Experimental Study on Adsorption of Methylene Blue in Wastewater by Bamboo Leaves Powder

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Keywords: Dye Wastewater, Adsorption, Bamboo Leaves, Software Simulation, Software Optimization.

Abstract: Adsorption is one of the common methods for treating dye wastewater. The existing adsorbents still have the problems of high cost, difficult disposal and secondary pollution. Agricultural and forestry wastes with low price and rich sources can be applied to the treatment of dye wastewater. The effect of bamboo leaf powder on the adsorption of methylene blue was studied. The effects of particle size, dosage, temperature, adsorption time and pH on the adsorption effect were studied. The results showed that the optimal single factors were: the particle size is less than 0.25mm, the dosage is 0.4g/100mL, the temperature is 20 ℃, the adsorption time is 50min and pH is 8. Study on the adsorption kinetics indicated that pseudo-second-order kinetic model could better describe the kinetic behaviour. Comprehensively considering the interaction among multiple factors, the optimization experiment was designed by the Optimal mode of Design-Expert software, and the factors were optimized by the response surface method. The results showed that the optimal conditions were: the dosage is 5.37g/L, the adsorption time is 65.47min, the temperature is 25.17 ℃, and pH is 6.7. The results showed that bamboo leaf is a potential natural adsorbent for methylene blue in wastewater.

1 INTRODUCTION

Most of the dyes contained in dye industrial wastewater are synthetic dyes with stable molecular structure and poor biodegradability. It is an important factor causing water pollution. Fenton oxidation, catalytic oxidation, biodegradation and adsorption are often used in the treatment of dye wastewater. Among them, adsorption is widely recognized because of its low cost, simple treatment method and high treatment efficiency (Gao 2018, Donkadokula 2020).

Among the existing adsorbents, activated carbon has good adsorption properties, but it is not suitable for the primary treatment of dye wastewater because of its high cost and difficult regeneration. Mineral adsorbents, coal and slag adsorbents have a wide range of raw materials and low cost, but the subsequent disposal is difficult and the secondary pollution is also a problem that cannot be ignored. The adsorption resin has good effect, easy regeneration and simple operation, but the high cost limits its large-scale use. As a possible adsorbent, agricultural and forestry wastes have the advantages of low price and rich sources, and can be applied to the treatment of dye containing wastewater.

China is the country with the most abundant bamboo resources in the world. Bamboo can not only be used for physical processing and making appliances, but also for extracting chemicals from bamboo knots, leaves, branches and roots. Bamboo charcoal and bamboo vinegar can be prepared by pyrolysis. Bamboo charcoal can be used as fuel, and to make activated carbon and then applied in environmental pollution control, and as conductive materials (Zhang 2020, Isa 2016, Jiang 2021). Bamboo vinegar can be used as soil disinfectant, composting accelerator, feed additive and plant growth regulator (Zhang 2021). As a kind of forestry waste, bamboo leaves have the potential to adsorb dye molecules in wastewater. At present, there are few reports on the adsorption of dyes in wastewater by bamboo leaves (Wu 2019, Yang 2017).

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In this study, bamboo leaves were used as raw materials and methylene blue aqueous solution was used to simulate the wastewater containing methylene blue. The effects of adsorption conditions such as bamboo leaf powder particle size, dosage, temperature and pH on the adsorption effect of methylene blue in wastewater were studied, and the mechanism of adsorption process was explained by fitting the adsorption kinetics. Finally, the Optimal model of Design-Expert was used to optimize the experimental design of response surface, and the optimal reaction conditions of bamboo leaf powder adsorbing methylene blue were obtained.

2 MATERIALS AND METHODS

2.1 Materials

The Moso bamboo leaves were collected from the campus and washed with tap water, then rinsed with distilled water, and dried in an oven at 60 °C. The dried bamboo leaves were then grinded into powder, screened to obtain powder with particle sizes of < 0.25mm, 0.25-0.4mm, 0.4-0.5mm, 0.5-0.8mm and 0.8-1.0mm, and then stored in sealed bags. The main reagents used in the experiment are methylene blue (analytical purity), HCl (analytical purity) and NaOH (analytical purity).

2.2 Instruments

Main instruments used in this study contains thermostatic oscillator (SKY-200B), ultraviolet spectrophotometer (UV2450) and pH meter (PB-10).

2.3 Methods

2.3.1 Effect Evaluation of Bamboo Leaf Powder Adsorbing Methylene Blue Title

(1) Standard curve of absorbance and methylene blue solution concentration

Methylene blue solution with concentration of 100mg/L was prepared and diluted to 2, 4, 5, 6 and 8mg/L, respectively. After zeroing with distilled water at 662nm on the ultraviolet spectrophotometer, the absorbance of the diluted solutions was measured. The standard curve was drawn with the concentration of methylene blue solution as the abscissa and the absorbance as the ordinate, and the standard curve equation was obtained.

(2) Evaluation of adsorption effect

The absorbance of methylene blue aqueous solution before and after adsorption was determined by ultraviolet spectrophotometer, the concentration of methylene blue was calculated according to the standard curve, and the adsorption effect was evaluated by the removal efficiency:

$$\eta=\frac{c_0-c_i}{c_0}\times 100\% \tag{1}$$

Where: η is the adsorption removal efficiency; c_0 and c_i are the concentration of methylene blue in solution before and after adsorption, mg/L.

2.3.2 Effect of Adsorption Factors on the Adsorption

Several conical bottles with volume of 250mL were prepared, 100 mL methylene blue solution with a certain concentration and bamboo leaf powder with a certain quality were added into each conical bottle, and then stirred at a speed of 150r/min for a certain time at a certain temperature. After stirring, the mixture in the conical flask was filtered with 0.45 μm filter membrane to obtain filtrate. The original methylene blue solution and the filtrate were diluted and their absorbance were determined by the ultraviolet spectrophotometer at the wavelength of 662nm, the concentration of methylene blue were calculated and the adsorption effect was evaluated. Factors such as particle sizes and dosage of bamboo leaf powder, temperature, adsorption time, pH and the initial concentrations of methylene blue solution were taken into consideration when discuss the effect of adsorption conditions on the adsorption.

2.3.3 Adsorption Kinetics

The concentration of methylene blue in the solution at different times of adsorption reaction was determined, and the results were fitted by the pseudo-first-order kinetic equation and the pseudo-second-order kinetic equation (Travin 2019, Francis 2021), then the kinetic equation most suitable for the behaviour of bamboo leaf powder adsorbing methylene blue in water is obtained by comparison.

The pseudo-first-order kinetic equation:

$$\ln(q_e\text{-}q_t)=\ln q_e-k_1 t \tag{2}$$

The pseudo-second-order kinetic equation:

$$\frac{t}{q_t}=\frac{1}{k_2 q_e^2}+\frac{t}{q_e} \tag{3}$$

Where: q_e is the equilibrium adsorption capacity of bamboo leaf powder for methylene blue, mg/g; q_t is the adsorption capacity of bamboo leaf powder for methylene blue at time t, mg/g; t is the reaction time, min; k_1 is the adsorption rate constant, min^{-1}; k_2 is the adsorption rate constant, g/mg · min.

2.3.4 Response Surface Optimization Test

Four factors of bamboo leaf powder dosage, adsorption time, temperature and pH were selected in the response surface optimization test. The range of each factor level included the corresponding value with the highest removal efficiency in the test results in 2.3.2. The removal efficiency of methylene blue by bamboo leaf powder was taken as the response value, the response surface test was designed by Design-Expert 8.0.6 software. The factors were optimized through the test results, and the best process parameters were obtained.

3 RESULT AND DISCUSSION

3.1 Effect of Adsorption Conditions on the Adsorption

3.1.1 Effect of Bamboo Leaf Powder Particle Size on the Adsorption

The effect of bamboo leaf particle sizes on the adsorption is shown in Figure 1. The particle sizes were determined as <0.25mm, 0.25-0.4mm, 0.4-0.5mm, 0.5-0.8mm and 0.8-1.0mm, respectively, the dosage is 0.5g, and the initial concentration of the methylene blue solution was 100mg/L. As shown in Figure 1, as the particle size gradually increases, the removal efficiency gradually decreases. When the particle size is less than 0.25mm, the removal efficiency reaches 93% and removal effect is the most significant. When the particle size increased to 0.8-1mm, the removal efficiency was only 69.4%. This is because the specific surface area of solid adsorption materials increases with the decrease of particle size. Therefore, bamboo leaf powder with particle size less than 0.25mm was selected as the material for subsequent tests.

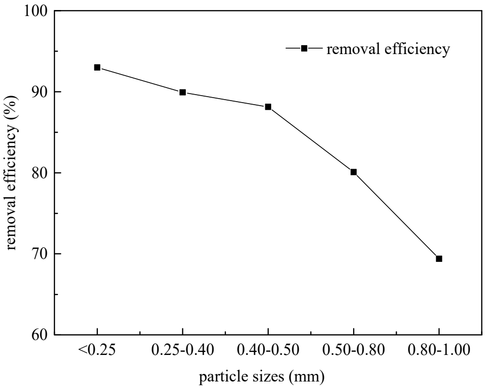

Figure 1: Effect of particle size on the removal efficiency.

3.1.2 Effect of Bamboo Leaf Powder Dosage on the Adsorption

The effect of bamboo leaf powder dosage on the adsorption is shown in Figure 2. In this test, different dosage of bamboo leaf powder with particle size less than 0.25mm was added into methylene blue solution with initial concentration of 100mg/L, and the dosage was determined as 0.1, 0.2, 0.3, 0.4, 0.6, 0.8 and 1.0g/100mL, respectively. It can be seen from Figure 2 that the removal efficiency increases with the increase of the dosage. When the dosage increases from 0.1 to 0.4g/100mL, the removal efficiency increases obviously. Then, when the dosage increases furtherly, the removal efficiency changes little and the adsorption tends to be saturated. Therefore, the optimum dosage of bamboo leaf powder under this condition is 0.4g/100mL.

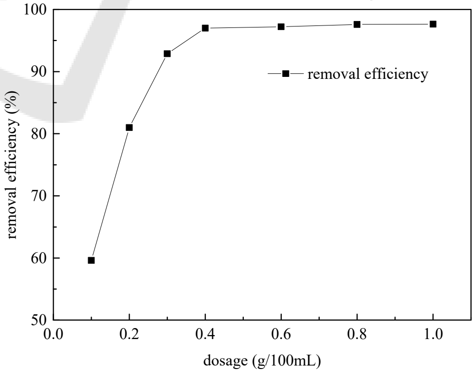

Figure 2: Effect of dosage on the removal efficiency.

3.1.3 Effect of Temperature on the Adsorption

The effect of temperature on the adsorption is shown in Figure 3. In this test, 0.4g bamboo leaf powder with particle size less than 0.25mm was added into the methylene blue solution with concentration of

100mg/L. The temperatures were determined as 5, 10, 15, 20, 30, 40, 50 and 60 °C respectively. In Figure 3, with the increase of temperature, the removal efficiency increases in the range of 5-20 °C, and then decreases slowly with the increase of temperature, and reached a maximum of 96.8% at 20 °C. Therefore, the optimum temperature for the adsorption is 20 °C.

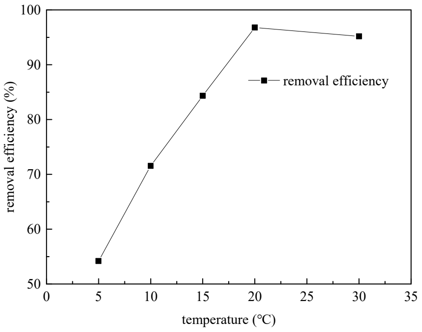

Figure 3: Effect of temperature on the removal efficiency.

3.1.4 Effect of Time on the Adsorption

The effect of time on the adsorption is shown in Figure 4. This test was carried out with temperature of 20 °C, bamboo leaf powder dosage of 0.4g with particle size less than 0.25mm, and with an initial concentration of 100mg/L. The adsorption reaction time was 20, 40, 60, 80,100,120, 140 and 160 min, respectively. It can be seen from Figure 4 the removal efficiency increases obviously in the first 50 min during the process and then tends to be stable, and the adsorption basically reaches the saturation state.

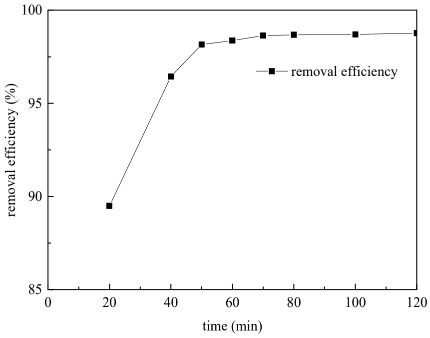

Figure 4: Effect of adsorption time on methylene blue removal efficiency.

3.1.5 Effect of pH on the Adsorption

The effect of pH on the adsorption is shown in Figure 5. The test was carried out with temperature of 20 °C, bamboo leaf powder dosage of 0.4g with particle size less than 0.25mm, and with an initial concentration of 100mg/L. The pH of the reaction system was adjusted by diluted HCl and diluted NaOH solution. It can be seen from Figure 5 that the removal efficiency increases with the increase of pH in the range of pH 3-8 and reaches the maximum at about pH 8. After that, the removal efficiency decreased gradually with the increase of pH.

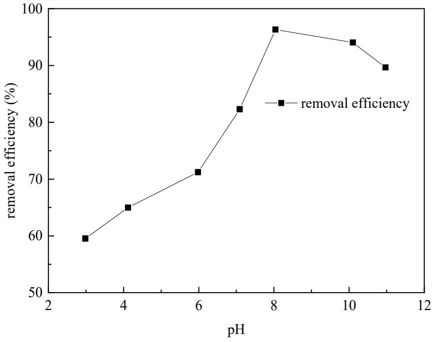

Figure 5: Effect of pH on the removal efficiency.

3.2 Adsorption Kinetics

The relationship between adsorption capacity and adsorption time in pseudo-first-order kinetic equation and pseudo-second-order kinetic equation is shown in Figure 6 and Figure 7. The parameters of the kinetic equation are shown in Table 1.

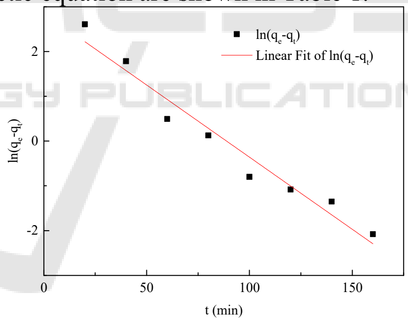

Figure 6: Fitting for the pseudo-first-order kinetic equation.

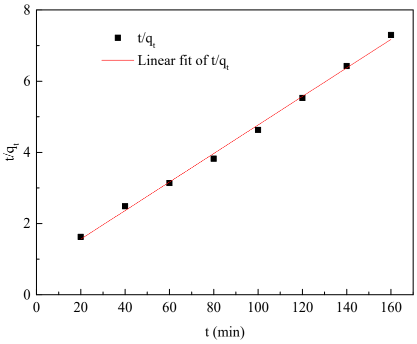

Figure 7: Fitting for the pseudo-second-order kinetic equation.

Table 1: Parameters of the kinetic equation.

Pseudo-first-order equation			Pseudo-second-order equation		
q_e (mg/g)	k_1 (min^{-1})	R^2	q_e (mg/g)	K_2 (g/mg·min)	R^2
17.52	0.0322	0.959	24.94	0.0021	0.997

Table 1 reveals that the linear correlation of pseudo-first-order and pseudo-second-order kinetic model fitting are relatively good, and the coefficients are 0.959 and 0.997, respectively. In terms of the linear correlation coefficient, pseudo-second-order kinetic model can better describe the kinetic behaviour of the adsorbing.

3.3 Response Surface Test

3.3.1 Test Scheme and Results

In order to determine the best combination of experimental variables through fewer experiments, the response surface method (RSM) was applied. The coupling effect of main reaction factors on removal efficiency was studied by test designed by the Optimal mode in Design-Expert software, and the value of independent variables was optimized. The optimum conditions were obtained by optimization with the highest adsorption removal efficiency as the goal. The resume of RSM experimental design is shown in Table 2.

Table 2: Parameters of the kinetic equation.

Factor	Name	Dimension	min	max	Mean value	Std. Dev.
A	Dosage	g/L	3	7	5.278	1.738
B	Time	min	40	80	59.31	15.75
C	Temperature	℃	10	40	24.56	13.05
D	pH		4	10	6.952	2.556

The tests are carried out with an initial concentration of methylene blue of 100mg/L, a particle size of bamboo leaf powder of less than 0.25mm and a stirring speed of 150r/min. The test scheme and result are shown in Table 3. The tests are carried out three times, and the experimental results are expressed as the average value.

Table 3: RSM test scheme and result.

No.	Independent variable				Response variable
	A	B	C	D	Removal efficiency, %
1	7.00	72.80	40.00	4.00	82.78
2	5.06	40.00	20.80	10.00	88.78
3	7.00	80.00	20.80	6.89	89.84
4	3.00	60.60	25.60	9.57	88.37
5	6.80	52.00	24.90	6.50	92.53
6	7.00	60.80	10.00	10.00	86.93
7	3.00	40.00	10.00	7.09	72.09
8	3.00	80.00	10.00	10.00	74.23
9	3.00	59.20	20.97	4.00	78.86
10	4.60	64.00	40.00	7.64	91.12
11	3.00	40.00	40.00	10.00	79.12
12	3.74	40.00	40.00	4.00	72.72
13	7.00	40.00	10.00	4.00	75.3
14	7.00	80.00	40.00	10.00	69.34
15	7.00	40.00	10.00	4.00	74.63
16	4.90	80.00	10.00	4.00	87.53
17	3.00	80.00	40.00	4.00	86.18
18	7.00	72.80	40.00	4.00	86.28
19	7.00	40.00	40.00	8.95	83.62
20	7.00	60.80	10.00	10.00	84.12
21	4.74	62.60	12.70	7.36	92.37

3.3.2 Parametric Model and Analysis of Variance

The main parameter data of the model fitted for response variable and the analysis of variance are shown in Table 4 and Table 5, respectively. According to the results, the quadratic model is selected. The significant coefficient p of the model is

0.0001, and the p of the lack of fit term is 0.6848 (>0.05), indicating that the model is significant, but the lack of fit is not significant. The determination coefficient R^2 is 0.9500, which shows that the model fits well with the actual situation and the error is small. It can accurately analyse and predict the removal efficiency. The F value of the model is 28.13, while the F value of the lack of fit is 0.5449, indicating that the model is significant, the mismatch term is not significant, and 68.48% of the F value of the mismatch term may be caused by noise. The coefficient of variation of the model is 1.9689% (<5%), indicating that the model has high reliability and sufficient experimental data. The precision is 17.31 (>4), which shows that the signal of the fitting model is sufficient and the fitting is reasonable. Figure 8 shows that the normal distribution linearity of the residual and the fitting effect of the model are good. The above results show that the fitting model has high reliability and precision.

Table 4: The main parameter data of the model.

Resource	p	p(Lack of fit)	R^2	
Linear	0.8300	0.0139	0.1455	
2FI	0.2563	0.0154	0.0515	
Quadratic	0.0001	0.6848	0.9500	Suggested
Cubic	0.6848	-	0.9352	Aliased

Table 5: The analysis of variance.

Variance source	Sum of Squares	DF	Mean Square	F	p (prob>F)	Significance
Model	1044.09	14	74.58	28.13	0.0003	**
A	69.66	1	69.66	26.27	0.0022	*
B	54.89	1	54.89	20.70	0.0039	**
C	5.08	1	5.08	1.91	0.2157	*
D	3.46	1	3.46	1.30	0.2969	**
AB	47.70	1	47.70	17.99	0.0054	*
AC	63.19	1	63.19	23.83	0.0028	
AD	21.86	1	21.86	8.24	0.0284	*
BC	4.77	1	4.77	1.80	0.2283	
BD	216.79	1	216.79	81.76	0.0001	*
CD	13.66	1	13.66	5.15	0.0637	
A^2	55.43	1	55.43	20.91	0.0038	**
B^2	57.27	1	57.27	21.60	0.0035	**
C^2	56.60	1	56.60	21.35	0.0036	**
D^2	86.49	1	86.49	32.62	0.0012	**
Residual	15.9091	6	2.65			
Lack of Fit	5.6116	3	1.87	0.5449	0.6848	
Pure Error	10.30	3	3.43			
Cor Total	1060.00	20				
R^2	0.9850					
C.V. %	1.9689					
Adeq. Precision	17.31					

Note: $*p<0.05$, significant, $**p<0.01$, extremely significant.

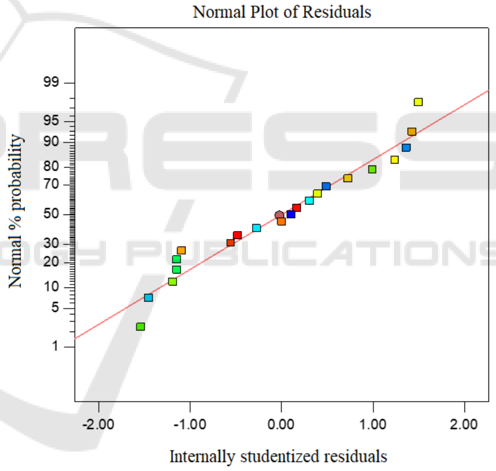

Figure 8: Normal distribution diagram of residual probability.

3.3.3 Single Factor Effect Analysis

In RSM test, the fluctuation diagram of A (dosage), B (time), C (temperature) and D (pH) factors is shown in Figure 9. The independent variables in this experiment have square effect, linear effect and interaction with the response variables. With the increase of factor level, the response value first rises and then decreases slowly. From the fluctuation range of response variable, the fluctuation of each factor from high to low is A, B, D, and C. Therefore, the influence of various factors on the removal efficiency is A, B, D and C from high to low.

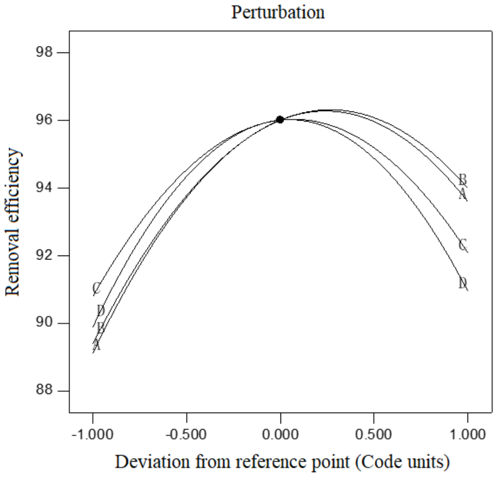

Figure 9: The fluctuation diagram of single factor.

3.3.4 Analysis of Interaction among Factors

The reaction surface analysis diagram is a three-dimensional diagram composed of reaction values and any experimental factors, showing the influence of the other two variables on the test efficiency when two of the factors A, B, C and D are at the intermediate level. However, when the two factors interact, the influence of one factor on the other factor is different at different levels. The response surface of this study basically presents an open form, that is, with the increase of factor level, the removal efficiency shows a trend from high to low. The contour map is a curve formed on the lower surface with the same factor value on the surface. The closer the contour is to the circle, the smaller the interaction between them. The closer the contour of the ellipse is, the greater the interaction between the two. The density of the contour line reflects the factors affecting the removal efficiency. The denser the contour, the greater the effect on the removal efficiency.

The pairwise interaction between the factors is shown in Figure10. The contour map of the interaction between A and B is shown in Figure10(a). The oval contour indicates that there is interaction between A and B. The contour density of A is higher than that of B, indicating that A has a greater impact on the removal efficiency than B. The response surface showed that the removal efficiency increased rapidly with the increase of A, and then begins to decline slowly when the dosage exceeds a certain value (5-6g/L). With the increase of B, the removal efficiency increases slowly and then decreases slowly, and reaches the maximum when the time during 56-72 min. The contour map of the interaction between A and C is shown in Figure10(b). The oval contour lines indicate that there is interaction between A and C. The contour density of A is higher than that of C, indicating that the impact on removal efficiency of A is greater than that of C. The response surface diagram shows that the removal efficiency increases slowly with the increase of A, and then decreases slowly when the dosage exceeds a certain value (5-6g/L). With the increase of C, the removal efficiency first increases slowly and then decreases slowly, and reaches the maximum when the temperature reaches 22-27℃. The contour map of the interaction between A and D is shown in Figure10(c). The oval contour line indicates that there is interaction between A and D. The contour density of A is greater than D, indicating that A has a greater impact on the removal efficiency than D. The response surface showed that the removal efficiency increased slowly with the increase of A level, and then begins to decline slowly when the dosage exceeds a certain value (5-6 g/L). The removal efficiency increases slowly with the increase of D, and begins to decline slowly when pH exceeds a certain value (6.5-7.5). The contour map of the interaction between B and C is shown in Figure10(d). The contours of B and C are close to circular, indicating that the interaction between B and C is not obvious. The density of B is greater than C, indicating that the effect of B on adsorption is greater than C. The response surface showed that the removal efficiency increased slowly with the increase of B, and then begins to decrease slowly when the time exceeds a certain value (60-70min). With the increase of C, the removal efficiency increases slowly and then decreases gradually, and it reaches the maximum when the temperature reaches 22-27℃. The contour map of the interaction between B and C is shown in Figure10(e). The oval contour indicates that there is interaction between B and D. The contour thickness of B is greater than D, indicating that B has a greater impact on the removal efficiency than D. The reaction surface showed that the removal efficiency increased rapidly with the increase of B, and then decreased slowly after the time exceeding a certain value (60-70min). The removal efficiency increased rapidly with the increase of D, and then begins to decline slowly when pH exceeds a certain value (6.5-7.5). The contour map of the interaction between C and D is shown in Figure10(f). The contour lines of C and D are close to circular, indicating that the interaction between C and D is not obvious. The contour line thickness of D is greater than C, indicating that D has a greater impact on the removal efficiency than

C. The reaction surface showed that the removal efficiency first increased slowly and then decreased slowly with the increase of C. The removal efficiency reaches the maximum when the temperature reaches 24-28°C. With the increase of D, the removal efficiency increased slowly and then begins to decline slowly when pH exceeds a certain value (6.5-7.5).

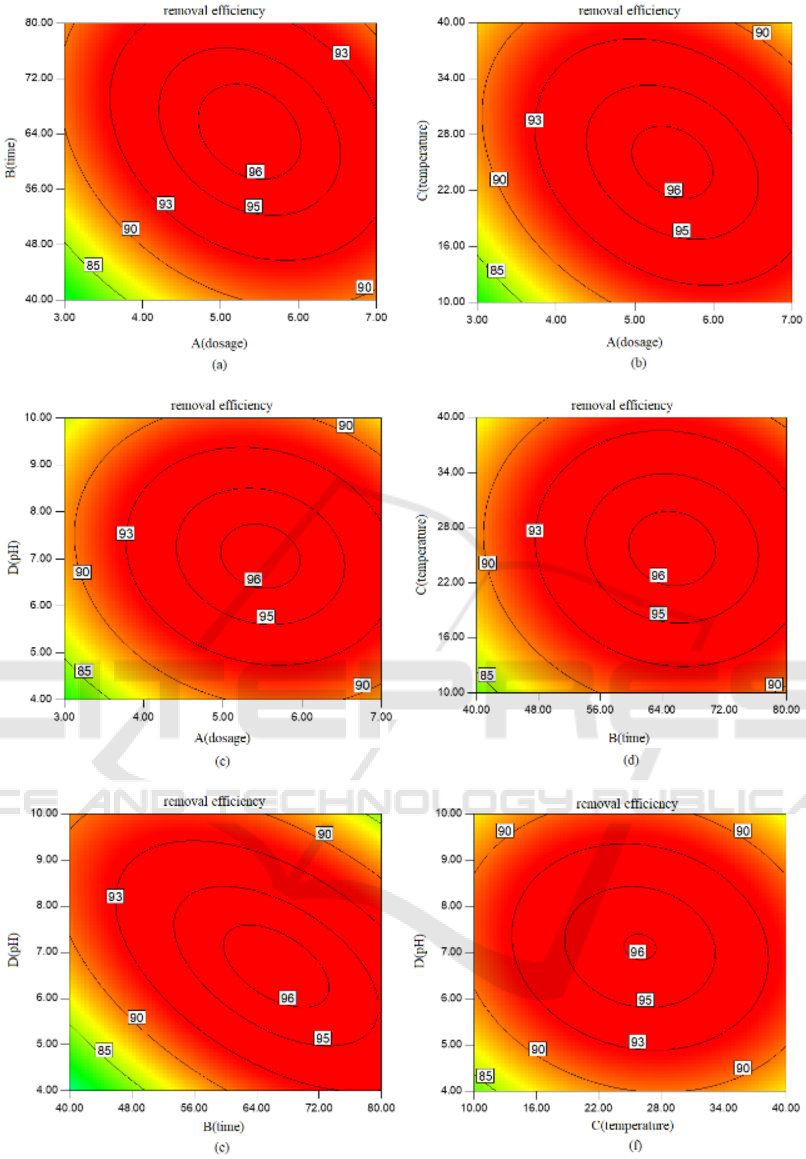

Figure 10: Pairwise interaction between the factors on removal efficiency.

3.3.5 Optimization of Adsorption Conditions

Two adsorption schemes are recommended by RSM optimization. The optimal adsorption conditions are: dosage of 5.37g/L, adsorption time of 65.47min, temperature of 25.17 ℃, and pH of 6.7. The theoretical predicted value of the removal efficiency is 96.5145%, and the expected value reaches 1.0, as shown in the Table 6.

Table 6: The optimal adsorption schemes.

No.	A	B	C	D	Removal Efficiency (%)	Desirability
1	5.37	65.47	25.17	6.70	96.5145	1
2	5.36	65.69	25.11	6.68	96.5142	0.999989

4 CONCLUSIONS

(1) Bamboo leaf is a potential natural adsorbent for methylene blue in wastewater.

(2) The best adsorption effect could be obtained when the particle size of bamboo leaf powder was less than 0.25mm, the dosage was 0.4g/100mL, the temperature was 20 ℃, the adsorption time was 50min and the pH was 8, respectively.

(3) Pseudo-second-order kinetic model can better describe the kinetic behaviour of bamboo leaf powder adsorbing methylene blue in wastewater.

(4) Comprehensively considering the influence of various factors on the adsorption effect, the optimal conditions for the adsorption were as follows: the dosage is 5.37g/L, the adsorption time is 65.47min, the temperature is 25.17 ℃, and pH is 6.7. The theoretical predicted value of removal efficiency is 96.5145%.

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