

# Modification of Wrinkle Resistance of Cotton Fabric

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Received 2 October 2004; accepted 25 June 2005

DOI 10.1002/app.23170

Published online 19 January 2006 in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** Cotton fabrics were treated with montmorillonite (MMT) so as to improve the wrinkle-resistant properties of the cotton fabrics. The MMT in emulsion form was applied by padding method to the cotton fabrics, and the wrinkle-resistant properties of the MMT-treated cotton fabrics and the wrinkle-resistant properties of MMT-treated cotton fabrics were consequently improved. Furthermore, different instrumental methods were used for studying the

distribution of MMT particles on the cotton fabric surface. It was noted that near nano-scale MMT particles were adhered on the fiber surface, and in addition, the particles size played an important role in influencing the wrinkle-resistant properties of the cotton fabric. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 99: 3700–3707, 2006

**Key words:** fibers; surfaces; nanoparticles; FTIR

## INTRODUCTION

Montmorillonite (MMT with chemical formula:  $(\text{Na,Ca})_{0.33}(\text{Al,Mg})_2\text{Si}_4\text{O}_{10}(\text{OH})_2 \cdot n\text{H}_2\text{O}$ ) is an inorganic natural clay mineral that typically exists as layered silicate minerals, which has  $\text{SiO}_4$  tetrahedral sheets arranged into a two-dimensional network structure.<sup>1</sup> The MMT can provide thermal resistance, wrinkle resistance, and antibacterial properties on textiles.<sup>1–7</sup> This paper was aimed to investigate the effect of the MMT on the wrinkle-resistant properties of the cotton fabrics. In this paper, an ultrasonic crashing machine was used for reducing the particle sizes of MMT. The reduced particles were then made into an emulsion form with dispersing agent and then padded onto the cotton fabrics. Not only physical testing was carried to evaluate the effect of the MMT treatment on the wrinkle-resistant properties of the cotton fabrics but also the instrumental methods were conducted to measure the particle sizes of the reduced MMT particles so as to characterize the surface morphology and chemical composition of the MMT-treated cotton fabrics.

## EXPERIMENTAL

### Fabric

One hundred percent twill cotton fabrics with yarn density of 50 picks/cm and 24 ends/cm were used. The fabrics had been desized, scoured, and bleached.

### Determination of the concentration of dispersing agent for preparing the MMT emulsion

Unless otherwise stated, all chemicals used for preparing the MMT emulsion were of AR grade. To prepare a good MMT emulsion, it is important that the tiny particles of MMT should be well dispersed in a medium such that the tiny particles of MMT will not be aggregated again to form large molecules. In this paper, a nonionic dispersing agent, Matexil DN-VL, with different concentrations were used for dispersing the MMT clay to form an emulsion. The emulsion was prepared by adding 1.0 g of MMT into 50 mL of dispersing agent with four different concentrations, i.e., 10, 50, 70, and 100% respectively. Each emulsion was then crashed by an ultrasonic crashing machine (Ningbo Scientz Biotechnology Co., Ltd.) for 15 min. After crashing, the behavior of the emulsions with different concentration of dispersing agent was observed carefully at different time intervals, i.e., 5, 10, 15, and 30 min.

### MMT emulsion preparation and its application on cotton fabric

MMT emulsions were prepared with 1.0 g MMT and 50 mL of 70% dispersing agent. The MMT emulsions were then subjected to an ultrasonic crashing machine to reduce the particle sizes. The emulsions were crashed to 8, 14, 20 numbers of cycles for 1, 2, and 3 h, respectively. Each crashing cycle represented that the emulsion was crashed for 99 times by the ultrasonic wave.

In addition, two more MMT emulsions were prepared that were (i) 2 mL of the binder mixed with 48 mL of the dispersing agent; and (ii) 8 mL of the binder mixed with

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**TABLE I**  
The Experimental Procedure for Preparing the MMT Emulsion and its Application on Cotton Fabric

Runs	MMT (g)	70% Matexil DN-VL (dispersing agent; mL)	Acramin SL (binder; mL)	Number of crashing cycle	Time duration (h)	Padding times	Wet pickup (%)
1	1	50	—	8	1	3	55
2	1	50	—	8	1	6	65
3	1	50	—	14	2	3	55
4	1	50	—	14	2	6	65
5	1	50	—	20	3	3	55
6	1	50	—	20	3	6	65
7	1	48	2	20	3	3	55
8	1	48	2	20	3	6	65
9	1	42	8	20	3	3	55
10	1	42	8	20	3	6	65

Padder pressure, 3 kg/cm<sup>2</sup>; speed of padder, 5 rpm; drying condition, 90°C for 10 min; curing condition, 160°C for 3 min.

42 mL of the dispersing agent. Each MMT emulsion was then subjected to crashing by the ultrasonic crashing machine to reduce the particle sizes. The emulsions were crashed up to 20 numbers of cycles for 3 h.

After preparing the MMT emulsions, the MMT emulsion of reduced particle size was padded onto the cotton fabric by a padding machine (Labortex Co. Ltd.). Because of the poor dispersing properties of the MMT particles, this padding process should be carried out as soon as possible, i.e., not longer than 30 min after finishing the crashing process, so as to prevent the reduced particles from aggregating again to form large molecules. The emulsion was padded onto the cotton fabrics by the padder, with the pressure of 3 kg/cm<sup>2</sup> and speed of 5 rpm. Two padding times were investigated, including 3 padding times and 6 padding times, and subsequently achieved the wet pickup of 55 and 65%, respectively. Finally, the MMT-treated cotton fabrics were dried at 90°C for 10 min in the oven and cured at 160°C for 3 min. Table I summarized the experimental procedures for preparing the MMT emulsion and its application on cotton fabric.

#### Evaluation of wrinkle-resistant properties of the fabrics

The wrinkle-resistant properties of the cotton fabrics were evaluated by the AATCC Test Method 66–1998.

#### Surface morphological study

The surface morphology of the MMT-treated cotton fabric was investigated by a scanning electron microscope (SEM; JEOL, Model No: JSM-6335F), with magnifications of 1000× and 30,000×.

#### Particle size analysis

Particle size analysis of the MMT emulsion was conducted in accordance with ISO 13,320–1:1999, which is

a laser diffraction method. The particle size of the MMT emulsion was measured by a Laser Diffraction Particle Size Analyzer (Beckman Coulter, Model No.: LS 13,320).

#### Chemical composition measurement

The chemical compositions of the treated fabrics were studied using a Perkin–Elmer System 2000 of Fourier transform infrared spectrophotometer (FTIR), with the scanning range between 4000 and 650 cm<sup>-1</sup>. The characteristic infrared bands of silicon compounds to be studied are summarized in Table II.

## RESULTS AND DISCUSSION

#### Dispersing properties of MMT

Since MMT clay was poorly dispersed in water, thus dispersing agent was required to form an emulsion with these particles. The dispersing properties of MMT with four different concentrations of dispersing agent are summarized in Table III.

The results in Table III clearly showed that the MMT clay could be well dispersed and stable for a longer

**TABLE II**  
Characteristic Infrared Bands of Silicon Compounds<sup>8,9</sup>

Frequency (cm <sup>-1</sup> )	Assignment
3700–3200	Si—OH stretching
2250–2100	Si—H stretching
1280–1255	Si—CH <sub>3</sub> symmetric deformation
1250–1200	Si—CH <sub>2</sub> —R stretching
1150	Si—C <sub>6</sub> H <sub>5</sub>
1130–1000	Si—O—Si asymmetric stretching
1110–1000	Si—O—R asymmetric stretching
970–920	Si—O—C <sub>6</sub> H <sub>5</sub>
950–800	Si—H stretching
860–760	Si—C stretching

**TABLE III**  
**The Dispersing Properties of MMT with Four Different Concentrations of Dispersing Agent at Different Time Duration**

Time (min)	Concentration of dispersing agent (%)			
	10	50	70	100
5	The particles started to sediment within 1 min and the emulsion started to separate into two layers after 4 min	The particles started to sediment after 5 min	The particles were well dispersed within 5 min	The particles were well dispersed within 5 min
10		The emulsion started to separate into two layers after 10 min	The particles were well dispersed within 10 min	The particles were well dispersed within 10 min
15		After 15 minutes, the emulsion was separated into two clear layers	The particles were well dispersed within 10 min	The particles were well dispersed within 10 min
30			The emulsion started to separate into two layers after 30 min	The emulsion started to separate into two layers after 30 min

time in dispersing agent with concentration of 70–100%. However, 100% dispersing agent was too viscous, and therefore, the dispersing agent with concentration of 70% was chosen and used.

#### Wrinkle-resistant properties of the fabrics

The wrinkle-resistant properties of the MMT-treated fabrics have been evaluated and the results obtained are shown in Table IV.

From Table IV, it was obvious that wrinkle-resistant properties of the MMT-treated fabrics in runs 1–4 did not have significant improvement when compared with those of the untreated fabric. However, when increasing the number of crashing cycles to 20 for 3 h as shown in runs 5 and 6, the wrinkle-resistant properties of the MMT-treated fabrics were improved. The recovery angle of the fabric treated with 3 times of padding increases 4.7%, while that of the fabric treated with 6 times of padding increases 6.0%. Therefore, it revealed that a longer crashing time used, better

would be the wrinkle-resistant properties of the cotton fabrics obtained.

When 2 mL of binder was added to the MMT emulsion as shown in runs 7 and 8, the recovery angle of the fabric treated with 3 times of padding increases 7.0%, while that of the fabric treated with 6 times of padding increases 8.8%. On the other hand, if 8 mL of binder was added to runs 9 and 10, the recovery angle of the fabric treated with 3 times of padding increases 9.1%, while that of the fabric treated with 6 times of padding increases 10.4%. These results clearly showed the addition of binder in the MMT emulsion could significantly improve the wrinkle-resistant properties of the cotton fabrics. It further addressed that a larger amount of binder used would impart a better wrinkle-resistant properties to the cotton fabrics. The reason might be due to the presence of the binder, which could enhance the coating of the MMT particles on the cotton fabric, and hence more MMT particles could be attached on the cotton fiber surfaces. Generally speaking, the MMT treatment could improve the wrinkle-resistant properties of cotton fabric to certain extent, depending on the application conditions.

**TABLE IV**  
**Wrinkle-Resistant Properties of MMT-Treated Fabrics**

Run	Times of padding	Recovery angle (W + F; °)	% Improvement
Untreated	—	212.0	—
1	3	212.0	0
2	6	214.0	0.9
3	3	214.0	0.9
4	6	215.3	1.6
5	3	222.0	4.7
6	6	224.7	6.0
7	3	226.7	7.0
8	6	230.7	8.8
9	3	231.3	9.1
10	6	234.0	10.4

#### Surface morphological study of MMT-treated cotton fabric

Figures 1–7 demonstrated a series of SEM images of (i) the untreated fabric (Fig. 1), (ii) MMT-treated fabric without binder (Figs. 2 and 3) and (iii) MMT-treated fabric with 2 or 8 mL binder as shown in the runs 4–10 (Figs. 4–7) with the magnification of 1000× respectively. The SEM results of the cotton fabrics treated with binder clearly showed that the fibers are coated with large amount of substances forming crosslinkages between the adjacent fibers. This phenomenon does not exist in both the untreated fabric and the

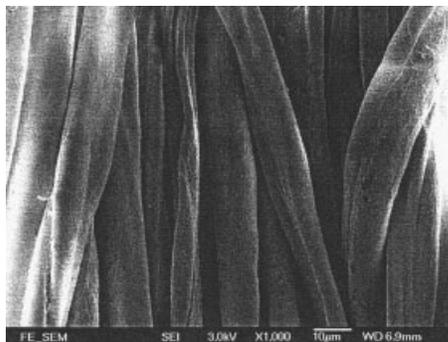


Figure 1 SEM image of untreated fabric (×1000).

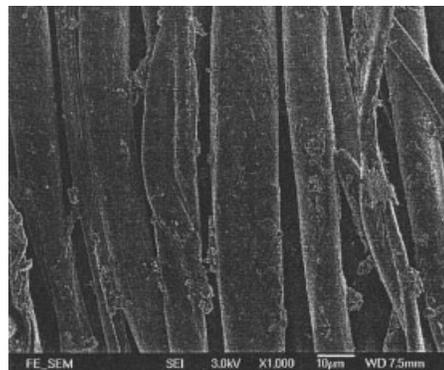


Figure 4 SEM image of MMT-treated fabric with 2 mL binder after 3 times of padding (×1000).

fabrics treated without binder. Hence, it was believed that the binder could enhance the coating of the MMT particles on the cotton fiber surface, resulting in affecting the wrinkle-resistant properties of the treated fabrics as shown in the runs 4–10. In addition, when the padding times were increased from 3 to 6, more MMT particles could be coated on the fiber surface.

Furthermore, Figures 8–14 demonstrated the set of SEM images of (i) the untreated fabric (Fig. 8), (ii) MMT-treated fabric with no binder (Figs. 9 and 10),

and (iii) MMT-treated fabric with 2 or 8 mL binder as shown in the runs 5 to 10 (Figs. 11–14) with a magnification of 30,000×, respectively. All SEM images revealed that some tiny particles of around 100 nm, which are at nano-particle size,<sup>1,3,6</sup> are attached on the fiber surfaces of the MMT-treated fabrics when compared with that of the untreated fabric. These tiny particles could be regarded as nano-MMT particles. In addition, larger particles (>100 nm) are also present

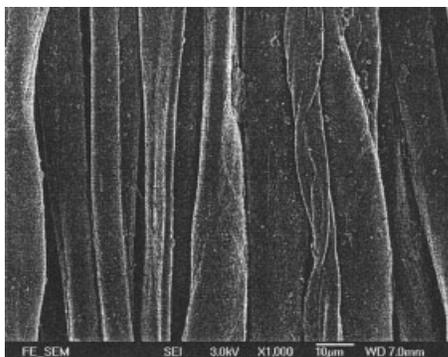


Figure 2 SEM image of MMT-treated fabric with no binder after 3 times of padding (×1000).

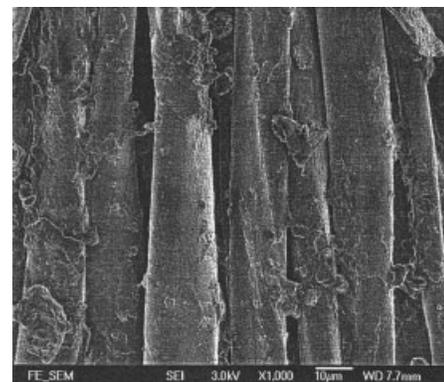


Figure 5 SEM image of MMT-treated fabric with 2 mL binder after 6 times of padding (×1000).

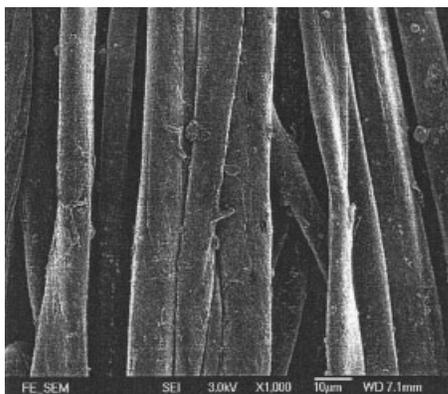


Figure 3 SEM image of MMT-treated fabric with no binder after 6 times of padding (×1000).

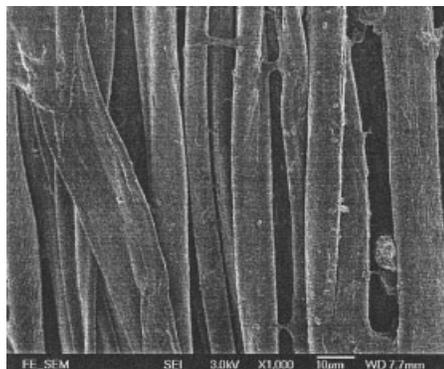
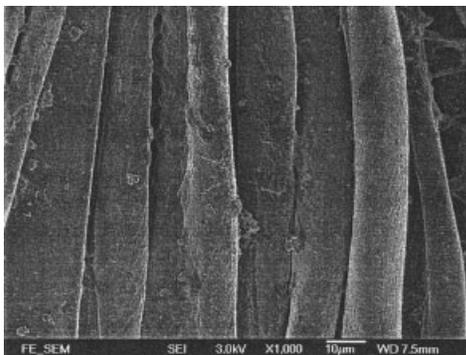


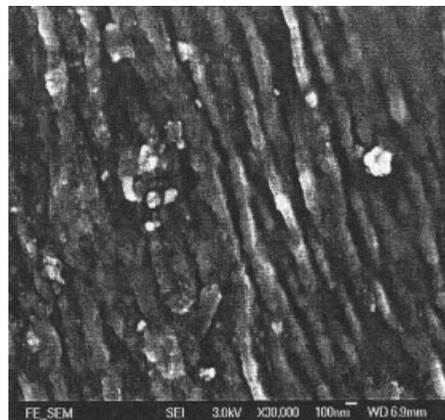
Figure 6 SEM image of MMT-treated fabric with 8 mL binder after 3 times of padding (×1000).



**Figure 7** SEM image of MMT-treated fabric with 8 mL binder after 6 times of padding ( $\times 1000$ ).

on the fiber surfaces, which may be considered as micro-MMT particles.

Figures 11–14 demonstrated that there are more MMT-particles present on the fiber surfaces of the fabrics, which were treated with binder. However, only a small amount of them is present on the fiber surfaces of those treated without binder as shown in Figures 9 and 10. Hence, the binder can be regarded as

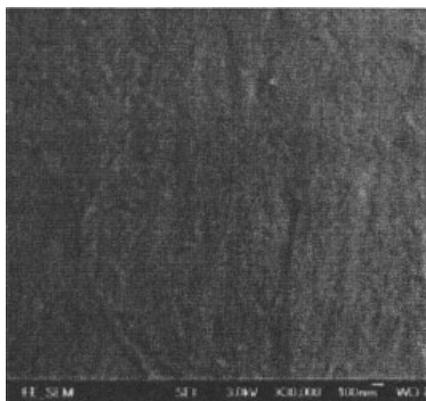


**Figure 10** SEM image of MMT-treated fabric with no binder after 6 times of padding ( $\times 30,000$ ).

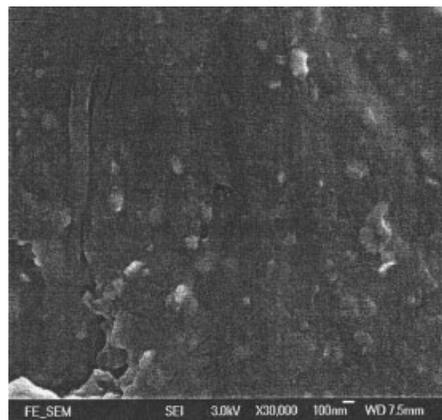
a medium enhancing the attachment of MMT particles in the cotton fiber surface.

#### Particle size analysis

The size distribution of the particles in the MMT emulsions was shown in Figure 15, in which the majority of



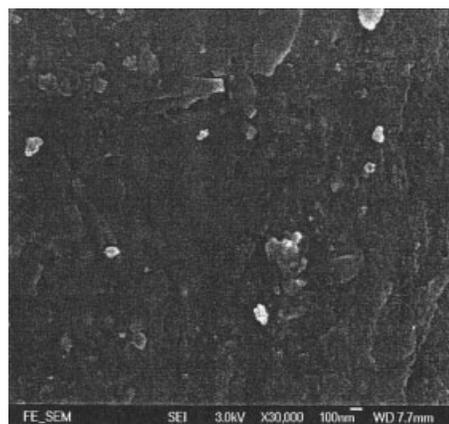
**Figure 8** SEM image of untreated fabric ( $\times 30,000$ ).



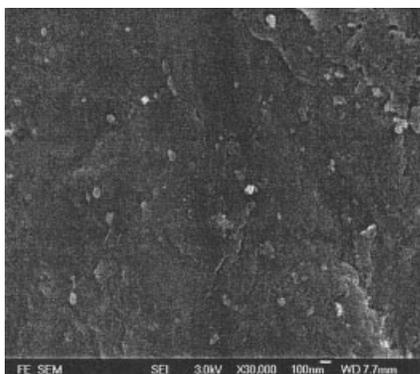
**Figure 11** SEM image of MMT-treated fabric with 2 mL binder after 3 times of padding ( $\times 30,000$ ).



**Figure 9** SEM image of MMT-treated fabric with no binder after 3 times of padding ( $\times 30,000$ ).



**Figure 12** SEM image of MMT-treated fabric with 2 mL binder after 6 times of padding ( $\times 30,000$ ).



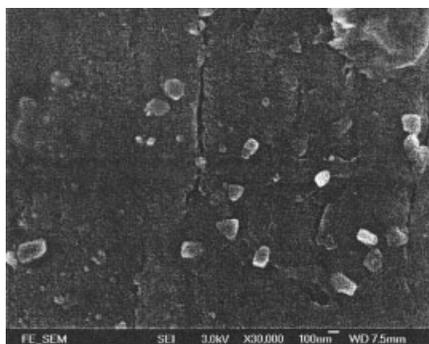
**Figure 13** SEM image of MMT-treated fabric 8 mL binder after 3 times of padding ( $\times 30,000$ ).

the particle size in the MMT emulsion is around  $0.4 \mu\text{m}$ , i.e.,  $400 \text{ nm}$ .

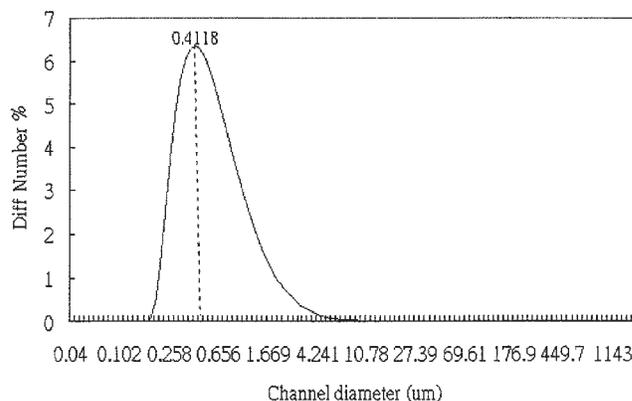
Although the SEM results proved that some nanoparticles attached on the fiber surfaces, the result of the particle size analysis revealed that the majority of the particle sizes in the MMT emulsion could reach to a near nano-particle scale. This may be a reason to explain why the MMT treatment in the present experimental condition cannot give a very significant wrinkle-resistant effect on the cotton fabrics. In addition, the inadequate crashing cycles and time, i.e., 20 crashing cycles for 3 h, would be another reason for reducing the MMT clay particles to nano-scale. As shown in Table IV, it was noted that when the crashing cycles and times increased, the wrinkle-resistant properties of MMT-treated cotton fabric increased correspondingly. Therefore, if crashing cycles and times were prolonged, the wrinkle-resistant properties would be further improved.

#### FTIR spectroscopy analysis

The FTIR spectra of both the untreated fabric and the MMT-treated fabrics without the use of binder were shown in Figure 16. The FTIR results obtained from



**Figure 14** SEM image of MMT-treated fabric with 8 mL binder after 6 times of padding ( $\times 30,000$ ).



**Figure 15** Result of particle size analysis.

the MMT-treated fabrics show that three absorption bands appear at  $802.4$ ,  $841.3$ , and  $1107.4 \text{ cm}^{-1}$  but none of them exist in the untreated fabric.

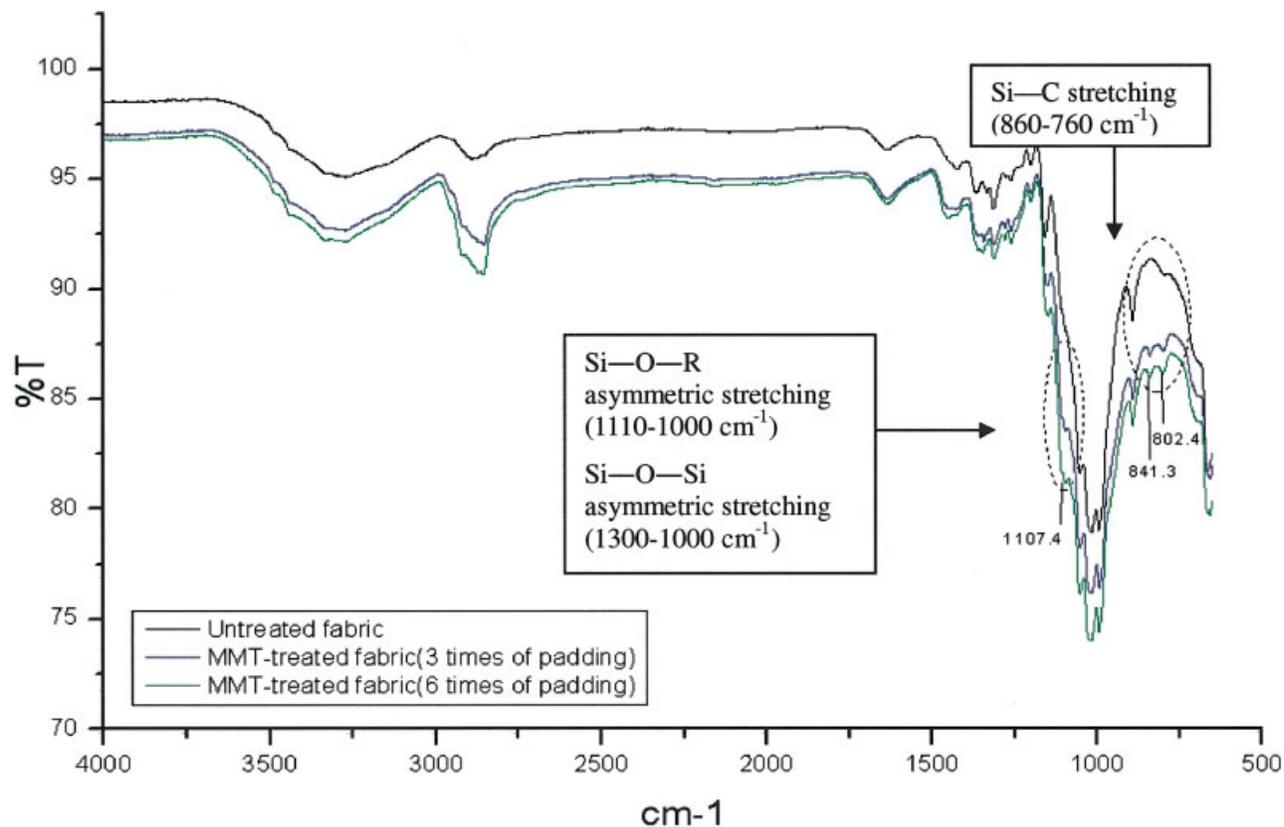
According to Table II, the MMT-treated fabrics are characterized to have Si—C stretching at  $802.4$  and  $841.3 \text{ cm}^{-1}$ , and Si—O—R asymmetric stretching and Si—O—Si asymmetric stretching at  $1107.4 \text{ cm}^{-1}$ . Since the major component of the MMT is silicon oxide, it is believed that such absorption bands are produced by the attachment of the MMT particles on the treated fabric. The interactions of MMT particles with cotton fibers could explain why the wrinkle-resistant properties of the treated fabrics were improved.

In addition, the absorption bands of Si—C stretching, Si—O—R asymmetric stretching, and Si—O—Si asymmetric stretching are stronger as the times of padding were increased. This implies that the amount of MMT particles attached on the treated fabrics is increased by the higher pickup percentage.

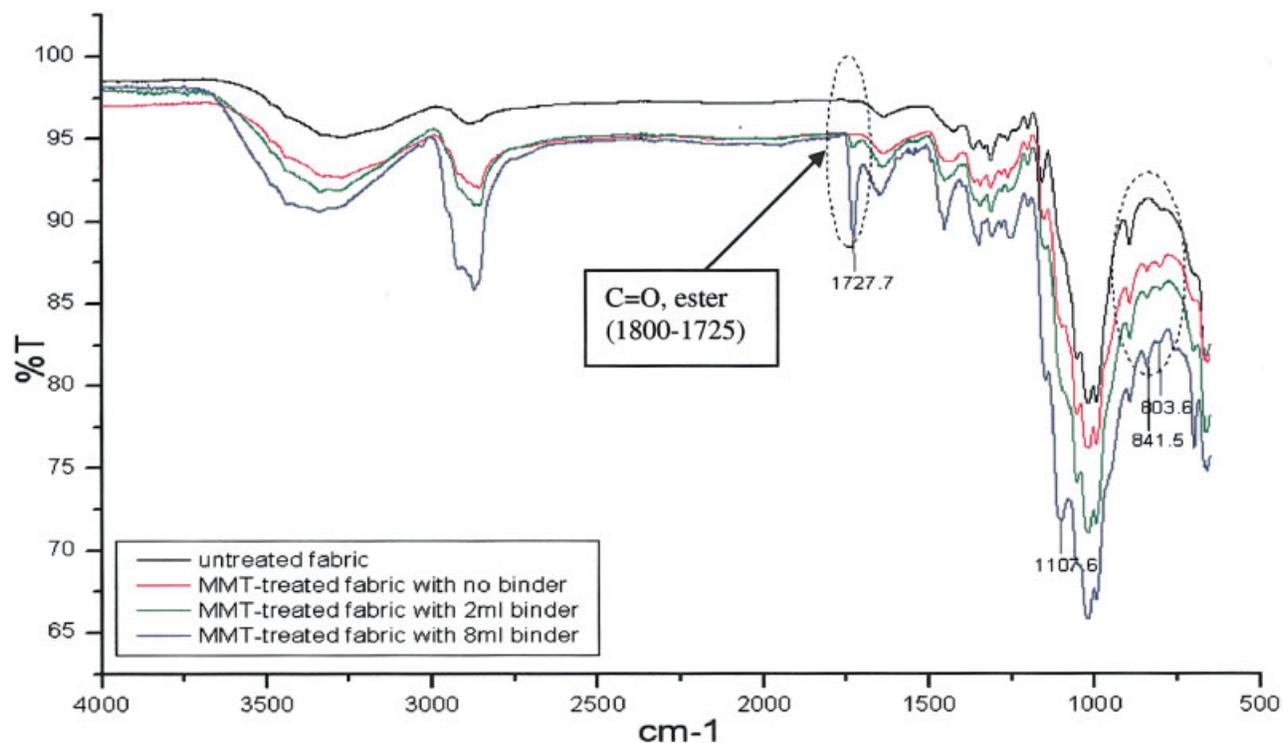
Figure 17 showed the FTIR spectra of the MMT-treated fabric with or without binder after 3 times of padding, while Figure 18 showed those of the MMT-treated fabric with or without binder after 6 times of padding.

The FTIR results obtained from the fabrics treated with binder show that an absorption bands appears at around  $1727$ – $1728 \text{ cm}^{-1}$ , but none of them exist in the untreated fabric and the fabrics treated with no binder. This band is characterized to be C=O bond of ester compound. The absorption band becomes stronger as the amount of the binder used is increased. Therefore, it is believed that this absorption band is produced by the addition of binder.

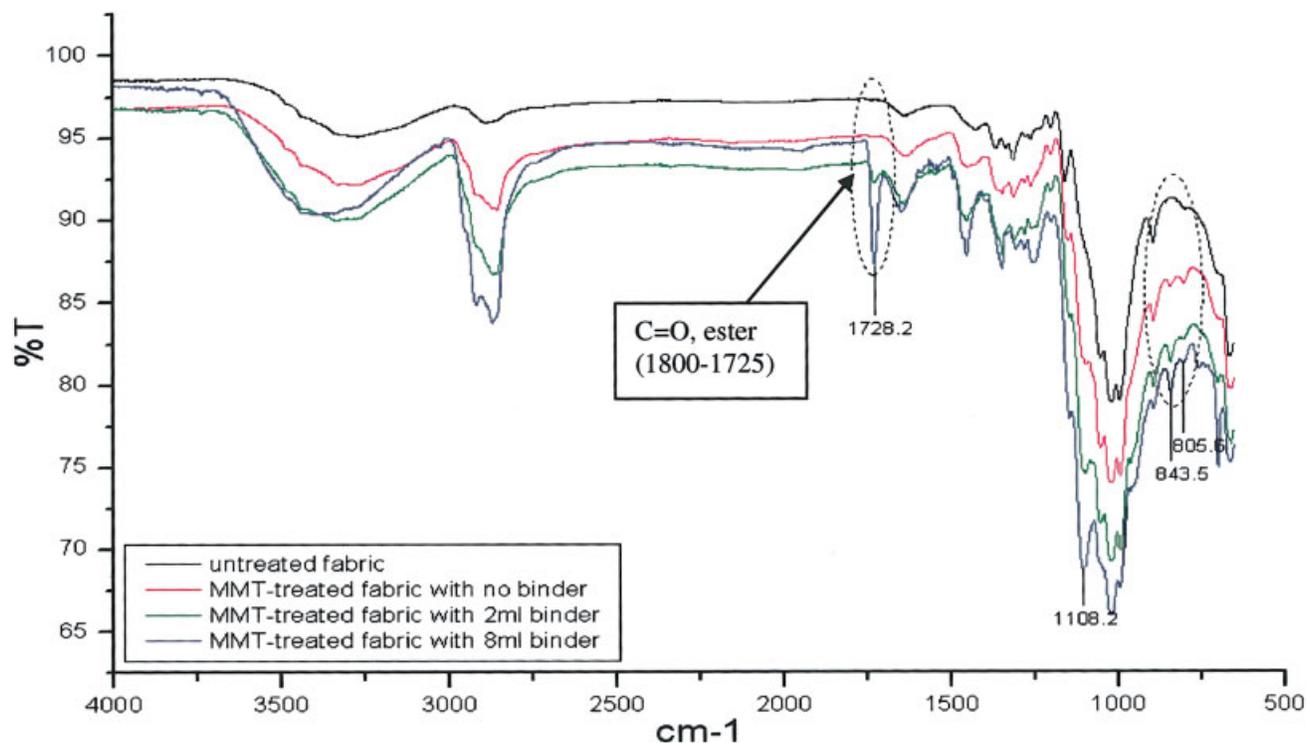
Furthermore, the absorption bands at  $803.6$ – $805.6$ ,  $841.5$ – $843.5$ , and  $1107.6$ – $1108.2 \text{ cm}^{-1}$  have been characterized as Si—C stretching, Si—O—R asymmetric stretching, and Si—O—Si asymmetric stretching, respectively. These absorption bands of the fabrics treated with binder are stronger than those treated without binder. In addition, the bands become stron-



**Figure 16** FTIR results of the MMT-treated fabrics without binder. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



**Figure 17** FTIR results of the MMT-treated fabrics (3 times of padding). [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



**Figure 18** FTIR results of the MMT-treated fabrics (6 times of padding). [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

ger as the amount of binder used is increased. This can explain why more MMT particles can be attached on the fiber surfaces using a larger amount of binder.

### CONCLUSIONS

In this paper, investigation had been conducted to study the effect of the MMT on the wrinkle-resistant properties of cotton fabrics. The sizes of the MMT particles were reduced by the ultrasonic crashing machine, with a condition of 20 crashing cycles for 3 h and the effect of the binder on the MMT treatment has also been investigated.

Recovery angle test has been used for evaluating the wrinkle-resistant properties of the MMT-treated fabrics. The test results showed that the wrinkle-resistant properties of the MMT-treated fabric were improved much in the presence of binder.

SEM, particle size analysis method, and FTIR spectroscopy had also been used for characterizing the MMT emulsion and the MMT-treated fabrics. The SEM results characterized that MMT particles with nano-particle size were attached on the fiber surfaces of the MMT-treated fabrics. However, the result of the particle size analysis revealed that the majority of the particle sizes of MMT in MMT emulsion were around 400 nm, which were near nano-scale.

The FTIR results demonstrated that the MMT particles could establish the interactions with the cotton

fibers. These interactions were also intensified by the addition of binder and the increase in the pickup percentage. However, the particle size of the MMT was the major factor to affect the wrinkle-resistant treatment for cotton fabric in this paper. The possible reason for the particle size problem might be due to inadequate crashing cycles and time. Twenty crashing cycles for 3 h was actually not enough to reduce the MMT particles to the nano-scale.

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