RESEARCH

Optimization of dyeing parameters of cotton standardized samples for laundry test of dye transfer inhibition program

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Abstract

In order to prevent light-colored clothes from being stained by dyes released from dark clothes during the washing process, some new-type washing machines have developed the dye transfer inhibition washing program. However, there is no certified reference materials for the test of dye transfer inhibition function. To this end, cotton fabric and reactive dyes are used as experimental materials to prepare standardized samples to evaluate the dye transfer inhibition function of washing machines. Firstly, the single factor analysis method is used to analyze the significance of the dyeing parameters including dye dosage, dyeing temperature, sodium sulfate dosage and sodium carbonate dosage. Secondly, a 4-factor 5-level experimental design and theoretical prediction of the best dyeing parameters are successively carried out through central composite design and response surface method. Two evaluation indicators, the dye release amount of the standardized sample of dyed fabric and the color difference value of the standardized sample of white fabric after washing, are proposed as the response values for response surface analysis to search the optimal dying parameters in theory. The optimal dyeing parameters obtained through response surface analysis are that the dosage of dye is 5.63% (owf), the dyeing temperature is 60 °C, the dosage of sodium sulfate is 93.60 g/L, and the dosage of sodium carbonate is 15 g/L. Experimental results indicate that the standardized samples prepared with optimal dyeing parameters can effectively distinguish the dye transfer inhibition function of washing machines.

Keywords: Cotton fabric, Standardized samples, Dye transfer inhibition program, Response surface method, Dyeing processing, Color difference

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Introduction

Nowadays, most clothes on sale are mainly made of cotton fiber. Cotton-based clothing is not only light and soft in texture, but also has good breathability and moisture absorption, which is suitable for human daily wear (Han 2019). Reactive dyes are often used to dye cotton fabrics due to their own advantages (Ristic et al. 2012). However, after the clothes are worn, they need to be washed to achieve the purpose of maintaining the beauty and extending the service life. Inevitably, during the washing process, dark-colored clothes are prone to fading. This is because not all reactive dyes have chemical reactions with cotton fibers, and some of them rely on van der Waals forces and hydrogen bonds to bond with cotton fibers. These weak dyes on clothes will dissolve in water during washing. And if light-colored clothes are washed at the same time, then these dyes dissolved in the water will have the opportunity to have an adsorption effect with them, which will cause stains on the light-colored clothes after washing, which will seriously affect their appearance and service life (Rathinamoorthy 2019). In order to improve the staining of white clothes during mixed washing, some household washing machines equipped with "color protection" and "dye transfer inhibition" washing programs have emerged. At present, Europe has released a relevant standard for testing the dye transfer inhibition performance of laundry detergents. However, the evaluation of dye transfer inhibition performance in washing machine–related standards is still in a blank stage. In China, there is no scientific regulation on the evaluation of dye transfer inhibition performance in the washing process, and it is impossible to evaluate and distinguish the washing machines equipped with dye transfer inhibition function on the market today. Therefore, given the lack of standardized samples for the evaluation of washing machine dye transfer inhibition washing programs, Wuxi Little Swan Electrical Co., Ltd and Jiangnan University are preparing certified reference materials for the group standard "household and similar electrical washing machine—technical requirements and performance test methods for dye transfer inhibition washing." In our previous work, the difference in the fading performance of the dyed fabric samples prepared with 6 different types of reactive dyes under two dyeing processes are discussed (Guo et al. 2020). And one of the dyes was selected to prepare the standardized sample of dyed fabric. However, in our previous work, the optimization of the dyeing parameters is not discussed.

Therefore, how to find an efficient and reasonable dyeing process has become the focus of this article. Orthogonal experimental design method can find the best combination of factor levels, but it cannot find a clear functional expression between factors and response values over the entire area given, then cannot predict the best dyeing process parameters (Xu et al. 2019). Therefore, it is not suitable for us to adopt. The response surface method is to design a reasonable test plan and analyze the test data, and then give a regression fitting equation and make the optimal process prediction for the expected response value (Kalpana et al. 2014). The central composite design is widely used experimental design method due to the selection of appropriate pivot points and the retention of the rotatability and sequentiality of the experimental data, which greatly improves its prediction accuracy (Baaka et al. 2015).

In this work, cotton fabric and reactive dye were used as materials to prepare the standardized sample of dyed fabric. Firstly, the four dyeing process parameters of dye dosage, dyeing temperature, sodium sulfate dosage and sodium carbonate dosage were

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analyzed by single factor experiment. Furthermore, the central composite design was used to design a 4-factor 5-level test plan by taking of the dye dosage, dyeing temperature, sodium sulfate dosage and sodium carbonate dosage as factors. And then, two evaluation parameters, i.e. the dye release amount (mg/ml) of the standardized sample of dyed fabric in the washing system and the color difference value of the standardized sample of white fabric after washing were proposed as the response value. Then, the response surface method was used to simulate the relationship between the dyeing parameters and the two proposed evaluation, and a prediction model was obtained. The optimal solution of the model is used as the optimal dyeing process parameter. The standardized samples were prepared with the best dyeing process parameters, and the distinguishability of the standardized samples was tested.

To prepare the standardized samples, cotton fabric and reactive dye were used as materials. In Sect. 1, the experiment materials and methods are demonstrated. The experiment method used in this work includes the single factor analysis, central composite design and response surface method. In Sect. 2, the effect of the dyeing parameters, dye dosage, dyeing temperature, sodium sulfate dosage and sodium carbonate dosage, on the dye release amount of the dyed fabric and the color difference value of the white fabric are discussed. The conclusions are presented in Sect. 3.

Methods

Materials

Bleached plain fabric is used as the raw material for the preparation of the standardized sample of dyed fabric and that of white fabric. The fabric used is 100% cotton in fiber content, the warp yarn is (21 ± 2) count, the weft yarn is (21 ± 2) count, the warp and weft yarn density is (235 ± 5) root/10 cm, and the areal density is (140 ± 5) g/m^2.

Reactive red 21 dye is used as the dye for the preparation of the standardized sample of dyed fabrics. Its reactive base type is vinyl sulfone type (Siddiqua et al. 2017). The dye is purchased from Feinuo Dyestuff Chemical (Wuxi) Co., Ltd. Dye information is shown in Table 1.

The chemical reagents used were anhydrous sodium sulfate (analytical grade; Sinopharm Chemical Reagent Co., Ltd.), anhydrous sodium carbonate (analytical grade; Sinopharm Chemical Reagent Co., Ltd.), glacial acetic acid (analytical grade; Sinopharm Chemical Reagent Co., Ltd.) And soaping agent (A-502F; Suzhou Liansheng Chemical Co., Ltd.). The main instruments and equipment used included an LHS-80HC-II constant temperature and humidity box (Shanghai Yiheng Scientific Instrument Co., Ltd), a Datacolor 650 colorimeter (Datacolor), Ahiba infrared dyeing small prototype (Datacolor), a TU-1900 dual-beam ultraviolet–visible spectrophotometer (Beijing General

Table 1 The information about dyes

Dye name	Formula weight	CAS	Structural formula
Reactive Red 21	838.7	11,099–79-9	

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Analysis General Instrument Co., Ltd), and a TM27BK electric sewing machine (Brother Business Co., Ltd).

Experimental method
Fabric pretreatment

To prepare the standardized samples, we first put the purchased experimental fabrics in a drum washing machine for cleaning. When these fabrics are washed, the weight of fabric is 5 kg, 50 g of standard detergent is added, and the cotton fabric washing program is used for washing. After washing, the fabric is rinsed twice, and, after dehydration, it is dried with a dryer for standby.

Standardized sample preparation

First, prepare the standardized sample of white fabric: cut the pretreated fabric into a white fabric sample with a size of 26 cm × 26 cm. While cutting the fabric, it should be ensured that there are no defects on the fabric and the distance from the fabric should be 15 cm or more. Then lock the trimmed sample with a width of 0.5 cm to ensure that the size of the standardized sample of white fabric is 25 cm × 25 cm. The standardized sample of white fabric was ironed and flattened, then vacuum-packed, and placed in an environment of 0–5 °C and protected from light (Guo et al. 2020).

Then, prepare the standardized sample of dyed fabric: in the experiment, the four factors of dye dosage (owf), dyeing temperature (°C), sodium sulfate dosage (g/L) and sodium carbonate dosage (g/L) are considered as the main factors (Miljković et al. 2007). The fabric after pretreatment is dyed with Ahiba infrared dyeing small prototype machine. That is, put a certain weight of the fabric and the prepared dye solution into the dyeing tank at the same time in the 1:10 bath ratio, and the dyeing solution are fully shaken and then put into the dyeing equipment for dyeing. Before placing the fabric in the dye cup, ensure that it is dried at 60 °C for 2 h. We set the dyeing process as follows: the initial dyeing temperature is 30 °C, then the heating rate of 2 °C/min is used to raise the temperature to the required dyeing temperature, and the dyeing is maintained at this temperature for 80 min. Then the temperature was lowered to 30 °C at a cooling rate of 4 °C/min. At this time, the fabric was taken out and washed twice, then neutralized with glacial acetic acid with a concentration of 2 g/L for 1 min, and then washed once. Finally, after soaping with a soaping-agent(A-502F) concentration of 1 g/L at 95 °C for 15 min, the fabric was taken out, washed once and dehydrated, and dried the sample in a flat manner. The experimental water involved in the entire dyeing process is deionized water with a bath ratio of 1:10 (Kai et al. 2013; Yan 2014). The specific dyeing process is shown in Fig. 1. After the fabric dries naturally, cut it into a size of 25 cm × 25 cm for use.

Simulation of washing environment

The water consumption of the main wash program of a household washing machine is generally about 20 L (Beemkumar et al. 2015).According to the proposed plan, 16 standardized sample of dyed fabrics and 8 standardized sample of white fabrics are required for the washing test of transfer inhibition program under this water consumption (the number of samples can be changed according to the water consumption).These standardized samples are placed in the washing machine in the order of ABAABA…ABA,
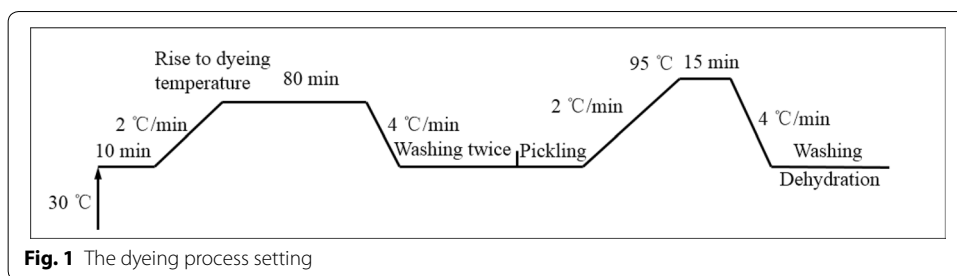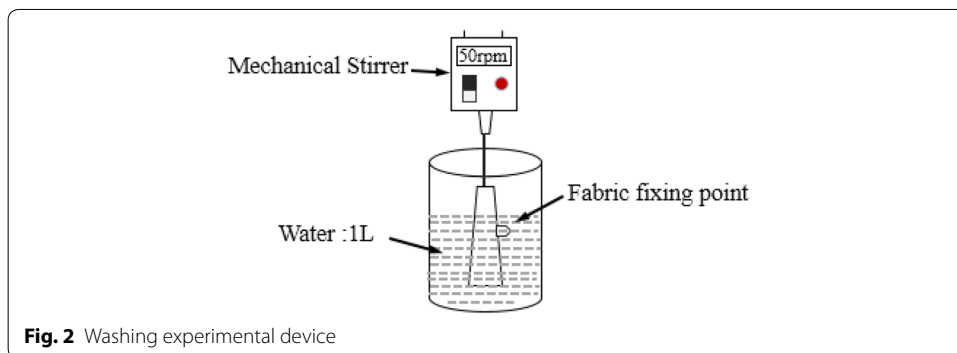
Fig. 1 The dyeing process setting

Fig. 2 Washing experimental device

where A represents the standardized sample of dyed fabric and B represents the standardized sample of white fabric. This kind of experiment requires a lot of manpower and material resources, and it is extremely difficult to complete it under laboratory conditions alone. Therefore, the experimental plan is simulated as follows: Use a combination of 1 L water with a dyed fabric and half a white fabric to perform the test. Among them, the standardized sample of dyed fabric and the standardized sample of white fabric are put in separately, that is, the washed standardized sample of dyed fabric is first taken out, and then the liquid in the 30 mL washing system is collected, and then the standardized sample of white fabric is put in. Each washing time is 30 min and the rotation speed is 50 rpm. The advantage of this scheme is that it is convenient to collect the washing residue after washing the standardized sample of dyed fabric, calculate the dye release amount of a single standardized sample of dyed fabric, and finally, dry the washed standardized sample of white fabric for use when testing the color difference. The simulated washing experiment device is shown in Fig. 2.

Establishment of standard curve

When we build the standard curve of Reactive Red 21, we need to accurately weigh 0.1 g of Reactive Red 21 and add an appropriate amount of deionized water to fully hydrolyze it. Then the volume is set in a 100-mL volumetric flask to obtain a mother liquor concentration of 1 mg/mL. Next, we used a pipette to accurately pipette 1 mL of mother liquor and 9 mL of deionized water in order to mix them evenly, and the concentration of the mother liquor was diluted to 0.1 mg/mL. Finally, we used a pipette to dilute the dye solution with a concentration of 0.1 mg/mL to the dye concentrations of 0.01, 0.015, 0.02, 0.025, 0.03, 0.035, 0.04, 0.045, and 0.05 mg/mL and used a spectrophotometer to measure its absorbance (Guo et al. 2020). We used the dye concentration as the abscissa

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and the absorbance value as the ordinate to fit the standard curve of Reactive Red 21, as shown in Fig. 3.

Determination of dye concentration in the washing system

After washing each sample, this paper took 30 mL of the washing system residue, filtered it with a 0.45-µm filter head, and then measured the absorbance of the residue at 510 nm, and calculated the average of the three results as the final test result. Then we calculated the dye concentration in the washing residue based on the standard curve drawn.

Color difference test

Datacolor650 color measurement and matching instrument is used to collect the color data of the sample and then evaluated the color difference of the sample (Jun et al. 2018; Yang et al. 2019). The specific color difference test method is specified as follows: the sample to be tested is placed at a temperature of 20 °C and a relative humidity of 65% at constant temperature and humidity for 24 h, under the test conditions of D65/10° for the illuminator and the observer. After folding the sample into four layers and flattening it, a transparent plate with an aperture of 30 mm was selected to measure the sample. Each cloth sample was measured at four points (two points were symmetrical to each other), and then the average value was taken as the color data of the sample. Finally, a piece of standardized sample of the white fabric is used as the control sample, the color difference $\Delta E_{CMC(l:c)}$ between the test sample and the control sample will be calculated, where the value of l is 2.0 and the value of c is 1.0.

Precision, reproducibility and stability test

Five standardized samples prepared in the same dyeing process, dried at 60 °C for 2 h, were taken out and quickly weighed, and the calculated standard deviation was 0.282%.
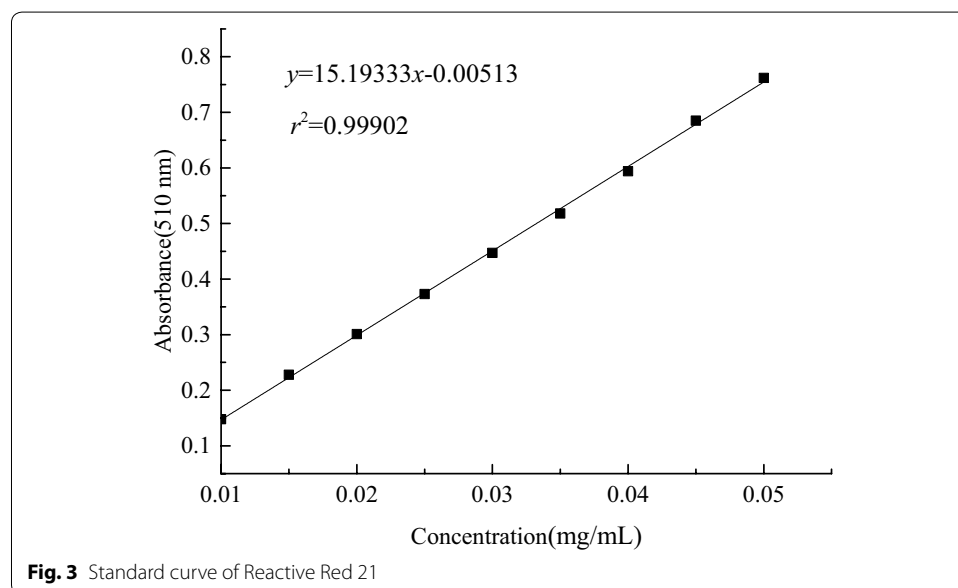

Fig. 3 Standard curve of Reactive Red 21

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It showed that the dry weight of the standardized samples prepared by strictly following the preparation process is consistent, which ensured that our simplified experimental method is highly reliable and representative.

A certain concentration of reactive red 21 control sample dye solution was repeatedly measured for absorbance at a wavelength of 510 nm for 5 times, and the calculated standard deviation was 0.154%. According to the method specified in 1.2.5 Determination of dye concentration in the washing residue, collected a test sample solution and place it indoors for 0, 12, 24, 36, 48 h. Then the absorbance was measured at a wavelength of 510 nm and the standard deviation was calculated to be 0.386%. This showed that the test sample solution is relatively stable within 48 h, and the precision and reproducibility are satisfactory (Liang et al. 2019).

Determination of fabric dyeing evenness

Eight points on the surface of the fabric sample (four symmetrical points on the front and back) were selected, and then the reflectance was tested on Datacolor650 (the test condition refers to the color difference test). The reflectance of each point is measured in the visible spectrum ($\lambda = 390$–700 nm), and the spectral interval is 10 nm. And then the measurement of fabric dyeing evenness is calculated by relative unevenness index (RUI). When the RUI value is less than 0.2, it means that the level dyeing property of the fabric is very good; the RUI value greater than 0.2 and less than 0.5 indicates that the level dyeing property of the fabric is good, and the RUI value greater than 0.5 indicates that the level dyeing property is poor. The calculation method is shown in formula (1):

$$RUI = \sum_{\lambda=390}^{700} \left(\frac{S_\lambda}{\overline{R}} \right) V_\lambda \tag{1}$$

In the formula: S_λ is the standard deviation of the reflectance of eight measuring points under a certain wavelength; $\overline{R}$ is the average reflectance of the eight measuring points under the corresponding wavelength; V_λ is the relative luminous rate function.

Single factor test

During the preparation of standardized samples for dye transfer inhibition washing program testing, the single factor test program we designed is as follows: different dye dosage (2–6% (owf)), different dyeing temperature (40–80 °C), different sodium sulfate dosage (50–100 g/L) and different sodium carbonate dosage (5–30 g/L), and analyzed the influence of different factors on the two evaluation indicators proposed.

Response surface test   On the basis of the single factor test, we took four factors of dye dosage, dyeing temperature, sodium sulfate dosage and sodium carbonate dosage as independent variables. And taking the two evaluation indicators proposed as the response value, then the Design-Expert 11.0.4 software was used to design a central composite design test with 4 factors and 5 levels, and the data was analyzed.

Distinguishability test of standardized samples   The color difference value can be used to distinguish the dye transfer inhibition ability of different washing machines. At the same time, it can also characterize the distinguishability of standardized samples. After

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the standardized samples are washed, the smaller the color difference of the standardized white fabric is, the stronger the dye transfer inhibition ability of the washing machine is. However, when calculating the color difference, we found that the value of the color difference is relatively discrete, which is different from the simple and easy-to-understand classification method. To this end, we converted the color difference between the test sample and the control sample of the standardized white fabric into the level of inhibiting dye transfer for visual evaluation. In this experiment, the dye transfer inhibition level is divided into three levels, namely, level 1, level 2, and level 3. Among them, when the dye transfer inhibition level is 1, the dye transfer inhibition effect is the best. When the test result of the tested washing machine is not within the specified level range, the dye transfer inhibition performance is not qualified (Guo et al. 2020). The relationship between color difference and the dye transfer inhibition grade of the washing machine is shown in Table 2.
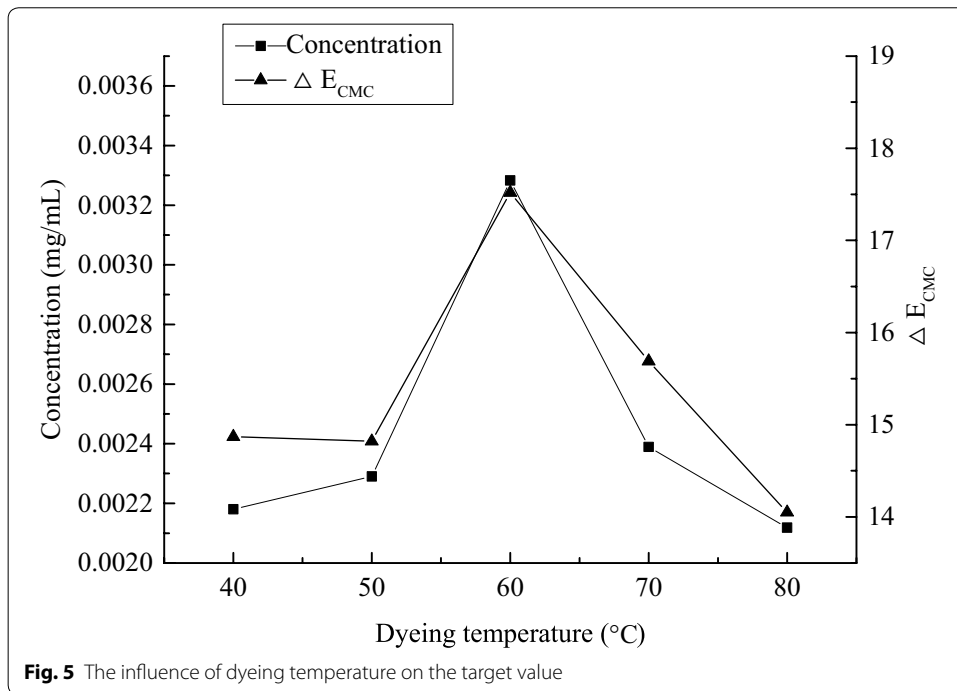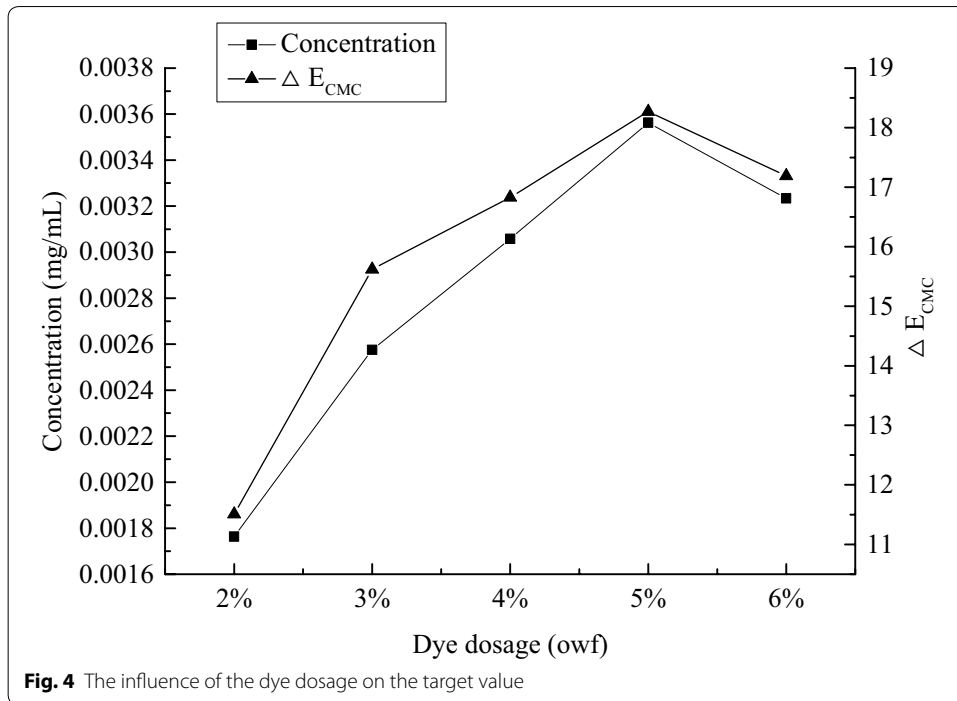
Results and discussion
Single factor test results
The influence of the dye dosage on the target value
According to the dyeing process specified in the article, under the condition that the dyeing temperature is 60 °C, the sodium sulfate dosage is 70 g/L and the sodium carbonate dosage is 15 g/L. We investigated the influence of different dye dosages on the target value (the target value is the two evaluation indicators proposed). It can be seen from Fig. 4 that as the dye dosage increases, the two evaluation indicators gradually increase. We found that when the dye dosage is 5% (owf), both reach the maximum value, and continue to increase the dye dosage, both of which decrease. This is because when reactive dyes dye cotton fabrics, they rely on the reaction between the dyes and fibers to form covalent bonds (Muhammed et al. 2020; Shu et al. 2019). At the same time, some dyes will also rely on hydrogen bonds or van der Waals forces to adsorb on the surface of cellulose fibers, or gather on the surface of cellulose fibers. In the pores between cotton fibers, as the dye dosage increases, the dye that can act on the fabric gradually reaches the maximum. When these dyed fabrics are washed with water, those binding methods that are not strong in force may easily cause the dye to fall off. Similarly, the more dyes that fall into the water, the greater the chance of adsorption with standardized sample of white fabric. We considered the experimental results and economic factors, so we chose the dye dosage of 5% (owf) as the central value.

Table 2 The dye transfer inhibition grade of the washing machine

Color difference *X*	Dye transfer inhibition grade
0–0.50	1
0.51–1.00	2
1.01–1.50	3

Fig. 4 The influence of the dye dosage on the target value

Fig. 5 The influence of dyeing temperature on the target value
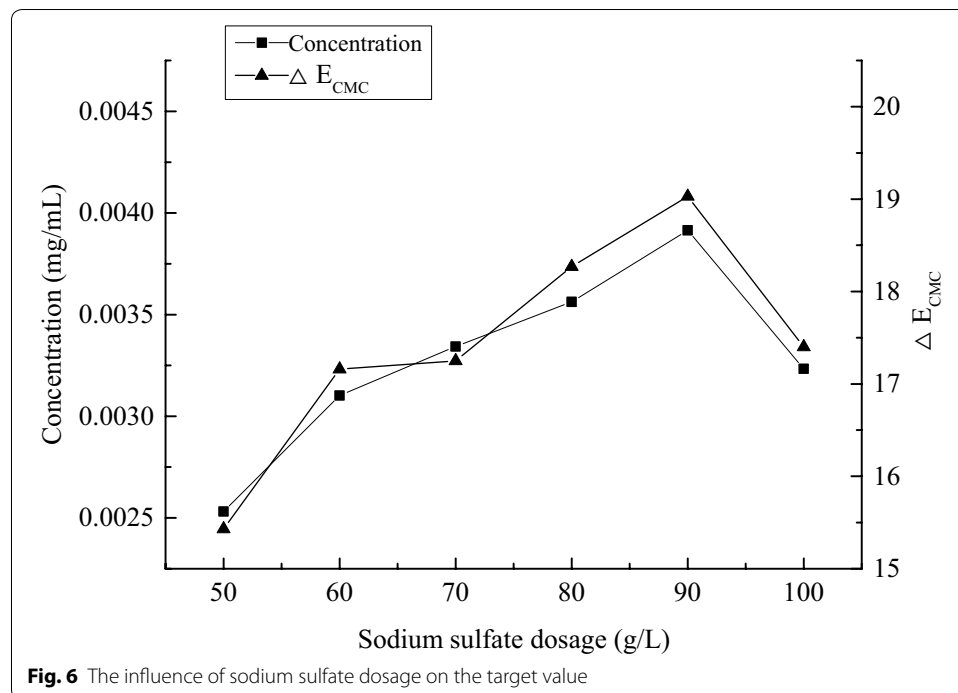
The influence of dyeing temperature on the target value

Under the condition that the dye dosage is 5% (owf), the sodium sulfate dosage is 70 g/L and the sodium carbonate dosage is 15 g/L, we investigated the influence of different dyeing temperatures on the target value. It can be seen from Fig. 5 that as

the dyeing temperature increases, the two evaluation indicators both reach the maximum when the dyeing temperature is 60 °C. Generally speaking, as the temperature increases, the reaction speed increases. However, too high temperature will cause the hydrolysis reaction speed of the dye to be faster than the reaction speed of the dye and the fiber, thereby reducing the utilization rate of the dye (Paul et al. 2017). When the temperature is right, more dyes can be combined with the fabric by means of chemical reaction, adsorption, and so on, so the probability of dye falling during washing is greater. Therefore, the dyeing temperature of 60 °C is selected as the central value.

The influence of sodium sulfate dosage on the target value

Under the conditions of dye dosage of 5% (owf), dyeing temperature of 60 °C and sodium carbonate dosage of 15 g/L, we investigated the influence of different sodium sulfate dosages on the target value. It can be seen from Fig. 6 that with the gradual increase in the amount of sodium sulfate, the two evaluation indicators gradually increase. When the sodium sulfate dosage is 90 g/L, both reach the maximum. Continue to increase the amount of sodium sulfate, and both have declined. The sodium sulfate in the dye liquor will play a role in promoting the dyeing. However, if the concentration of sodium sulfate in the dye liquor is too high, it will cause the dye to accumulate in the dye liquor, which is not conducive to dyeing and fixing (Sun et al. 2017). When the concentration of sodium sulfate in the dyeing solution is appropriate, the dye reaches the maximum degree of dyeing, so that the amount of dye falling during washing is the largest, and the color difference value is the largest after washing. Therefore, the central value of 90 g/L sodium sulfate is selected.

Fig. 6 The influence of sodium sulfate dosage on the target value

The influence of sodium carbonate dosage on target value

Under the conditions of dye dosage of 5% (owf), dyeing temperature of 60 °C and sodium sulfate dosage of 90 g/L, we investigated the influence of different sodium carbonate dosages on the target value. It can be seen from Fig. 7 that with the gradual increase in the amount of sodium carbonate, the two evaluation indicators both reach the maximum when the sodium carbonate dosage is 15 g/L. This is because as the concentration of sodium carbonate in the dye liquor increases, the ratio between cellulose anions and hydroxyl anions continues to decrease, which improves the reactivity of dyes and fibers to a certain extent. However, continue to increase the amount of sodium carbonate, and the number of cellulose anions will continue to increase, which will have a certain repelling effect on the reactive dye anions that further diffuse to the fiber surface and reduce the dye uptake percentage. At the same time, it will aggravate the hydrolysis reaction of the dye, causing uneven dyeing. Therefore, 15 g/L of sodium carbonate was selected as the central value.

Response surface method to optimize dyeing process parameters

Response surface test design and results

Based on the single-factor test results, we selected the level range determined by the four factors of dye dosage, dyeing temperature, sodium sulfate dosage and sodium carbonate dosage, and the two evaluation indicators were the response values, and marked as R_1 and R_2. Design-Expert software was used to design the central combination experiment with 4 factors and 5 levels to determine the dyeing process parameters when the response values R_1 and R_2 were maximized. The factors and levels of the central composite test design are shown in Table 3, and the test results are shown in Table 4.
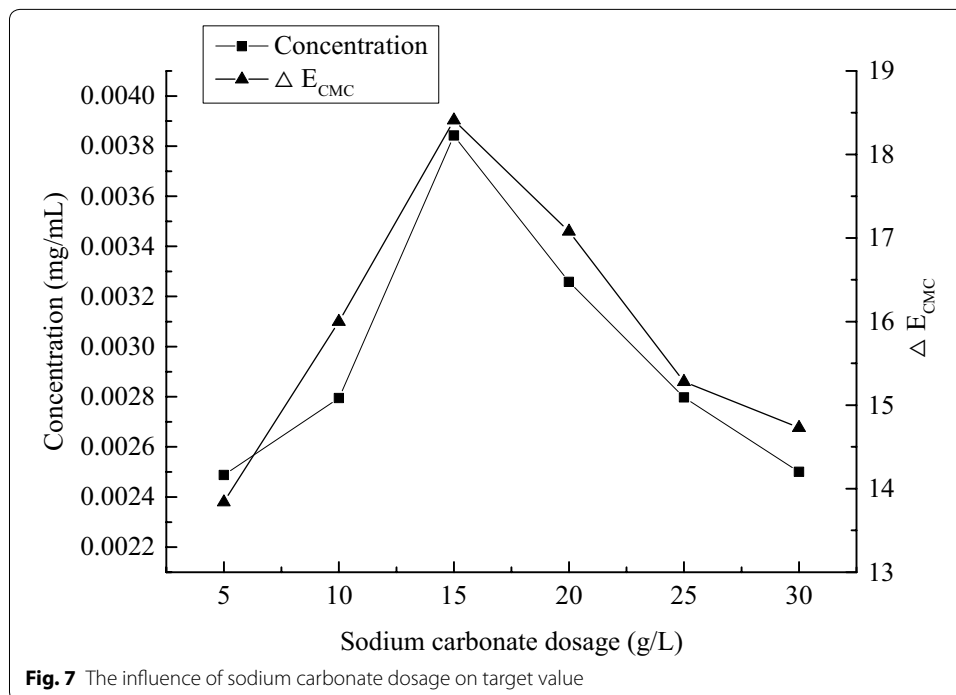

Fig. 7 The influence of sodium carbonate dosage on target value

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Table 3 The factors and levels of the central composite experiment design

Factor	Unit	Code	Coding level				
			− 2	− 1	0	1	2
Dye dosage	% (owf)	A	3	4	5	6	7
Dyeing temperature	°C	B	40	50	60	70	80
Sodium sulfate dosage	g/L	C	70	80	90	100	110
Sodium carbonate dosage	g/L	D	5	10	15	20	25

Table 4 The design and results of the central composite experiment

Run	A: Dye dosage (%(owf))	B: Dyeing temperature (°C)	C: Sodium sulfate dosage (g/L)	D: Sodium carbonate dosage (g/L)	R_1 (mg/mL)	R_2
1	5	40	90	15	0.00177	13.34
2	5	60	90	15	0.00379	17.4
3	5	60	90	15	0.00358	17.13
4	6	70	100	10	0.00295	16.67
5	4	50	100	10	0.00216	14.28
6	5	60	90	25	0.00285	16.48
7	4	70	80	20	0.00205	14.07
8	6	50	100	20	0.0029	16.53
9	4	70	100	20	0.00216	14.45
10	6	50	80	20	0.00264	15.48
11	5	80	90	15	0.00176	13.18
12	7	60	90	15	0.00314	16.79
13	4	70	80	10	0.00211	14.11
14	3	60	90	15	0.00193	13.85
15	4	50	80	10	0.00222	14.61
16	5	60	90	15	0.00368	17.29
17	6	50	100	10	0.00279	16.47
18	6	70	80	10	0.00279	16.24
19	5	60	90	5	0.00265	15.52
20	4	70	100	10	0.00196	14.03
21	5	60	110	15	0.00306	16.77
22	5	60	90	15	0.00375	17.52
23	6	70	80	20	0.0026	15.34
24	4	50	100	20	0.00238	14.66
25	5	60	90	15	0.00369	17.71
26	5	60	70	15	0.00271	16.06
27	4	50	80	20	0.0022	14.61
28	6	70	100	20	0.00266	15.77
29	6	50	80	10	0.00242	15.24
30	5	60	90	15	0.00389	17.16

Establishment and analysis of regression model

The purpose of establishing an empirical model is to better describe the factors that affect the dyeing process within the scope of the experiment (Ganguly et al. 2018). The forecast statistics of different models are shown in Table 5. Table 5 shows that for the response values R_1 and R_2, the P values of both are less than 0.0001 when using the

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Table 5 The forecast statistical results of different models

Source model	Sequential p-value	R^2	Adjusted R^2	Predicted R^2	
R_1					
Linear	0.2695	0.1808	0.0497	− 0.0163	
2FI	0.9989	0.1960	− 0.2272	− 0.4142	
Quadratic	< 0.0001	0.9868	0.9744	0.9443	Suggested
Cubic	0.4384	0.9942	0.9762	0.8501	Aliased
R_2					
Linear	0.0580	0.2965	0.1839	0.0719	
2FI	0.9909	0.3242	− 0.0314	− 0.2001	
Quadratic	< 0.0001	0.9744	0.9506	0.8729	Suggested
Cubic	0.0702	0.9945	0.9774	0.8778	Aliased

quadratic regression equation. The R^2 values predicted by the model are 0.9443 and 0.8729 respectively, and the adjusted R^2 values are 0.9744 and 0.9506, respectively. And the difference between the predicted R^2 value and the adjusted R^2 value is less than 0.2. This shows that the model is most suitable for using the quadratic regression model, and the higher R^2 value also confirms that the quadratic regression model is very reliable in processing experimental data.

The dye release amount R_1 was used as the response value, and regression analysis was performed on the data in Table 4. The obtained quadratic regression equation with the R_1 as the objective function is shown in formula (2). A indicates the dye dosage, B indicates the dyeing temperature, C indicates the sodium sulfate dosage and D indicates the sodium carbonate dosage.

$$
\begin{aligned}
R_1 = {} & -0.042899 + 0.002664A + 0.000616B + 0.000393C + 0.000372D \\
& + (5.81250E - 06)AB + (4.81250E - 06)AC - (6.12500E - 06)AD \\
& - (3.56250E - 07)BC - (1.08750E - 06)BD + (3.62500E - 07)CD \\
& - 0.000307A^2 - (4.99063E - 06)B^2 - (2.19063E - 06)C^2 - 0.000010D^2
\end{aligned} \tag{2}
$$

The R_2 is used as the response value, and the quadratic regression equation obtained by analysis is shown in formula (3).

$$
\begin{aligned}
R_2 = {} & -56.48021 + 4.04583A + 1.27429B + 0.396333C + 0.662583D \\
& + 0.011250AB + 0.019500AC - 0.028250AD - 0.000525BC \\
& - 0.002625BD + 0.000825CD - 0.526875A^2 - 0.010419B^2 \\
& - 0.002531C^2 - 0.014275D^2
\end{aligned} \tag{3}
$$

Analysis of variance is used to evaluate the significance of the effects of all variables and their interactions on the response value (Aminoddin et al. 2016). The results of analysis of variance and significance test of the response surface regression model are shown in Table 6. The significance of the impact of each variable on the response value in the regression model is determined by the F test. The smaller the P value, the higher the significance of the corresponding variable. It can be seen from Table 6 that the regression effect of the regression equation in response to the R_1 and R_2 is highly significant. Values of Prob > F less than 0.05 imply that the model terms are significant at the 95%

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Table 6 The analysis of variance and significance test results

Source	R_1			R_2		
	F-value	*P*-value Prob > *F*	Significance	*F*-value	*P*-value Prob > *F*	Significance
Model	79.84	< 0.0001	**	40.85	< 0.0001	**
A	191.99	< 0.0001	**	162.63	< 0.0001	**
B	0.8095	0.3825		1.06	0.3188	
C	10.62	0.0053	*	9.65	0.0072	*
D	1.39	0.2565		0.6407	0.4360	
AB	5.19	0.0378	*	2.24	0.1555	
AC	3.56	0.0789		6.72	0.0204	*
AD	1.44	0.2488		3.53	0.0800	
BC	1.95	0.1831		0.4870	0.4959	
BD	4.54	0.0501		3.04	0.1015	
CD	0.5043	0.4885		0.3007	0.5915	
A^2	247.32	< 0.0001	**	84.08	< 0.0001	**
B^2	655.43	< 0.0001	**	328.80	< 0.0001	**
C^2	126.29	< 0.0001	**	19.41	0.0005	*
D^2	168.20	< 0.0001	**	38.58	< 0.0001	**
Lack of Fit	0.8909	0.5919		2.25	0.1921	

P > 0.05 is not significant; *P* ≤ 0.05 is significant, represented by *; *P* ≤ 0.0001 is highly significant, represented by **

confidence level. The factors that have significant effects on the response value R_1 are *A*, *C*, *AB*, A^2, B^2, C^2, D^2. The factors that have significant effects on the response value R_2 are *A*, *C*, *AC*, A^2, B^2, C^2, D^2. The model lack-of-fit term represents the probability that the predicted value of the model does not fit the actual value. The *P* values of the model lack-of-fit items in Table 6 are 0.5919 and 0.1921 respectively, which are both greater than 0.05, indicating that the model lack-fit items are not significant. The regression equation established by the model can better explain the response results and predict the optimal dyeing process parameters.

The actual forecast graph shows how well the model fits the data (Rajkumar et al. 2017). The diagonal line indicates that the predicted value and the actual value have the same position. If all the points are on or around this diagonal, it proves that the predicted value is more representative. The relationship between the predicted value and the actual value of the response is shown in Fig. 8. The Fig. 8 shows that there is sufficient consistency between the actual data and the data obtained from the model.

Response surface analysis and optimal process prediction

In order to better understand the influence of the interaction between different factors on the target value, the Design-Expert software is used to draw a three-dimensional response surface curve (Hamza et al. 2020). Figure 9a–c shows a three-dimensional surface diagram that has a strong interaction on the amount of dye release, and Fig. 9d, e shows a three-dimensional surface diagram that has an interactive effect on the color difference value.

Figure 9a shows that under the condition that the dosage of sodium sulfate is 90 g/L and the dosage of sodium carbonate is 15 g/L, when the dyeing temperature is around 60 °C, the dye dosage within a certain range can make the response value larger. This

Fig. 8 Experimental (actual) values versus predicted values for **a** dye release and **b** color difference value
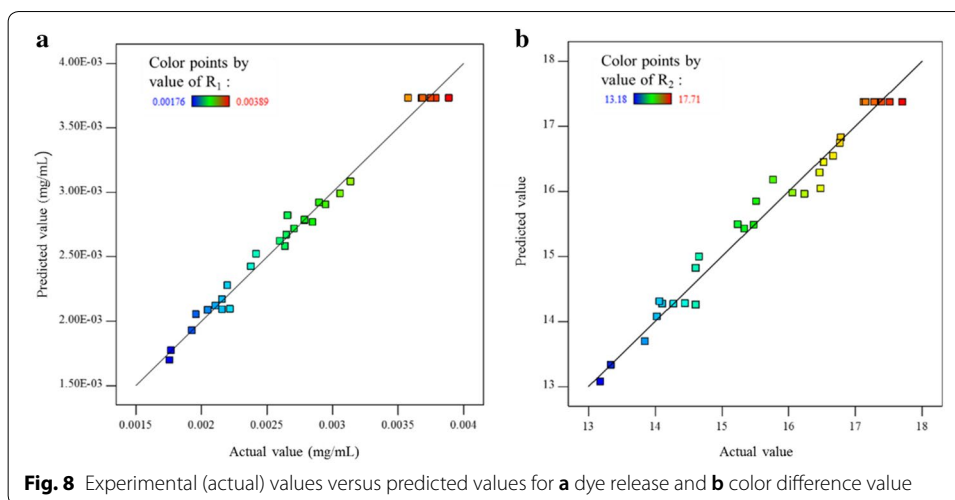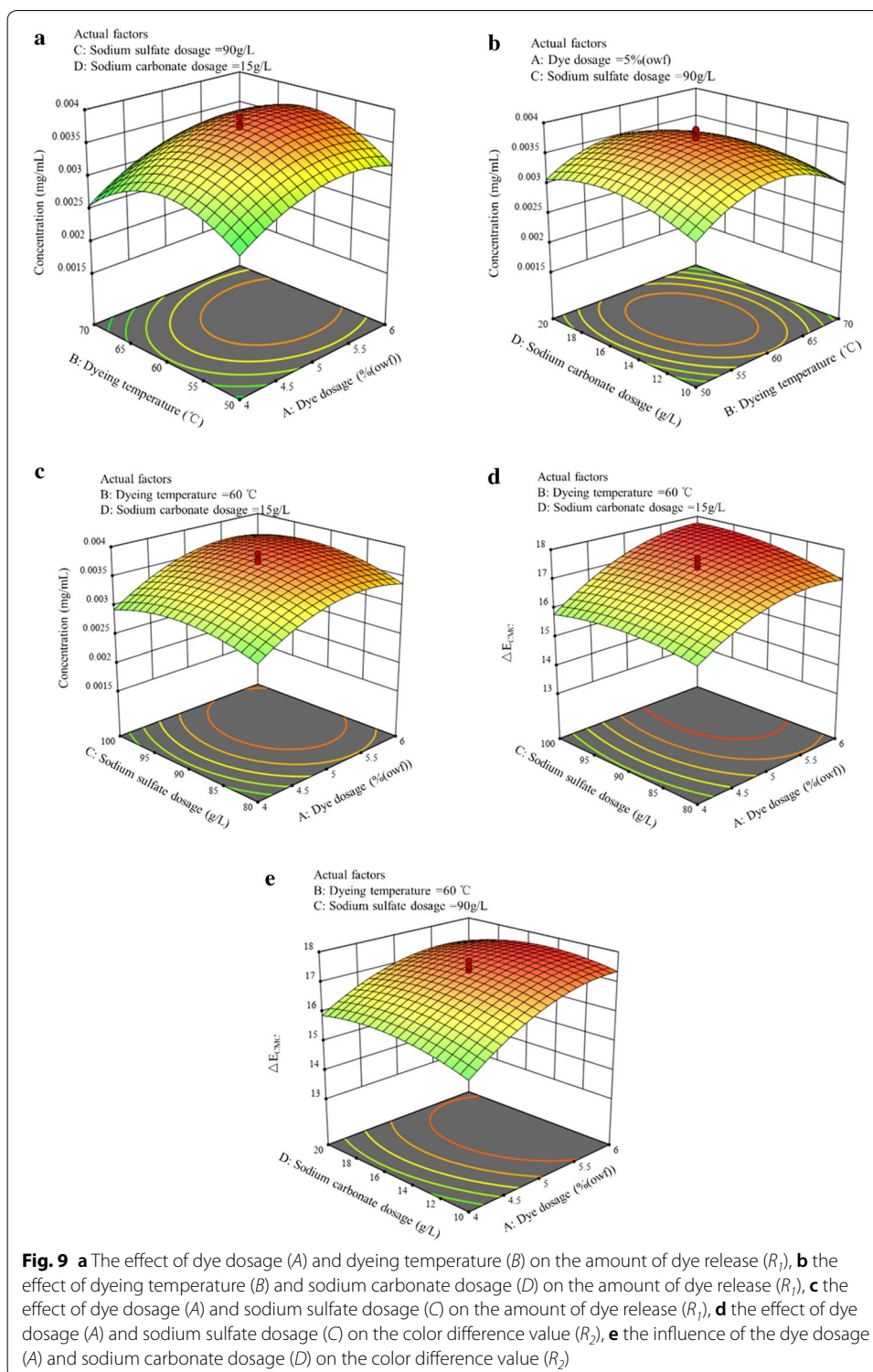
proves that the interaction between the dyeing temperature and the amount of dye is obvious, and the dyeing temperature has a more obvious effect on the value of the dye release. Figure 9b shows that the effect of dyeing temperature and sodium carbonate dosage on the amount of dye released is similar to that of Fig. 9a. This phenomenon is also closely related to the type of reactive groups of the dye, indicating that vinyl sulfone reactive dyes are greatly affected by temperature during dyeing, and are suitable for dyeing at around 60 °C. As can be seen in Fig. 9c, when the dyeing temperature is 60 °C and the sodium carbonate dosage is 15 g/L, and when the dye dosage is large, the sodium sulfate dosage within a certain range can make the response value larger. It shows that the interaction between the dosage of dye and the dosage of sodium carbonate is obvious, and the dye dosage has a more obvious effect on the value of dye release. Figure 9d shows that under the conditions of a temperature of 60 °C and a sodium carbonate dosage of 15 g/L, and when the dosage of sodium sulfate is constant, the color difference value increases with the increase of the dosage of dye. It shows that the dosage of dye has a more obvious influence on the value of color difference. It can also be seen in Fig. 9e that the effect of the dye dosage and sodium carbonate dosage on the color difference is similar to that of Fig. 9d. Therefore, appropriately increasing the dosage of dye is beneficial to increase the color difference value.

We took the maximum value of the response values R_1 and R_2 as the optimization direction, and set a value that cannot be reached in this experiment to limit their value range to ensure the accuracy of the optimization results. Through optimization, the optimal dyeing process parameters are as follows: the dye dosage is 5.634% (owf), the dyeing temperature is 60.008 °C, the sodium sulfate dosage is 93.593 g/L, and the sodium carbonate dosage is 15.002 g/L. At this time, the amount of dye released was 0.004 mg/mL, and the color difference was 17.733.

Response surface prediction model accuracy test and standardized sample dyeing evenness determination

In order to verify the predicted results, we considered the actual test conditions, and then made corrections under the optimized process conditions. The revised dyeing

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Fig. 9 **a** The effect of dye dosage (*A*) and dyeing temperature (*B*) on the amount of dye release (*R₁*), **b** the effect of dyeing temperature (*B*) and sodium carbonate dosage (*D*) on the amount of dye release (*R₁*), **c** the effect of dye dosage (*A*) and sodium sulfate dosage (*C*) on the amount of dye release (*R₁*), **d** the effect of dye dosage (*A*) and sodium sulfate dosage (*C*) on the color difference value (*R₂*), **e** the influence of the dye dosage (*A*) and sodium carbonate dosage (*D*) on the color difference value (*R₂*)

process parameters are: dye dosage is 5.63% (owf), dyeing temperature is 60 °C, sodium sulfate dosage is 93.60 g/L and sodium carbonate dosage is 15 g/L. Then the test was repeated 3 times under the dyeing process conditions, and the average value was taken as the final experimental result. The dye release amount of the standardized sample of

dyed fabric was 0.00396 mg/mL, and the standardized sample of white fabric's color difference after staining was 17.725. The experimental results are in good agreement with the theoretical predictions, indicating that the model is reasonable and effective. Examples of images of dyed fabric, white fabric, and white fabric after staining are shown in Fig. 10.

The modified dyeing process parameters is used to make the standardized sample of dyed fabric, and according to the fabric dyeing evenness test method specified in 1.2.8 for dyeing evenness inspection. We randomly selected 10 pieces of standardized sample of dyed fabric, used Datacolor650 to measure the reflectivity and calculate by formula (1), then the relative unevenness index (RUI) of each sample was within 0.2. This shows that the standardized sample of dyed fabric developed under the optimized dyeing parameter is very good and have no unevenness, which further proves the rationality of the dyeing process parameters.

Distinguishability test of standardized samples

We used the developed standardized samples as test materials, and selected three washing machines of different brands that all claim to have the function of protecting the color of clothes for dye transfer inhibition function test to verify the distinguishability and feasibility of this set of standardized samples. The test programs of the three prototypes are respectively denoted as Program 1, Program 2 and Program 3. The main wash water intake of the three washing machines is 20 L. First of all, we only add the standardized sample of white fabric to the washing machine and run it once to obtain the control sample of the of white fabric. Then, each washing machine was put into the same batch of 16 dyed fabric (A) and 8 white fabric (B) and each program was run three times. Thus, we obtained test samples of the standardized sample of white fabric after washing through different dye transfer inhibition programs.

We used the Datacolor650 to collect the color data of the control sample and test sample of the washed standardized sample of white fabric, and then the test samples were evaluated according to the color difference test method, and the test result was marked as X. The dye transfer inhibition performance test was run three times, and the color difference results were denoted as X_1, X_2 and X_3, and the arithmetic average of the three test results was taken as the test result of the dye transfer inhibition performance test.
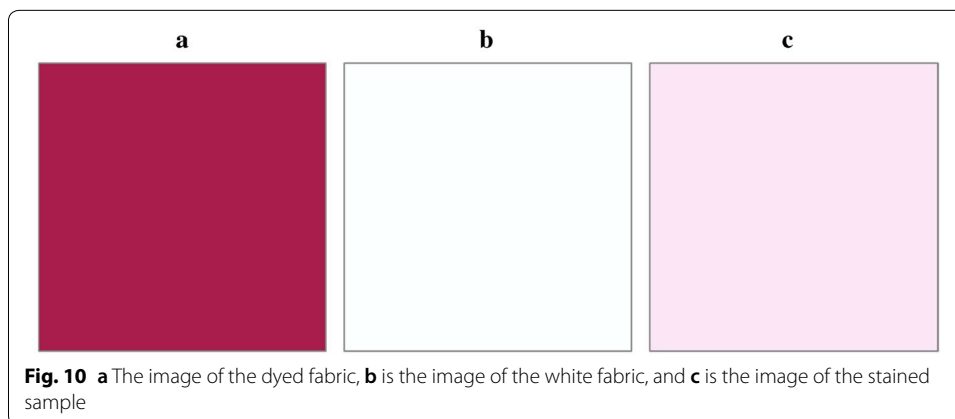

Fig. 10 **a** The image of the dyed fabric, **b** is the image of the white fabric, and **c** is the image of the stained sample

Table 7 The test results of the standardized sample of white fabric

Program	Color difference	Dye transfer inhibition grade
Program 1	0.93	Level 2
Program 2	1.48	Level 3
Program 3	3.47	Unqualified

The color difference of the test sample of the standardized sample of white fabric and the corresponding dye transfer inhibition grade are shown in Table 7.

By analyzing the test data in Table 7, we find that the color difference test results of the three test programs are 0.93, 1.48, and 3.47. Two programs are within the range of dye transfer inhibition level, and the test result of the other one is unqualified. This shows that this set of standardized samples can well distinguish the dye transfer inhibition function of different washing machines, and has good operability and feasibility.

Conclusion

In our experiment, a set of standardized samples prepared using cotton fabric and reactive red 21 dye can effectively distinguish different dye transfer inhibition washing programs. Single factor analysis showed that the four parameters of dye dosage, dyeing temperature, sodium sulfate dosage and sodium carbonate dosage have significant effects on both indicators, the dye release amount of the standardized sample of dyed fabric and the color difference value of white fabric. Through response surface analysis, it is found that the dosage of dye has the most significant impact on the response value, followed by the dosage of sodium sulfate. A quadratic regression model was constructed through response surface analysis, and the maximum value of the response value was taken as the optimization direction of dyeing parameters. The corrected optimal dyeing process parameters were as follows, the dye dosage is 5.63% (owf), the dyeing temperature is 60 °C, the sodium sulfate dosage is 93.60 g/L, and the sodium carbonate dosage is 15 g/L. The standardized samples prepared by these dyeing parameters can successfully realize the evaluation of the dye transfer inhibition function of three different washing programs.

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