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The effect of washing parameters on the quantity of dye discharge from clothes

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article

Abstract

In this paper, the effect of washing parameters on the quantity of dye discharge from fabric dyed in highly saturated color (*FSC*) was discussed. Firstly, the *FSC* was prepared with reactive red 195 to represent the dark clothes. Secondly, the effect of the washing parameters, including washing time, washing temperature, washing water volume, the weight of *FSC*, and the dosage of detergent, on dye discharge was analyzed through the single factor test. Thirdly, the mathematical relationship between the quantity of dye discharge (*DDQ*) and the four washing parameters was discussed through the center combination experiment and response surface method. And a quadratic model was built to predict the quantity of dye discharge when the *FSC* were washed with different washing parameters. The experimental results indicated that the quantity of dye discharge was affected by the four selected washing parameters and the optimal washing parameters to reduce the quantity of dye discharge can be deduced from the quadratic model. The optimized parameters effectively reduced 0.99 mg dye discharge per liter, and the *DDQ* reduction rate was up to 53.5%.

Keywords: Washing parameters, Dye discharge, Single-factor, Response surface, Reactive red 195

Introduction

Cotton fabrics are made of natural cellulose fibers, which have the advantages of moisture absorption, breathability, comfort, and mild handle, and are favored by consumers. With the improvement of people's living standards, the color requirements of cotton fabrics are also increasing. Reactive dyes have the advantages of various colors and low cost and become the first choice for cotton fabric dyeing (Lewis, 2014). However, due to their structural characteristics, reactive dyes are easily hydrolyzed and shed by temperature, sunlight, washing, and other effects (Lu et al., 2014). Due to the defects of deep chroma, poor biodegradability, and large diffusibility, the shedding reactive dyes cause large fluctuation of water quality and high chemical oxygen demand (COD), which seriously affects the ecological environment and human health (Bezerra et al., 2021).

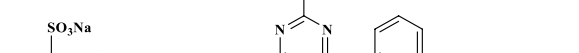
In daily washing, people often mix a large weight of Fabric dyed in highly saturated color (*FSC*) and light-colored clothes for the convenience of washing (Guo et al., 2020). Due to the defects of the dye itself and the improper post-treatment process of dyeing and finishing, the floating color on the surface of the fabric accumulates, which

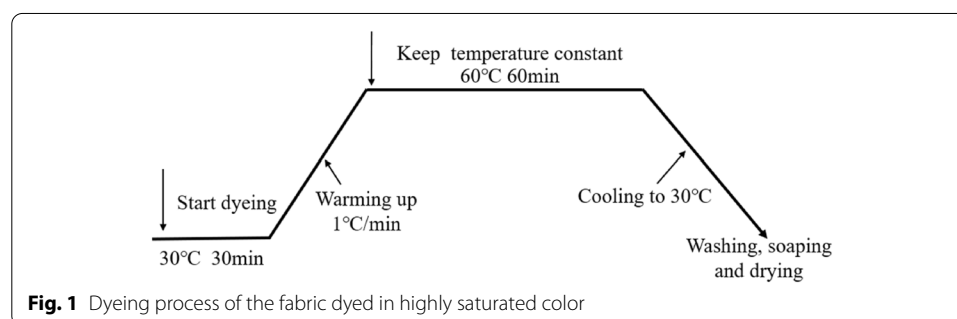
leads to low colorfastness to washing. Under the mixed action of washing parameters, the dye shedding is aggravated (Kongliang et al., 2006; Wei et al., 2016). On the one hand, the dye content in the washing wastewater is high, resulting in wastewater pollution, on the other hand, it causes cross-color of light-colored fabrics. Previous studies have shown that the dye discharge of fabric dyed in highly saturated color (*FSC*) is related to pH, the type of dyes, fiber type, temperature, water hardness, mechanical strength, and so on (Gorenšek 1999; Hu & Fan, 2013). Fan et al. studied and analyzed the reasons for poor color fastness to wet rubbing of reactive dyes, which mainly described that alkaline dyeing of reactive dyes led to the formation of hydrolyzed dyes attached to fibers, which reduced the color fastness to wet rubbing (Fan et al., 2006). Bilisik et al. studied and analyzed that the dye uptake of *FSC* is high, the dye dispersion density is high, and there are still many floating color residues after a series of post-dyeing treatments. In addition, due to the hydrolysis of the dye itself, the covalent bond between the dye and the fiber is lost, so the dye will fall off into the washing solution when washing the *FSC* (Bilisik & Yolacan, 2011).

Therefore, how to find a set of washing parameters that can effectively reduce the quantity of dye discharges (*DDQ*) in the washing wastewater and the prediction model of the quantity of dye discharges (*DDQ*) become the focus of this study. The traditional orthogonal experimental design method can find the best combination of factor levels affecting the test results, but its disadvantage is that it can not give a comprehensive expression of the relationship between factors and response values in a certain region, that is, the regression equation, so it can not optimize the best combination of factors (Tang & Cai, 2006). Therefore, it is particularly important to find a test method that can effectively reduce the number of experiments, shorten the test period, obtain the regression equation of factor-response value, and obtain the influence of interaction between various factors on the response value (Kalpana & King, 2014). In this study, the central composite experimental design method was selected to design 30 experimental points with alternative combinations of various factors. The pivot point selectivity of central composite experimental design, the spatial and rotatable nature of experimental data, and the combination of response surface analysis could greatly improve the accuracy of the prediction model (Baaka et al., 2015).

Therefore, to deeply simulate and study the influence of the combination of washing parameters on the dye discharge of *FSC* in household washing, five groups of single-factor experiments were carried out on washing time (*A*), washing temperature (*B*), washing water volume (*C*), the weight of *FSC* and the dosage of detergent (*F*). The parameters that significantly affected the dye discharge were determined, as well as the distribution of significant intervals. Then, the effects of the interactions among the four washing parameters on the dye stripping quantity of *FSC* were analyzed by central composite design, and a Quadratic model was established to fit the experimental data to obtain a quadratic regression equation, to predict the effects of washing parameters on the dye stripping weight of *FSC*. Through the fitting optimization and verification test, the parameter collocation of effectively reducing dye shedding and the most dye shedding was obtained, which provided a theoretical reference for the prevention and control of dye shedding pollution in household clothes washing.

Table 1 Property of the C.I.Reactive Red 195

Dye	CAS	Molecular formula	Molecular weight	λ_{nm} (max)
Reactive Red 195	93050-79-4	$\text{C}_{31}\text{H}_{19}\text{ClN}_7\text{Na}_5\text{O}_{19}\text{S}_6$	1136.32	542
				



Methods

Materials

The bleached cotton fabric was used to prepare the *FSC*, the linear density of the warp and weft yarn of which are both (27.8 ± 2.5) tex, with both (235 ± 5) picks/10 cm in the warp and weft directions.

Reactive Red 195 was purchased from Zhejiang Run Tu Co, Ltd. the dye information is shown in Table 1. Anhydrous sodium sulfate and anhydrous sodium carbonate were purchased from Sinopharm Group Chemical Reagent Co, Ltd., and A-502F soaping agent was purchased from Suzhou Lian sheng Chemical Company Limited.

Preparation of fabric dyed in highly saturated color

Dyeing process

The FSC was prepared through a one-bath one-step method with reactive red 195, and the dying process is shown in Fig. 1. The dosage of reactive red 195 is 0.2% (*o.w.f*), the concentration of sodium sulfate is 50 g/L, and the concentration of sodium carbonate solution is 18 g/L. the dyeing bath ratio is 10:1.

Reflectance test of the FSC

To ensure the *FSC* were homogeneously prepared, the reflectance of each sample was measured using the color measuring and matching instrument (Datacolor, USA) before washing. First, the spectrogram of Reactive Red 195 was measured with an ultraviolet–visible spectrophotometer (Datacolor, USA). As shown in Fig. 2, the maximum absorption wavelength of Reactive Red 195 is at 542 nm. Then the medium aperture and 100% UV filter are selected in the color measuring and matching instrument, the reflectivity of four points on the front and back of the fabric is tested under the CIE standard light

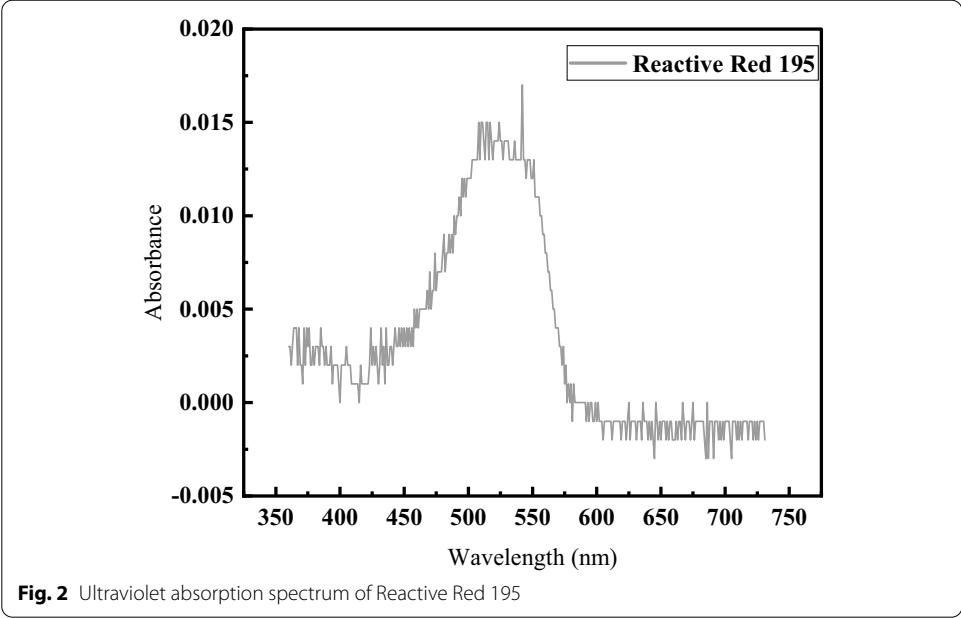


Table 2 Reflectance test results of fabric dyed in highly saturated color

Group	Reflectance values of different batches of dyed samples (%)			
	1st (542 nm)	2nd (542 nm)	3rd (542 nm)	4th (542 nm)
1	21.00	20.93	20.83	20.78
2	20.77	21.01	20.87	20.76
3	20.89	20.97	20.89	21.00
4	20.87	20.86	20.87	20.89
5	20.79	20.77	21.03	21.01
6	21.00	20.91	20.91	21.04
7	21.02	21.00	20.77	20.98
8	20.79	20.88	21.03	20.94
Median value	20.83	20.82	20.99	20.95

Between-group deviation [− 0.25%, +0.25%]

source, and the average value is calculated to obtain the reflectivity data of the *FSC*. In our experiment, the median value of the reflectance of the same and different batches of samples shall be both within [− 0.25%, +0.25%] at the maximum absorption wavelength. The test results are shown in Table 2.

Standard curve of Reactive red 195

Reactive red 195, 0.1 g, was added to deionized water and fixed volume to 100 mL used as dye mother solution with a concentration of 1 mg/L after stirring and dissolving. The dye mother solution was diluted to 0.0001, 0.0005, 0.001, 0.0015, 0.002 mg/mL respectively, and then the absorbance at the maximum absorption wavelength was measured to plot the standard curve of reactive red 195. Using the dye concentration as the abscissa and the absorbance at the maximum absorption wavelength as the ordinate (Tao et al.,

2015), as shown in Fig. 3. The regression equation of the standard curve was fitted according to the dye concentration-absorbance value, and the fitting coefficient R^2 was more than 0.999, which indicated that the fitting degree was good.

The determination of dye discharge under actual washing condition

TD100-163W MUIATD front-load washing machine with drum (Wuxi Little Swan Electric Co., Ltd) was used in the washing test. In the experiment, *FSC* and light fabrics were washed together, with washing time, washing temperature, washing water volume, and the weight of *FSC* as test variables. The weight of the light white cloth was fixed at 50 g, and the fabric was cut into 25 cm × 25 cm. Collect the washing residual liquid after washing. The absorbance of the washing solution was measured with an ultraviolet spectrophotometer after filtering using a 0.45 μm aqueous filter head. The measurement results were ternarily averaged. Then, the concentration of the dye was calculated according to the standard curve. And the total dye discharge after washing can also be calculated if the water volume of the washing machine is given.

Results and Discussion

To remove the pollutants that may remain in the drum of the washing machine, the washing machine needs to complete four complete washing pretreatment procedures before the formal test. According to the requirements of ISO 105-C10:2006 Textiles—Tests for colorfastness—Part C10: Colour fastness to washing with soap or soap and soda (Krishnanand et al., 2016), the pretreatment water temperature shall be kept at $(30 \pm 2) ^\circ\text{C}$, and the test water pressure connected to the water inlet pipe of the washing machine shall be kept at (0.24 ± 0.05) MPa, to ensure that the water inlet of the washing machine is stable within the specified time. The hardness of the test water is guaranteed to be 2.4–2.6 mmol/L. In the first operation, there is no test load, and 50 g of standard

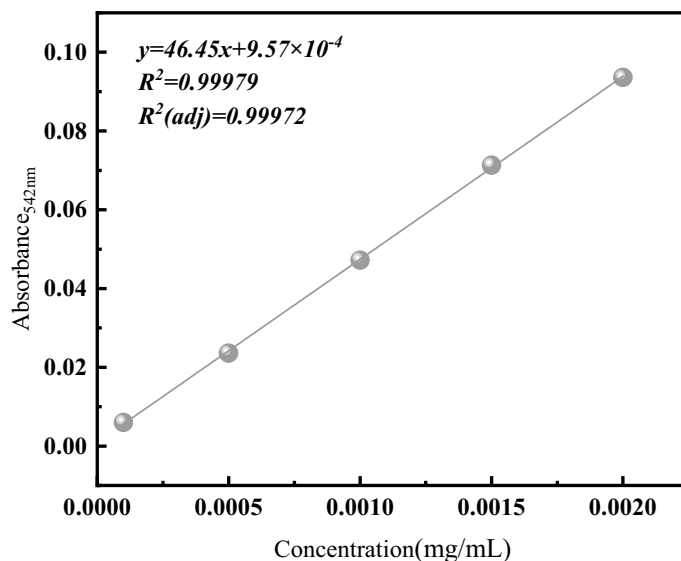


Fig. 3 Standard curve of Reactive Red 195

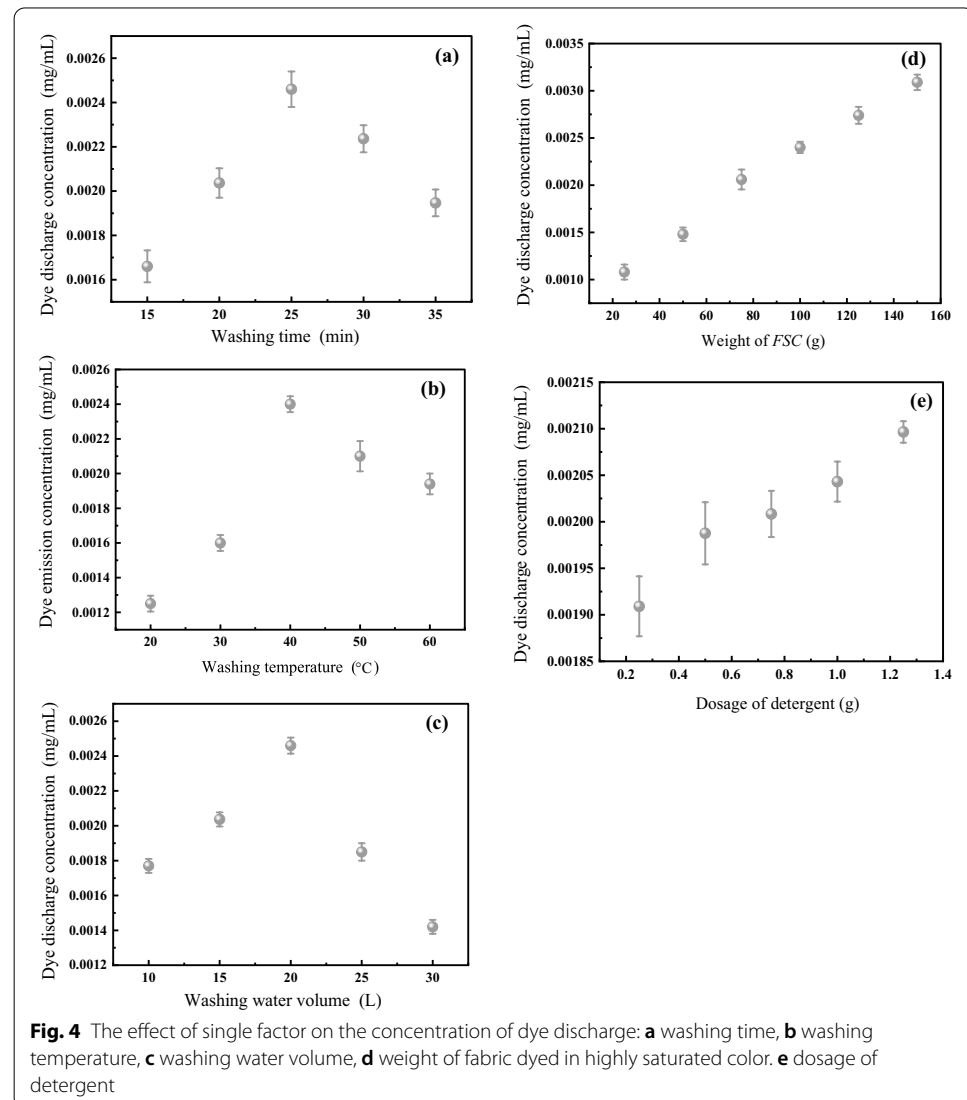
detergent conforming to ISO 105-C10:2006 is used. 2nd, 3rd, and 4th run without test load and detergent. After the fourth washing, ensure that the washing machine is placed in the test environment for at least 2 h before the test.

Single factor experiment

In the single factor test, the effects of four washing parameters on the dye concentration in the washing wastewater were investigated by taking the washing time, washing temperature, washing water volume, and the weight of FSC as factors and data from three parallel experiments are shown in Fig. 4.

The effect of washing time


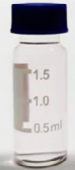

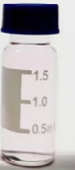
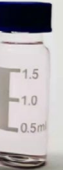
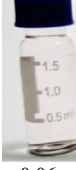
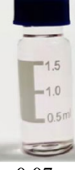
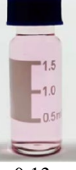
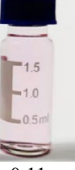


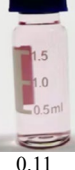
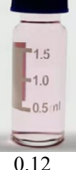
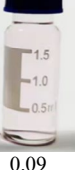
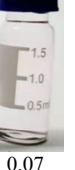

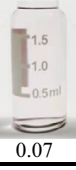

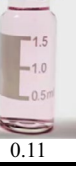

The effect of washing time on the dye discharge concentration in the washing wastewater is illustrated in Fig. 4a. The washing time was set as 15 min, 20 min, 25 min, 30 min, and 35 min, respectively, the other washing conditions were fixed as 75 g of



FSC and 50 g of light color fabric, the washing temperature was set as 40 °C, and the washing water volume was 20 L, the dosage of detergent was 0.75 g. It can be seen from Fig. 4a that the maximum discharge concentration is 0.00246 mg/mL when the washing time is 25 min, and the dye discharge concentration is 0.00166 mg/mL when the washing time is 15 min. The difference of *DDQ* in the drum reaches 16 mg/20 L, and the dye discharge increases by 48.2%. When the washing time was 35 min, the dye discharge was reduced by 21.1%. When the washing time was 25 min, the *DDQ* reached the peak. As the washing time continues to increase, the *DDQ* in the washing system decreases, because the dye discharge and saturates when the washing time is 25 min. If the washing time continues to increase, the quantity of dye adsorbed by the light-colored fabric will increase. According to the quantity of dye adsorbed by 50 g mixed washed light-colored fabric, the quantity of dye adsorbed after 30 min was increased by 4.468 mg compared with that after 25 min, so the quantity of residual dye in the washing system after 25 min was reduced.

To further screen the washing parameter range, each of the five groups of tests was repeated three times, the total *DDQ* for three times was collected, and the dyes of the same specification were weighed and configured in the sample bottle. As shown in Table 3, under the D65 light source, the dye absorbance was larger when the washing

Table 3 The absorbance of washing residue in three parallel experiments

Factor	Total absorbance for three parallel experiments				
Washing time/min	15 min	20 min	25 min	30 min	35 min
					
Absorbance	0.07	0.08	0.12	0.10	0.09
Washing temperature/°C	20 °C	30 °C	40 °C	50 °C	60 °C
					
Absorbance	0.06	0.07	0.13	0.11	0.09
Washing water volume/ L	10 L	15 L	20 L	25 L	30 L
					
Absorbance	0.08	0.11	0.12	0.09	0.07
Weight of <i>FSC</i> /g	25 g	50 g	75 g	100 g	125 g
					
Absorbance	0.05	0.07	0.10	0.11	0.13

time was 25 min, and the absorbance was lighter when the washing time was 15 min. Therefore, 15–35 min was selected for the central combination test.

The effect of washing temperature

The effect of washing temperature on the dye discharge concentration in the washing wastewater is illustrated in Fig. 4b. The washing temperature was set as 20 °C, 30 °C, 40 °C, 50 °C, and 60 °C, respectively. The other washing conditions were fixed as follows: 75 g of FSC and 50 g of light fabric were put in, the washing time was set at 25 min, the volume of washing water was 20 L, the dosage of detergent was 0.75 g. The results showed that with the increase of washing temperature, the discharge concentration of the dye increased first and then decreased, the maximum concentration of dye discharge was 0.00240 mg/mL at 40 °C, and the concentration of dye discharge was 0.02 mg/mL at 20 °C, with a difference of 23 mg/20 L and an increase of 92.0%. When the temperature continues to increase, the dye in the washing wastewater is reduced by 18.8% at 60 °C, which is related to the structural effect of temperature on reactive dyes. Within a certain temperature, with the increase of temperature, the chemical reaction and adsorption reaction between the dye falling off and the light-colored fabric in the washing drum are accelerated. When the washing temperature continues to rise, the DDQ reaches saturation, the dye adsorption rate of light-colored fabrics is accelerated, and the dye diffusion rate is also accelerated, resulting in the reduction of residual dye in the washing system. However, the dyes that do not react with the cotton fabric are desorbed during the washing process, and there is a charge repulsion force between the desorbed dyes and the fiber surface, so the possibility of re-adsorption is very small, and the adsorption of light-colored fabrics increases (Hang & He, 2013). According to the test, on 50 g light-colored fabric, the quantity of dye adsorption at 50 °C increased by 5.9 mg compared with that at 40 °C, which verified the reason why the DDQ in the washing system decreased with the increase of washing temperature.

The total DDQ for three times was collected, weigh the dyes of the same specification, and preparing them in the sampling bottle. It can be seen from Table 3 that the absorbance difference is obvious. Therefore, in the central combination test, the influence interval of washing temperature on the DDQ is set as 20 °C to 60 °C.

The effect of washing water volume

The effect of washing water volume on the dye discharge concentration in the washing wastewater is illustrated in Fig. 4c. The washing water volume was set as 10 L, 15 L, 20 L, 25 L, and 30 L, respectively. The other washing conditions were fixed as follows: 75 g of FSC and 50 g of light fabric were put in, the washing time was set at 25 min, the washing temperature was 40 °C, the dosage of detergent was 0.75 g. When the volume of washing water is 20 L, the dye concentration in the washing wastewater reaches the maximum value of 0.00246 mg/mL, and when the volume of washing water is 10 L, the dye concentration in the washing wastewater is 0.00177 mg/mL. In the range of 10–20 L, with the increase of washing water volume by 10 L, the DDQ in the cleaning wastewater increases by 31.5 mg, increasing by 38.9%. When the volume of water is 30 L, the concentration of dye discharge is 0.00142 mg/mL, which is 6.6 mg less than the peak value, and the discharge is reduced by 13.4%. When the volume of washing water is 20 L, the DDQ is the

largest, and when the volume of washing water continues to increase, the *DDQ* reaches saturation, and the dye concentration in the washing system decreases. The dye adsorption capacity of light-colored fabric gradually slowed down with the increase of washing water. The dye adsorption capacity increased by 2.95 mg when the washing water was 25 L compared with 20 L, and increased by 3.65 mg when the washing water was 30 L compared with 25 L.

Therefore, when the washing water volume is in the range of 10–30 L, both the dye discharge concentration has a trend of increase and decrease, and the effect is more significant. As shown in Table 3, the total *DDQ* for 3 times was collected and the difference in dye concentration was obvious. Therefore, in the central combination test, the range of the influence of the volume of washing water on the *DDQ* was selected to be 10 L to 30 L.

The effect of the weight of fabric dyed in highly saturated color

Figure 4d shows the effect of the weight of *FSC* applied on the dye discharge concentration. Fixed 50 g of light color fabric, the washing time was set as 25 min, the washing temperature was 40 °C, the volume of washing water was 20 L, the dosage of detergent was 0.75 g. The input weight of *FSC* was set as 25 g, 50 g, 75 g, 100 g, and 125 g in turn. Since the input weight of *FSC* was not a fixed parameter of the washing machine, an additional set of tests was added to explore the overall impact, and the input weight was increased to 150 g.

As shown in Fig. 4d, with the increase of the weight of *FSC*, the dye concentration in the washing residue increases, in turn, showing a positive correlation. When the weight of *FSC* is 25 g, the dye discharge concentration is the lowest, which is only 0.00108 mg/mL. When the weight of *FSC* increased to 75 g, the dye discharge concentration reached 0.00206 mg/mL, and the dye discharge increased by 19.6 mg/20 L, with an increase of 90.7%. The dye discharge concentrations were 0.00240 mg/mL, 0.00274 mg/mL and 0.00309 mg/mL, respectively, when the weight of *FSC* was 100 g, 125 g, and 150 g. Therefore, the dye discharge concentration continued to increase with the increase of the weight of *FSC*, but the increase rate slowed down. Therefore, when the input weight of *FSC* is within [25, 125], the dye discharge concentration of the washing residual liquid increases from rapid to slow, and the test phenomenon is more obvious. The *DDQ* in three parallel tests within [25, 125] is collected, and the quantity of dye discharged in three cumulative tests is prepared as the washing waste liquid. It can be seen from Table 3 that the concentration difference is significant. Therefore, in the central combination test, the influence interval of the weight of *FSC* discharged on the *DDQ* is set as [25, 125].

The effect of the dosage of detergent

Figure 4e shows the effect of the dosage of detergent applied on the dye discharge concentration. According to IEC 60456:2010, the dosage of detergent shall not exceed one percent of the washing load (Stamminger et al., 2016). In the detergent single factor experiment, 125 g of fabric was placed in the drum, so the dosage of detergent was set as 0.25 g, 0.5 g, 0.75 g, 1 g, and 1.25 g respectively. The other washing conditions were fixed

as follows: 75 g of *FSC* and 50 g of light fabric were put in, the washing time was set at 25 min, the volume of washing water was 20 L and the washing temperature was 40 °C.

As shown in Fig. 4e, the dye concentration in the washing residue increases slowly with the increase of the dosage of detergent. When the dosage of detergent was increased from 0.25 g to 1.25 g, the dye discharge concentration increased only by 9.9%, and the *DDQ* increased by 3.8 mg/20 L. The *DDQ* from 0.5 g to 1.25 g increased by 2.18 mg/20 L, an increase of 5.5%. To some extent, the detergent has a slight effect on the *DDQ*. The ordinary detergents are mainly composed of non-ionic surfactants and anionic surfactants, while reactive dyes belong to anionic dyes, and anionic surfactants can increase the discharge of reactive dyes under the action of charge repulsion while removing stains. Therefore, with the increase of the dosage of detergent, the *DDQ* has an upward trend, but under the action of non-ionic surfactant in detergent, the dye discharge is inhibited, in general, the upward trend is small and the discharge of dyes is not significant. Nonionic surfactants can form a layer of effective adhesion membrane on the surface of *FSC* in the washing process, which can reduce the discharge of certain dyes (Musnickas et al., 2005). In the central composite experimental of washing parameters, the ones that have a significant impact on the *DDQ* are washing time, washing temperature, washing water volume, and the weight of *FSC*, which will be set as the four washing parameters of the central composite experimental.

Central composite design and response surface optimization

Central composite experimental design and result analysis

Based on the results of the single-factor experiment, a central composite design with four factors, five levels, and thirty points was designed according to the principle of central composite design, in which the independent variables were washing time (A, min), washing temperature (B, °C), washing water volume (C, L) and the weight of *FSC* (D, g). (*R*, mg) is the response value, washing time (15–35), washing temperature (20–60), washing water volume (10–30), and the weight of *FSC* (25–125), and the factors and levels are arranged in Table 4. The test scheme and test results are shown in Table 5.

Prediction model of *DDQ*

In the experiment, the linear, two-factor interaction, quadratic and cubic models are chosen for the prediction model of *DDQ*. The prediction results of different models in Table 6, and the Quadratic model has a high degree of fitting when the sequential *p*-value and *R*² are

Table 4 Factors and coding level of center combination experiment

Factor	Unit	Coding level				
		– 2	– 1	0	1	2
A: Washing time	min	15	20	25	30	35
B: Washing temperature	°C	20	30	40	50	60
C: Washing water volume	L	10	15	20	25	30
D: Weight of <i>FSC</i>	g	25	50	75	100	125

Table 5 Central combination test design scheme and test results

Run	A/min	B/°C	C/L	D/g	R/mg
1	1	− 1	− 1	1	34.67
2	1	1	− 1	1	37.65
3	1	− 1	1	− 1	23.92
4	0	0	0	0	46.20
5	1	1	1	− 1	22.94
6	0	0	2	0	40.60
7	0	0	0	0	49.53
8	2	0	0	0	38.80
9	− 1	− 1	− 1	− 1	26.60
10	− 1	− 1	1	1	41.64
11	− 1	− 1	1	− 1	24.76
12	− 1	1	− 1	1	32.56
13	0	0	− 2	0	21.70
14	0	0	0	2	52.20
15	0	− 2	0	0	28.00
16	1	− 1	− 1	− 1	22.43
17	− 1	1	1	− 1	19.96
18	0	2	0	0	38.80
19	− 1	1	1	1	38.34
20	− 1	− 1	− 1	1	30.22
21	1	− 1	1	1	35.01
22	0	0	0	0	49.69
23	0	0	0	0	51.80
24	0	0	0	0	53.60
25	1	1	− 1	− 1	25.62
26	0	0	0	0	49.20
27	1	1	1	1	40.19
28	− 2	0	0	0	33.20
29	0	0	0	− 2	21.60
30	− 1	1	− 1	− 1	22.68

Table 6 Forecast results of different models

Model source	Sequential p-value	R^2	
Linear	0.0162	0.3742	
Two-factor interaction	0.9878	0.4017	
Quadratic	<0.0001	0.9114	Suggested
Cubic	0.5451	0.9570	Aliased

both used as evaluation criteria. So the quadratic regression equation is used to predict and fit the data. Correspondingly, the quadratic model is shown in Eq. (1).

$$\begin{aligned}
 R = & -257.4683 + 8.01392A + 3.33058B + 9.29283C \\
 & + 0.79015D + 0.025063AB - 0.027375AC + 1.925 \times 10^{-3}AD \\
 & - 0.010613BC + 3.42750 \times 10^{-3}BD + 0.012915CD \\
 & - 0.16945A^2 - 0.48864B^2 - 0.021795C^2 - 6.41817 \times 10^{-3}D^2
 \end{aligned} \tag{1}$$

In Eq. (1) A is the washing time, B is the washing temperature, C is the washing water volume, D is the weight of *FSC*, AB is the interaction between the washing time and the washing temperature, AC is the interaction between the washing time and the Washing water volume, AD is the interactions between the wash time and the weight of *FSC*, BC is the interactions between the wash temperature and the washing water volume, BD is the interaction between the wash temperature and the weight of *FSC*, and CD is the interaction between the washing water volume and the weight of *FSC*.

Robustness test of the prediction model of DDQ

Before determining the final prediction model, it is necessary to verify the robustness of the prediction model, to ensure the prediction accuracy.

Figure 5a is the normal residual diagram. The residual is the standard deviation between the experimental value and the predicted value, in which the probability can characterize whether the obtained residual can obey the conventional normal distribution. The distribution of experimental points on a straight line shows that there is no obvious normality problem, and the model has good adequacy (Korbahti & Rauf, 2008).

At the same time, the actual value and the predicted value of the *DDQ* can be compared through response surface analysis, to reflect the fitting degree of the Quadratic model, as shown in Fig. 5b. Where the diagonal lines of the image represent a high degree of agreement between the predicted values of the model and the actual values. The distribution around the diagonal line is more concentrated, and the fitting degree of the model is higher, which indicates that the predicted value under the model is representative.

According to the results of variance analysis of the *DDQ* in Table 7, $P < 0.0001$, the loss of fit is less than 0.05, the difference was not significant, indicating that the fitting model has extremely significant differences and small error. According to the model fitting data in Table 5, the fitting coefficient R^2 of the model is 91.1%, indicating that the model can be used to predict the impact of washing parameters on the *DDQ*.

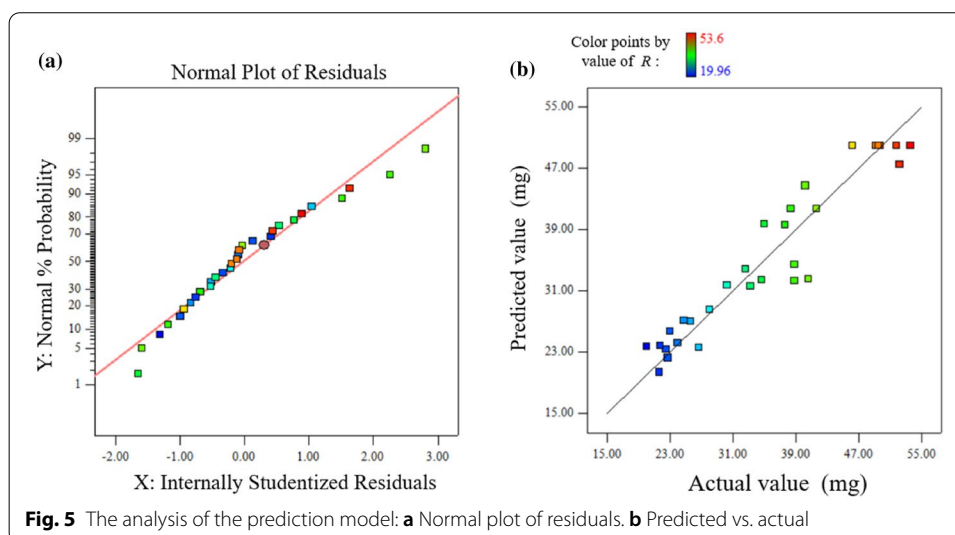


Table 7 Analysis of variance and significance test

Source	<i>R: DDQ</i>					
	Sum of squares	df	Mean square	F value	P-value Prob > F	Significance
Model	3036.79	14	216.91	11.02	< 0.0001	**
A	11.86	1	11.86	0.60	0.4498	
B	20.70	1	20.70	1.05	0.3214	
C	113.23	1	113.23	5.75	0.0299	*
D	1101.21	1	1101.21	55.93	< 0.0001	**
AB	25.13	1	25.12	1.28	0.2764	
AC	7.49	1	7.49	0.38	0.5465	
AD	0.93	1	0.93	0.047	0.8312	
BC	4.51	1	4.51	0.23	0.6393	
BD	11.75	1	11.75	0.60	0.0420	
CD	41.70	1	41.70	2.12	0.0062	
A ²	492.25	1	492.25	25.00	0.0002	*
B ²	654.90	1	654.90	33.26	< 0.0001	**
C ²	814.35	1	814.36	41.36	< 0.0001	**
D ²	441.35	1	441.35	22.42	0.0003	*
Residual	295.33	15	19.69			
Lack of fit	263.73	10	26.37	4.17	0.0641	
Pure error	31.60	5	6.32			
Cor total	3332.12	29				

$P < 0.05$ is significant, marked with *

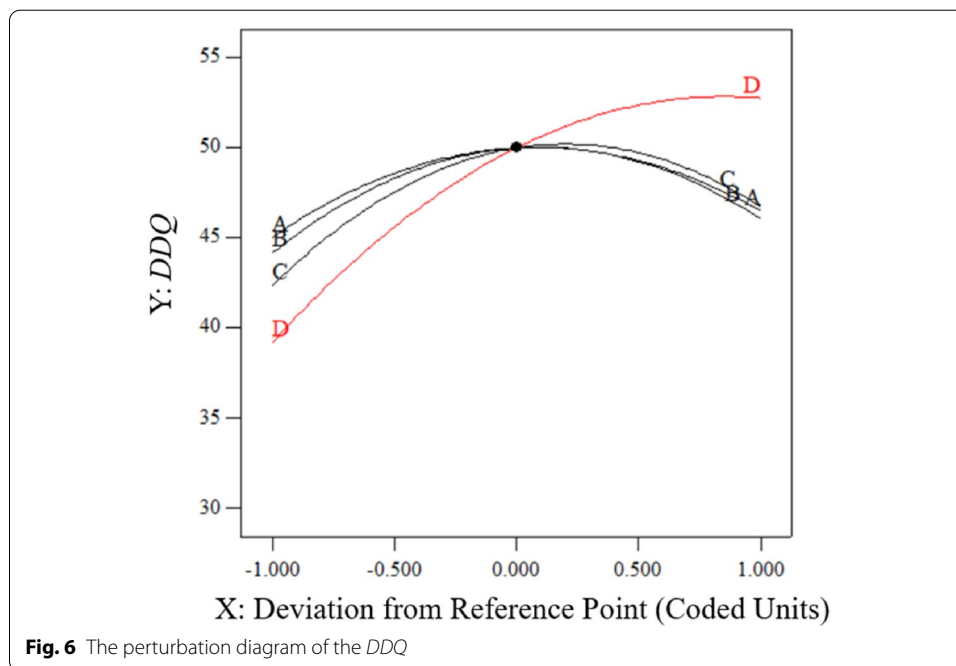
$P > 0.05$ is not significant, not marked; $P < 0.001$ is highly significant, marked with **

The P -value of factor C was less than 0.05, and the P -value of factor D was less than 0.001, indicating that factor C had a significant impact on the DDQ , factor D had an extremely significant impact, and factor A and B had no significant impact ($P > 0.05$). Among them, A^2 , B^2 , C^2 , and D^2 were significant ($P < 0.05$), BC and BD were also significant ($P < 0.05$), and other interactions were not significant. Therefore, the influence of A, B, C, and D on the DDQ in the washing wastewater is not only a linear relationship but also a quadratic term and an interaction term. The order of the four factors affecting the DDQ is D, C, A, B, and the order of the interaction is CD, BD, AB, AC, BC, AD.

When the washing time is 25 min, the washing temperature is 40 °C, the Washing water volume is 20 L, and the weight of FSC is 75 g, the perturbation diagram of the DDQ is shown in Fig. 6. The function of the perturbation diagram is to make a significant comparison of all factors at the selected experimental point. If the curve is flat, it means that the influence on the DDQ in the design space is not significant (Rajkumar & Mauthukumar, 2017). The weight of FSC (D) has the most significant influence on the DDQ in the design space, which also verifies the significant test results in Table 6.

Response surface analysis

The response surface of the interaction between each two of the four washing parameters on the dye discharges, as shown in Fig. 7. According to the contour density, the horizontal point distribution of each factor on the response value can be seen. According to

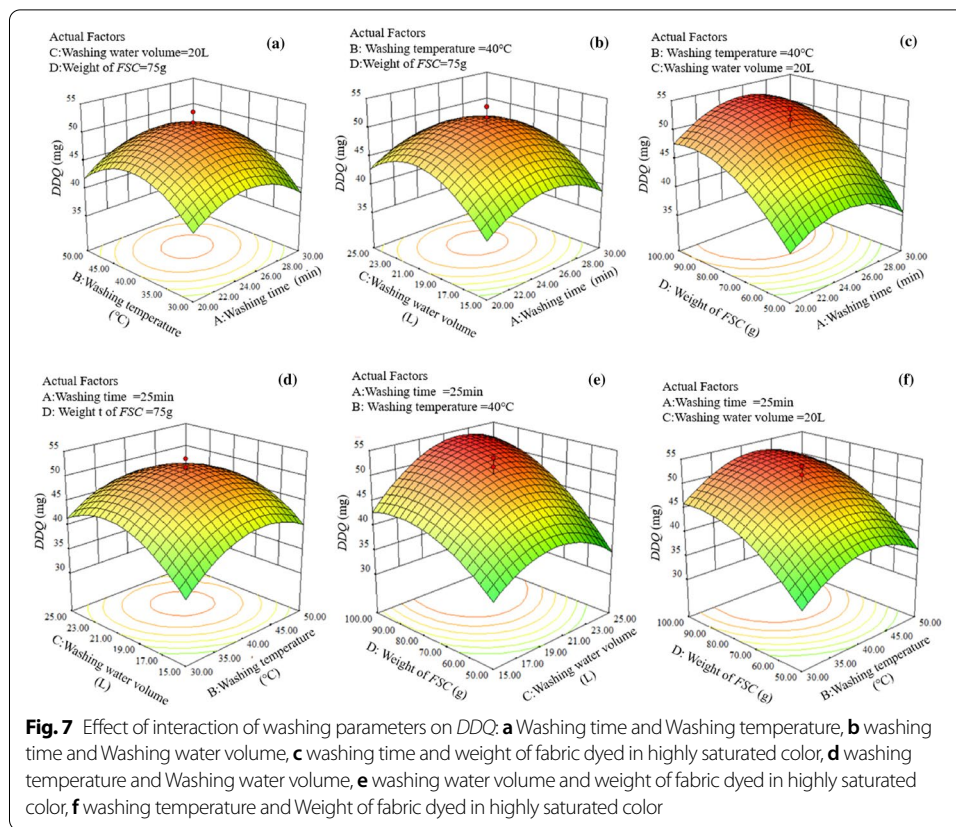


the steepness of the response surface, the significance of the interaction between the two factors on the response value can be seen (Tushar et al., 2013).

It can be seen from Fig. 7f that the interaction of the washing water volume (C) and the weight of *FSC* (D) have the most significant impact on the *DDQ*. The slope of the response surface is steep, and the contour line is oval. When the volume of washing water is 20 L and the weight of *FSC* is 75 g, it is denser, and the peak color is also darker. It can be seen from Fig. 7c that the interaction between washing temperature (B) and washing water volume (C) has little effect on the *DDQ*, the slope is gentle, and the contour line is approximately circular. Figure 7a,b are the response surfaces of the interaction of washing time (A) and washing temperature (B) with washing time (A) and washing water volume (C) respectively, and the contour lines are nearly circular, which have low significance on the response value. Therefore, according to the shape of the response surface, the contour distribution, and the analysis of variance, it can be concluded that the influence of the interaction of washing time, washing temperature, washing water volume, and the weight of *FSC* on the *DDQ* in the washing wastewater is $CD > AB > AC > BC > AD$.

Verification test

The minimum washing parameters T_1 optimized as follows: washing time was 20 min, the washing temperature was 50 °C, washing water volume was 15 L, and the weight of *FSC* was 50 g, and the predicted value of dye discharge was 22.29 mg. The washing parameters T_2 with the maximum dye discharge are as follows: washing time was 25.45 min, washing temperature was 41.73 °C, washing water volume was 21.61 L and the weight of *FSC* was 98.27 g, under which the predicted *DDQ* was 53.62 mg. To facilitate the experimental operation, T_2 was adjusted to a washing time of 25 min, a washing

**Table 8** Verification results of optimized parameters

	Number	Predicted value/mg	Test value/mg	Test average	Range of prediction error rate
T_1	1	22.29	24.17	24.70	[− 2.14%, + 1.82%]
	2	22.29	25.15		
	3	22.29	24.77		
T_2	1	53.62	51.29	53.10	[− 3.53%, + 2.98%]
	2	53.62	53.39		
	3	53.62	54.63		

temperature of 40 °C, a washing water volume of 20 L, and the weight of FSC of 98 g. The verification test results are shown in Table 8. The inter-group prediction error rate of T_1 optimization parameters was [− 2.14%, + 1.82%], and the inter-group prediction error rate of T_2 optimization parameters was [− 3.53%, + 2.98%], both of which were less than [− 5%, + 5%], indicating that the optimization prediction model was more reliable and the response value prediction was more accurate. In addition, under the model, the DDQ from household laundry was effectively suppressed, and the optimized parameters for discharge reduction could effectively reduce the dye discharge by 0.99 mg per liter, with an average discharge reduction rate of 53.5%.

Conclusions

In this research, the effect of washing parameters on dye discharge of *FSC* by reactive dyes has been discussed. The main factors affecting the *DDQ* and the significant interval distribution of factor variables were determined by a single factor experiment. Washing time, washing temperature, washing water volume, and the weight of *FSC* had a significant effect on the dye discharge concentration. Under a certain washing load, the effect of the dosage of detergent on the dye discharge concentration was not significant. According to the central composite design and variance analysis, the Quadratic model was found to be the most suitable model to predict the impact of washing parameters on the *DDQ*, and the regression model between washing parameters and dye discharges was established. The results of variance analysis and response surface distribution showed that the order of significant single factor effect on the *DDQ* was the weight of *FSC*, washing water volume, washing time, and washing temperature, and the order of significance of single factor interaction on *DDQ* was washing water volume and weight of *FSC*, washing temperature and weight of *FSC*, washing time and washing temperature, washing time and washing water volume, washing temperature and washing water volume, washing time and weight of *FSC*.

The optimal washing parameters were obtained as the following: washing time was 20 min, washing temperature was 50 °C, washing water volume was 15 L, and the weight of *FSC* was 50 g, and the predicted *DDQ* was 22.29 mg. The washing parameters with the maximum of *DDQ* are as the following: washing time was 25.45 min, washing temperature was 41.73 °C, washing water volume was 21.61 L and the weight of *FSC* was 98.27 g, under which the predicted of the *DDQ* is 53.62 mg. The optimized washing parameters can effectively reduce the dye discharge of 0.99 mg/L when compared with the unoptimized washing model, and the average emission reduction rate is up to 53.5%, which has been validated through three groups of tests.

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Authors' contributions

XZ is responsible for the conception of the whole study, the design and implementation of the experiment, the collection and analysis of data and the drafting of the first draft, LJ is responsible for the planning of the paper, QBY and MX are responsible for coordinating the research tasks and data analysis, CLZ and JLL are responsible for data analysis and the revision of the first draft. All authors read and approved the final manuscript.

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Availability of data and materials

Please contact author for data requests.

Declarations

Competing interests

The authors declare that they have no competing interests.

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