THERMAL PROPERTIES AND WATER REPELLENCY OF COTTON FABRIC PREPARED THROUGH SOL-GEL METHOD

by

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Cotton fabrics were treated by one-step sol-gel method. The pure silica hydrosol and phosphorus-doped hydrosol were prepared with the addition of a hydrophobic hexadecyltrimethoxysilane to decrease the surface energy of cotton fabric. The thermal properties and water repellency of treated cotton fabric were characterized by thermo-gravimetric analysis, micro combustion, limiting oxygen index, and contact angle measurement. The results showed that cotton fabric treated by phosphorus-doped silica hydrosol had excellent flame retardance, and the water repellence was apparently improved with the addition of hexadecyltrimethoxysilane.

Key words: *cotton fabric, thermal properties, water repellency, sol-gel method, phosphorus-doped*

Introduction

As a significant natural fiber, cotton is used in a large range of applications such as T-shirt, curtain and beddings [1]. The reasons why cotton appeals consumers' appetite are its excellent properties, including softness, air permeability, hygroscopicity, and comfortableness. However, some drawbacks confine its further application and development. On the one hand, it can be easily ignited and rapidly burns out, which may cause fire disaster and threaten people's life and wealth. On the other hand, the concentrated hydroxyl groups on cotton surface make the fabrics easily be stained by dirty water. Consequently, it is necessary to improve the flame retardance and hydrophobicity of cotton fabrics for some specific application.

As an effective surface modification technology, sol-gel method can endow textiles with water repellency, flame retardancy, UV protection and antimicrobial property, which has been proved by the researchers' massive investigations [2-5]. In order to improve the flame resistance and hydrophobicity of cotton fabric, a one-step sol-gel process was used in this work. The pure silica hydrosol and phosphorus-doped silica hydrosol was prepared via sol-gel method with methyltrimethoxylsilane and tetraethoxysilane as precursors and ammonium biphosphate as an additive in the presence of acid catalyst and cationic/nonionic surfactants in aqueous solution. Then, the synthesized sol was modified by hexadecyltrimethoxysilane (HDTMS) to decrease the surface energy of cotton fabric. The cotton fabric was dipped and padded with the prepared hydrosol and cured under suitable temperature. The thermal property, combustion

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stability and water repellency of cotton fabric treated with silica hydrosol (cotton_Si) and phosphorus-doped silica hydrosol (cotton_Si/P) were investigated.

Experimental analysis

Thermal gravimetric analysis

The thermal stability of the treated and untreated cotton fabrics was evaluated by thermal gravimetric analysis from 50-600 °C with the heating rate of 10 °C per minute in nitrogen atmosphere. Figure 1 illustrates the thermogravimetric analysis (TG) curves of pure cotton (cotton_control), cotton_Si, and cotton_Si/P. It can be seen that all of them had only one thermal degradation step in nitrogen atmosphere. The maximum mass loss of them involved two simultaneous conditions: the dehydration of glycosyl units and the deploymerization of such units into volatile species. In case of cotton_Si/P, there was a fast decrease period around 200-300 °C, then the weight loss rate became slow and finally the weight residue remained the value of 29.7%, which was higher than cotton_control 13.3% and cotton_Si 11.4%, tab.1. This result indicated that phosphorus-doped silica coating contributed to the formation of physical char barrier which can prevent the heat transfer and limit the production of inflammable substances, then retarding the hydrolysis reactions. However, compared with cotton_control, a slight sensitiveness was found for treated cotton [6] (*e. g*. 320 °C for cotton_control and 296 °C for cotton_Si, both of the values measured at $T_{onset\ 10\%}$). Additionally, cotton_Si did not present satisfactory result owing to the existence of flammable long chains (16 C) which would accelerate the degradation of cotton fabric.

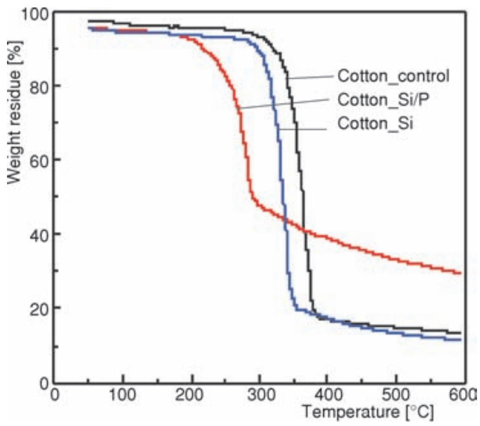

Figure 1. The TG curves of cotton samples

Table 1. The TG data of cotton samples

Sample	$T_{onset\ 10\%}$ [°C]	Residue at 600 °C [%]
Cotton_control	320	13.3
Cotton_Si	296	11.4
Cotton_Si/P	217	29.7

Micro combustion performance

The flame behavior of cotton samples was measured via micro combustion calorimeter and limiting oxygen index instrument. The results of heat release capacity (HRC), peak of heat release rate (PHRR), total heat release (THR), and limiting oxygen index (LOI) value are presented in tab. 2. Taking cotton_Si/P into consideration, the LOI value reached 29.4% which was much higher than 18.0% of pure cotton, indicating the flame retardancy of cotton_Si/P was improved effectively. The micro combustion calorimeter (MCC) data also illustrated this characteristic, the THR value of cotton_Si/P was 3.8 kJ/g, which was lower than 12.1 kJ/g of cotton_control. In case of cotton_Si, a slight increase of HRC and PHRR value occurred compared with cotton_control. This result might be attributed to the breakage of SiO_2 film under high temperature causing the leakage of volatile species. However, in terms of THR value of cotton_Si, there was a 0.6 kJ/g lower than cotton_control. This phenomenon interpreted that the silicon as a flame retardant element still had certain positive influence on cotton flame retardancy.

Table 2. Combustion data and contact angle of cotton samples

Sample	HRC [J·g·K^{-1}]	PHRR [W·g^{-1}]	THR [kJ·g^{-1}]	T_{max} [°C]	LOI [%]	Contact angle [°]
Cotton_control	235	235	12.1	369.7	18.5	–
Cotton_Si	268	268.7	11.5	337.5	17.4	129.4
Cotton_Si/P	82	81.46	3.8	261.1	29.4	134.6

Figure 2 presents the HRR curves of treated and untreated cotton fabrics. It can be seen that the PHRR of cotton_Si/P at 261 °C obviously decreased by 152.2 W/g compared with cotton_control at 369.7 °C. Besides, the ignition temperature of cotton_Si/P was lower than that of cotton_control which may be due to the addition of flammable long chain. However, the HRR of cotton_Si/P rapidly decreased after 261 °C, which also demonstrated the good flame retardancy of treated cotton. This was attributed to the existence of phosphorus-doped silica layer on cotton surface promoting formation to char and blocking the heat flue.

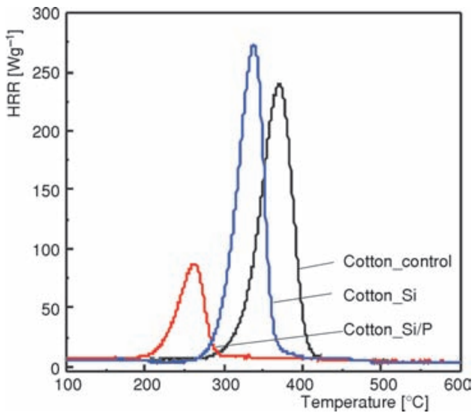

Figure 2. The HRR curves of cotton samples

Surface morphology and water status of cotton samples

The morphology of cotton samples before and after burning was observed by scanning electron microscope (SEM). Figure 3 shows six images: (a) cotton_control, (b) cotton_control burning, (c) cotton_Si, (d) cotton_Si burning, (e) cotton_Si/P, and (f) cotton_Si/P burning. It can be vividly seen that after burning cotton_control totally lost the original form and became ashes. In contrast, the cotton treated with phosphorus-doped silica sol completely retained its original structure. Furthermore, after burning the treated cotton emerged a rough cover which was considered as the char layer formed at high temperature with the existence of phosphorus and silicon on cotton surface. In case of cotton_Si, it still remained original shape after burning but it became thinner compared with cotton_Si/P burning, which revealed the synergistic effect of Si/P.

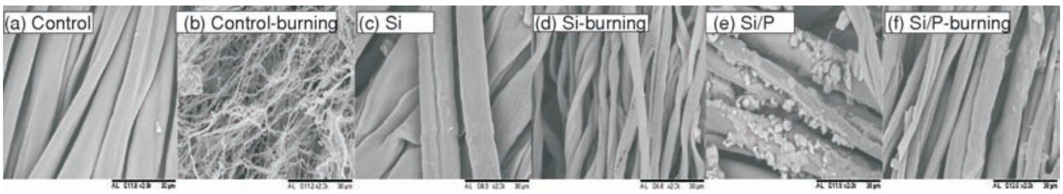

Figure 3. The SEM images of cotton samples

From fig. 4 and tab. 2, it can be seen that water drops remains on cotton surface modified by HDTMS (cotton_Si and cotton_Si/P, whose contact angle are 129.4° and 134.6°, respectively). This result confirmed that the addition of HDTMS had a positive effect on improving water repellency of cotton fabric.

Figure 4. Water status of treated and untreated cotton

Energy dispersive spectra test

Figure 5 shows the energy dispersive spectra of cotton samples. From fig. 5, it can be seen that after treated by phosphorus-doped silica hydrosol, P and Si elements can be found on cotton's surface. In the same way, Si element appeared on the cotton fabric after treated by pure silica hydrosol. In conclusion, the silica sol treated cotton fabric was successfully obtained.

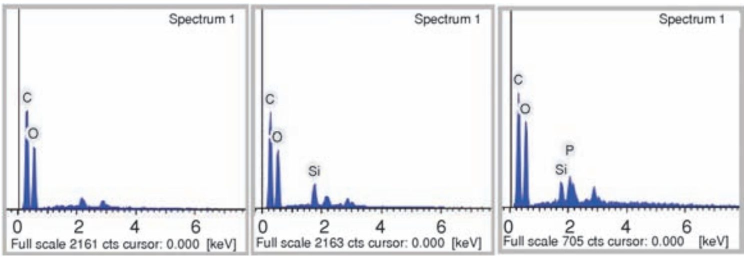

Figure 5. The electronic data systems of treated and untreated cotton

Conclusion

Phosphorus-doped silica hydrosol could effectively improve the flame retardancy of cotton fabric. The composite sol presented a good synergy effect on promoting thermal stability. The HDTMS played a positive role on hydrophobicity of treated cotton fabric.

Acknowledgment

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