Carbohydrate Polymers 74 (2008) 934–937

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Effect of microwave irradiation on the physical properties and morphological structures of cotton cellulose

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ARTICLE INFO

Article history:
Received 29 April 2008
Accepted 20 May 2008
Available online 27 May 2008

Keywords:
Cotton fabric
Microwave
Crystallinity
Preferred orientation

ABSTRACT

Microwave heating has been proved to be more rapid, uniform and efficient, and easily penetrate to particle inside. To investigate the effect of microwave irradiation on the physical property and morphological structure of cotton cellulose, cellulose fabric was treated with microwave irradiation at different conditions. The physical properties of the treated cellulose fabric were investigated. The morphological structures and thermal stabilities of the untreated and treated cellulose were investigated with differential scanning calorimetry (DSC) and X-ray diffraction. The results show that the physical properties of the treated cellulose fabrics were improved and the recoverability had not significant change. The thermal stability of the treated cellulose was changed. The crystallinity and preferred orientation of the treated cotton cellulose increased.

1. Introduction

Cellulose is one of the most widespread natural polymers. For all the possible chemical modifications of natural products, the reactions based on cellulose are the most important (Antova, Vasvasova, & Zlatanov, 2004; Xie, Hou, & Sun, 2007a; Xie, Hou, & Wang, 2008). Much attention has been paid on modification and utilization of cellulose, due to its good biodegradability and biocompatibility. Modifications of cellulose with graft copolymer or compounds expand the applications as functional hybrid materials. In recent years, modifications and dry of some materials have been conducted under microwave irradiation condition (Liu, Li, Fang, & Chen, 2005; Nakai, Yoshimizu, & Tsujita, 2005; Nurjaya & Wong, 2005). Microwave irradiation is one of powerful techniques of non-contact heating, because the dielectric substances with large dielectric loss constant vigorously fever by vibration and rotation of permanent dipoles in microwave field. Microwave has been used for reacting, heating and drying cellulose materials.

Microwave-assisted organic synthesis (Biswal et al., 2007; Takano, Ishikawa, Kamitakahara, & Nakatsubo, 2007) has gained popularity in recent years because microwave irradiation was found to accelerate remarkably a wide variety of reactions. Particularly, a solvent-free microwave-assisted reaction provides an opportunity to work with open vessels thus avoiding the development of high pressure and provides a possibility of up-scaling the reaction on a preparative scale and helps the induction of the reaction under dry conditions. Considerable efforts have also been devoted to investigate the modification of polymer materials (Satge et al., 2002; Shogren & Biswas, 2006).

The microwave irradiation has been used in the dyeing processing of cellulose fabric. In the conventional processing of fabric, a large amount of energy is consumed. Some new techniques and methods for saving energy were investigated (Liu, Li, Li, & Fang, 2004; Xie, Liu, & Li, 2007b, 2007c). 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 and morphological structure of cellulose, including some physical properties. The report of the effect of microwave irradiation on the physical properties and morphological structure of cellulose was scarce.

In this paper, cellulose fabric was treated with microwave irradiation. The effect of microwave irradiation on the morphological structure of cellulose was investigated with X-ray diffraction (XRD). The thermal stability and physical properties of the treated cellulose was also discussed.

2. Experimental

2.1. Materials

Desized, scoured and bleached cotton cellulose fabric was obtained from Beijing Textile Company (Beijing, China).

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doi:10.1016/j.carbpol.2008.05.011

2.2. Microwave irradiation treatment of cellulose fabrics

A microwave dyeing pilot, YK-01, used in this study had a total capacity of 800 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.

The cellulose fabrics were immersed in the water bath at certain temperature, the liquor ratio being 1:10. The samples were irradiated different time, 1, 10 and 30 min, at different temperatures, respectively. After irradiation, the fabrics were removed and slowly cooled under vacuum for 24 h.

2.3. Physical property measurements

Fabric tensile strength was determined by using a H10KS Tensile Testing Machine (Hounsfield SDL Co.). Six specimens (three for warp and three for weft) were tested at a gauge length of 200 mm with a stain rate of 30 mm/min. The width of the specimen was 50 mm. The tensile and tear properties of the fabric were measured according to ISO13934.1-1994 and ISO 13937.1-1995, respectively.

The conditional wrinkle recovery angle (WRA) was measured by using a P500570 (SDL. Co.) according to AATCC Test Method 66-2003.

2.4. XRD

The untreated and treated cotton fabrics were cut into powder and carded into loose fibers, respectively. The X-ray diffraction patterns of the samples were measured with a D/max-2550 PC X-ray Diffractometer (Rigaku Corporation, Japan), which used Cu-K target at 40 kV, 300 mA and $\lambda = 1.54056$ Å.

2.5. DSC

A DSC 822e differential scanning calorimeter (Mettler/Toledo, Greifensee, Switzerland) was used. Samples of about 5 mg, placed in a DSC pan, were heated from 25 to 400 °C at a scanning rate of 10 °C/min, under a constant flow of dry nitrogen.

3. Results and discussion

3.1. Physical properties of the cotton cellulose fabric treated with microwave

Physical properties of the cotton cellulose fabric treated with microwave irradiation may be changed. Treating temperature and time under microwave irradiation condition may also impact the physical properties of cotton fabric. The tensile and tearing strength of the cotton cellulose fabrics untreated and treated with microwave at different temperature and time are presented in Tables 1 and 2, respectively. Table 1 shows that compared with the untreated cellulose fabric, the tensile strength of the treated cellulose with microwave irradiation was higher and increased with treating temperature and time. It can be seen from Table 2 that tearing property of the treated cellulose fabric with microwave irradiation was also higher and increased with treating temperature and time. These changes of physical properties were mainly because the crystallinity of the cellulose fabric treated with microwave might be increased.

The wrinkle recovery angle (WRA) of the cellulose fabrics untreated and treated with microwave is shown in Table 2. It exhibits that the WRA of the treated cellulose fabric was smaller than that of the untreated cellulose fabric. But the WRA of the treated cellulose fabric had not significant increase with treating temperature and time. It is found that the treatment of cellulose fabric with microwave irradiation had not significant influence on the recoverability.

Table 1
Tensile property of the cotton fabrics untreated and treated with microwave irradiation

Temperature (°C)	Time (min)	Tensile strength (N)	Elongation at break (mm)
40	1	397.0	24.255
	10	404.7	22.842
	30	415.3	21.842
60	1	418.7	21.704
	10	427.5	20.964
	30	434.8	20.502
Untreated sample		385.1	25.531

Table 2
Tearing and WRA properties of the cotton fabrics untreated and treated with microwave irradiation

Time (min)	Temperature (°C)	Tearing strength (N)	WRA (°, w + f)
1	40	16.16	183.8
	50	17.33	184.6
	60	18.73	186.2
10	40	17.31	190.8
	50	18.44	191.8
	60	18.06	190.2
Untreated sample		16.02	209.4

3.2. Thermal properties

Fig. 1 shows the DSC curve of the untreated cellulose fabric. Figs. 2 and 3 show the DSC curves of the cellulose fabrics treated 1 and 10 min at 60 °C with microwave irradiation, respectively. For the untreated cellulose fabric, the endothermic peak initiated at 329.66 °C, finished at 380.36 °C, the major endothermic peak at 362.99 °C, the enthalpy of decomposing 167.03 J/g. For the cellulose fabric treated 1 min at 60 °C with microwave, the endothermic peak initiated at 327.56 °C, finished at 368.65 °C, the major endothermic peak at 353.55 °C, the enthalpy of decomposing 274.68 J/g. For the cellulose fabric treated 10 min at 60 °C with microwave, the endothermic peak initiated at 320.21 °C, finished at 365.34 °C,

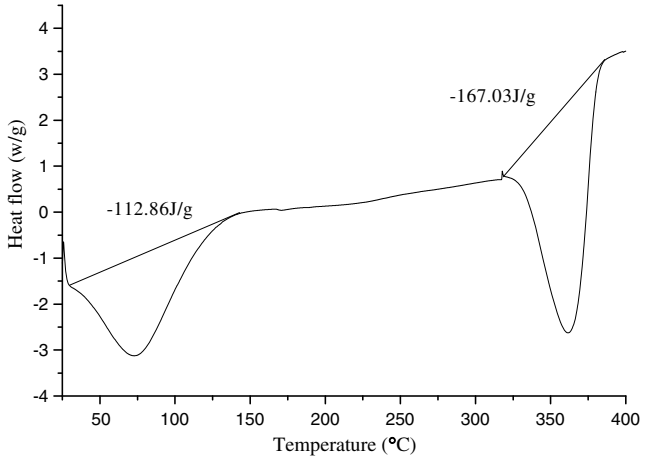

Fig. 1. DSC plot of the untreated cotton.

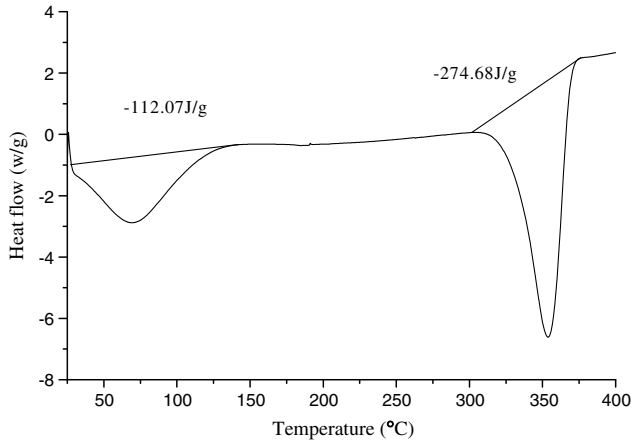

Fig. 2. DSC plot of the cotton treated 1 min at 60 °C with microwave.

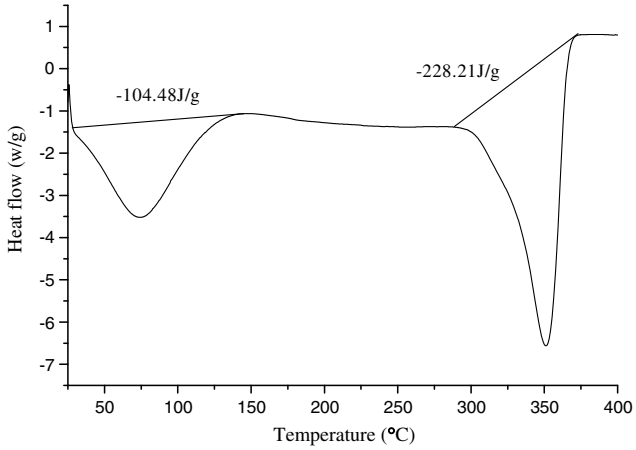

Fig. 3. DSC plot of the cotton treated 10 min at 60 °C with microwave.

the major endothermic peak at 351.06 °C, the enthalpy of decomposing 228.21 J/g. The position of the endothermic peak of the treated fabric was lower than that of the untreated cellulose fabric, and decreased slightly with treating time. But the enthalpy of decomposing of the treated fabric was much higher than that of the untreated cellulose fabric. It means that the microwave irradiation affected the crystallinity of cellulose fabric.

3.3. XRD analysis

In order to investigate the influence of the treatment with microwave on the cellulose fiber fine structure, experiments with cellulose fabrics under microwave 1 min and 10 min at 60 °C were carried out, respectively. The X-ray diffraction analyses of the crystallinity of the cellulose samples untreated and treated 1 and 10 min at 60 °C with microwave are listed in Tables 3–5, respectively.

It can be seen that compared with the untreated cellulose, the crystallinity of the treated cellulose with microwave increased. With treating time, from 1 to 10 min, the crystallinity of the cellulose treated with microwave also slightly increased.

The preferred orientation (PO) of the cotton untreated, treated 1 and 10 min at 60 °C were measured, respectively. The PO of the cotton untreated, treated 1 and 10 min at 60 °C were 67.3%, 68.9%, 70.5%, respectively. The results show that the PO of the treated cotton was slightly increased. The increase of the crystallinity and PO of the treated cotton fabric made the physical property improve.

Table 3
Half-peak breadth of diffraction peaks, spacing *d* of diffraction planes, crystal size, and crystallinity of the untreated cotton

2θ (°)	Spacing *d* (Å)	Half-peak breadth (Å)	Crystal size (Å)	Crystallinity (%)
14.577	6.0714	1.908	42	74.27
16.437	5.3884	1.481	54	
20.360	4.3583	1.420	57	
22.554	3.9390	1.352	60	
29.849	2.9909	4.795	17	
34.089	2.6279	2.001	42	
37.414	2.4016	6.122	14	
41.820	2.1582	1.952	44	
45.709	1.9832	4.507	19	

Table 4
Half-peak breadth of diffraction peaks, spacing *d* of diffraction planes, crystal size, and crystallinity of the cotton treated 1 min at 60 °C

2θ (°)	Spacing *d* (Å)	Half-peak breadth (Å)	Crystal size (Å)	Crystallinity (%)
14.679	6.0298	1.921	42	78.16
16.466	5.3789	1.297	62	
20.311	4.3686	1.553	52	
22.647	3.9231	1.352	60	
30.246	2.9525	4.370	19	
34.209	2.6189	2.123	39	
37.660	2.3865	4.905	17	
41.835	2.1575	2.227	38	
46.516	1.9507	4.487	19	

Table 5
Half-peak breadth of diffraction peaks, spacing *d* of diffraction planes, crystal size, and crystallinity of the cotton treated 10 min at 60 °C

2θ (°)	Spacing *d* (Å)	Half-peak breadth (Å)	Crystal size (Å)	Crystallinity (%)
14.625	6.0519	2.053	39	79.33
16.453	5.3834	1.260	64	
20.372	4.3557	2.840	28	
22.590	3.9328	1.308	62	
29.489	3.0266	6.009	14	
34.113	2.6261	1.892	44	
37.448	2.3995	7.117	12	
41.743	2.1621	2.140	40	
46.433	1.9540	4.942	17	

4. Conclusions

The microwave irradiation could affect the morphological structure of cellulose. Compared with the untreated cellulose fabric, the tensile strength of the treated cellulose with microwave irradiation was higher and increased with treating temperature and time. The tearing property of the treated cellulose fabric with microwave irradiation was also higher and increased with treating temperature and time. The wrinkle recovery angle (WRA) of the cellulose fabrics was smaller than that of the untreated cellulose fabric. The physical property of the cellulose fabric treated with microwave was improved, but the recoverability had not significant change. The thermal stability of the treated cellulose was changed. The crystallinity and PO of the treated cellulose with microwave increased.

References

Antova, G., Vasvasova, P., & Zlatanov, M. (2004). Studies upon the synthesis of cellulose stearate under microwave heating. *Carbohydrate Polymers, 57*, 131–134.

Biswal, J., Kumar, V., Bhardwaj, Y. K., Goel, N. K., Dubey, K. A., Chaudhari, C. V., et al. (2007). Radiation-induced grafting of acrylamide onto guar gum in aqueous

medium: synthesis and characterization of grafted polymer guar-g-acrylamide. *Radiation Physics and Chemistry, 76*, 1624–1630.
Liu, L., Li, Y., Li, Y., & Fang, Y. (2004). Rapid N-phthaloylation of chitosan by microwave irradiation. *Carbohydrate Polymers, 57*, 97–100.
Liu, L., Li, Y., Fang, Y., & Chen, L. (2005). Microwave-assisted graft copolymerization of caprolactone onto chitosan via the phthaloyl protection method. *Carbohydrate Polymers, 60*, 351–356.
Nakai, Y., Yoshimizu, H., & Tsujita, Y. (2005). Enhanced gas permeability of cellulose acetate membranes under microwave irradiation. *Journal of Membrane Science, 256*, 72–77.
Nurjaya, S., & Wong, T. W. (2005). Effects of microwave on drug release properties of matrices of pectin. *Carbohydrate Polymers, 62*, 245–257.
Shogren, R. L., & Biswas, A. (2006). Preparation of water-soluble and water-swellable starch acetates using microwave heating. *Carbohydrate Polymers, 64*, 16–21.
Satge, C., Verneuil, B., Branland, P., Granet, R., Krausz, P., Rozier, J., et al. (2002). Rapid homogeneous esterification of cellulose induced by microwave irradiation. *Carbohydrate Polymers, 49*, 373–376.
Takano, T., Ishikawa, J., Kamitakahara, H., & Nakatsubo, F. (2007). The application of microwave heating to the synthesis of 6-amino-6-deoxycellulose. *Carbohydrate Research, 342*, 2456–2460.
Xie, K., Hou, A., & Sun, Y. (2007a). Chemical and morphological structures of modified novel cellulose with triazine derivatives containing cationic and anionic groups. *Carbohydrate Polymers, 70*, 285–290.
Xie, K., Hou, A., & Wang, X. (2008). Dyeing and diffusion kinetics of modified cellulose with triazine derivatives containing cationic and anionic groups. *Carbohydrate Polymers, 72*, 646–651.
Xie, K., Liu, Y., & Li, X. (2007b). Synthesis and properties of the novel surface-active dyes containing fluorocarbon groups. Part 1: Synthesis and dyeing properties of the novel surface-active dyes on silk fabric. *Materials Chemistry and Physics, 105*, 199–203.
Xie, K., Liu, Y., & Li, X. (2007c). Synthesis and properties of the novel surface-active dyes containing fluorocarbon groups. Part 2: Dyeing diffusion kinetics of the novel surface-active dyes on silk fabric. *Materials Chemistry and Physics, 105*, 204–207.