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Reactive Dyeing of Wool Fabric Using Recycled Dyeing Wastewater

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Abstract: Aiming to solve the problem of large discharge and severe pollution of reactive dyeing wastewater for wool fabrics, peroxodisulfate (SPS) was used for the degradation and recycling of dyeing wastewater containing reactive dye Lanasol Red CE. The process of degrading the reactive dye was determined by using the dye residual rate as the evaluation index. The feasibility of reactive dyeing of wool fabrics using recycled dyeing wastewater was confirmed by measuring the dye uptake, exhaustion and fixation rates, as well as color parameters and fastness of the dyed fabrics. The results showed that the appropriate conditions for degrading Lanasol Red CE were 0.2 g/L SPS, an initial pH value of 3 and 100 ℃ for 30 min. Under these conditions, the dye degradation rate was as high as 93.14%. When the recycled dyeing wastewater was used for dyeing of wool fabrics, the exhaustion rate of Lanasol Red CE exceeded 99%, and the fixation rate was higher than that achieved by the conventional dyeing process. Under the same dyeing conditions, the recycled-dyed fabrics appeared darker. When the number of cycles was fewer than five, the effect on color fastness was not obvious. Although the color fastness to rubbing and washing of the fabrics dyed in the 10th cycle decreased by half a grade and 1 grade, respectively, compared to that of the fabrics dyed with the conventional dyeing process, they still met the production requirements.

Keywords: wool fabric; reactive dye; dyeing wastewater; recycling; dyeing

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0 Introduction

Wool products are highly regarded by consumers because of their exceptional elasticity, luxurious feel and excellent moisture absorption properties[1]. Wool reactive dye is designed specifically for wool fibers, offering advantages such as vibrant colors, easy application, uniform dyeing and excellent fastness[2]. However, the wool fiber dyeing process generates substantial amounts of dyeing wastewater, causing severe environmental pollution.

Printing and dyeing wastewater exhibits deep color, high alkalinity, high organic pollutant contents, and significant variations in water quality, making it one of the most challenging types of industrial wastewater to treat[3-4]. To meet the requirements for the reuse of printing and dyeing wastewater, the primary treatment targets include reducing chemical oxygen demand (COD), biochemical oxygen demand (BOD) and chromaticity[5]. Three main treatment technologies are employed for dealing with dyes in printing and dyeing wastewater: physical methods (such as adsorption, membrane separation and coagulation-flocculation), chemical methods (including chemical oxidation, photocatalytic oxidation and electrochemical processes), and biological methods (comprising anaerobic, aerobic and combined anaerobic-aerobic treatments)[6-7].

Current methods for treating printing and dyeing wastewater include adsorption, membrane separation and advanced oxidation[8]. The adsorption method can capture various toxic and hazardous substances, making it suitable for treating different types of printing and dyeing wastewater[9-10]. Additionally, the cost of preparing adsorbents is relatively low. Nevertheless, adsorbents need periodic replacement, and the waste adsorbents can cause secondary pollution. The research demonstrated that activated carbon adsorption treatment of printing and dyeing wastewater achieved a COD removal rate of 66% with a theoretical adsorption capacity of 2.11 mg/g after static adsorption for 30 min[11]. However, this method exhibits poor adsorption effects for water-soluble dyes and low regeneration rates. The membrane separation method is simple to operate and can recover reusable substances[12-14]. Sun et al.[15] prepared a thiourea-modified graphene oxide membrane and investigated its removal efficiency of methyl orange, rhodamine B and methylene blue, which reached 94.01%, 87.07% and 99.67%, respectively. However, in practice, membranes are prone to clogging due to contaminants and incur high production cost.

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The advanced oxidation method exhibits high oxidation potential, rapid degradation rates, high efficiency in degradation and minimal secondary pollution, making it a standout method for treating printing and dyeing wastewater[16]. Advanced oxidation is the degradation of dye molecules in printing and dyeing wastewater with the help of oxidation techniques such as Fenton oxidation, ozone oxidation and persulfate oxidation[17]. Among these oxidation techniques, Fenton oxidation offers mild and efficient reaction conditions and is relatively easy to operate. However, it requires the use of large amounts of H_2O_2. Additionally, the side reaction between Fe^{2+} and H_2O_2 reduces the utilization of H_2O_2, and the high cost of H_2O_2 adds to the overall expense[18]. Although ozone can effectively remove organic matter and color from printing and dyeing wastewater, it struggles to achieve deep treatment effects or to meet reuse standards when used alone[19-20]. Persulfate oxidation produces free radicals with strong oxidizing ability during the reaction process, which decomposes pollutants with complex structures that are difficult to degrade into CO_2, H_2O and inorganic salts[21-23]. The generated inorganic salts can serve as dyeing promoters, but the relevant studies on recycling them from dyeing wastewater for reuse in wool fabric dyeing have rarely been reported.

This paper focuses on the degradation and recycling treatment of dyeing wastewater containing Lanasol Red CE through the thermal activation of peroxodisulfate (SPS). The effects of three factors, namely the temperature, pH value and SPS mass concentration on the dye degradation were discussed. Subsequently, the recycled dyeing wastewater was used as a dyeing medium for wool fabric dyeing. The dye uptake, exhaustion and fixation rates, color parameters and color fastness of the dyed fabrics were compared with those of the fabrics dyed with the conventional dyeing process.

1 Materials and Methods

1.1 Materials

Lanasol Red CE was purchased from Huntsman Chemical Co., Ltd. (Shanghai, China). Glacial acetic acid (analytic reagent) was purchased from Tianjin Oubokai Chemical Co., Ltd. (Tianjin, China). Anhydrous sodium carbonate (analytic reagent) was purchased from Tianjin Baishi Chemical Co., Ltd. (Tianjin, China). Soap flakes were obtained from Shanghai Textile Industry Technical Supervision Institute (Shanghai, China). SPS was purchased from Tesco Chemical Co., Ltd. (Hubei, China). Other chemicals were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Wool fabrics with an area density of 280 g/m^2 were provided by Shandong Ruyi Woolen Garment Group Co., Ltd. (Shandong, China).

1.2 Experimental methods

1.2.1 Preparation of simulated dyeing wastewater

Simulated dyeing wastewater was prepared using Lanasol Red CE at a mass concentration of 0.05 g/L. The simulated dyeing wastewater (100 mL) was used to investigate the effects of various reaction conditions on the dye residual rate by varying the degradation temperatures (25 ℃ to 100 ℃), pH values (3 to 11) and SPS mass concentrations (0.2 g/L to 0.4 g/L).

1.2.2 Reactive dyeing process of wool fabrics

The dyeing process of wool fabrics was carried out according to the process curve shown in Fig. 1, with a dye dosage of 2% (on the mass of fabrics) and a bath ratio of 1:40.

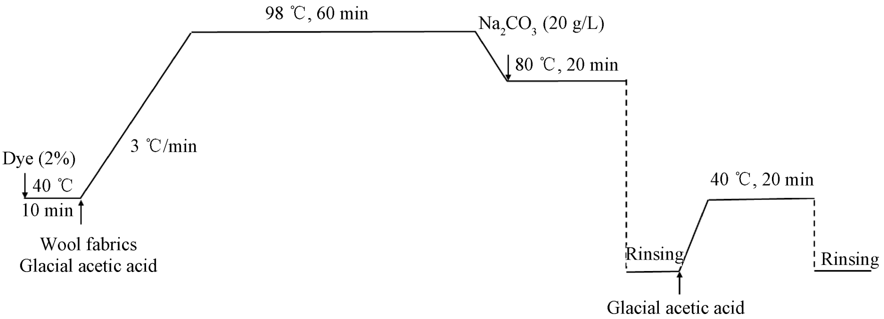

Fig. 1 Dyeing process of wool fabrics with Lanasol Red CE

Wool fabrics were dyed using an AS-I rotary room-temperature dyeing machine (Guangdong Heshan Hongfa Dyeing & Finishing Machinery Manufacturing Co., Ltd., China). After the dyeing process, dyeing wastewater was heated to 100 ℃, and SPS (0.2 g/L) was added and treated the dyeing wastewater for 30 min to obtain reusable water. Subsequently, Lanasol Red CE was added to prepare the dyeing solution for the next round of dyeing. The above process was repeated for 10 cycles, with the wastewater of each dyeing cycle collected separately. The color parameters of the dyed fabrics were measured after each dyeing cycle, and the exhaustion and fixation rates were determined after each dyeing cycle.

1.3 Analyses and measurements

1.3.1 Degradation of dyeing wastewater

According to Lambert-Beer's law, the absorbance *A* of a parallel beam of monochromatic light passing through a uniform, non-scattering and light-absorbing substance is directly proportional to the mass concentration *C* of the substance and the thickness of the absorbing layer.

Therefore, with a constant thickness, A is proportional to C. Hence, the dye residual rate S and the first-order dye degradation rate constant k are

$$S = \frac{C_{t_1}}{C_0} \times 100\% = \frac{A_{t_1}}{A_0} \times 100\%, \tag{1}$$

$$k = -\ln(C_{t_1}/C_0)/t_1, \tag{2}$$

where t_1 is the degradation time; C_{t_1} and A_{t_1} represent the mass concentration and absorbance of the dyeing solution at time t_1, respectively; C_0 and A_0 represent the mass concentration and absorbance of the original dyeing solution, respectively.

1.3.2 Dye uptake measurement

The absorbance of the original dyeing solution and the residual dyeing solution was measured separately. The dye update D of the reactive dye is

$$D = \frac{A_0 - A_{t_2}}{A_0} \times 100\%, \tag{3}$$

where t_2 is the dyeing time; A_{t_2} represents the absorbance of the residual dyeing solution at time t_2.

1.3.3 Exhaustion rate and fixation rate measurements

The exhaustion rate E of the reactive dye is

$$E = \frac{A_0 - A_e}{A_0} \times 100\%, \tag{4}$$

where A_e represents the absorbance of the residual dyeing solution at the end of the dyeing process.

The dyed fabrics were washed at a bath ratio of 1∶50 with soap flakes (2 g/L) to get the soaped residue liquor. Absorbance measurements were performed on the soaped residue liquor, the residue dyeing solution and the original dyeing solution. The fixation rate F of the reactive dye is

$$F = \left(1 - \frac{A_e + A_2}{A_0}\right) \times 100\%, \tag{5}$$

where A_2 represents the absorbance of the soaped residue liquor.

1.3.4 Color performance analysis

The color parameters of the fabric samples were measured using a Color i5 colorimeter (Datacolor Corporation, USA) at a 10° viewing angle with D_{65} illumination. The measured L^*, a^*, b^*, C^*, h and K/S values were averaged. L^*, a^*, b^*, C^*, h and K/S are the brightness, red-green index, yellow-blue index, color saturation value, hue value and apparent color depth value of the dyed fabrics, respectively.

The color difference ΔE^* of the dyed fabrics is

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}, \tag{6}$$

where ΔL^* is the difference of the brightness; Δa^* is the difference of the red-green index; Δb^* is the difference of the yellow-blue index.

1.3.5 Color fastness measurement

The color fastness of dyed fabrics to dry and wet rubbing was tested according to GB/T 3920—2008 Textiles—tests for color fastness—color fastness to rubbing. Color fading and staining were assessed using a gray scale to measure the color change. Washing fastness was tested according to GB/T 3921—2008 Textiles—tests for color fastness—color fastness to washing, using a washing color fastness tester (Wenzhou Darong Textile Instrument Co., Ltd., China) under the following conditions: 5 g/L soap flakes, 40 ℃, 30 min, and a bath ratio of 1∶50.

1.3.6 Determination of SPS mass concentration

The standard curve of SPS mass concentration vs. absorbance was plotted by potassium iodide chromatography, and the absorbance was then calculated to obtain the mass concentration of the remaining SPS in the dyeing wastewater. An SPS solution with a mass concentration of 250.000 mg/L was prepared and successively diluted to obtain SPS standard solutions with mass concentrations of 125.000, 62.500, 31.250 and 15.625 mg/L. Then, the SPS standard solutions (1 mL) were taken and reacted with potassium iodide solution (100 g/L) for 15 min. Subsequently, the absorbance was measured at 400 nm using a UV-3200 UV-Vis spectrophotometer (Shanghai Mapada Instrument Co., Ltd., China) and the standard curve was plotted as shown in Fig. 2 (y represents the SPS mass concentration, x represents the absorbance, and R represents the correlation coefficient).

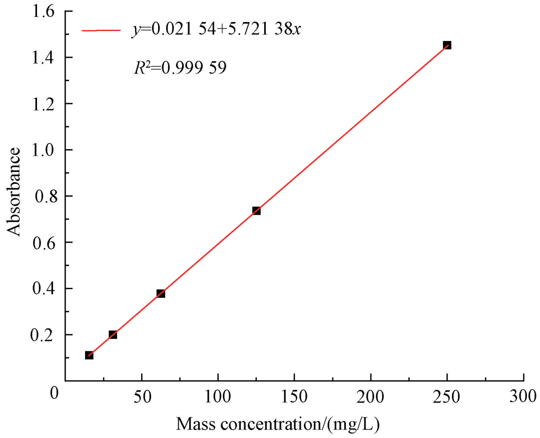

Fig. 2 Standard curve of SPS mass concentration vs. absorbance

Figure 2 illustrates a strong correlation between SPS mass concentration and absorbance, indicating that absorbance can serve as a reliable metric for quantifying the SPS mass concentration.

2 Results and Discussion

There are two prerequisites for recycling dyeing wastewater from the reactive dyeing of wool fabrics: one is the efficient treatment of colored wastewater and the other is the application of recycled wastewater to dyeing without affecting the dyeing behavior of the dye. Dyeing wastewater contains hydrolyzed dye, inorganic salts and alkali, and may impact the effectiveness of wastewater treatment[24]. Therefore, the dye residual rate was used as the evaluation index to explore the impact of the degradation temperature, pH value and SPS mass concentration on the degradation of the reactive dye.

2.1 Influencing factors of dye degradation

2.1.1 Degradation temperature

To investigate the effect of the degradation temperature on the treatment of dyeing wastewater, experiments were conducted under neutral conditions. The mass concentration of SPS was set at 0.8 g/L, and the degradation temperatures were set at 25, 50, 80, 90 and 100 ℃, respectively, for degradation time ranging from 0 to 30 min. The dye residual rate and the first-order dye degradation rate constant of Lanasol Red CE are shown in Fig. 3.

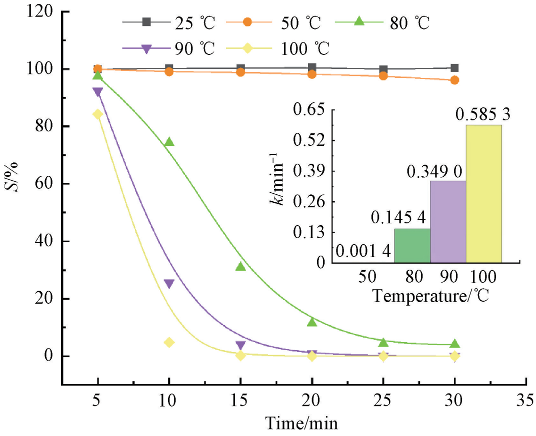

Fig. 3 Effect of degradation temperature on dye residual rate

As shown in Fig. 3, when the degradation temperatures are set at 25 ℃ and 50 ℃, the dye residual rates are higher than 96.0% even after treating the dyeing wastewater for 30 min, which means that dye degradation could not be achieved using SPS below 50 ℃. When the degradation temperature increases to 80 ℃ and the degradation time extends from 5 min to 30 min, the dye residual rate gradually decreases from 97.5% to 4.0%. When the degradation temperature exceeds 80 ℃, the dye residual rate decreases significantly. This suggests that at the same degradation time, higher degradation temperatures result in lower dye residual rates, indicating more dye degradation. In addition, from the effect of temperature on the dye degradation rate constant in Fig. 3, it can be seen that when the degradation temperature increases from 50 ℃ to 100 ℃, the dye degradation rate constant increases from 0.001 4 min^{-1} to 0.585 3 min^{-1}, indicating that increasing the temperature is favorable for the degradation of the dye by SPS. The mentioned phenomenon is attributed to the fact that increasing the temperature activates SPS, which generates a large number of free radicals (·OH and $SO_4^{-}\cdot$) and destroys the structure of Lanasol Red CE. To ensure the complete degradation of the reactive dye, the degradation temperature is set at 100 ℃ in subsequent experiments.

2.1.2 Initial pH value

To reveal the influence of the initial pH value on the degradation of dyeing wastewater, the pH value was adjusted to 3, 7 and 11, respectively. The mass concentration of SPS was maintained at 0.8 g/L, and the degradation was conducted at 100 ℃ for 0–30 min. The dye residual rate and the first-order dye degradation rate constant of Lanasol Red CE are shown in Fig. 4.

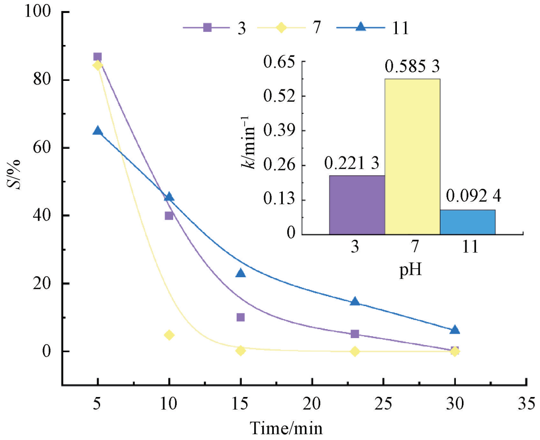

Fig. 4 Effect of initial pH value on dye residual rate

As shown in Fig. 4, when the pH value is 7, the dye residual rate decreases with degradation time, indicating that the majority of the reactive dye has been degraded. When the pH value is adjusted to 11 with Na_2CO_3 solution, the dye degradation rate slows down. The result suggests that a longer degradation time is necessary under the alkaline condition to completely degrade the reactive dye. When the pH value is 3, the dye residual rate decreases to 10.01%. As illustrated in Fig. 4, the dye degradation rate constant increases from 0.092 4 min^{-1} to 0.585 3 min^{-1} when the pH value decreases from 11 to 7. Then, it subsequently decreases to 0.221 3 min^{-1} when the pH value is further adjusted to 3. However, the degradation rate is excessively rapid under neutral conditions, making it challenging to control the reaction process. Considering the complete degradation of the reactive dye and the reuse of dyeing wastewater as a medium for dyeing wool fabrics, the initial pH value is set at 3 for subsequent experiments.

Wool fabric dyeing does not necessitate the addition of salt, resulting in dyeing wastewater that is free of salt. The addition of Na_2CO_3 to the dyeing wastewater affects not only the rate of dye degradation but also the residual rate corresponding to 30 min of degradation. This effect is attributed to the scavenging action of CO_3^{2-} in Na_2CO_3 on hydroxyl radicals in the solution. When CO_3^{2-} is present in the dyeing wastewater, it competes with dye molecules for hydroxyl radicals (·OH) in the solution, reacting to form carbonate radicals ($CO_3^- \cdot$)[25]:

$$CO_3^{2-} + \cdot OH \rightarrow OH^- + CO_3^- \cdot. \qquad (7)$$

Thus the number of active radicals is reduced, leading to a significant decrease in degradation effect.

2.1.3 SPS mass concentration

During the dye degradation process, the number of free radicals determines the dye degradation effect, and the SPS mass concentration is closely related to the number of free radicals. To identify the minimum effective SPS mass concentration that achieves efficient dye degradation while maintaining cost effectiveness, a lower SPS mass concentration of 0.2–0.4 g/L was used at a pH value of 3, and the degradation was carried out at 100 ℃ for 30 min. The effect of SPS mass concentration on dye residual rate and the first-order dye degradation rate constant of Lanasol Red CE is shown in Fig. 5.

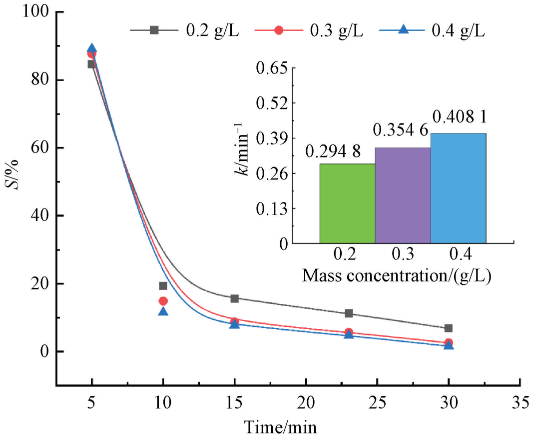

Fig. 5 Effect of SPS mass concentration on dye residual rate

As shown in Fig. 5, Lanasol Red CE degrades rapidly in the first 10 min. After 10 min, the dye residual rate decreases slowly with the prolongation of the degradation time. The higher the mass concentration of SPS, the lower the dye residual rate at the same treatment time. The dye residual rate decreases from 6.86% to 1.65% when the SPS mass concentration increases from 0.2 g/L to 0.4 g/L, and the dye degradation rate constant increases from 0.294 8 min^{-1} to 0.408 1 min^{-1}. At the end of the degradation, the dyeing wastewater is clarified and transparent, which can achieve a good degradation effect and can be used for subsequent recycled dyeing. Excessive mass concentration of SPS may lead to its incomplete decomposition, and SPS will remain in the dye solution, affecting the subsequent dyeing. Therefore, 0.2 g/L SPS was chosen for the subsequent experiments.

2.2 Recycling reactive dyeing of wool fabrics

SPS is converted to sodium sulfate salt, which remains in the degradation solution. Meanwhile, thermally activated reactive dyes are degraded into small molecules. The dyeing behavior of Lanasol Red CE may be affected by the use of recycled dyeing wastewater. To reveal the dyeing behavior of reactive dye for wool fabrics, cycle dyeing experiments were conducted.

2.2.1 Dye uptake

To adequately degrade the reactive dye in the dyeing wastewater, the dyeing wastewater was treated with 0.2 g/L SPS at 100 ℃ for 30 min. Subsequently, the dyeing of wool fabrics was carried out following the dyeing method outlined in the experimental section. The results of the dye uptake are shown in Fig. 6.

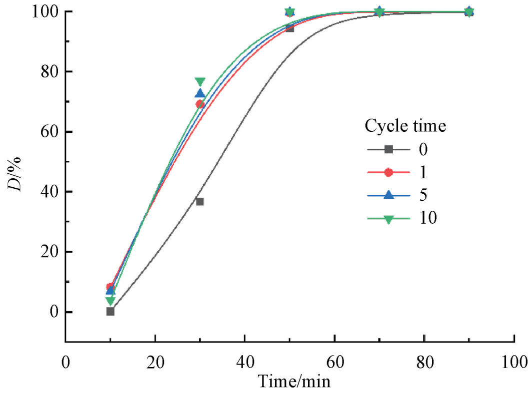

Fig. 6 Effect of cycle times on dye uptake

In the conventional dyeing process (cycle time 0), dye uptake increases slowly with the dyeing time, which reaches 94.39% at 50 min and eventually stabilizes at 99.62% at 70 min. When the dyeing wastewater is used for dyeing, the dye uptake increases compared to that of the conventional dyeing process, and the increase becomes more noticeable with each subsequent cycle. Taking dyeing for 30 min as an example, the dye uptake is 36.65% when dyeing with a conventional dyeing process. The dye uptakes of 1, 5 and 10 cycle times are 69.07%, 72.55% and 76.92%, respectively. Although the cycle time changes the dye uptake, it does not impact the final exhaustion rate. Compared to conventional dyeing process, the dyeing wastewater contains inorganic salts. These salts facilitate the dye molecules to detach from the dye solution and penetrate into the wool fiber, leading to a high dye uptake at the beginning of the dyeing process[26].

2.2.2 Exhaustion rate and fixation rate

Figure 7 shows that the exhaustion rate of Lanasol Red CE is almost unaffected by the cycle times, reaching over 99.00%, while the fixation rate shows a trend of initially increasing and then stabilizing. The fixation rate of conventional dyeing is 82.90%, which is lower than that of the recycling dyeing. The fixation rate suggests that using recycled dyeing wastewater for wool fabric dyeing can achieve higher fixation rates.

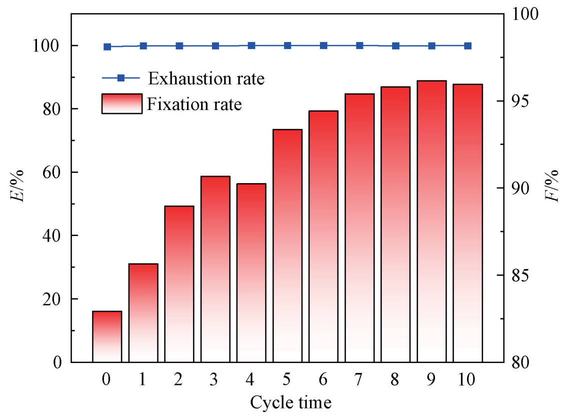

Fig. 7 Exhaustion rate and fixation rate of Lanasol Red CE

2.2.3 Color parameters and color fastness

To investigate the dyeing effect of Lanasol Red CE in two different dyeing media, the color parameters of the dyed fabrics were tested. The results are shown in Table 1. The brightness value L^* of the dyed fabrics using the recycled dyeing wastewater decreases. This indicates that the dyed fabrics are darker, which coincides with the increase in the apparent color depth K/S value by 0.47 to 2.43. The increase in the red-green index a^*, yellow-blue index b^*, color saturation value C^* and hue value h indicates that the color of the cycle-dyed fabrics exhibits enhanced reddish, bluish and bright hues. At the same dye dosage, the apparent color depth K/S value of the dyed fabrics increases, indicating that the dyed fabrics have higher color yields, which coincides with the higher color fixation rate shown in Fig. 7. Compared to that of the conventional dyeing process, the color differences ΔE^* for the 1st, 5th and 10th dyeing cycles are 0.93, 2.25 and 4.02, respectively. This indicates that the color difference increases with the increasing dyeing cycle times. The dyed fabric with ΔE^* between 2.00 and 5.00 is considered as a conditionally accepted product. Although there are slight color variations, the color consistently matches that of the conventional dyeing process. The color parameters are slightly different but can be corrected by adjusting the dyeing process variables[27].

Table 1 Color parameters of dyed fabric

Cycle time	L^*	a^*	b^*	C^*	h	K/S	ΔE^*	Color block of picture pattern
0	40.69	59.13	12.36	60.41	11.80	18.50	—	🟥
1	40.36	58.78	13.16	60.02	11.67	18.97	0.93	🟥
5	40.67	59.39	14.59	61.16	13.79	20.16	2.25	🟥
10	40.07	59.57	16.31	61.77	15.31	20.93	4.02	🟥

The color fastness to rubbing and washing of the dyed fabrics is presented in Table 2. The color fastness to rubbing is the same as that of conventional dyeing when the cycle times are less than 5, while the color fastness to washing shows a decrease of half a grade. The color fastness to rubbing and washing of the 10th cycle dyed fabrics decreases by half a grade and 1 grade, respectively, but all of them meet the production requirements.

Table 2 Color fastness to rubbing and washing of dyed fabrics

Cycle time	Rubbing fastness		Washing fastness		
	Dry	Wet	Dyed fabric discoloration	Wool fabric stained	Cotton fabric stained
0	4	4	4–5	4–5	4–5
1	4	4	4	4	4
5	4	4	4	4	4
10	3–4	3–4	3–4	3–4	3–4

3 Conclusions

As for the dyeing wastewater of Lanasol Red CE, the appropriate degradation conditions were 0.2 g/L SPS, an initial pH value of 3 and 100 ℃ for 30 min. The dye degradation rate reached as high as 93.14%. Compared with conventional dyeing, when using recycled dyeing wastewater for wool fabric dyeing, the dye exhaustion rate exceeded 99.00%, and the fixation rate was higher than that achieved by the conventional dyeing process. The K/S value of wool fabrics dyed using the recycled dyeing wastewater increased by 0.47 to 2.43. The rubbing and washing fastness of the 10th cycle dyed fabrics were reduced by half a grade and 1 grade, respectively, but all of them met the production requirements.

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回用染色废水用于羊毛织物活性染料染色

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摘　要：针对毛用活性染料染色废水排放量大、污染严重的问题，利用过二硫酸钠（SPS）对毛用活性染料兰纳素红 CE 染色残液进行降解处理和循环再利用。以染料残留率为评价指标，确定了毛用活性染料降解工艺。通过测试循环染色过程中的染料上染率、固色率及染色织物颜色参数和色牢度，证实了利用活性染料染色废水实现羊毛织物循环染色的可行性。结果表明：处理毛用活性染料兰纳素红 CE 所适宜的条件是 0.2 g/L SPS，初始 pH 值为 3，100 ℃处理 30 min，此时染料降解率高达 93.14%。在利用降解后的染色废水进行循环染色时，染料上染率均高于 99.00%，固色率较传统染色工艺高。在染色条件相同的情况下，循环染色织物表观颜色更深。循环次数低于 5 次时，对色牢度影响不明显。尽管第 10 次循环染色织物的耐摩擦色牢度和耐洗色牢度分别降低半级和 1 级，但织物仍满足生产要求。

关键词：羊毛织物；活性染料；染色废水；循环利用；染色