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Enhancement in flame retardancy of cotton fabric by using surfactant-aided polymerization

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ABSTRACT

In this study, a commercially available phosphorus-based methacrylate ester was polymerized on cotton fabric by a variation of admicellar polymerization to make it flame retardant. The resulting film of polymerized phosphorus monomer formed on the cotton surface was characterized by Fourier Transform Infrared spectroscopy and Scanning Electron Microscopy. Thermal properties were studied by Thermogravimetrical analyses which showed that the treated cotton fabric has higher char yield and lower decomposition temperature than untreated cotton fabric. Auto 45° flammability test was performed to understand the burning behaviour of treated cotton fabrics. Untreated cotton fabric burned the entire length of a 6.5 cm sample to ashes in 41 s. The treated cotton fabrics did not ignite during test and passed the flammability test with a residual char of 1 cm length. The durability of treated cotton fabric with flame retardant property was also investigated by repeated home-launderings.

1. Introduction

Cotton is a natural polymer which is the principal fibre used in making the world's clothing because of being lightweight, cool, comfortable and absorbent. Many people describe cotton as a fabric that “breathes”. Therefore, the field of applications of cotton in the form of finished fabrics is very wide including defence, industrial purposes, space research, fire fighters' clothing and various textiles. But, increases in fire accidents caused by the burning of cotton fabrics have motivated research in textiles with flame retardant properties [1]. Therefore, to avoid hazards in the future there is a requirement of better flame retardancy for textiles made of cotton [2].

A flame retardant fabric can be defined as a textile, which does not propagate the flame, although it may burn or char when the material is subject to any form of heat. In order to impart flame retardancy in cotton, the burning process must be interrupted by using compounds which alter the course of decomposition, prevent the formation of flammable volatiles, and increase char formation [3,4]. Product distributions from combustion and pyrolysis of cellulose are very complex with over 40 different species including alcohols, ketones and furans [5,6]. The thermal degradation of cotton cellulose follows two pathways [7,8], at low temperature (below 300 °C), cellulose undergoes dehydration, depolymerisation and oxidation with release of CO, CO_2 and carbonaceous residue whereas at high temperature (above 300 °C) it produces combustible volatile compounds like levoglucosan [9] which are responsible for propagation of fire. A lot of attempts have been made to impart flame retardancy to fabrics by methods including chemical treatment [10], pad-dry cure [11], ionized radiation graft polymerization [12] and plasma induced graft polymerization [13], nanoparticles adsorption [14], sol gel process [15], layer by layer deposition [16] and dual cure processes [17]. However, most of them require special equipment or involve working with a gas phase, which can be difficult to control, along with other disadvantages such as low fabric strength, and high fabric stiffness. A versatile technique, admicellar polymerization [18] conducted in the liquid phase can overcome these disadvantages. The admicellar polymerization process forms ultra thin films [19] of thicknesses on the order of 10 nm, which helps in maintaining desirable fabric properties like softness, pliability and air permeability. Admicellar polymerization has been successfully used to form various types of polymeric thin films on different substrates, such as polystyrene and poly methyl methacrylate over silica, alumina and cotton [20–23]. Recent work has shown that admicellar polymerization can be used to impart properties like flame retardancy and water repellency to cotton fabric [24,25].

The admicellar polymerization process can be described in four main steps: admicelle formation, monomer adsolubilization,

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polymeric film formation, and surfactant removal as shown in Fig. 1. In the outer surfactant layer, the amphiphilic molecules are oriented with their ionic head groups in contact with the aqueous solution, while the long hydrophobic tails interact to form a hydrophobic inner core (Fig. 1). An inner layer oriented with the head groups in contact with the substrate completes the surfactant bilayer called an admicelle. When a typical hydrophobic organic monomer is added into the solution, it will preferentially partition into the core of the admicelle in a process called adsolubilization. In the presence of a thermal initiator, the monomer in the admicelle will undergo a polymerization reaction to form a polymeric layer on the substrate surface. After the polymerization, surfactant in the upper layer may be removed by washing to expose the polymeric layer on the substrate surface. In this study, interfacial polymerization of an anionic, relatively hydrophilic monomer (phosphoric acid 2-hydroxy ethyl methacrylate ester, PHEME) was carried out after creating a positively charged surface by adsorption of a cationic surfactant and employing the hydrophobic initiator (azobis isobutyronitrile, AIBN). The coating proved to be an effective flame retardant, prepared with a commercially available monomer.

2. Experimental

2.1. Materials

Interlock knit cotton fabric was purchased from Alamac American Knits (Lumberton, North Carolina, USA). The surface density for cotton was 284 g/m^2. Monomer phosphoric acid 2-hydroxy ethyl methacrylate ester (PHEME) ($C_6H_{13}O_7P$), initiatior azobis isobutyronitrile (AIBN) ($C_8H_{12}N_4$) and binding agent N-(hydroxymethyl) acrylamide solution ($C_4H_7NO_2$) [26] were purchased from Sigma Aldrich (USA). Hexadecyl trimethyl ammonium bromide (CTAB) was purchased from Acros Organics (USA) and ammonium hydroxide (NH_4OH) was purchased from EM Science (USA). Cotton fabric weighing 2.84 g was repeatedly scoured with warm water in a washing machine until it was free from any remaining surfactant prior to use. All chemicals were used as received without further purification.

2.2. Fabric treatment by admicellar polymerization process

A 6 inch × 2.5 inch (20 cm × 5 cm) piece of cotton fabric weighing 2.84 g was placed in a 32 ml reactor vial with a solution of 1.5 ml of 0.45 mM CTAB and 3.6 mM/g N-hydroxymethyl acrylamide as binding agent [26]. Ammonium hydroxide was added to maintain solution pH before sealing and the vial was placed in a shaker bath. After 2 h at 80 °C for surfactant adsorption, liquid monomer (100 mM) and 3 ml AIBN at 0.01 M concentration dissolved in isopropyl alcohol were added. The reaction was continued for another 2 h to allow monomer adsolubilization and polymerization. After polymerization, the vial was cooled and the fabric was removed for drying in an oven overnight at 65 °C. Samples were tested for flame retardancy and durability using different analytical techniques described below.

Table 1
Description of samples.

S. No.	Sample	Description of samples
Without binding agent		
1	UCF	Untreated cotton fabric
2	TCF	Treated cotton fabric with phosphorus monomer, initiator and surfactant
3	TCF-1	TCF after first home laundering
With binding agent		
4	TCF-BA	Cotton fabric treated with phosphorus monomer, initiator and surfactant with binding agent
5	TCF-BA-1	TCF-BA after first home laundering
6	TCF-BA-2	TCF-BA after second home laundering
7	TCF-BA-3	TCF-BA after third home laundering

To evaluate the washing durability of flame retardant treated cotton fabric, the cotton fabric samples were home laundered in a washing machine using detergent (Tide) and water at room temperature for 30 min. The samples were then dried in a conventional tumble dryer at 60 °C and stored in a desiccator until required. The washed fabrics were tested for flame retardent behaviour. The fabrics which were burnt after the flammability test were not tested further. The fabrics which were not burnt were home laundered and tested for flame retardency and the process was repeated until the fabric was burnt. The samples prepared by admicellar polymerization are mainly of two types (Table 1): treated cotton fabric samples in absence of binding agent and treated cotton fabric samples containing binding agent. For three fabric samples: UCF, TCF and TCF-1, Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM), elemental analysis, thermal analysis and flammability studies were carried out. For the four samples containing binding agent: TCF-BA, TCF-BA-1, TCF-BA-2, TCF-BA-3, durability and flammability studies were carried out.

2.3. Surface characterization by fourier transform infrared spectroscopy (FTIR)

FTIR spectra of untreated cotton, treated cotton and treated cotton after home laundering were recorded by the KBr pellet testing techniques using Shimadzu IR affinity-I 8000 FTIR spectrophotometer in the wavenumber range from 4000 to 400 cm^{-1}

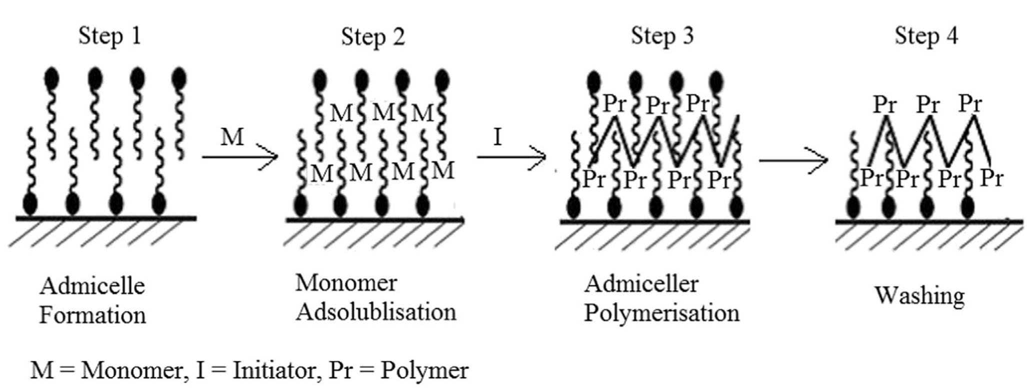

Fig. 1. Steps of admicellar polymerization process.