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Multi-functional textile design using in-situ Ag NPs incorporation into natural fabric matrix

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ABSTRACT

The multifunctional natural fabrics as colored, antibacterial and UV-protective fabrics were effectively prepared by in-situ incorporation of silver nanoparticles (Ag NPs) into the fabric matrix. Cotton and wool fabrics were used as natural fabrics and trisodium citrate was utilized as reductant for Ag^+ and stabilizer for Ag NPs. The formation of Ag NPs in the residual solutions was confirmed by UV/Vis spectra. Ag NPs on fabrics were shown by scanning electron microscopy and their size was in range of 20–90 nm and 70–150 nm for cotton and wool fabrics, respectively. X-ray photoelectron spectroscopy confirmed the stabilization action of citrate. As a result of surface plasmon resonance of Ag NPs, colored fabrics were produced with good fastness properties. Functional properties of treated fabrics indicate that, the in-situ incorporation of Ag NPs into fabric matrix was simultaneously imparted multifunctional properties (colorant, antibacterial action and UV-protection) into natural fabrics concurrently.

1. Introduction

Great potentials of nanoparticles (NPs) can be efficiently utilized for production of functional textiles [1]. Many studies reported the application of nanoparticles and metal oxides as surface modifiers of fabrics for imparting antimicrobial, self-cleaning and UV protective properties [2–18]. Due to high volume to surface area ratio of nanoparticles (Ag, Au, TiO_2, ZnO … etc.), great researches were interested in their preparation and characterization, as each of metal nanoparticles have different colors according to their Surface Plasmon Resonance peaks [19].

Lately, a new approaches for applying the noble metal nanoparticles as multifunctional agent imparting coloration and antimicrobial properties for textile fabrics have been reported. Based on literature, three main approaches have been applied in the coloration of textile fabrics by using noble metal nanoparticles. Firstly, noble metal nanoparticles colloid solution with different shapes and colors will be synthesized at first step and then, the textile fabrics are impregnated by dipping them in the noble metal nanoparticles colloid solution as dyeing process [20–22]. Secondly, the noble metal nanoparticles are synthesized in-situ the fabrics [23–27]. In the third method, a polymer-nanoparticles composite is synthesized followed by spinning process [28].

Herein, we present an approach for realizing multifunctional finishing (coloration, antibacterial and UV protection properties) for different natural fabric by in-situ incorporation of Ag NPs into fabric matrix. Ag NPs were synthesized and incorporated directly into fabrics matrix by using trisodium citrate as reducer, stabilizing and/or linking agent. The mechanism of assembling Ag NPs on the fabrics was discussed. The existence of Ag NPs on the modified fabrics was confirmed by SEM, EDX and XPS. The color of fabrics was measured under different reaction conditions. The antibacterial activity and UV protection properties of Ag treated fabrics were all investigated.

2. Experimental

2.1. Materials and chemicals

Scoured and bleached Cotton fabric (142 g/m^2) was purchased from Misr Spinning and Weaving Company, Mahalla El- Kobra, Egypt. Before using, the fabric was washed with a solution containing 5 g/l non-ionic detergent (Hostapal CV, Clariant), at 95 °C for 4 h. Pure Australian merino scoured Wool woven fabric (210 g/m^2), supplied by Misr Company for Spinning and Weaving, El-Mehalla El-Kobra – Egypt, Silver nitrate (99.5%, from Panreac,

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Barcelona – Spain), and Trisodium citrate (99% from Sigma–Aldrich – Germany) were all reagents used without any further purification.

2.2. Procedure

In typical experiment, weight of 10 × 16 cm^2 cotton or wool fabrics were immersed in 250 mL of silver nitrate solution (200, 400, 800 and 1200 ppm) using liquor ratio 1/50 and heated in water bath till boiling. A 25 mL from an aqueous solution of (1, 3 and 5%) trisodium citrate was added dropwise to the mixture. After that, the fabrics were taken out, squeezed and rinsed by tap water. The fabrics were dried at room temperature prior to analysis and characterization. The absorbance of all supernatants was measured after the immersion time.

2.3. Measurements

2.3.1. UV–visible spectra

The absorption spectra of the residual solutions after the treatment step was measured using UV–visible multi-channel spectrophotometer (T80 UV/VIS, d = 10 mm, PG Instruments Ltd, Japan) from 250 to 600 nm. The transmission of ultraviolet (UV) radiation through fabrics was evaluated with a Varian Cary 300 ultraviolet–visible spectrophotometer (Mulgrave, Victoria 3170, Australia) at a wavelength range of 320–400 nm.

2.3.2. Scanning electron microscope (SEM) and Dispersive X–ray spectroscopy (EDX)

ZEISS LEO 1530 Gemini Optics Lens scanning electron microscopy (SEM) with 30 kV scanning voltages was employed to observe the morphologies of untreated and treated fabrics. Zeiss LEO 438 VP with Oxford Instruments EDX with INCA software system. EDX measurement conditions, 20 kV accelerating voltage, 21 mm working distance, 1 nA sample.

2.3.3. X-ray photoelectron spectroscopy (XPS)

XPS spectra were measured using a Kratos Ultra system. The Following acquisition parameters were applied: Sample neutralization by low energy electrons (2.6 eV), hybrid mode (using electrostatic and magnetic lenses), take-off angle of electrons: 0°, pass energy: 20 eV (detection of high resolution spectra); 160 eV (detection of survey spectra), excitation of photoelectrons by monochromatic $Al_{k\alpha}$ radiation.

2.3.4. Color measurements

The colorimetric analysis of the colored fabrics was recorded using a spectrophotometer with pulsed xenon lamps as light source (Ultra Scan Pro, Hunter Lab, USA) 10° observer with D65 illuminant, d/2 viewing geometry and measurement area of 2 mm. All measurements were occurred at λ_{425} nm wavelength. The corresponding color strength value (K/S) was assessed by applying the Kubelka Munk [29] (Eq. (1)).

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

Where R is the decimal fraction of the reflection of the colored fabric, K is the absorption coefficient and S is the scattering coefficient.

2.3.5. Fastness properties

The colored fabrics samples were tested according to ISO standard methods. The specific tests were ISO 105-X12 (1987), color fastness to rubbing; ISO 105-C02 (1989), color fastness to washing and ISO 105 E04 (1989), color fastness to perspiration [30].

2.4. Antibacterial test

The antibacterial activity of colored fabrics was tested against *Escherichia coli* AATCC 2666 as gram negative bacteria. The antibacterial tests were performed quantitatively using the standard test method according to the AATCC test method 100–1999 for Bacterial Counting [31]. All treated fabric samples were kept at controlled temperature 35 °C prior to test. After incubation step, weighted fibers were transferred into 100 mL of nutrient broth (1:500) (ca. 1.5 × 108 colony forming unit per mL) and then shaken vigorously for 1 min. A normal saline solution that was prepared with 0.9% (w/v) was serial diluted and then plated onto Mac Conkey agar Eosin Methylene Blue (EMB) agar plates for *E. coli*. For all plates, the bacteria were incubated at 37 °C for 24 h and then the colonies were counted. The antibacterial activity of fabrics was estimated by calculating the reduction percentage of bacterial colonies using equation (2).

$$R\% = [(B - A)/B] \times 100 \quad (2)$$

Where **R %** is the reduction percentage of bacterial colonies, **A** is the number of bacterial colonies surviving on the agar plate with treated fabric samples and **B** is the number of bacterial colonies surviving on the agar plate for control.

3. Results and discussion

Different techniques were been used to synthesize functional fabrics/fibers using nanoparticles. Such methodologies include multi-steps (nanoparticles colloidal solutions preparation followed by loading on the fabrics/fibers) [32–36], expensive chemicals [36], more chemicals consumption [33,37] and using of crosslinkers for improving fastness [32,34]. The current research considered with designing of multifunctional fabrics by concurrent synthesis and incorporation of AgNPs into the fabric matrix using quite simple technique with low chemical consumptions and no need of use any binding polymers. Two different natural fabrics were been used based on their different resources and in turn different in chemical structure and physical properties namely; cotton as cellulosic fabric (from plant) and wool as protein based fabric (from animal). They were used in order to ensure that our simple methodology is effective and applicable for different types of fibers/fabrics.

3.1. Mechanism of in-situ Ag NPs incorporation into fabric matrix

Preparation of metal nanoparticles using sodium citrate as a reducer and stabilizing agent has been previously reported [38,39]. The excess of trisodium citrate controls the particle growth of the Ag particles and produces a stable nanosilver colloid [40–43].

The schematic mechanism for the in-situ incorporation of Ag NPs in the natural fabrics is presented in Fig. 1. Once silver nitrate added to fabric, ion exchange interaction and/or complexation between silver ions and chelating groups of cotton (COOH and OH groups) [16,17,44–46] or wool (COOH, NH_2 and SH) are supposed to take a place [47,48]. After addition of sodium citrate, citrate is oxidized to acetone carboxylate and Ag^+ is in turn reduced to Ag^0 nanoparticles at high temperature (Fig. 1). Then, both of citrate and building units of fabrics acts in stabilizing the produced nanoparticles as they both chelate the NPs (i.e. citrate – AgNPs – fabrics).

Finally, the high surface area to volume ratio of the stabilized AgNPs may suppose to be the template for building up silver