



Photocatalytic discoloration of denim using advanced oxidation process with H₂O₂/UV

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ABSTRACT

Nowadays, various physical and chemical methods have been presented by researchers around the World for discoloration of denim fabrics. Although they have their own advantages and disadvantages, some of them have been used in garment industry for many years. In the present study, indigo dyed denim was discolored with one of the most popular advanced oxidation procedures (AOP) using combination of H₂O₂/UV. We observed a very effective discoloration ($\Delta E^* = 17.44$ after 150 min UV exposure). UV-vis spectra showed the disappearance of absorption peaks of indigo due to decomposition of chromophores and aromatic rings. Based on the FTIR spectra from discolored denim, we concluded that photolysis reactions occurred on denim following by generation of OH $\cdot$ and OOH $\cdot$ radicals. Cotton fibers in discolored denim samples showed disorientation, wrinkling and defibrillation depending on UV exposure duration. Hence, H₂O₂/UV process could be recommended to as a simple and effective method for denim discoloration.

1. Introduction

Denim garments are elegant fashionable textile products with very high level of market all over the World. It has been well-known that unfinished denim textiles are uncomfortable to wear due to their heavy and rigid structures, therefore, it is necessary to achieve a soft handle, an enhanced comfort as well as a desirable worn-out look by finishing [1,2]. Great efforts have been hailed to explore different chemical and physical finishing procedures for discoloration and improving the handle of denim. They include oxidizing-bleaching treatment [3], stonewashing [4], electrochemical reduction [5], bio-washing [6–13], plasma treatment [14–18], laser irradiation [19–25] and combination of nanoparticles with enzymes [26,27]. Oxidizing agents and stone washing with pumice were first introduced in garment industry for aged-look denim finishing [3]. However, several negative impacts were observed on finished products and washing machines by these two methods. To overcome their drawbacks, electrochemical reduction technique has been later adopted to bleach denim fabrics by in-situ producing reactive radical scavengers, but it has not yet been a simple scale-up procedure [5]. Enzymatic treatment of textiles has also been demonstrated as an environmentally friendly method for textile finishing [28–32]. In this regard, great efforts have been devoted for denim finishing by pure laccase [33], mixture of laccase/cellulase [6],

pure acid cellulase [7], pure neutral cellulase [8], mixture of amylase/cellulase/laccase [9], mixture of cellulase/methacrylate copolymer [10] and mixture of liquid ammonia/cellulase [11]. It is worth mentioning that plasma and laser etching methods are recently introduced as environmentally friendly physical approaches to reduce the chemical agents and water consumption usage in textile industry [22,23,34–38]. Several research groups have found that stone-washing technique can be replaced by low temperature plasmas of O₂, air, argon and helium gases for removing the oxidized indigo from denim surface [14–18]. Laser has also been known as another environmentally friendly approach for discoloration of denim in garment industry. However, considerations should be undertaken to control laser procedure parameters in order to avoid damaging in denim structure and preventing reduction in the tensile strength [19–25].

Montazer and his group recently introduced a new method for discoloration of indigo on denim using nanoparticles and reducing agents. In this regard, they reduced silver nitrate on denim in the presence of starch and/or glucose in alkali media. They showed that glucose is able to reduce indigo dye, resulting to an old-look denim yellow garment. They also interestingly observed an antibacterial activity on denim against *E. coli* and *S. aureus* bacteria [39]. Furthermore they developed a nano/bio-finishing process on denim using combination of nanoclay and enzymes. This method proved multifunctional

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Table 1
Major chemical and physical methods for discoloration of denim.

Methods	Advantages	Disadvantages	References
Oxidizing-bleaching treatment	● Strong discoloration power ● Simple application	● Relatively high cost ● Possibility to reduce mechanical properties ● Yellowing effect ● Not environmentally friendly ● Toxic chemicals usage ● Corrosion of equipment ● Unpleasant odor in working environment	[3]
Stone-washing	● Desired vintage effect ● Desired softness ● Desired worn-out look	● Corrosion of equipment ● Time-consuming stone dust removing ● Possible to damage denim ● Not environmentally friendly	[4]
Electrochemical reduction	● Versatility and high-energy efficiency ● Environmentally friendly	● Not available in scale-up ● Relatively high cost	[5]
Bio-washing	● Mild processing conditions ● Environmentally friendly ● Low risk in denim damage ● Recyclability of enzymes	● Necessity of enzyme neutralization ● Chances of back-staining in enzyme treatment ● Inability to create different designs	[6–13]
Plasma treatment	● Environmentally friendly ● Fast discoloration ● No solid waste and air pollution ● Dry operation ● No negative effect on bulk properties	● Necessity to have operational skills ● Necessity to optimize process parameters for efficient discoloration ● Technical challenges in scale-up ● Yellowing effect	[4–18]
Laser irradiation	● Environmentally friendly ● Fast discoloration ● Dry operation ● Repeatability of designs	● Necessity to control laser parameters ● Necessity to have operational skills ● Yellowing effect ● Possibility to reduce the mechanical properties	[19–25]
Combination of nanoparticles with enzymes	● Additional functionality on denim depending on nanoparticle type ● No negative impact on mechanical properties	● Possibility to reduce the mechanical properties ● Not yet commercialized. ● More experiments for optimizing are needed.	[26,27,39]
AOP	● No negative impact to the environment ● No corrosion to equipment ● No yellowing effects ● Efficient discoloration ● No need of stabilizers, acetic acid, caustic soda,... ● No neutralization is required.		[40–43]

ability on denim including higher light fastness, softness and handle as well as antibacterial and antifungal properties [26,27].

In the last few decades, UV catalysis discoloration of dyes in the presence of H_2O_2 has been considered as a simple and cost-effective procedure in textile wastewater treatments. In this context, researchers named this method as AOP for degradation of acid, basic and reactive dyes in water under UV radiation [40–43]. The reaction mechanism for discoloration is proposed based on the formation of H_2O_2 free radicals as active species. However, as far as we know, there is no research program explaining UV-induced aged-look denim in the presence of H_2O_2 [14]. For the first time, the present study introduces AOP method for denim finishing. Table 1 demonstrated a comparison between our proposed method and other previously reported denim discoloration methods.

2. Experimental

2.1. Materials

Desized denim fabric was used with warp yarn count of 468 denier, weft yarn count of 603 denier and area density of 389 g/cm^2 . H_2O_2 was 30% (w/w) from Merck Company. Nonionic detergent (Diadavin UN from Resin Saveh (Saveh, Iran) was used to wash denim fabrics after discoloration.

2.2. Methods

In this study, seven denim samples were first dipped in H_2O_2 solution 30% (40 ml/lit) for 5 min, then rinsed to remove additional solution. A treated sample was kept as a reference and we exposed remaining samples to UV light (PHILIPS TUV 15W G15 T8 Germicidal

Fluorescent Light Bulb, 54 V, 0.34 A, UV-C and $\lambda_{\text{max}} = 253.7 \text{ nm}$, Poland) at different durations including 30, 60, 90, 120 and 150 min. To provide uniform discoloration of fabrics, the humidity was kept constant during the process. Finally, the fabrics were washed with non-ionic detergent, rinsed and completely dried.

Absorption of indigo was performed after discoloration to investigate the possible changes in the chemical structure. The used instrument was UV-vis spectrophotometer from Varian Canadian Company (model CARY 300) and the absorption spectra were measured from 200 to 400 nm. We used deionized water for calibration and dissolving indigo in quartz cells.

FTIR analysis was conducted using a Hartmann & Braun spectroscopy (Bomem-MB100, Canada). It was utilized to investigate the chemical changes occurred on denim before and after discoloration.

The reflectance spectra of samples were measured using Texflash Spectrophotometer (Data Color, Switzerland). The CIELAB color coordinates (L^* , a^* , b^* , and h) were assessed at include mode with geometry of 0/d for a 10° observer and a D65 illuminant source. ΔE as color difference was also measured from the reflectance spectra of samples before and after discoloration with Eq. (1):

$$\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2} \quad (1)$$

where $\Delta L = L^*$ before discoloration – L^* after discoloration; $\Delta a = a^*$ before discoloration – a^* after discoloration; $\Delta b = b^*$ before discoloration – b^* after discoloration.

We also measured the yellowness index (YI) of samples according to Eq. (2):

$$YI = 100 (1.3013 X - 1.1498 Z)/Y \quad (2)$$

Scanning electron microscope was used to investigate the surface morphology of cotton fiber (EM3200, KYKY, China) with acceleration

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