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# Improvement of dyeing and antimicrobial properties of nylon fabrics modified using chitosan-poly(propylene imine) dendreimer hybrid



Mousa Sadeghi-Kiakhani<sup>a,\*</sup>, Siyamak Safapour<sup>b</sup>

<sup>a</sup> Institute for Color Science and Technology, Department of Organic Colorants, Tehran, Iran<sup>b</sup> Faculty of Carpet, Tabriz Islamic Art University, P.O. Box 51385-4567, Tabriz, Iran

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#### ABSTRACT

Nylon fabric surface modification using chitosan-poly(propylene imine) dendreimer hybrid (CS-PPI) as a novel eco-friendly finishing agent has been reported. Effects of some operational parameters such as pH, temperature, time and CS-PPI concentration on grafting yield were examined through dye up-take using two commercial reactive dyes in terms of color strength (*K*/*S*). FTIR, SEM, and DSC data confirmed the grafting of CS-PPI onto nylon substrate. Optimal grafting values obtained were pH 4, temperature 60 °C, time 6 h and 2.5 g/L CS-PPI concentration. The performance of CS-PPI grafted nylon was investigated in terms of its dyeability, color fastness, and antimicrobial properties.

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## Introduction

A wide range of dyes are nowadays available in the market for dyeing of nylon (polyamide) textiles and among them acid, metal complex and reactive dyes are being extensively used [1,2]. All these dye are anionic which can be absorbed by cationic sites (amino groups) of nylon polymer chains. Some advantages of reactive dyes over other dye classes are their brilliancy, high wet fastness, convenient usage, wide range of hues, and high applicability. However, there are some challenges with the use of these dyes which limit their application. Reactive dyes need almost a large amount of electrolyte as auxiliary agents in dyeing, demonstrate low dyeing ability, and generate high volume of discharged wastewater [3-5]. In addition, color yield of reactive dyes is generally low, depending on the different natures of the both dye and textile materials used. In general, 40-50% of the reactive dyes remain unexhausted in dye bath after dyeing [6,7]. The discharge of such colored textile wastewater into the environment has therefore generated serious environmental and ecological problems [8].

Considering nylon textiles as target material for dyeing, on the other hand, the number of terminal amine groups as dye absorbing

sites on nylon is restricted, and therefore, heavy shades can be hardly obtained [9]. The improvement of dyeing ability of nylon textiles with reactive dyes has been investigated by some researchers [10–19]. Of the various methods examined, it is well accepted that surface modification may enhance some physical and chemical properties nylon such as dye-ability, antimicrobial properties, color fastness, wettability, and shrink-proof characteristics. Moreover, by surface modification the load of hazardous materials in the dyeing waste water can be appreciably decreased.

Recently, chitosan and its derivatives have been extensively used in surface modification of textiles. One of promising chitosan derivative, having numerous terminal amine groups, is chitosanpolypropylene imine dendrimer (CS-PPI) which its potential has been reported as efficient compound in dye removal from wastewater [20], antimicrobial finishing agent for textiles [21], and salt-free reactive dyeing of wool and cotton [22,23].

To the best our knowledge, thus far, the surface modification of nylon using CS-PPI hybrid has not been reported. Therefore, the aim of this study was to determine whether the surface modification with chitosan-poly(propylene imine) dendreimer hybrid (CS-PPI) could improve the dye-ability of the nylon 6 with reactive dyes. Hence, CS-PPI was applied on nylon fabrics according to the dip-dry-cure method. Then, the modified nylon were dyed with two commercial reactive dyes by conventional exhaustion method in order to explore the optimal key parameters of treatment such as CS-PPI concentration, pH, temperature and

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<sup>\*</sup> Corresponding author. Tel.: +98 21 22969774; fax: +98 21 22969774. *E-mail address:* sadeghi-mo@icrc.ac.ir (M. Sadeghi-Kiakhani).

time. Then, color fastness and antibacterial activity against two common pathogen bacteria, namely, *Escherichia coli* and *Staphylococcus aureus* of dyed nylon modified at optimal conditions were investigated and deliberated.

# Experimental

# Materials and apparatus

Chitosan with degree of deacetylation (DD): 98.5% and  $M_W$  = 200 kDa and poly(propylene imine) (PPI) dendrimer (Generation 2,  $M_W$  = 770 g/mol) were obtained from Kitotak Co. (Iran) and Ciba Ltd. (Switzerland), respectively. Polyamide (Nylon 6) knitted fabric (101 g/m<sup>2</sup>) was used. Nonionic detergent (Lotensol, Hansa) was utilized for scouring of fabrics. Two commercial reactive dyes, C.I. Reactive Black 5 (RB5) and C.I. Reactive Red 198 (RR198), were provided from Dystar, and used for nylon dyeing as received without further purification. All other reagents were of analytical laboratory grade.

The dyeing of treated and pristine nylon fabrics was accomplished in acidic media (pH 5) (adjusted by acetic acid) using a laboratory HT dyeing machine. UV-visible absorption spectra of samples were recorded using a Cecil 9200 double beam spectrophotometer. The reflectance characteristics of the dyed samples were measured on a Macbeth spectrophotometer Color-Eye 7000A, color eye reflection spectrophotometer (D65 illumination. 10° observer). Attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) spectra of the samples were recorded in a Nicolet FTIR spectrophotometer (Madison, USA). 2 mg of samples were grinded with100 mg dry potassium bromide (KBr), and pressed into a mold in a uniaxial hydraulic press under a load of 0.9 MPa to obtain IR-transparent pellets. The spectra were then collected in transmittance mode in the region of 4000-400 cm<sup>-1</sup> with resolution of 4 cm<sup>-1</sup>. Morphological analyses of nylon fabrics were carried out with a LEO 1455VP scanning electron microscope (SEM). Fabrics were coated with gold (Au) in a high-resolution sputter coater. Differential scanning calorimetry (DSC) was measured under constant nitrogen purge on a TA Instrument in pierced aluminum pan in the range of 0-300 °C with the scanning rate of 10 °C/min.

# Preparation of CS-PPI hybrid

(5 g) Chitosan (1, Scheme 1) was dissolved in 80 mL water/ methanol 1/1 (v/v) and 1.5 mL acetic acid solution, and then 0.5 mL ethyl acrylate was added to the solution. After stirring at 50 °C for 10 days, the reaction mixture was quenched and precipitated in 80 mL acetone saturated with NaHCO<sub>3</sub>. The precipitate was separated by filtration, and then the filtrates dispersed in 20 mL water saturated with NaHCO<sub>3</sub>. The resulting mixture was dialyzed against 4 L water, and lyophilized to give *N*carboxyethyl chitosan ethyl ester (2, Scheme 1). For the preparation of *N*-carboxyethyl chitosan (3, Scheme 1), the prepared compound (2, Scheme 1) was added to 50 mL NaOH solution; the mixture was stirred for 2 h, dialyzed, and lyophilized as above. The precipitated powders were obtained in quantitative yield of 95%.

(100 mg) Compound (3, Scheme 1) was dispersed in 50 mL methanol, poly(propylene imine) (PPI; G = 2) was added to the prepared suspension and the mixture was stirred at room temperature. After three days, the mixture was evaporated to dryness, dispersed in NaOH solution (0.2 M) at room temperature for 2 h, dialyzed, and lyophilized to yield CS-PPI. The summarized preparation of CS-PPI is shown in Scheme 1, and was explained in detail in our previous work [20,21].

#### Preparation of nylon fabrics

Nylon fabric was scoured with 5 g/L nonionic detergent at 60  $^{\circ}$ C for 20 min, liquor ratio (L:R) of 40:1, rinsed and air dried. A fine powder of CS-PPI was dissolved in citric acid solution (pH 4) and the grafting of nylon fabrics with CS-PPI was carried out according to the dip-dry-cure method as follows:

The scoured fabrics were immersed in CS-PPI solutions at various conditions. The treated fabrics were then dried for 5 min at 70 °C, and cured in the curing chamber at 120 °C for 5 min. In this study, the effects of some important operational parameters such as CS-PPI concentrations (1, 2.5, 7.5 and 12.5 g/L), treatment temperature (40, 60 and 80 °C), treatment time (2, 6 and 12 h), and pH (4, 6 and 7) were evaluated on the grafting process.

### Dyeing method

Pristine and treated nylon fabrics were dyed with 4% owf (on weight of fiber) reactive dye at pH 4 using a buffer solution produced from acetic acid and sodium acetate mixture, the procedure which is recommended by dye manufacturer for dyeing at 60 °C. All dyeing trials were performed using a rapid laboratory dyeing machine at L:R (20:1). The fabrics were wetted for 5 min in the dye bath at 30 °C before the addition of dye. The temperature of dye bath was then gradually raised from 30 °C to 60 °C at rate of 2 °C/min. Dyeing was continued for 60 min at this temperature. After completion of dyeing cycle, the fabrics were rinsed with hot and then cold water, and then air dried.

Effects of operational parameters such as CS-PPI concentration, pH, temperature, and time on the grafting yield were monitored by changes in dye up-take ability of substrates as expressed by color strength (*K*/*S*) values. *K*/*S* values were calculated at  $\lambda_{max}$  (wavelength of maximum absorption) using Kubelka–Munk equation (Eq. (1)) as follows:

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

where *R* is the reflectance, *K* and *S* are the absorbance and scattering coefficients, respectively.

#### Fastness properties

Wash fastness was measured by the standard ISO 105 C06 C2S:1994 (E) method. The washing was conducted for 30 min at 60 °C, rinsed with cold water, air dried, and analyzed with grey scale. Light fastness test ISO 105 B02:1988 (E) was evaluated with the xenon arc lamp using blue reference samples. The rub fastness test was performed according to ISO 105-X12:1993 (E) standard using a crockmeter. For the wet rub test, the testing squares were thoroughly immersed in distilled water; the rest of the procedure was the same as in the dry test. The staining on the white test cloth was assessed according to the gray scale.

The perspiration fastness was assessed in acidic and alkaline media according to the procedure prescribed in the ISO 105-E04:1994 (E) standard. The samples were prepared by stitching a piece of dyed wool fabric between two pieces of adjacent fabrics, all of the same length, and then immersed in the acid and alkaline solutions for 30 min. The staining on the adjacent fabrics was evaluated according to the gray scale.

# Antimicrobial activity of the treated fabrics

Antimicrobial properties of treated fabrics were examined according to ASTM E2149-01, a quantitative antimicrobial test method performed under dynamic contact conditions using test Download English Version:

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