

RESPONSE SURFACE OPTIMIZATION BASED ON FREEZE-THAW CYCLE PRETREATMENT OF POPLAR WOOD DYEING EFFECT

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ABSTRACT

To improve the permeability of dye solution in wood, poplar was pretreated by freeze-thaw cycle. The effects of three parameters, such as freezing time, thawing time and circulation times on the dyeing effect of pretreated poplar were investigated by single factor method. On this basis, pretreatment conditions were optimized by response surface methodology. The optimum conditions of this treatment were freezing time of 11.9 h, thawing time of 7.2 h and two cycles. Under these conditions, the dye uptake and chromatic aberration of the treated wood were 25.77% and 21.14%, respectively, which were close to the theoretical predicted values. The results showed that freeze-thaw cycle pretreatment could effectively improve the permeability of poplar and enhance the dyeing effect of wood.

KEYWORDS: Poplar, freeze-thaw cycles, response surface, dye uptake, chromatic aberration.

INTRODUCTION

In 2020, China put forward the goal for "double carbon", which triggered the thinking of various industries. China is one of the countries with relatively short timber resources. In the future, the development direction of wood conservation should be more closely integrated with the national goal of "double carbon" and strive to realize "zero carbon industry"(Barecka et al. 2021). Poplar has the characteristics of short rotation period, wide planting area and strong adaptability, and has become one of the four fast-growing timber forest species in China. However, as a short-period industrial material, poplar is loose in material, low

in density and monotonous in color, and can only be used as inferior material for a long time, resulting in many restrictions in the field of medium and high-grade furniture (Li et al. 2021). Wood dyeing is the most common wood processing technique. The low-grade wood processed by dyeing not only has the advantages of natural wood, but also can simulate the color and texture of high-grade wood, thus improving the decorative effect and added value (Hu et al. 2020). The dyeing is a process in which dye molecules permeate, adsorb and fix in wood (Liu et al. 2021). Improving the permeability of wood is an important link to improve the dyeing effect of wood, and the pretreatment of wood is the key to improving the permeability.

Research by domestic and foreign scholars on improving wood permeability revolves around the following three aspects: physical, chemical, and biological methods. Physical methods mainly involve increasing the permeability of channels by virtue of external or mechanical forces that cause varying degrees of damage to the grain pore membrane and thin-walled cells of wood (Yang and Xu 2021). Commonly used methods include microwave pretreatment (Ethaib et al. 2017), steam pretreatment (Kumar et al. 2012), freezing pretreatment (Babiński et al. 2011), and micro-explosion pretreatment (Kvist et al. 2020). The chemical method, on the other hand, is based on the reaction of chemicals such as alkalis, hot water, and organic solutions with extractive in the wood that hinder penetration, thus dissolving and reducing extractive and increasing fluid flow channels (Modenbach and Nokes 2014). Biological methods use microorganisms and enzymes, among others, to break down the lignin in wood and erode the wood tubular cells with marginal grain pores as well as the cellular grain pores to achieve an increase in wood permeation channels (Lehringer et al. 2009). Among them, chemical method involves the substantial problems of reagent cost, wood performance and ecological protection, such as deepening the wood color, producing waste liquid, and corroding equipment. The biological method has the limitations of low efficiency, poor controllability and uneven permeability change. The principle of freeze-thaw cycle pretreatment is to improve the smoothness of the fluid channel by virtue of the water in the wood under low temperature environment condensing into ice, and the volume expansion causes cracks or damage to the pit membrane (Torgovnikov and Vinden 2010). Its process and principle are as shown in Fig. 1. It has little impact on the performance of wood, is environmentally friendly, and conforms to the national "double carbon" goal.

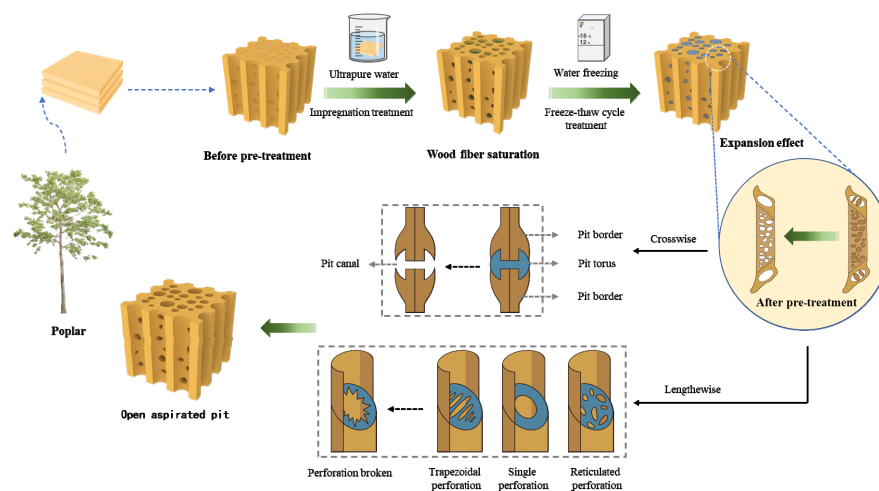


Fig. 1: Freeze-thaw cycle pretreatment process and principle.

At this stage, domestic and foreign research on freeze-thaw cycling of wood is mainly focused on wood permeability (Kumar et al. 2016), drying rate (Yin and Liu 2021), and reduction of wrinkling rate (Yang and Liu 2021), and the research on improving the effect of wood dyeing with natural dyes is still slightly insufficient. Therefore, in this paper, poplar wood, one of the four fast-growing timber in China, was used as the research object, and the freeze-thaw cycle method was applied as a pretreatment. Based on the previous single factor exploration, the effects of freezing time, thawing time and number of cycles on the effect of natural peanut dye on dyed pretreated wood were investigated. The dye uptake and chromatic aberration were used as the response values to optimize the pretreatment process using the response surface method, so as to provide reference for the application of freeze-thaw cycle pretreatment technology to in natural dyes dyed wood (Wang and Zhou 2023).

MATERIAL AND METHODS

Test materials and equipment

Poplar (*Populus tomentosa*) was obtained from plantation forest, originated in Shijiazhuang, Hebei Province, with an air-dry density of 0.386 g cm^{-3} and 12% moisture content. The selected specifications were $40 \times 40 \times 2 \text{ mm}$ (L×W×T), without defective material. Homemade peanut coat natural dyes were used.

Ultrasonic cleaner KH-300DE (Kunshan Hechuang Ultrasonic Instrument Co., Ltd.) was used for extracting peanut coat dye and dyeing the poplar wood. Colorimeter Ci6x (Shanghai Keheng Industrial Development Co., Ltd.) was used for measuring the chromatic aberration of poplar wood before and after dyeing. UV-visible spectrophotometer U-3900 (Hitachi High-Technologies Science Co., Ltd.) was used for measuring the absorbance of the dye solution.

Freeze-thaw cycle processing method

Poplar wood was selected with a relatively flat surface and similar appearance and 800-grit sandpaper was used to remove the surface burrs, spikes and number them. The test material was soaked in ultra-pure water for 24 h, and it was taken out after reaching the saturation point of wood fiber. The moisture on the surface of the test material was wiped off and sealed with a self-adhesive film to prevent the water evaporation of the test material. It was placed in a refrigerator with a temperature of -18 to -20°C to freeze for the corresponding time, then taken out and placed in a constant temperature box at 28°C to thaw for the corresponding time, then taken out and placed in the refrigerator to freeze, and cycled for the corresponding number of times (Boháček et al. 2021). Six samples were tested in each group, and each group was repeated for three times. The specific factor levels of the single factor experiment are shown in Tab. 1.

Tab. 1: Freeze-thaw cycle pretreatment one-way test factor level.

Level	Factor		
	Freezing time (h)	Thawing time (h)	Number of cycles
1	3	1	1
2	6	3	2

3	12	6	3
4	18	9	4
5	24	12	5

Peanut coat dye extraction and wood dyeing methods

Natural dyes generally refer to dyes obtained from natural plants, animals or other material resources, which are basically not treated with chemical solvent (Shahid and Mohammad 2013). Compared with chemical dyes, it has the advantages of soft color, green and harmless, no carcinogen, and environmental friendliness (Ferreira et al. 2004).

The extraction process is: peanut coating → drying (45 °C, 24 h) → crushing → sieving (40 mesh) → ultrasonic-assisted extraction → extraction and filtration → pigment filtrate → rotary evaporation and concentration → vacuum drying.

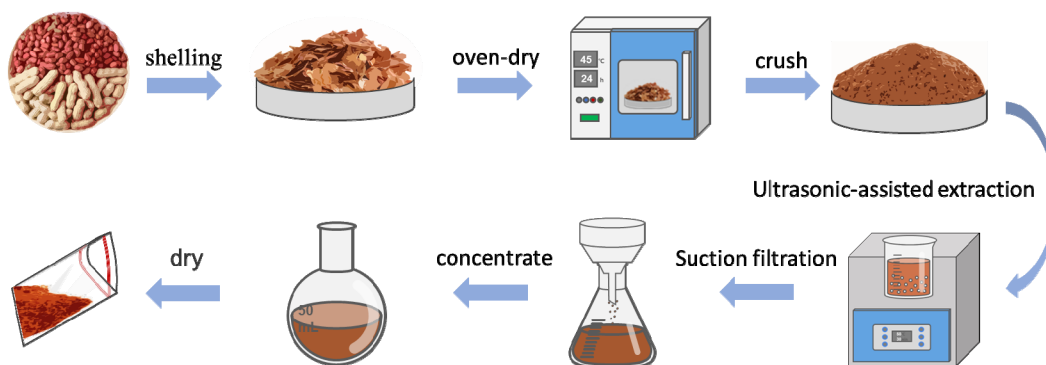


Fig. 2: Flow chart of peanut coat dye extraction.

The peanut coat dye solution with a concentration of 1% was accurately configured, and 50 mL of the dye solution was poured into a beaker. After sealing it with a self-adhesive membrane, the poplar wood was dyed in an ultrasonic cleaning machine at 70°C and 240 W for 3 h. The surface was rinsed with distilled water, and the sample material was drained and then air-dried. The dye uptake and chromatic aberration were to be measured, and the average value of 3 test pieces was taken as the result.

Dye uptake measurement method

After the dyeing was completed, the test materials were taken out. The dyeing solution was cooled to room temperature and supplemented with deionized water until the volume of the dyeing solution was 50 mL, and shaken well. The absorbance at the maximum absorption wavelength of each group of dyeing solution before and after dyeing was measured separately by UV-visible spectrophotometer. The dye uptake was calculated by Eq. 1:

$$U(\%) = \frac{(A_0 - A_1)}{A_0} \times 100 \quad (1)$$

where: U - the dye uptake (%); A_0 - the absorbance of the maximum absorption wavelength of the dye solution before dyeing; A_1 - the absorbance of the maximum absorption wavelength of the dye solution after dyeing (Wang et al. 2016).

Chromatic aberration measurement method

Five marking points were selected on the front of each veneer, and the circle marking part was measured with a colorimeter. CIE (1976) L^* , a^* , b^* chromaticity parameters were used to calculate ΔL^* , Δa^* , Δb^* and ΔE^* . The larger the ΔE^* , the greater the difference in color of the wood before and after treatment. Conversely, a smaller ΔE^* indicates a smaller difference in color between the wood before and after treatment. Using Eq. 2 to calculate the chromatic aberration:

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

where: $\Delta L^* = L_1^* - L_0^*$; $\Delta a^* = a_1^* - a_0^*$; $\Delta b^* = b_1^* - b_0^*$; ΔE^* indicates the veneer chromatic aberration in NBS; ΔL^* reflects the veneer brightness; Δa^* reflects the veneer red and green axis color saturation; Δb^* reflects the veneer yellow and blue axis color saturation; L_0^* , a_0^* , b_0^* represent the pretreatment specimen chromaticity value before; L_1^* , a_1^* , b_1^* represent the chromaticity values of the specimens after pretreatment (Yang et al. 2021).

Response surface experimental design

The response surface method is an optimized statistical method. It was first proposed by R.A. Fisher. After obtaining certain data through reasonable experimental design, the optimal process parameters are found by simulation with multivariate quadratic regression equation. It has the advantages of fewer testing times, short testing period, high accuracy and good prediction performance, and has been widely used in many fields.

Based on the results of the previous single-factor test, the Box-Behnken central combination design was used, with freezing time (A), thawing time (B), and number of cycles as variables (C), and each independent variable was coded at three levels: high (1), medium (0), and low (-1), as shown in Tab. 2. The response surface analysis test with three factors and three levels for a total of 17 test points was designed with the upper dye uptake (U) and chromatic aberration (E) as response values.

Tab. 2: Response surface test factor level coding table.

Factor	Level		
	-1	0	1
Freezing time (A)	6	12	18
Thawing time (B)	3	6	9
Number of cycles (C)	1	2	3

RESULTS AND DISCUSSION

Single-factor test results and analysis

Effect of freezing time on dye uptake and chromatic aberration of poplar wood

The effects of freezing time on the dye uptake and chromatic aberration of poplar wood under the conditions of thawing temperature of 28°C, thawing time of 6 h and two cycles are shown in Fig. 3a. It could be seen that the dye uptake and chromatic aberration had a similar

trend with the extension of freezing time, increasing first and then decreasing. When frozen for 12 h, the dye uptake and chromatic aberration reached the maximum, which were 25.13% and 17.46%, respectively. The freezing time continued to increase, the dye uptake and chromatic aberration decreased instead of increasing. This was due to the water molecules not frozen in the poplar cell wall moving from the cell wall pit membrane to the pit during the process of water freezing and heat transfer inside the poplar wood, causing the cell wall to shrink and offset the expansion effect caused by ice crystals (Xu et al. 2021). So, the dye uptake and chromatic aberration were optimum when the freezing time was 6 h to 18 h.

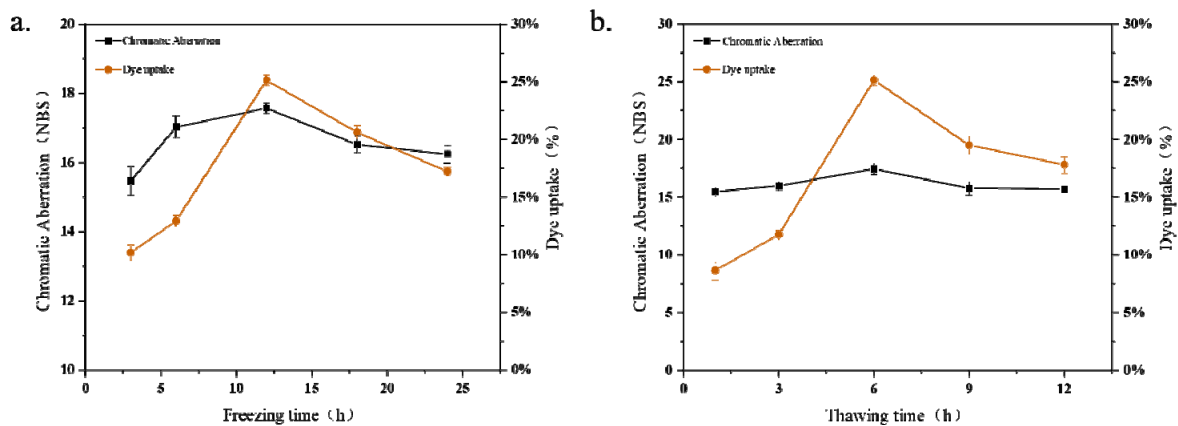


Fig. 3: a) The effect of freezing time on dye uptake and color aberration, b) the effect of thawing time on dye uptake and chromatic aberration.

Effect of thawing time on dye uptake and chromatic aberration of poplar wood

The effect of thawing time on the dye uptake and chromatic aberration of poplar wood under the conditions of thawing temperature of 28°C, freezing time of 12 h and two cycles is shown in Fig. 2b. The best results were obtained at 12 h of thawing, with a dye uptake of 25.12% and a chromatic aberration of 17.57. When water turned to ice, a large number of bubbles formed. After the ice melted, these bubbles existed in the form of particles, and some cells also had bubbles in their cavities (Jakes and Stone 2015). The reason for this increase and then decrease was that when the thawing time was insufficient, the capillary tension generated by the free water migration process was not enough to cause the bubble expansion, and the cyclic freezing and thawing caused very limited extrusion damage to the tracheids, wood rays, and pits (Adzka et al. 2020). When the thawing time was too long, it would cause the wood to evaporate some of the surface water into the air, and then migrated some of the internal water from the cell structures such as pits and tracheids to the surface of poplar (Poncsák et al. 2006). Capillary tension was transferred to the cell wall through the discharged free water, which would lead to wood shrinkage, resulting in poor freeze-thawing effects (Nguyen et al. 2021). The most suitable thawing time was between 3 h and 9 h.

Effect of number of cycles on dye uptake and chromatic aberration of poplar wood

Under the conditions of thawing temperature of 28°C, freezing time of 12 h and thawing time of 6 h, the influence of the number of cycles on the poplar dye uptake and chromatic aberration is shown in Fig. 4. After two cycles, the dye uptake and chromatic aberration reached

the optimum values of 25.12% and 18.63%. When the number of cycles continued to increase, the dye uptake decreased and the chromatic aberration did not change much. This was due to the fact that too few cycles caused less damage to the microstructure inside the wood and the dyeing effect was poor (Jakes et al. 2019). Excessive circulation led to the migration of free water from the microfibrils, pits and tracheids to the outside, and the reduction of water weakened the expansion effect after freezing, resulting in poor freeze-thaw effects (Rooni et al. 2017). In a word, it was reasonable to choose the cycle times range of 1 to 3 times.

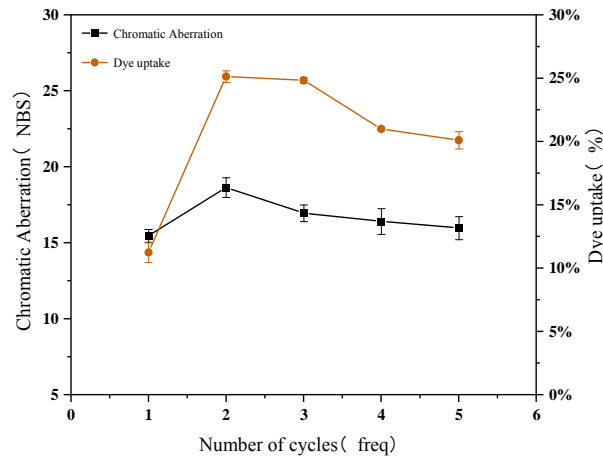


Fig. 4: The effect of the number of cycles on the dye uptake and chromatic aberration.

Response surface test results and analysis

Box-Behnken test design and results

The response surface test protocol and results are shown in Tab. 3. Multivariate analytical regression and quadratic term fitting of the results were performed using Design-Expert 10.0 software, and the regression equation models for dye uptake (U) and chromatic aberration (E) were obtained as Eq. 3 and Eq. 4:

$$U = 0.249 - 0.0178A + 0.0082B + 0.0034C - 0.0002AB + 0.0164AC - 0.0131BC - 0.0341A^2 - 0.0066B^2 - 0.0103C^2 \tag{3}$$

$$E = 20.0857 - 0.5076A + 0.7888B - 0.1842C - 0.0767AB - 0.5988AC + 0.555BC - 0.79A^2 - 1.2554B^2 - 1.4137C^2 \tag{4}$$

Tab. 3: Experimental design and results of response surface analysis.

Test N ^o .	Coded			Actual			Dye uptake (%)	Chromatic aberration (NBS)
	A	B	C	A Freezing time	B Thawing time	C Number of cycles		
1	-1	-1	0	6	3	2	21.69	17.63
2	0	0	-1	18	3	2	18.29	16.80
3	0	-1	-1	6	9	2	23.40	19.43
4	0	0	0	18	9	2	19.92	18.30
5	0	0	1	6	6	1	23.57	17.96
6	0	-1	1	18	6	1	16.60	18.12
7	0	1	-1	6	6	3	21.03	18.85
8	-1	1	0	18	6	3	20.61	16.60

9	-1	0	1	12	3	1	20.78	17.43
10	0	-1	0	12	9	1	25.00	17.82
11	1	0	1	12	3	3	24.03	15.90
12	0	0	-1	12	9	3	23.01	18.51
13	1	0	0	12	6	2	24.70	20.13
14	1	0	-1	12	6	2	24.93	20.09
15	0	0	0	12	6	2	25.00	20.10
16	1	1	0	12	6	2	24.95	20.09
17	0	0	0	12	6	2	24.90	20.02

Regression modeling and significance testing

As seen from the ANOVA of the regression equations in Tab. 4: the p-values were less than 0.0001 and both models were highly significant. The lack of fit terms were not significant and were greater than 0.05 (Putra et al. 2018). The coefficients of variation for the dye uptake (U) and chromatic aberration (E) models were 0.51 and 0.32, respectively, with R^2 of 99.91% and 99.92%, respectively. This indicated that the models could reasonably fit the relationship between the response values and the factors with high experimental accuracy (Kaymaz and McMahon 2005). The significance test showed that the order of the factors affecting the dye uptake of poplar wood was $A > B > C$, that is, freezing time $>$ thawing time $>$ number of cycles. The order of the primary and secondary factors affecting the chromatic aberration of poplar wood was $B > A > C$, that is, thawing time $>$ freezing time $>$ number of cycles.

Tab. 4: Analysis of variance for dye uptake (U) and chromatic aberration (E) regression models.

Source	Quadratic sum		Free degree		Mean square		F-value		p-value		Significance	
	U	E	U	E	U	E	U	E	U	E	U	E
Model	0.0108	29.6102	9	9	0.0012	3.2900	897.00	927.99	<0.001	<0.001	**	**
A	0.0025	2.0611	1	1	0.0025	2.0611	1902.12	581.36	<0.001	<0.001	**	**
B	0.0005	4.9779	1	1	0.0005	4.9779	398.86	1404.08	<0.001	<0.001	**	**
C	0.0001	0.2714	1	1	0.0001	0.2714	69.94	76.54	<0.001	<0.001	**	**
AB	0.0019×10^{-4}	0.0236	1	1	0.0019×10^{-4}	0.0236	0.14	6.64	0.7179	0.0366		*
AC	0.0011	1.4340	1	1	0.0011	1.4340	802.75	404.48	<0.001	<0.001	**	**
BC	0.0007	1.2319	1	1	0.0007	1.2319	512.66	347.47	<0.001	<0.001	**	**
A²	0.0049	2.6280	1	1	0.0049	2.6280	3664.63	741.25	<0.001	<0.001	**	**
B²	0.0002	6.6356	1	1	0.0002	6.6356	136.74	1871.67	<0.001	<0.001	**	**
C²	0.0004	8.4154	1	1	0.0004	8.4154	334.16	2373.66	<0.001	<0.001	**	**
Residual	0.0009×10^{-2}	0.0248	7	7	0.0001×10^{-2}	0.0035						
Lack of fit	0.0004×10^{-2}	0.0183	3	3	0.0001×10^{-2}	0.0061	1.01		0.4741	0.1162	Insign.	Insign.
Pure error	0.0005×10^{-2}	0.0065	4	4	0.0001×10^{-2}	0.0016						
Cor total	0.0108	29.6351	16	16								

Note: * * indicates extremely significant difference ($P < 0.01$); * indicates significant difference ($0.01 < p < 0.05$).

Interaction analysis of response surfaces

Figs. 5 and 6 show the response surfaces and contour plots of each factor on the dye uptake and chromatic aberration of poplar wood, respectively, which visually reflect the influence of each factor and their interaction on the response values. From Fig. 5a, under the condition of

a certain number of cycles, the interaction between freezing time and thawing time on wood dye uptake was not significant, which may be due to the volume expansion having little effect on microstructure during the first freeze-thaw process. From Fig. 5 and Fig. 6 the contour lines of freezing time, thawing time and number of cycles were all dense ellipses, which indicated that the interaction between all other factors reached significance (Alberti et al. 2014). The response surface plot opened downward and there were positive and negative coefficients in the regression equation, indicating that the model had a stable point, and the extreme value point was its stable point (Nepote et al. 2004).

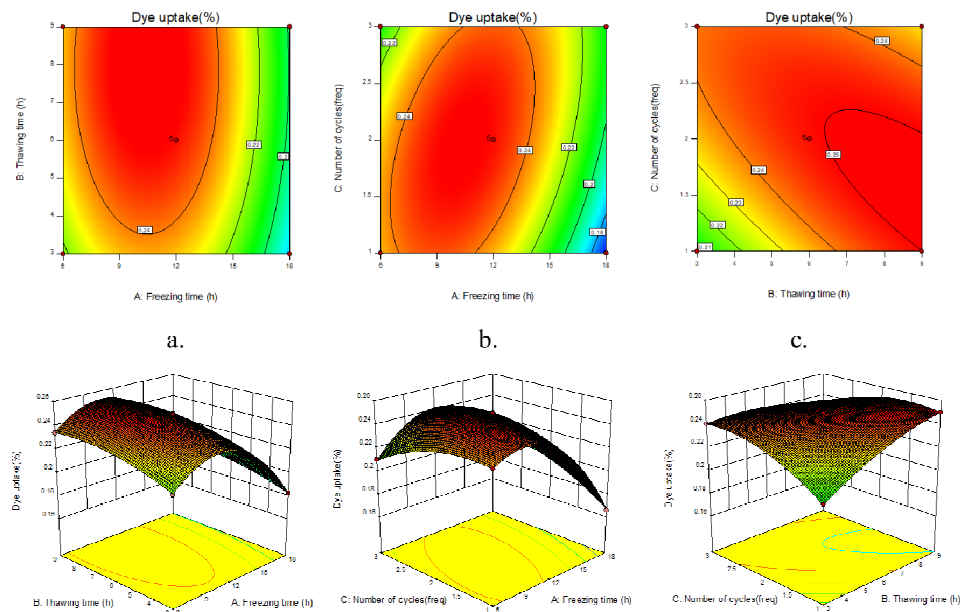


Fig. 5: The interaction of various factors on the dye uptake of poplar.

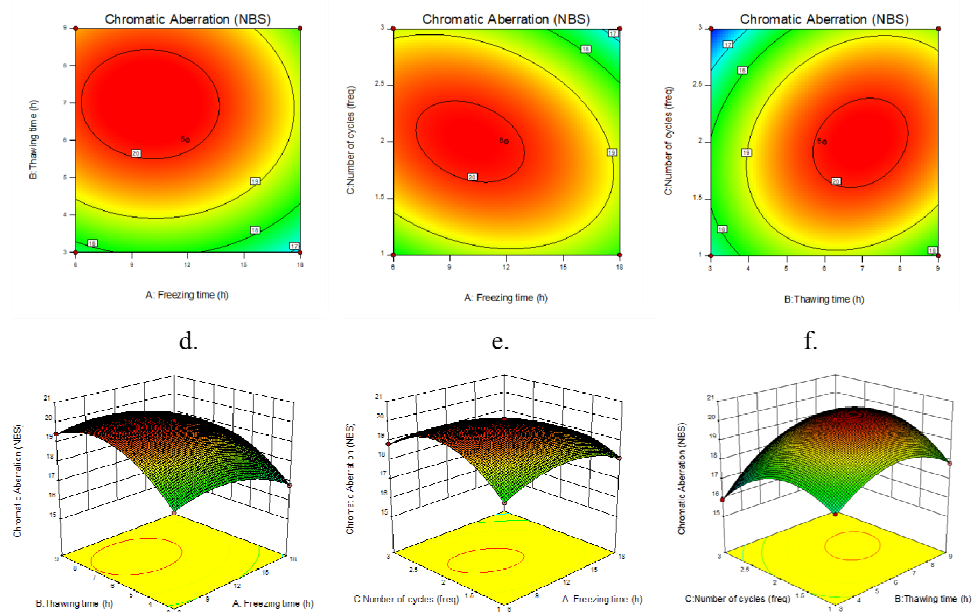


Fig. 6: The interaction of various factors on poplar chromatic aberration.

Response surface optimization and validation

According to the optimization analysis by Design-Expert software, the best pretreatment conditions were freezing for 11.87 h, thawing for 7.21 h, and cycling times for 2.01 times. Under these conditions, the poplar treated by ultrasonic dyeing for 3 hours predicted that the dye uptake and chromatic aberration could reach 25.15% and 20.21%. Considering the actual test operation, the parameters were adjusted as follows: freezing time was 11.9 h, thawing time was 7.2 h, and the number of cycles was 2 times. Under the optimized parameters, three validation tests were conducted and the average values were taken. The dye uptake and chromatic aberration of poplar wood were 25.77% and 21.14, resp., with the relative errors of 2.59% and 4.60% between the actual and theoretical values, which showed that the model was reasonable and accurate and the treatment process parameters were reliable.

CONCLUSIONS

The freezing time, thawing time and number of cycles were important factors influencing the improvement of dye uptake and chromatic aberration of poplar wood in plantation forests by freeze-thaw cycle pretreatment. Among them, the interaction between freezing time and thawing time on the dye uptake was not significant, while the rest of the factors showed significant interaction. With the increase of the level of each factor, the dye uptake and chromatic aberration showed a trend of increasing first and then decreasing.

The optimum conditions for the freeze-thaw cycle pretreatment of poplar wood were 11.9 h freezing time, 7.2 h thawing time, and 2 cycles. The dye uptake and chromatic aberration of poplar wood after freeze-thaw cycle pretreatment were significantly increased, which effectively improved the effect of natural dye dyed wood. In order to promote the green modification of wood and provide new ideas for the development of the veneer dyeing industry towards the goal of "zero carbon".

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