



Developing a novel UV protection process for cotton based on layer-by-layer self-assembly

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ABSTRACT

A new approach for UV protection of cotton fabrics based on electrostatic self-assembly (ESA) technique was studied in this paper. Three fluorescent brightening agents (FBAs) and poly(diallyldimethylammonium chloride) (PDDA) were stepwisely fabricated on cationized cotton fabrics through direct layer-by-layer (LbL) deposition technique. Dyeing of the assembled cotton fabrics with anionic dyes shows regular and identifiable “odd–even” changes in color depth (*K/S* value), indicating the variation of surface polarities of the cotton substrates due to the alternate deposition of FBA and PDDA on them. The stepwise increases in UPF of treated cotton fabrics further revealed the growth of these LbL deposition multilayers. The assembled cotton fabrics could obtain excellent UV protection ratings of UPF > 40 after several bilayers of FBA/PDDA were fabricated, depending on the type of FBAs. Excellent durability to washing of the (FBA/PDDA)_{*n*} multilayers was obtained, which indicates good adhesion between the multilayer coatings and the cotton surfaces.

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1. Introduction

In recent years, there is a progressive increase in UV radiation on human skin caused by the depletion of the ozone in the earth's atmosphere. As long-term exposure to UV light can result in a series of negative health effects such as acceleration of skin ageing, photodermatitis (acne), erythema (skin reddening), and even severe skin cancer, developing textiles with UV protection functionality has been widely researched so far (Reinert, Fuso, Hilfiker, & Schmidt, 1997; Schindler & Hauser, 2004). UV radiation on or near the earth's surface is an invisible component of sunlight, divided into UV-A (315–400 nm), UV-B (280–315 nm) and UV-C (200–280 nm). Fortunately, the very high energy of the UV-C photons is mostly absorbed by ozone in the higher regions of the atmosphere, decreasing their relative intensity on the earth surface to almost zero. Therefore, to be useful in protecting the wearers from solar UV radiation, the main UV rays that should be blocked by textiles are UV-A and UV-B. To date, many approaches have been investigated to improve the UV protection function of cotton fabrics because cotton textiles are the most regular summer clothes but having the least UV-blocking ability. Among them, organic UV absorbers (Akrman & Prikryl, 2008; Czajkowski, Paluszkiwicz,

Stolarski, Kazmierska, & Grzesiak, 2006; Riva, Algaba, & Pepio, 2006; Tragoonwichian, O'Rear, & Yanumet, 2008), nanoscaled inorganics such as ZnO (Becheri, Durr, Lo Nostro, & Baglioni, 2008; Vigneshwaran, Kumar, Kathe, Varadarajan, & Prasad, 2006) and titanium hydrosols (Xin, Daoud, & Kong, 2004; Xu, Liu, Wang, & Chen, 2006), dyestuffs (Gorensek, Sluga, & Urbas, 2007; Gorensek, Urbas, Strnad, & Parac-Osterman, 2007; Kim, 2006; Veatch & Gatewood, 2002), fluorescent brightening agents (Grancaric, Tarbuk, Dumitrescu, & Biscan, 2006; Pusic & Soljagic, 2008) and miscellaneous (Cheng, Li, Yuen, & Hu, 2006; El-Tahlawy, El-Nagar, & Elhendawy, 2007; Vigneshwaran et al., 2006) have been widely studied. Some UV absorbers with reactive groups that can covalently bond with cellulosic hydroxide groups have been industrially applied.

Much research on layer-by-layer (LbL) electrostatic self-assembly (ESA) of polyelectrolyte multilayers on charged surfaces has been conducted during the past decades, aiming at providing surfaces and materials with specific properties and functionalities (Decher & Schlenoff, 2003; Decher, Hong, & Schmitt, 1992; Lvov, Decher, & Moehwald, 1993). The polyelectrolytes with opposite charges could bond to each other with ionic bonds when the charged substrate is alternatively dipped in polyion solutions, forming multilayer ultrathin films on the substrate. ESA processing has advantages including no limitation on the requirement of size, shape and topography of the charged substrates, controllable functionalization through flexible utilization of polyelectrolytes or other charged substances, as well as a simple, cost-

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and energy-saving and eco-friendly process (Chen & McCarthy, 1997).

It should be noted that this new technique is not limited to polyelectrolytes. Some organic molecules with positive or negative charges have also been integrated into multilayers via LbL self-assembled deposition (Ariga, Lvov, & Kunitake, 1997; Chang-Yen, Lvov, McShane, & Gale, 2002). In view of this, it is reasonable that the textiles could be functionalized via incorporating various functional materials into ESA multilayer assemblies on fiber surfaces. However, few researchers so far have focused on textile substrates for their self-assembly functionalization (Dubas, Kumlangdudsana, & Potiyaraj, 2006; Dubas, Limsavarn, Iamsamai, & Potiyaraj, 2006; Hyde, Rusa, & Hinestroza, 2005). As the commonly used polyelectrolytes such as poly(sodium 4-styrenesulfonate) (PSS), poly(acrylic acid) (PAA), poly(dimethyldiammonium chloride) (PDDA), poly(allylamine hydrochloride) (PAH) and polyethyleneimine (PEI) have no special functions transferred to textiles, non-polyelectrolytes with negative charges such as nanoparticles (Dubas, Kumlangdudsana, et al., 2006) and dyestuffs (Dubas, Limsavarn, et al., 2006) were integrated into the LbL self-assembled multilayers together with polyelectrolytes to modify the surface antibacterial properties and dyeability, respectively. The present objective of this work is to fabricate anti-UV multilayer coatings on cationized cotton fabrics using the ESA technique. The alternating LbL buildup of three fluorescent brightening agents (FBAs) with anionic groups and polyelectrolytes on cotton substrates was performed. Three important aspects of the ESA anti-UV functionalization of cotton, i.e., characterization, growth and durability of multilayer ultrathin films on cotton, were evaluated.

2. Experimental

2.1. Materials

Well-prepared 100% cotton woven fabric was used in all experiments. 3-Chloro-2-hydroxypropyltrimethylammonium chloride (CR-2000, Dow Chemical Co., USA) is commercially available as a 65% (w/v) solution in water and was used as received. Three fluorescent brightening agents, Phorwite BA (CAS No. 4193-55-9, C.I. Fluorescent Brightener 28), Phorwite BBU (CAS No. 16324-27-9, C.I. Fluorescent Brightener 114) and Uvitex NFW (CAS No. 27344-41-8, C.I. Fluorescent Brightener 351) were used as UV-blocking agents. Poly(dimethyldiammonium chloride) (PDDA, 20%, w/w, Mw 100,000–200,000, Aldrich) and poly(sodium 4-styrenesulfonate) (PSS, 30%, w/w, Mw 70,000, Aldrich) were used as positively and negatively charged polyelectrolytes, respectively. They were used as received without further purification. Direct Red 80 (CAS No. 2610-10-8, C.I. Direct Red 80) was used for dyeing without further purification. The chemical structures of polyelectrolytes, FBAs and dyes used in this experiment are shown in Fig. 1. All chemicals were obtained from Fisher Scientific (Raleigh, NC) unless stated otherwise.

2.2. Preparation of cationized cotton substrates

For the purpose of multilayer film fabrication using the 'layer-by-layer' technique, the cotton fabrics were cationized using a cationizing agent (CR-2000). A solution containing 50 g/l of CR-2000 and 18 g/l sodium hydroxide was padded on the well-prepared cotton woven fabric. Then the padded fabric was wrapped in plastic film, slowly rotated at room temperature for 24 h, rinsed several times with water and finally the residual alkali on the fabric was neutralized with a dilute acetic acid solution (Hauser & Tabba, 2001).

2.3. Fabrication of self-assembled multilayers on cationized cotton surfaces

2.3.1. Direct deposition of multilayers

Polyelectrolyte multilayers were directly fabricated on the surfaces of the cationized cotton fabrics at room temperature according to the following procedure. Cotton swatches (4 cm × 4 cm, approximately 0.2 g each) were first immersed into a solution containing 1.0 mg/ml FBA in a plastic dish for 5 min followed by rinsing in deionized water for 5 min. Then the FBA-masked samples were transferred to PDDA solution (0.01 M by repeat unit) for 5 min followed by rinsing in deionized water for 5 min. After this, two monolayers or one bilayer were assembled on the surfaces of cotton fibers. The odd number denoted FBA layers, while the even number was PDDA layers. The above procedure was repeated cyclically until a (FBA/PDDA)_n multilayer thin film with the desired bilayer number (*n*) was deposited on the surface of the cationized cotton fabric.

2.3.2. Pre-mixed deposition of multilayers

Since each FBA has fewer negative charges compared with common polyelectrolytes, a new path was designed for fabrication of multilayers incorporating FBAs on the cotton surface. Each anionic FBA was mixed with a polycation (PDDA) in advance according to varied ratios, to form an interpolyelectrolyte complex that bears a net positive charge, (PDDA–FBA)[⊕]. The mole ratios of FBA to PDDA monomeric unit were 1:80, 1:50 and 1:10, corresponding to concentrations of 0.12, 0.19, and 0.96 mg/ml for Phorwite BA, 0.13, 0.22, and 1.08 mg/ml for Phorwite BBU and 0.06, 0.10, and 0.52 mg/ml for Uvitex NFW, respectively. The cationized cotton specimens with same dimensions as above were LbL self-assembled with PSS solutions and these mixed PDDA–FBA solutions. The specimens were first dipped in PSS solutions for 5 min to invert the surface electric properties from positively charged to negatively charged. After a rinsing in deionized water bath for 5 min, the PSS-coated specimens were transferred to the PDDA–FBA interpolyelectrolyte solutions and left for 5 min. They were then removed to a deionized water bath and rinsed for 5 min. This process produced ultrathin PSS/(PDDA–FBA) bilayer films on the cotton surfaces. After the assembly procedure was repeated multiple times to form functional FBA multilayers on the substrates, the specimens were air-dried.

2.4. Dyeing characterization of self-assembled multilayers deposited on cationized cotton surfaces

The surface polarity of the cationized cotton substrate deposited with self-assembled multilayers of polyelectrolytes was investigated by monitoring the change in color depth of the dyed cotton fabrics according to our previous report (Wang & Hauser, 2009). Cotton specimens were dyed using 7.5% o/w anionic dye (Direct Red 80) with a liquor ratio of 150:1. The dyeing was performed in beakers without stirring at ambient temperature for 120 min to approach a dyeing equilibrium. All dyeing experiments were performed without additional auxiliary chemicals in order to evaluate dyeability without external interference.

The relative color depth of the dyed fabrics, expressed as *K/S*, was measured by the light reflectance technique using the Kubelka–Munk equation.

$$\frac{K}{S} = \frac{(1 - R)^2}{2R}$$

The reflectance (*R*) of the dyed fabrics was measured at the maximum absorbance wavelength on a Datacolor Spectraflash SF600 Plus spectrophotometer.

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