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Decolorization of Viscose Fabric Dye Bath Using UV/H2O2 Process and Reuse of Dyeing Water

Semiha Eren [1] 0000-0002-2326-686X
Aliye Akarsu Özenç [2] 0000-0001-5603-5913
Merve Öztürk [3] 0000-0001-8356-2321

[1]Bursa Uludag University Textile Engineering Department, Görükle, Nilüfer, Bursa, Türkiye
[2]Bursa Uludag University Textile Engineering Department, Görükle, Nilüfer, Bursa, Türkiye
[3]Bursa Uludag University Textile Engineering Department, Görükle, Nilüfer, Bursa, Türkiye

Corresponding Author: Semiha Eren, semihaeren@uludag.edu.tr

ABSTRACT

This study investigates the successful removal of color from wastewater generated during the dyeing of widely used viscose fabrics in the textile industry using the advanced oxidation process, UV/ H_2O_2. Subsequently, the usability of these treated waters for repeated dyeing is examined. No chemicals other than the dye were added during the dyeing processes. The results demonstrate that viscose samples can be dyed successfully up to the 3rd cycle without any significant loss in color difference and fastness values. While no change in pH values was observed until the 7th cycle, there was a decrease in conductivity values, resulting in the samples being dyed in progressively lighter colors. No significant losses in fastness and strength were detected in the samples until the 7th cycle. The textile finishing industry is known for its high water consumption and pollution, contributing to a rapid decrease in accessible clean water resources. This wastewater is discharged into the environment, posing significant environmental challenges. According to the findings of this study, the ability to reuse the same water for up to 3 cycles can offer substantial advantages in terms of both the environment and cost savings.

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KEYWORDS

Decolorization, UV/ H_2O_2, Recycling DyeBath

1. INTRODUCTION

Cellulose, a biopolymer, is considered one of the world's popular raw materials and renewable resources [1]. It finds extensive applications in the textile industry owing to its biodegradability, high moisture absorption, alkali resistance, and abundance on Earth [1]. Viscose, a fiber widely used in textiles, is produced from cellulose xanthate solution, a result of treating cellulose with carbon disulfide and sodium hydroxide [2]. Viscose fiber offers advantages like comfort, biodegradability, and high hydrophilicity, making it a preferred choice [3]. Worldwide, the consumption of viscose fiber has exceeded 6 million tons, with 85-90% of industrial cotton fiber waste being converted into viscose fiber. Due to synthetic fibers' limitations in comfort, viscose fiber production has been gradually increasing.

Water is a fundamental natural resource vital for sustaining life. Researchers have highlighted the gradual depletion of freshwater resources due to population growth and industrial expansion [4,5]. Furthermore, global warming has exacerbated drought conditions in the last 35 years [6]. The textile industry, known for its high water consumption and pollution, stands out as one of the sectors contributing significantly to these issues. Dyeing 1 ton of textile products requires approximately 200-350 m^3 of water [7]. The textile dyeing process involves the use of dyestuffs, alkalis, and salts, contributing to the pollution of water resources. Textile wastewater, with high dyestuff, chemical oxygen demand (COD), and biological oxygen demand (BOD) levels, poses severe environmental threats, endangers aquatic life, elevates river salinity, and poses health risks when released into the environment [8]. COD represents the amount of oxygen required to chemically oxidize organic and inorganic pollutants in water, whereas BOD refers to the oxygen demand needed by microorganisms to decompose organic matter. High COD and BOD levels indicate significant organic and chemical contamination, leading to oxygen depletion in aquatic ecosystems and severe environmental

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consequences. Consequently, treating wastewater from textile processes like bleaching and dyeing is crucial to mitigate environmental damage [9-11].

Textile wastewater treatment primarily employs physical, chemical, and biological processes [12, 13]. However, these conventional methods are often insufficient, resulting in substantial sludge formation [14, 15]. To address these challenges, advanced oxidation processes (AOPs) have gained prominence. AOPs aim to break down organic compounds and convert pollutants into less toxic substances by generating hydroxyl radicals [16, 17, 18]. Combinations of ozone (O_3), hydrogen peroxide (H_2O_2), and UV radiation fall under AOPs, and they have found extensive use in treating industrial wastewater and organic products [19,20,21].

Studies have demonstrated the remarkable effectiveness of advanced oxidation processes (AOPs) in wastewater treatment, particularly the UV/ H_2O_2 process [22, 23]. In this process, hydrogen peroxide and UV radiation combine to generate highly reactive hydroxyl radicals (2·OH radicals) with an oxidation potential of 2.80 eV [17, 24, 25, 26]. These hydroxyl radicals exhibit rapid reactivity with pollutants, ensuring thorough wastewater purification [22].

Numerous studies in the literature explore the recovery and reuse of wastewater employing advanced oxidation processes. Muthukumar et al. (2005) utilized Acid Red 88, Acid Red 18, Acid Orange 7, Acid Orange 10, and Acid Red 73 dyestuffs to remove color from wastewater using ozone gas at a flow rate of 2 L/min [24]. Silk fabrics were dyed twice with the recycled wastewater containing all dyes. The study revealed a decrease in COD values for the recycled wastewater. However, Acid Orange 7 dyestuff was found unsuitable for recycling, and fabrics dyed with the second recycled wastewater had higher ΔE values.

In a study conducted by Senthilkumar and Muthukumar (2007), cotton fabrics were dyed with Reactive Red 5MR and Reactive Golden Yellow MR dyestuffs at a dyeing concentration of 3% [29]. The wastewater produced after dyeing underwent color removal with ozone gas at a flow rate of 2 L/L. The wastewater was recycled a total of 5 times, with cotton fabric dyed at each recycling stage. Results indicated that the cotton fabrics became progressively darker with each recycling cycle. Basrık et al. (2021) dyed polyester yarns CI Disperse Orange 30 and CI Disperse Red 167 using a mix of dyestuffs. Wastewater generated during the dyeing process was ozonized for 45 minutes at an ozone flow rate of 5 L/min, effectively removing color. The wastewater was recycled three times and used for dyeing with the same dyestuffs. This study observed darker yarn colors in the 2nd and 3rd cycles, alongside a 17% reduction in COD values following the 1st cycle [30].

In this study, 100% viscose fabrics were subjected to a dyeing process using Reactive Red 195 dyestuff at a dyeing concentration of 0.1%. The wastewater resulting from the dyeing process was treated for color removal through the UV/ H_2O_2 advanced oxidation method. This color removal process was applied iteratively seven times, and subsequent dyeing processes were conducted seven times using the decolorized wastewater. Importantly, during these repeated dyeing cycles, only dyestuff was reintroduced into the wastewater that had been decolorized using the UV/ H_2O_2 advanced oxidation method.

2. EXPERIMENTAL

2.1. Materials

In this experimental study, 37 g/m² viscose fabric samples were utilized. For the dyeing of the fabrics, Reactive Red 195 dyestuff was employed. Reactive Red 195 (C.I. Reactive Red 195) is an azo-based reactive dye with a molecular mass of 1136.32 g/mol. It has a purity of ≥95%, is highly soluble in water, with a solubility exceeding 100 g/L at 50°C. (https://www.worlddyevariety.com/reactive-dyes/reactive-red-195.html). The molecular structure of the dye was given in Figure 1. The dyeing processes were conducted using soda and salt (Merck, Germany) in a laboratory-scale dyeing machine. Additionally, H_2O_2 (Merck, Germany) was utilized for bleaching the dye solutions.

For the UV-based experiments, an apparatus featuring 254 nm UV lamps was employed. This apparatus was equipped with a total of 18 lamps, providing a combined power output of 470 Watts. Figure 2 illustrating the UV cabin's appearance. These lamps were strategically positioned on both sides and the top of the UV cabinet. The dimensions of the UV cabinet used in the experiments measured 100 x 70 x 138 cm.

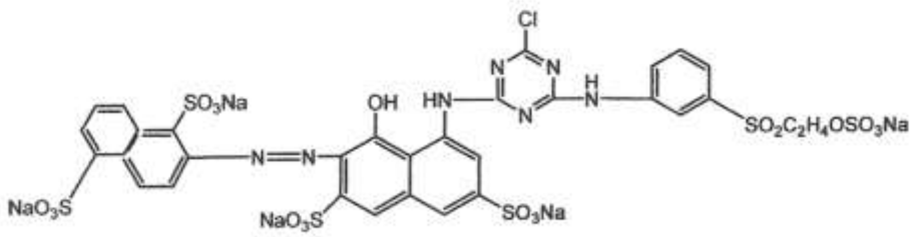


Figure 1. Reactive Red 195 Molecular Structure

2.2. Method

Fabric Dyeing

In the dyeing processes, fabric samples were dyed using a 0.1% dyestuff concentration. The dyeing was carried out in a bath with a solution ratio of 1:10, containing 4.5 g/L of salt and 3.75 g/L of soda, at a temperature of 60°C for a duration of 60 minutes. Each staining process was repeated three times. Following the initial dyeing, the dye solution was subjected to bleaching under UV light in the presence of H_2O_2. Subsequently, only a 0.1% dyestuff was added to the bleached solution, and the dyeing process was repeated. This cyclic process was repeated for a total of 7 cycles.

Bleaching the Dye Bath

Upon the completion of each dyeing process, the remaining solution in the dyeing tube was transferred to a glass container. To this solution, 40 mL/L of H_2O_2 was added, and the container was then exposed to UV light within the UV cabinet. Throughout each decolorization cycle, pH and conductivity values were recorded.

Figure 2. UV Cabinet with Dye Bath

Test Methods

Conductivity measurements were conducted using the WTW Cond 3210 model conductivity measuring device, while pH measurements were carried out with the WTW PH 3210 model pH measuring device.

Absorbance values of the decolorized dyebath were performed using a Spectroquant Pharo 300 model UV-visible spectrophotometer.

For COD measurements of conventional bleaching water and ozonation waters, the test was carried out for 2 hours at 148°C using Merck COD measurement kits and a WTW CR 2200 model thermoreactor. The test result was measured with a Spectroquant Pharo 300 model UV-visible spectrophotometer. This measurement was made according to the standard titrimetric method (Standard Methods 5220 C: Closed Reflux, Titrimetric Method, APHA, 19th ed., American Public Health Association, 1995).

The washing fastness of the dyed samples was assessed using the Test Laboratory Instrument (model 412 NB HT).

For color measurements and fastness evaluations (ΔE values) of the samples, a Konica Minolta CM3600D model reflectance spectrophotometer was employed.

Tensile strength tests were conducted using the SHIMADZU Model AG-Xplus (Kyoto, Japan) test device in accordance with ISO 13934-1.

Statistical Analysis: Data analysis was performed using SPSS Statistics Software (Version 23.0; IBM Corp). The data's distribution was assessed using the Shapiro-Wilk test. Parametric data were examined using the Student t-test. For all tests, significance was determined at $p < 0.05$.

3. FINDINGS

3.1. Bleaching the DyeBath

The decolorization of the dye bath was achieved through a process involving the presence of UV light and H_2O_2 in each cycle. The photooxidation efficiency was found to be high when the pH was below 6, but it decreased when the pH exceeded 6 due to a reduction in OH radical formation, as reported by Hameed and Mousa (2019), Galindo and Kalt (1999), and Chang et al. (2010) [31, 32, 33]. In this study, the pH values in Table 1 were observed to be 10 and above. Therefore, the UV processing time was set to 120 minutes, aligning with established practices in the literature as suggested by Muruganandham and Swaminathan (2004) and Eren (2018) [27,28]. Wavelength-absorbance values, after decolorization of the solutions at the end of each cycle, are depicted in Figure 3. As Figure 3 illustrates, the color of the dyeing wastewater was effectively eliminated at the conclusion of each cycle.

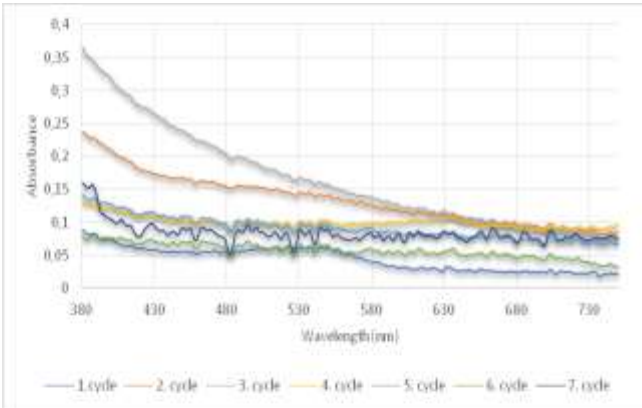


Figure 3. Wavelength-Absorbance Graphs of the Samples

3.2. pH and Conductivity Values

In Table 1, the pH and conductivity values of the dyebath before (in) and after (out) decolorization, measured in each cycle, are presented. These data reveal that there were no significant changes in pH values. It is noteworthy that the oxidation process is primarily driven by hydroxyl radicals (•OH) generated through the direct photolysis of H_2O_2 under UV irradiation, a well-documented phenomenon in the literature [34, 35].

$$H_2O_2 + hv \rightarrow 2OH\cdot \qquad (1)$$

Mitrovic et al. (2011) observed that there was no significant change in pH within strongly basic (pH 10-12) and strongly acidic (pH 2.08-3.2) environments during UV/H_2O_2 advanced oxidation processes [36]. They attributed this to the insufficient formation of weak organic acids required to neutralize the basic environment.

Multiple researchers, including Nagel-Hassemer et al. (2012) and Bezerra et al. (2021), have pointed out that the use of salt in the dyeing process increases the conductivity of the dye bath [37, 38]. A consistent reduction in conductivity values indicates a decrease in the dissolved salt content within the dye bath, which is a result of the color removal process. López-Grimau et al. (2012) noted that the color of the dyeing bath was electrochemically removed following reactive dyeing, resulting in decreased conductivity values after each treatment [39].

It's noteworthy that the conductivity value decreased by a similar amount after each decolorization application. However, with each recycling, the quantity of salt required to restore the conductivity to the initial dye bath level increased.

3.3. Color Measurements Results

At the conclusion of each cycle, the discolored samples were compared to the initial sample, and the color measurement results are detailed in Table 2. It was observed that at the end of the third cycle, the ΔE values exceeded 1. As previously mentioned, this outcome is linked to the decrease in the salt content in the solution. The literature supports the necessity of a basic dyeing medium and the use of salt to enhance the absorption of reactive dyestuffs by cotton samples [40]. Similarly, Imada and Harada (1992) noted that the success of dyeing relies on the degree of primary depletion, which is contingent on the quantity of inorganic salt in the dye bath [41]. Likewise, upon examining Figure 4, it becomes evident that the K/S_{sum} values of the cycle samples remained consistent until the third cycle but declined after the third cycle. In Figure 5, images of the samples are displayed. Remarkably, it was observed that ΔE remained below 1 when only dyestuff was added to the decolorized wastewater bath until the third cycle. No salt or any additional chemicals were introduced into the dye bath during these cycles. Bezerra et al. (2021) also suggested that cotton dye wastewater, decolorized using the H_2O_2/UV advanced oxidation method, could be used for three cycles, while textile wastewater required the addition of salt for the same purpose [38].

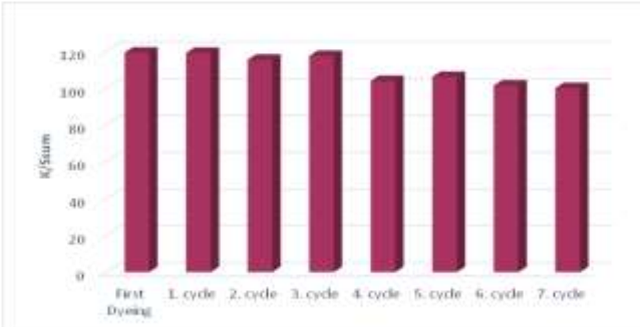


Figure 4. K/S_{sum} Graphs of the Samples

Table 1. pH and Conductivity Values of the Samples

Samples	pH_{in}	pH_{out}	$Conductivity_{in}$	$Conductivity_{out}$
1. cycle	10,8	10,7	47,65	46,75
2. cycle	10,8	10,7	45,35	42,75
3. cycle	10,8	10,7	43,50	42,30
4. cycle	10,8	10,7	31,50	30,45
5. cycle	10,8	10,8	19,50	19,45
6. cycle	10,7	10,5	15,85	15,75
7. cycle	10,6	10,5	11,85	11,40

Table 2. Color Measurement Results of Samples

Samples	DL	Da	Db	ΔE
1. cycle	0,1485	0,03	-0,047	0,431
2. cycle	0,04	0,7875	0,383	0,72
3. cycle	0,425	0,588	0,4255	0,9215
4. cycle	1,372	-0,8725	0,3725	1,844
5. cycle	0,8155	-0,399	-0,2005	1,5885
6. cycle	2,1695	0,4655	0,688	2,41
7. cycle	2,0595	1,3065	1,097	2,6915

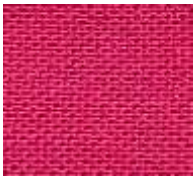
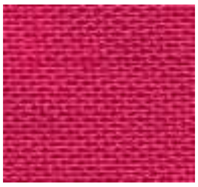
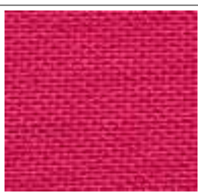
Cycles	Sample Images
First Dyeing	
1.cycle	
2.cycle	
3.cycle	
4.cycle	
5.cycle	
6.cycle	
7.cycle	

Figure 5. Images of samples

3.4. COD Test Results

COD is a test method that measures the oxygen value of organic matter in a water sample using a strong chemical oxidizer and is the most widely used method in wastewater characterization [42]. COD kits capable of measuring in the range of 300-3500 mg/L were used. In Figure 6, the COD values of the dyeing wastewater from the first dyeing cycle, the second dyeing cycle wastewater, the fourth dyeing cycle wastewater, and the seventh and final dyeing cycle wastewater are presented. When the results are examined, a decrease in COD values was observed as the conductivity values in the wastewater baths decreased.

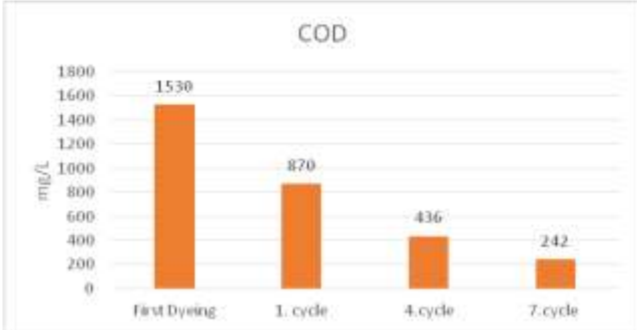


Figure 6. COD measurement results for wastewater cycles

3.5. Washing Fastness Results

The washing fastness values of the samples are presented in Table 3 in detail. As a result of the examinations, it was determined that there was no significant difference between the washing fastnesses of the samples during 7 cycles and the results obtained were in commercially acceptable standards. Likewise, in a study conducted by Rosa et al. (2020), it was reported that when cotton fabrics were dyed with mixed dyestuffs and subsequently treated with the H_2O_2/UV advanced oxidation method, there were no significant differences in the color fastness of the dyed fabrics even after 15 cycles [43].

3.6. Tensile Strength Results

The breaking load data for the samples are visually illustrated in Figure 7. Upon close examination of Figure 7, it becomes evident that there is no significant difference in the breaking loads among the samples. Statistical analysis using SPSS yielded no statistically significant difference in breaking loads among the samples (p= <0.001). The standard deviation values for the first and second measurements were 26.78 and 27.80, respectively, with corresponding coefficient of variation (CV) values of 9.44% and 9.87%

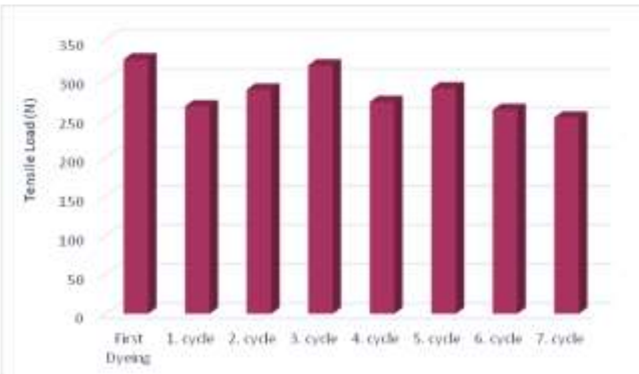


Figure 7. Breaking Load Graph of the Samples

Table 3. Washing Fastness Values for the Samples

Samples	Wool	Acrylic	Polyester	Polyamide	Cotton	Acetate
First Dyeing	4-5	4-5	4-5	4-5	4-5	5
1. cycle	4-5	4-5	4-5	4-5	4	4-5
2. cycle	4-5	4-5	4-5	4-5	4	4-5
3. cycle	4-5	4-5	4-5	4-5	4	4-5
4. cycle	4	4-5	4-5	4-5	4	4-5
5. cycle	4-5	4-5	4-5	4-5	4	4-5
6. cycle	4-5	4-5	4-5	4	4	4-5
7. cycle	4-5	4	4-5	3-4	4	4-5

4. CONCLUSION

This study delves into the effective removal of dyeing wastewater from viscose fabric, a prevalent material in the textile industry, using the UV/ H_2O_2 method—an advanced oxidation process. Additionally, it explores the feasibility of reusing this treated wastewater for subsequent dyeing processes without the introduction of any additional chemicals aside from dyestuff. The findings reveal that viscose samples can be dyed successfully without experiencing any notable discrepancies in color variation or fastness values, even up to the 3rd cycle. While the pH value remained unchanged throughout the 7 cycles, there was a reduction in conductivity values, resulting in the samples being dyed in progressively lighter shades. Importantly, no significant loss in fastness or tensile strength was detected in the samples up to the 7th cycle.

The textile finishing industry is recognized for its substantial water consumption and pollution, contributing to the rapid depletion of accessible clean water sources. The discharge of industrial wastewater into the environment compounds these concerns. As per the outcomes of this study, the ability to reuse the same water for up to 3 cycles presents significant advantages in both environmental and cost-saving contexts.

Author contribution SE, AAÖ and MÖ: conceptualization, methodology, investigation, writing — original draft, writing — review and editing.

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Data availability The data supporting this study’s findings are available on request.

Declarations

Ethical approval and consent to participate The author declares that the study has no human participants, human data, or human issues.

Consent for publication The authors do not have any person’s data in any form.

Competing interests The authors declare no competing interests.

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