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Preparation and properties of cotton fabrics treated with a novel polysiloxane water repellent and flame retardant

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ABSTRACT

Iodine butyl-*N*-sulfonate amino polysiloxane [(IB-*N*-SA) PDMS] was synthesized as a novel water repellent and flame retardant and it was characterized by FT-IR. The water repellency of treated cotton fabric with (IB-N-SA) PDMS was studied and the results showed that the water contact angle increased from 88.37° to 124.49°. The flame retardant properties of treated cotton fabric were evaluated by limiting oxygen index (LOI) and the vertical burning test. The treated cotton fabric obtained good flame retardancy with a LOI value of 30.9%. Furthermore, The combustion properties of treated cotton fabric were assessed by cone calorimeter and it demonstrated that the treated cotton fabric with (IB-N-SA) PDMS generated less combustion heat and obtained better flame retardancy which can be confirmed by the increase of TTI and FPI values and the decrease in the values of HRR, THR, EHC, Mass loss and CO_2/CO ratio.

1. Introduction

Cotton is tremendously popular in civilian and military applications (clothing, wall-hangings, bedding materials, firefighter apparel and military garments) due to its good mechanical properties, biodegradability and air permeability [1–3]. However, cotton faces certain shortcomings such as ease to ignition, microbial infection and poor water repellency [4–6]. Cotton-related burn injuries and financial losses have been a worldwide problem and many repeated attempts have been made to develop flame retardant cotton fabric [7].

The flame retardancy of cotton fabric can be achieved by chemical treatment and halogen-based and formaldehyde-based flame retardants have shown remarkable flame retardancy in the past decades [8,9]. However, formaldehyde is known as a carcinogenic compound by World Health Organization and halogen-containing compounds in the combustion will release hydrogen halide toxic and corrosive gases [10]. Therefore, government regulations for fire safety and concerns over the toxicological and environmental consequences of these flame retardants have fueled the search for environmentally friendly and cost-effective alternatives [11].

In recent years, silicone-based flame retardants have been a research hotspot in academic. These compounds possess good thermal stability, low fire hazard and biological compatibility [12,13]. The flame retarding mechanism is explained as that the flame retardants migrate to the material surface and produce silicaceous char layer during combustion. Furthermore, synergistic effect of silicon and other elements on enhancing flame retardancy of cotton fabric is previously observed [14].

The interest in improvement of water repellency of cotton has been increased recently. Cotton treated with fluorinated compounds can obtain high water repellency. However, fluorinated compounds have potential risk to humans and the environment [15]. Nowadays, silicon compounds have been a good option because they can create a hydrophobic surface and they are eco-friendly [16]. The development of cotton fabrics with multifunctional features is attracting more attention.

In this work, Iodine butyl-N-sulfonate amino polysiloxane [(IB-N-SA) PDMS] was synthesized as a novel agent with double functions of water repellency and flame retardancy using poly (4-iodobutoxy) methylsiloxane and guanidine sulfamate as raw materials. It was applied to cotton fabrics and it could combine to cotton fabrics with covalent bond due to the activity group. The water repellency and flame retardancy of treated cotton fabrics with (IB-N-SA) PDMS were evaluated by water contact angle and limiting oxygen index (LOI). The combustion behaviors of treated cotton fabrics were investigated by cone calorimeter.

2. Experimental

Materials: Scoured and bleached 100% plain-woven cotton fabric (14.75 × 14.75 tex^2, 122 g/m^2) was purchased from Weifang Qirong Textiles Co., Ltd.

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Poly (4-iodobutoxy) methylsiloxane was self-made in our laboratory. Guanidine sulfamate was supplied by Tangshan Sanding Chemicals Co., Ltd. Zirconium oxychloride was obtained from Aladdin Reagent Co., Ltd.

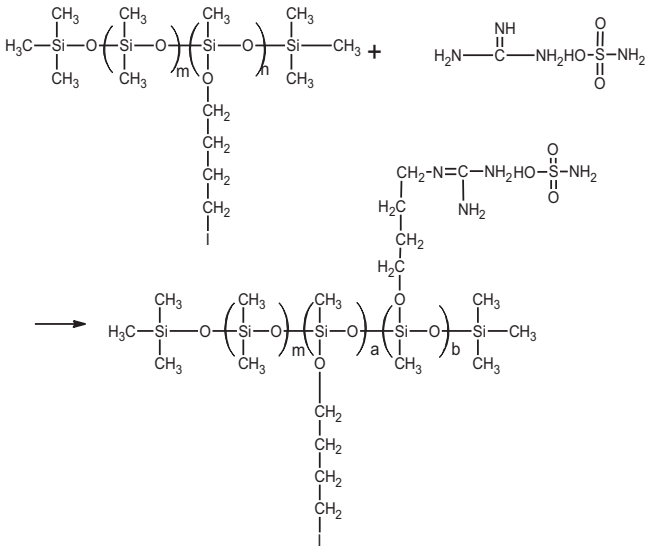
Preparation of (IB-N-SA) PDMS: Iodine butyl-N-sulfonate amino polysiloxane [(IB-N-SA) PDMS] was synthesized using poly (4-iodobutoxy) methylsiloxane and guanidine sulfamate as raw materials. The structure of (IB-N-SA) PDMS is shown in Scheme 1 and it is characterized by FT-IR.

FT-IR: the absorption peak at 3352–3198 cm^{-1} was symmetrical and asymmetrical stretching vibration of N–H and absorption peaks at 1667 cm^{-1} was assigned to stretching vibration of C═N; the absorption peak at about 1241 cm^{-1} was stretching vibration of C–N and the absorption peak at about 1053 cm^{-1} was stretching vibration of Si–O–Si.

Preparation of treated cotton fabrics: The cotton fabrics were soaked in finishing bath containing (IB-N-SA) PDMS, zirconium oxide chloride and urea at room temperature for 4 min under proper pH condition. Then the samples passed through a laboratory-scale padder with two dips and two nips to get a wet pick up of 100%. Finally, the samples were dried at 100 °C for 3 min and cured at 150 °C for 4 min.

The amount (wt% owf) of flame retardant added on cotton fabric was calculated as follows:

$$\text{Add-on}\% = \frac{W_f - W_b}{W_b} \times 100\%$$



Scheme 1. Preparation of (IB-N-SA) PDMS.

where W_b and W_f represent the weights of cotton fabrics before and after flame retardant treatment, respectively.

Characterization: The water repellent properties of the fabrics were evaluated in accordance with AATCC Test Method 22. Hydrophobic properties were also characterized by an Easy Drop video optical contact angle meter. The contact angle was calculated using the formula $\theta = 2\tan - 1\,(2\,h/d)$, where h is the height of the water droplet and d is the width of the droplet touching the film.

Limited oxygen index (LOI) tests were carried out on a digital display oxygen index instrument LFY-606 according to GB/T 5454-2009. The vertical burning test was carried out on CZF-3 instrument according to GB/T 5455-2009.

The combustion of cotton fabrics was investigated using a FTT0007 cone calorimeter (Fire Testing Technology Ltd.) under a heat flux of 30 kW/m^2 according to ISO 5660.

3. Results and discussion

Water repellency: The water repellent properties of the cotton fabrics treated with (IB-N-SA) PDMS were evaluated and the grade of water repellency of treated cotton fabric was 90. Furthermore, the water contact angle of treated cotton fabric increased from 88.37° to 124.49° as shown in Fig. 1. The reason of the improvement of water repellency was that (IB-N-SA) PDMS could react with cotton fiber and the groups which oriented on fiber surface, mainly methyl groups, formed a hydrophobic film, decreasing the critical surface tension of cotton fabric [17].

Flame retardant performance: The flammable properties of cotton fabrics were investigated and the results showed that the LOI value of treated cotton fabric increased from 18.0% to 30.9%. The upward burning behaviors of cotton fabrics were also determined by the vertical burning test and the results demonstrated that the untreated cotton fabric was completely destroyed while the treated cotton fabric obtained shorter char length (3.8 cm), shorter after-glow time (4 s) and no after-flame which indicated that the flammability of cotton fabric was retarded by (IB-N-SA) PDMS. The reason was that (IB-N-SA) PDMS decomposed during degradation and produced the incombustible gases which can dilute the concentration of the flammable gases and formed the foamed-char layers. Furthermore, (IB-N-SA)PDMS also produced silica and glassy char, which acted as a physical barrier and could protect cotton fabric from heat and oxygen transfer.

Combustion behaviors: Cone calorimeter was employed to investigate the combustion properties of cotton fabrics, the results are shown in Fig. 2 and the critical data related to the combustibility are presented in Table 1. The treated cotton fabric with (IB-N-SA) PDMS obtained a remarkable reduction in heat release rate (HRR), corresponding peak HRR (PHRR) and total heat release rate (THR)

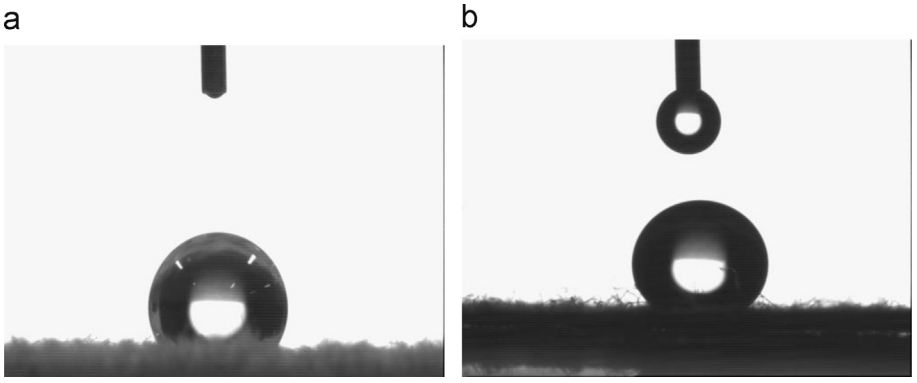


Fig. 1. Contact angles of the cotton fabrics: (a) the untreated fabric (θ=88.37°); (b) the treated fabric with (IB-N-SA) PDMS (θ=124.49°).