THERMAL PROPERTIES OF WOOL FABRIC TREATED BY PHOSPHORUS-DOPED SILICA SOLS THROUGH SOL-GEL METHOD

by

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Wool fabric was treated with silica sol and phosphorus doped silica sol by sol-gel method in order to improve its thermal properties and flame retardance. The thermal stability, combustion behavior, and smoke suppression of the control and finished wool fabric were analyzed using thermogravimetric analysis, limited oxygen index, micro combustion calorimeter, and smoke chamber. The results showed that wool fabric treated by phosphorus doped silica sol had excellent thermal properties and flame retardance with higher final char residue and LOI value. Furthermore, heat release rate and smoke density results indicated the safety performance of the treated wool fabric on fire.

Key words: *thermal properties, wool, thermal gravimetric analysis, heat release rate, smoke density*

Introduction

Wool fabric as a high-grade textile has many superior properties, such as good moisture absorption, soft hand and excellent warmth and become one of the most important raw materials for textile industry. Compared with other fibers, wool is naturally flame retardant (LOI = 25.2%) because of its highly nitrogen and sulfur content, but it still cannot satisfy the strict conditions. In order to meet the stringent demand on flame retardant, it is necessary and urgent to improve the thermal properties and flame retardancy of wool.

Sol-gel method is a new technology for material surface modification in recent years and can be an effective procedure to entrap organic and inorganic compounds with various functionalities on different surfaces [1]. Since the sols prepared is easy to be modified chemically or physically, sol-gel method with simple operation, low reaction temperature and inexpensive process has been widely applied in functional finishing for textiles [2-4]. Silicious flame retardant was environmental friendly and could obtained synergistic effect when acted with phosphorus flame retardant together [5, 6]. In the present work, the flame retardant sol system was prepared using tetraethyl-orthosilicate (TEOS) as precursor with or without dimethyl phosphate as additive, and then applied in wool fabric finishing. The nanoscale sols on the surface of treated fabric could transform into gels after heat-treatment which could adhere to the fabric firmly. The thermal properties of wool fabric treated with silica sol (Wool_Si) and phosphorus doped silica sol (Wool_Si/P) were investigated as well.

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Experimental analysis

Thermal gravimetric analysis

The thermal stability of treated wool fabric was measured by thermogravimetric analysis (TGA), fig. 1. It can be seen from fig. 1 that the weight loss of three samples were all slight at low temperature (before 210 °C) sharing the similar behavior. This stage was attributed to the loss of regain water. From 230~450 °C, this stage was responsible for the major weight loss. The temperature reaching the fastest weight loss rate of treated wool fabric was higher than the control wool, *i. e*. 67% of weight residue at 315 °C for treated wool while 67% at 295 °C for the control wool. At this stage, the increasing decomposition of wool fabric mainly derived from the breakage of hydrogen bond and disulfide bond between spiral peptide chains of the fiber structure and the oxidization of carbon. At nearly 800 °C, the weight loss rate became steady and the char residue remained 30.66%, 36.90%, and 14.34% for wool_Si, wool_Si/P and control wool fabric, respectively. This can be explained by the inorganic –Si-O-Si- network formed on the surface of the treated wool fabric which could act as barrier preventing heat transfer. The further introduction of dimethyl phosphite was helpful to form more non-flammable char residue caused the further improvement of the thermal stability of wool-Si/P.

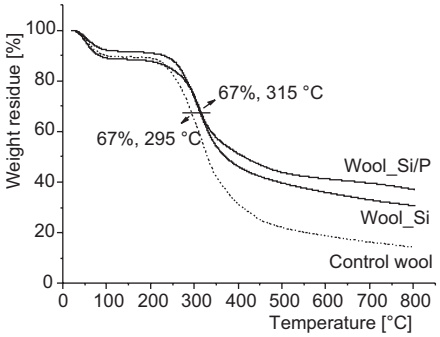

Figure 1. TG curves of wool fabric under nitrogen air

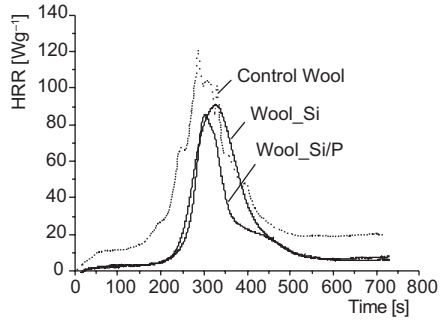

Figure 2. Heat release rate of wool fabric

Micro combustion performance

The flame retardance of treated fabric had been evaluated by micro combustion calorimeter and limiting oxygen index instrument. The results of measuring heat release capacity (HRC), heat release rate (HRR), total heat release (THR), the temperature when fiber decomposited mostly (*T*max) and LOI value were summarized in tab. 1 and HRR curves was showed in fig. 2 particularly.

Table 1. Combustion properties of the control and finished wool

Samples	HRC [J(gK)$^{-1}$]	HRR [Wg^{-1}]	THR [kJg^{-1}]	T_{max} [°C]	LOI [%]
Control wool	112	107.60	12.9	300.4	25.1
Wool_Si	92	83.05	11.6	309.9	26.6
Wool_Si/P	86	81.45	8.1	299.4	30.7

According to tab. 1, LOI value was 26.6% for wool_Si and 30.7% for wool_Si/P compared with 25.1% for control wool. It demonstrated that the treated wool fabric achieved better flame retardance than the control sample, which was due to the flame retardant based on silica and phosphorus. The flame retardance of wool_Si/P was even better than that of wool_Si. SiO_2 layer on the surface of wool fabric had the heat insulation and shielding effect, and phosphorus flame retardant could generate phosphorous acid diluting the flammable gas from the combustion. The LOI values remarkably enhanced especially when the two sub-

stances worked together, which accord with the micro-scale combustion data. Figure 2 shows the HRR of wool fabric, it can be seen that the shape of the HRR curves for treated wool fabric were narrower and smaller than control fabric, which meant that the treated fabric would release heat more slowly and the total heat release would be less while burning.

Smoke density test

The smoke densities of the control and treated wool were measured using smoke chamber and the result was showed in fig. 3. It could be seen that the treated fabric had smaller smoke density during the thermal decomposition process compared with control wool. Wool_Si/P had the excellent smoke suppression property whose maximum smoke density was 16.25 while 49.92 for wool_Si, which was helpful to reduce the risk on fire.

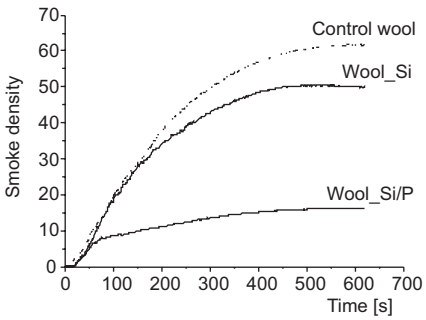

Figure 3. Variation of smoke density with time
Note: the data are the smoke density per unit of fabric weight [/g]

Conclusions

TGA and flame retardance measurements indicated the flame retardance of wool fabric was improved by sol-gel method. Wool fabric treated with phosphorus doped silica sol presented superior synergistic effect of phosphorus and silica.

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