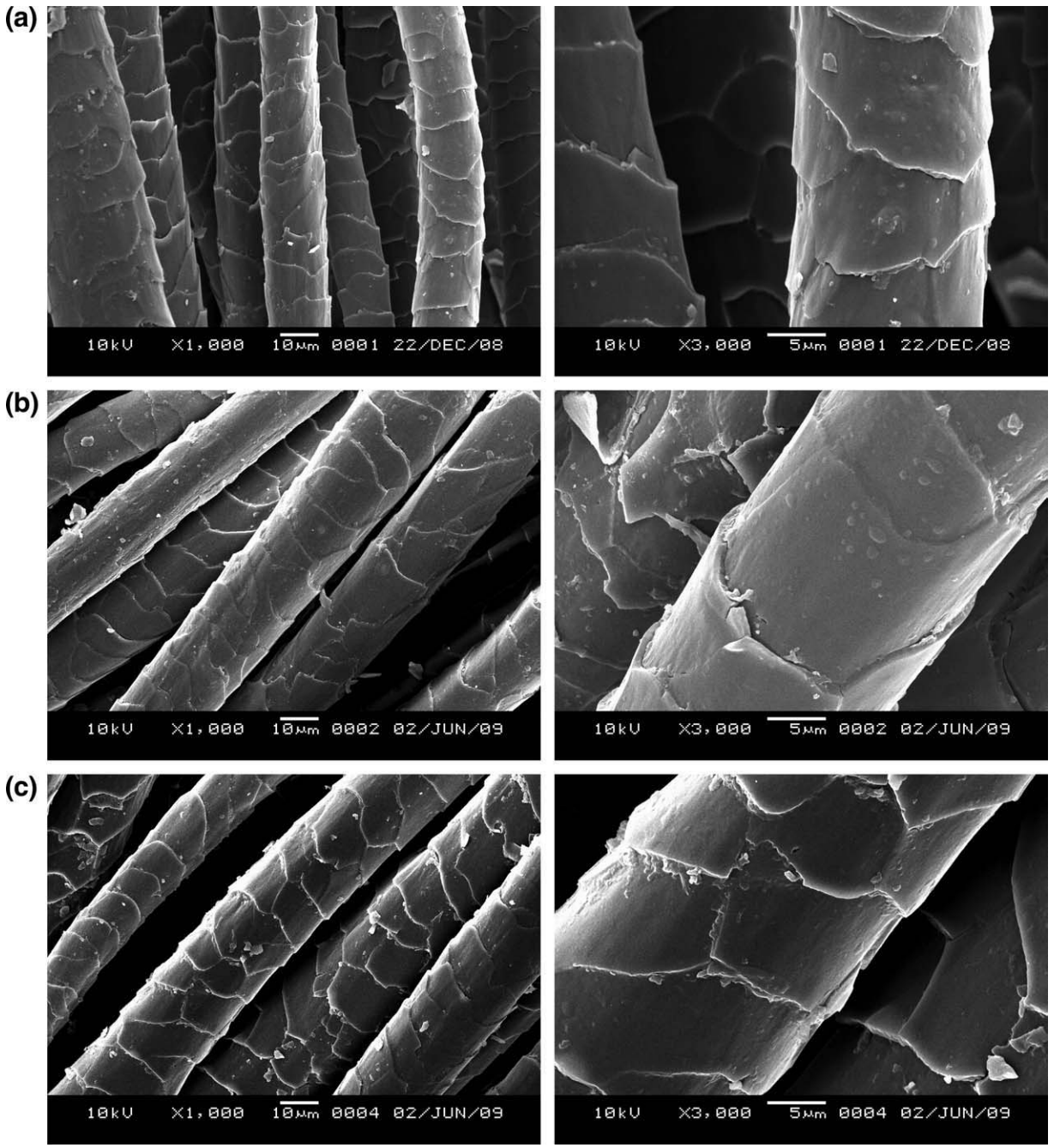

Figure 9 SEM micrographs of untreated wool and microwave-treated wool, (a) Untreated wool, (b) Treated nature humid wool at 300 W power, (c) Treated nature humid wool at 500 W power, (d) Treated nature humid wool at 700 W power, (e) Treated dry wool at 700 W power, and (f) Treated wet wool at 700 W power.

surface scale-like structure of wool, the great mass of the scales still remained on the fiber surface.

LRS analysis

To investigate the influence of the treatment with microwave on the concentration the S—S bonds, ordered degree for wool fine structure and the concentration of the a-helical conformation, experiments with wool fabric in different humid state under microwave irradiation 4 min was carried out. The Laser Raman spectroscopy curve of the wool samples untreated and treated with microwave are showed in Figure 10.

Raman spectroscopy techniques have been well-established used to analyze the structure transformation of wool fibers. The corresponding Raman spectrum of intense band component at 1652 cm^{-1} can be attributed to a-helical conformation. The peak at 1453 cm^{-1} can be considered the standard

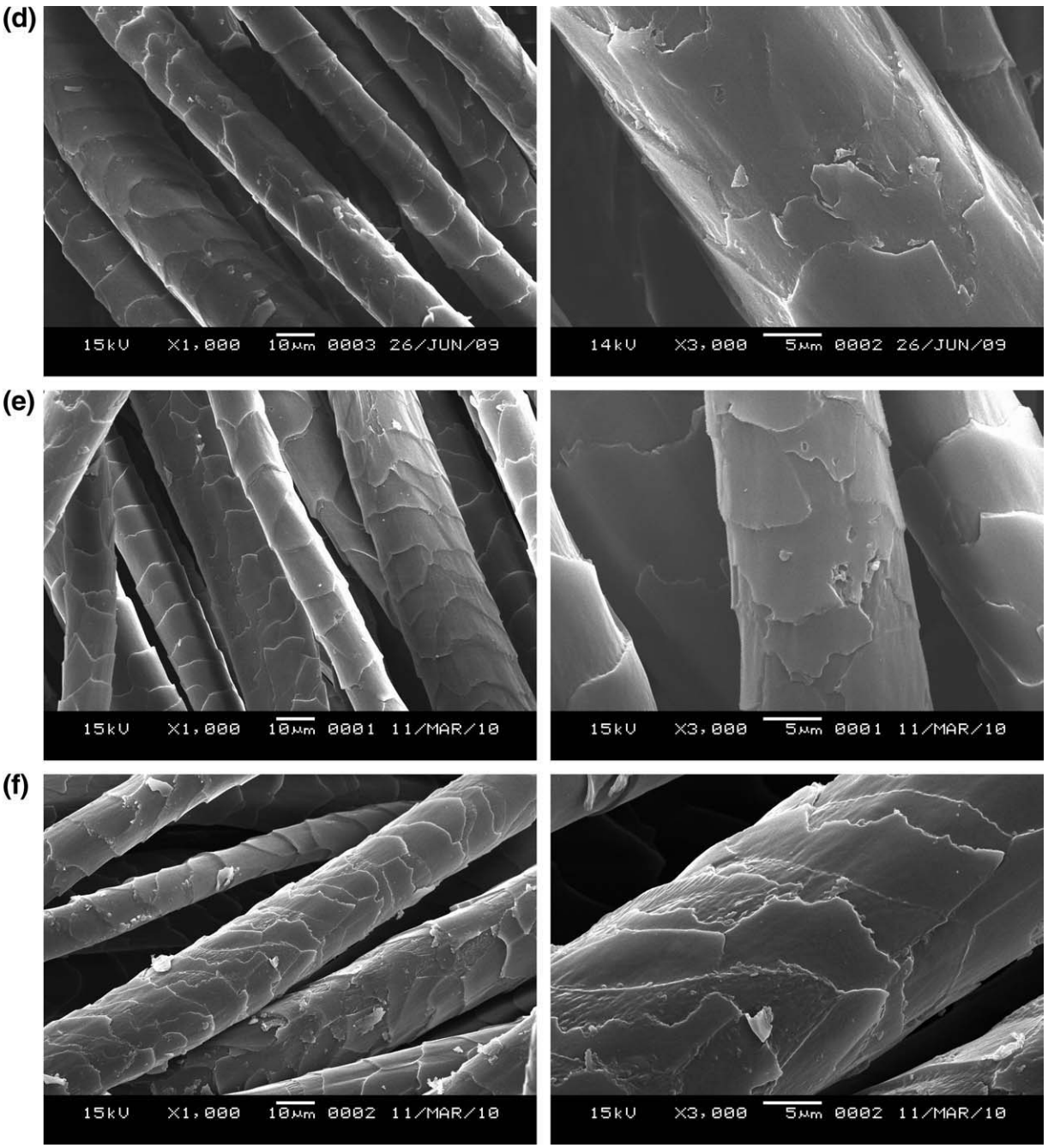

Figure 9 (*Continued*)

peak because it is largely associated with the amino acid side chains and thus not affected by the peptide backbone conformation. The intense band component at 1246 cm^{-1} can be attributed to disordered peptides. In addition, the bands near 510 cm^{-1} can be assigned to the S—S bonds.[18] The cystine S—S bond plays an important role in the tertiary intermolecular structure of keratin. According to Ref.[19–21], I_{510}/I_{1453} are used for calculating the concentration of the S—S bonds in wool fibers. The bigger the I_{510}/I_{1453}, the higher the concentration of the S—S bonds in wool fibers. The degree of disorder for fine structure is estimated with I_{1246}/I_{1453}. The bigger the I_{1246}/I_{1453}, the higher the degree of disorder for wool fine structure. I_{1652}/I_{1453} are used for calculating the concentration of the a-helical conformation. The bigger the I_{1652}/I_{1453}, the higher the a-helical conformation in wool fibers. I_{510}/I_{1453}, I_{1246}/I_{1453}, and I_{1652}/I_{1453} of untreated wool and microwave-treated wool in Raman spectroscopy is illustrated in Table III.

It can be seen from Table III that compared with the untreated wool, I_{510}/I_{1453} of the treated wool with microwave are lower. This means that the concentration of cystine S—S bonds for the treated wool fiber in different humid state is lower than that of the untreated wool fibers. The cystine S—S gradually decreases with an increasing irradiation power. This may contribute to structural rearrangement as a result of partial disulfide bond rupture. Microwave treatment of the wool fibers resulted in the reduction of the concentration the S—S bonds. I_{1246}/I_{1453} of the treated wool in nature humid state with microwave are lower than the untreated wool. I_{1246}/I_{1453} of the treated wool in dry state and wet state with microwave are higher than the untreated wool. Microwave treatment improves ordered degree for nature humid wool fine structure and decreases ordered degree for dry and wet wool fine structure, which is accorded with results of X-ray diffraction analysis. I_{1652}/I_{1453} of the treated wool fibers in nature humid state are decreased with 300 and 500 W irradiation power and increased with 700 W irradiation power compared to the untreated wool fibers. I_{1652}/I_{1453} of the treated wool in dry state and wet state with microwave are lower than the untreated wool. The change of the I_{1652}/I_{1453} of the treated wool fabric in different humid state made the concentration of the a-helical conformation increased or decreased.

TABLE III
Characteristic Raman Intensity of Untreated Wool and Microwave-Treated Wool

Wool samples	I_{510}/I_{1453}	I_{1246}/I_{1453}	I_{1652}/I_{1453}
Untreated wool	1.069	0.971	0.911
Treated nature humid wool at 300 W power	1.014	0.952	0.873
Treated nature humid wool at 500 W power	1.006	0.941	0.907
Treated nature humid wool at 700 W power	0.930	0.929	1.026
Treated dry wool at 700 W power	0.903	0.999	0.867
Treated wet wool at 700 W power	0.850	1.003	0.856

CONCLUSIONS

It can be concluded from the investigated report that microwave irradiation could impact the breaking strength and elongation of different humid state wool fabrics. The whiteness of wool fabrics subjected to microwave treatment was slightly decreased. It was also found that microwave irradiation also could affect the surface morphological structure and fine structure of wool. The chemical structure of microwave-treated wool had not obvious change. The microwave treatment caused an obvious damaging effect of the surface scale-like structure of the wool fibers. The concentration of cystine S—S bonds in microwave-treated wool fibers is lower than that of the untreated wool fibers. The crystallnity of the treated wool in nature humid state with microwave irradiation increased. The crystallnity of the treated wool in dry state and wet state with microwave irradiation decreased. After microwave irradiation, the concentration of a-helical conformation in wool fibers was also changed. Microwave heating is more efficient than conventional heating methods. During the conventional heating, the heat is generated outside the treated product and conveyed by conduction or convection. On the contrary, in microwave treatment, the heat is generated in a distributed manner inside of the material, allowing more uniform and faster heating. Microwave irradiation technique has significant potential for industrial application as microwave is a clean, environmentally friendly heating technology.

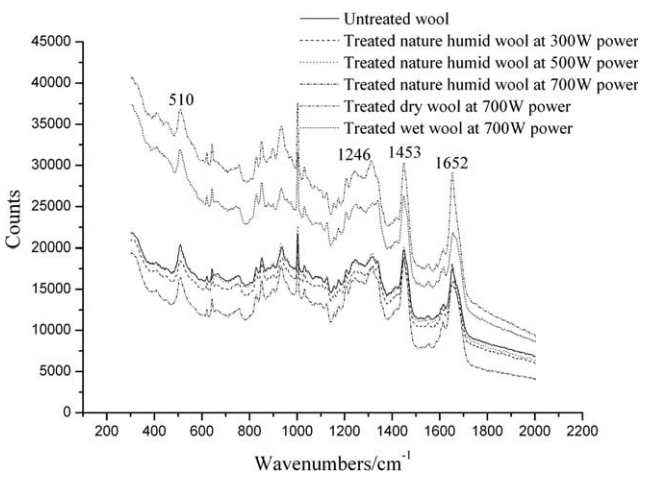

Figure 10 LRS spectra of untreated wool and microwave-treated wool.

References

1. Tsukada, M.; Islam, S.; Arai, T. Autex Res J 2005, 5, 40.
2. Yurika, Y. Seni Gakkaishi 2007, 63, 146.
3. Murugan, R.; Senthilkumar, M.; Ramachandran, T. J Inst Eng (India), Part TX: Text Eng Div 2007, 87, 23.
4. Li, J. P.; Lin, H. F.; Zhao, W. F.; Chen, G. H. J Appl Polym Sci 2008, 109, 1377.
5. Moharram, M. A.; Mahmoud, O. M. J Appl Polym Sci 2008, 107, 30.
6. Weifang, H. Application of Microwave in Textile Industry; China Textile Press: Beijing, 1990.
7. Delan, M. J. Text Chem Color 1972, 4, 119.
8. Xu, W. L.; Bao, J. J.; Zhang, J. C.; Shi, M. W. J Appl Polym Sci 1998, 70, 2343.

9. Jiang, P. Q.; Xu, W. L. J Donghua Univ (Eng Ed) 1999, 16, 74.
10. Hanna, H. L.; Abdou, L. A.; El-Khatib, E. M. Am Dyestuff Reporter 1985, 74, 42.
11. Lee, M.; Wakida, T.; Lee, M. S. J Appl Polym Sci 2001, 80, 1058.
12. Elke, B.; Ernst, C.; Eckhard, S. Rev Prog Color 2002, 32, 88.
13. Kan, C. W.; Yuen, C. W. M. J Appl Polym Sci 2006, 102, 5958.
14. Cai, Z. S.; Qiu, Y. P. J Appl Polym Sci 2008, 109, 1257.
15. Sakabe, H.; Ito, H.; Miyamoto, T.; Inagaki, H. Tex Res J 1987, 57, 66.
16. Pierlot, A. P. Tex Res J 1999, 69, 97.
17. Segal, L. Tex Res J 1959, 29, 786.
18. Liu, H. L.; Yu, W. D. J Appl Polym Sci 2007, 103, 1.
19. Salber, J.; Tillmann, W. The 10th International Wool Textile Research Conference, Aachen, 2000; FI-14.
20. Lucia, E. Text Res J 1995, 65, 593.
21. Huson, M.; Church, J.; Heintze, G. The 10th International Wool Textile Research Conference, Aachen, 2000; ST-5.