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STUDY ON PERMEABILITY OF *CUNNINGHAMIA LANCEOLATA* BASED ON STEAM TREATMENT AND FREEZE TREATMENT

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ABSTRACT

In order to improve the permeability of *Cunninghamia lanceolata,* the weight gain rate of *C. lanceolata* was taken as index. The effect of time, temperature and water content on the weight gain rate of impregnated wood was analyzed by frozen and steaming treatment. By comparing the weight gain rate under different modification methods, the optimal modification process was determined. The results indicate that the optimum parameters of *C. lanceolata* were saturated water content (-25°C and 8 h) at this time, the three-day gain rate of silica sol impregnated at normal temperature and pressure was 15.058%. After *C. lanceolata* is pre-treated by superheated steam, the weight gain rate of *C. lanceolata*, which in oven-dried specimen (120°C and 3 h) contents was the highest, at this time, the three-day gain rate of silica sol impregnated at normal temperature and pressure was 15.291%. By comparing the results of pre-freezing with the results of superheated steam treatment of *C. lanceolata*, the latter will increase the permeability of *C. lanceolata* better. Therefore superheated steam treatment should be chosen as an effective method for the pretreatment.

KEYWORDS: *Cunninghamia lanceolata,* fast-growing, freezing treatment, modification treatment, ANOVA, optimization.

INTRODUCTION

The traditional natural trees, as the main material of furniture production, have a long growth cycle while the market demand for furniture is increasing, causing the situation of shortage of supply gradually (Fahrenhorst and Altan 1992). Start with the source of materials and improve the performance of quick-growing materials. let it can meet the demand of furniture production, so as to improve the capacity of qualified wood and alleviate the problem of insufficient wood supply (Pittau et al. 2018).

Based on literature review, it was found that permeability is an important index to measure wood properties, so improving the permeability of wood was the key to improve the effect of wood modification (Zlem et al. 2018, Emaminasab et al. 2015). In order to improve the permeability of wood, the number and size of the micropores of the effective pore membrane, the content and distribution of amorphous material deposited in the inner wall and cell wall, and the content and distribution of resin in the cell cavity were the main factors affecting the permeability of wood (Bao 1992). As two commonly used methods of wood treatment, freezing treatment and steam treatment have a significant effect on improving the permeability of wood (Seo et al. 2015, Ogawa et al. 2016).

The principle of pre-freezing treatment on improving wood permeability is that The water in the saturated wood is frozen into ice. When the volume of water molecules increases, the cell wall and pore wall are squeezed, and part of the pore membrane is destroyed, which expands the pores of wood to a certain extent, so as to improve the permeability of wood (Lan et al. 2016, Lisiewska et al. 2007). In addition, according to the research, the pre-freezing treatment can also migrate part of the wood extracts to the cell wall, which can not only fill the cell wall, but also improve the elasticity of the cell wall, so as to improve the shrinkage phenomenon of wood during drying. (Abd-Elhady 2014, Tomas et al. 2013, Zhang et al. 2011).

Steam treatment improves wood permeability, as well. High temperature steam in steam pretreatment produces persistent pressure on wood, which destroys the pore film on the cell wall inside the wood, thus allowing liquid molecules to pass more smoothly through the pores and enter into the cell space and cell cavity, thus increasing the permeability of wood (Awoyemi et al. 2010, Missio et al. 2016). In addition, the free hydroxyl groups of cellulose and hemicellulose in the wood react to such reactions as cross linking during steam treatment heating, which results in a reduction in the number of hydrophilic groups, thus reducing the moisture absorption of wood and enhancing the dimensional stability of wood, so the moisture content uniformity of wood was higher and the dry shrinkage ratio was lower after steam treatment (Poonia et al. 2015, Bao and Zhou 2017).

It was understood that there are few reports on the effects of freezing and steam treatment on the permeability of *Cunninghamia lanceolata*. Steam treatment and pre freezing treatment can expand some internal pores of wood, improve the permeability of wood, improve the performance of fast-growing wood and relieve the pressure of wood energy shortage (Cortez and Roberto 2012, Tao 2006).

Thus, in this work, improving the permeability of *C. lanceolata* was in focus. Through the freezing and steam treatment of *C. lanceolata*, analyzes the impregnation weight gain rate under different parameters, and clarifies the optimal modification parameters of *C. lanceolata*, in order to provide theoretical guidance for advanced wood improvement technology.

MATERIAL AND METHODS

Test materials and equipment

Samples of fast-growing *Cunninghamia lanceolata* 20 × 20 × 100 mm (T × R × L) were used in this study. In each group, 8 repeated tests were arranged, that is, 8 pieces of test materials were tested under the same conditions, and the average value was finally taken. The equipment (Vlosky and Shupe 2004, Zhu et al. 2020) used in the test is shown in Tab. 1.

Tab. 1: Laboratory equipment.

Name of equipment	Model	Manufacturers
Electric blast dryer	DHG-9245A Type	Shanghai - Heng Scientific Instruments Co., Ltd.
Electronic balance (0.001g)	EL-3KJ Type	Changzhou Tianzhiping Instrument Equipment Co., Ltd.
Refrigerators	BC-101KT1Type	Guangdong Rongsheng Electric Co., Ltd.

Steam treatment test design

The three factors of temperature, time and moisture content were taken as the main experimental research parameters (Tab. 2). The orthogonal test of three levels was carried out (Tab. 3). Each group carries out eight repeated tests, and the final result takes its average value.

Tab. 2: Table of variable factors for ordinary superheated pre-stemming.

Level	Temperature (°C)	The moisture content (%)	Time (h)
1	100	Wet specimen	1
2	120	Oven-dried specimen	2
3	140	Air-dried specimen	3

Tab. 3: The weight gain rate of impregnation of untreated and superheated pre-steaming treatment C. lanceolata.

N°.	Temperature (%)	Moisture content (%)	Time (h)	WPG (%)	The improve of WPG (%)
1	100	Wet specimen	1	13.321	5.87
2	100	Oven-dried specimen	2	12.863	2.23
3	100	Air-dried specimen	3	15.072	19.78
4	120	Wet specimen	2	14.832	17.87
5	120	Oven-dried specimen	3	15.291	21.52
6	120	Air-dried specimen	1	14.530	15.47
7	140	Wet specimen	3	13.531	7.53
8	140	Oven-dried specimen	1	11.963	-4.93
9	140	Air-dried specimen	2	12.642	0.47
Untreated	——	——	——	12.583	——

The *C. lanceolata* with the same size and specification have been Pre-frozen treated in the orthogonal experiment, The process was as follows: *(1)* The oven-dried specimen was obtained by drying *C. lanceolata*; The air-dried specimen with moisture content about 15% was obtained under relative humidity 65 ± 3%, 20 ± 2°C; Soak *C. lanceolata* to saturated water content to get wet specimen. *(2)* The wood with different moisture content was placed in the steaming environment at a certain temperature for a period of time, and the relative humidity was maintained at 100%. *(3)* The steamed wood was placed at room temperature for a period of time until it is at normal temperature. *(4)* Dry the steam-treated wood until it was fully dry and wait for impregnation. *(5)* The steam-treated wood was impregnated at normal temperature and pressure. Combined with the impregnated condition of the fast-growing wood at normal temperature and pressure and the pre-freezing treatment test, the same impregnating time was used for three days and nights in the test. Then the weight gain rate of the steam-treated wood impregnated with silica sol was compared. *(6)* The weight gain rate of the impregnated wood reflects the permeability of the pre-treated wood to some extent. Therefore, the optimum steam treatment scheme and test parameters can be concluded by comparing the weight gain rate of the pretreated material with untreated material and comparing the increase of the permeability of steamed materials

Freeze treatment test design

Three factors of temperature, time and moisture content, were taken as the main experimental research parameters (Tab. 4). The orthogonal test of three factors and three levels was carried out (Tab. 5). Each group carries out eight repeated tests, and then the average value was calculated as the final result (Dong et al. 2018).

Tab. 4: Table of variable factors for pre-freezing process.

Level	Temperature (°C)	Time (h)	Moisture content (%)
1	-10	8	Oven-dried specimen
2	-15	12	Air-dried specimen
3	-20	16	Wet specimen

Tab. 5: The weight gain rate of impregnation and untreated of pre-freezing C. lanceolata.

N°.	Temperature (°C)	Time (h)	Moisture content (%)	WPG (%)	The improve of WPG (%)
1	-10	8	Oven-dried specimen	13.670	8.64
2	-10	12	Air-dried specimen	10.085	-19.85
3	-10	16	Wet specimen	13.204	4.93
4	-15	8	Air-dried specimen	7.429	-40.96
5	-15	12	Wet specimen	11.009	-12.51
6	-15	16	Oven-dried specimen	8.594	-31.70
7	-20	8	Wet specimen	14.758	17.28
8	-20	12	Oven-dried specimen	14.481	15.08
9	-20	16	Air-dried specimen	12.007	-4.58
Untreated	-	-	-	12.583	-

The *C. lanceolata* with the same size and specification have been Pre-frozen treated in the orthogonal experiment, The process was as follows: *(1)* The oven-dried specimen was obtained by drying *C. lanceolata*; The air-dried specimen with moisture content about 15% was obtained under relative humidity 65 ± 3%, 20 ± 2°C; Soak *C. lanceolata* to saturated water content to get wet specimen. *(2)* The wood with different moisture content was placed in the refrigerator with temperature of -10°C, -15°C and -20°C for some time. *(3)* The frozen wood was placed at room temperature for a period of time until it thawed. *(4)* Dry the pre-frozen wood until it was fully dry and wait for impregnation; *(5)* The wood that have been pre-treated was impregnated at normal temperature and pressure. Because the change of weight gain rate of two kinds of quick-growing materials was observed before, in order to compare the weight gain rate of each group of test materials after impregnation of silica sol, the same impregnation time was used for three days and nights (72 h). *(6)* The weight gain rate of the impregnated material reflects the permeability of the pre-treated *C. lanceolata* to some extent. Therefore, compare the weight gain rate of the pre-treated material with untreated material and compare the increase of the permeability of pre-frozen materials to find the optimum pre-frozen method and experiment parameter.

RESULTS AND DISCUSSION

Effect of steam treatment on the permeability

Experimental results of steam treatment on weight gain rate

According to the data in Tab. 4, the weight gain rate of *C. lanceolata* impregnated at normal temperature and pressure after three days without pretreatment was 12.583%, and the weight gain rate of *C. lanceolata* impregnated silica sol after three days after superheated steam treatment ranged from 11.963% to 15.291%, and the weight gain rate was - 4.93 - 21.52% over untreated wood. By comparing the weight gain rate between the superheated steam pretreatment and the untreated material, it was found that the permeability of most *C. lanceolata* was enhanced after superheated steam treatment, and a few was decreased. The permeability of *C. lanceolata* was best in the 5th group. That is, specimen was oven-dried, steam temperature was 120°C and steam treatment time was 3 h.

Range analysis (Cadambi et al. 2012) of weight gain rate of C. lanceolata treated by steam

Tab. 6 is an intuitive analysis of the weight gain rate of *C. lanceolata* impregnated material after superheated steam pretreatment. According to the data in the table, the range value, which mean the influence of superheated steam pretreatment on *C. lanceolata*, is temperature > time > moisture content. This shows that the temperature have the greatest influence on the permeability of *C. lanceolata,* followed by time, and the effect of moisture content was the least. With the increase of temperature, the weight gain rate of *C. lanceolata* impregnated material after superheated steam pretreatment increased first and then decreased, and the effect was the best when the temperature was 120°C. With the increase of treatment time, the weight gain rate of *C. lanceolata* impregnated material after superheated steam pretreatment increased first and then decreased, and the effect was the best when the treatment time was 3 h. The weight gain rate of material superheated steam treatment manifested as air-dried specimen > wet specimen > oven-dried specimen. When the temperature was 120°C ,the time was 3 h and moisture content was air dried condition the weight gain rate of *C. lanceolata* impregnated material after superheated steam pretreatment was the best, that is, the permeability of *C. lanceolata* after superheated steam pretreatment was the best.

Tab. 6: Maximum analysis of variables on weight gain rate of impregnation of superheated pre-steanming treatment C. lanceolata.

Properties	Level	Temperature (°C)	Moisture content (%)	Time (h)
WPG (%)	Mean1	13.752	13.895	13.271
	Mean2	14.884	13.372	13.446
	Mean3	12.712	14.081	14.631
	Range	2.172	0.709	1.360

ANOVA analysis (Didier et al. 2002) of weight gain rate of C. lanceolata steam treatment

Tab. 7 is the variance analysis and significance test of the influence of variable factors on the weight gain rate of *C. lanceolata* superheated steam impregnated material. From the data in the table, we can see that temperature and time have a significant effect on the weight gain rate of *C. lanceolata* after superheated steam pretreatment, and the moisture content during superheated steam pretreatment has little effect on the permeability of *C. lanceolata* after pretreatment.

Tab. 7: Variance analysis and significance test of designed parameters on WPG of impregnation of superheated pre-steaming treatment C. lanceolata.

Factor	Sum of squares	DF	F value	F critical value	Pr > F
Temperature	7.083	2	46.294	19	*
Water content	0.810	2	5.294	19	
Time	3.286	2	21.477	19	*
Error	0.15	2			

$\alpha = 0.05$.

Optimization and verification of steam treatment process of C. lanceolata

According to the results of orthogonal test of steam treatment of *C. lanceolate* (Alizadeh and Fujita 2010), the optimum parameters of steam treatment of *C. lanceolata* were 120°C, 3 h and air-dried. The weight gain rate of *C. lanceolata* impregnated material after superheated steam pretreatment was best in this condition. Among them, only moisture content had no significant effect on the weight gain rate of *C. lanceolata*. In the actual utilization of wood, the moisture content of wood was close to the air-dried, considering the actual use of wood and the cost of drying treatment (Kadam et al. 2011), under the condition that the temperature was 120°C, the time was 3 h and the moisture content was air-dried, the permeability of *C. lanceolata* was the best after superheated steam pretreatment.

Effects of freezing treatment on the permeability

Experimental results of freeze-treated C. lanceolata

Tab. 5 shows the weight gain rate of *C. lanceolata* after pretreatment and material impregnated with silica sol. From the data in the table, the weight gain rate of *C. lanceolata* without pretreatment was 12.583% after three days of normal temperature and pressure impregnation. The weight gain rate of *C. lanceolata* impregnated with silica sol after three days after pretreatment ranged from 7.429% to 14.758%, and the weight gain rate was in range

-40.96% to 17.28% over untreated wood. By comparing the weight gain rate of *C. lanceolata* impregnated with silica gel after pre-freezing treatment, it was found that the permeability of some *C. lanceolata* was enhanced after pre-freezing treatment, and the some was decreased, and the group 7 under the condition that *C. lanceolata* was wet specimen, the pre-freezing temperature of -20°C and pre-freezing of 8 h resulted in the best permeability of *C. lanceolata.*

Range analysis of weight gain rate of C. lanceolata frozen treatment

Tab. 8 is an intuitive analysis of the weight gain rate of *C. lanceolata* pretreated impregnated materials after pre-freezing treatment. According to the data in the table, the range values of three factors of pre-freezing treatment were: temperature > moisture content > time, so the influence of three factors in the pre-freezing treatment test was: temperature > moisture content > time. With the decrease of temperature, the weight gain rate of *C. lanceolata* after pre-freezing treatment first decreased and then increased. With the increase of pre-freezing treatment time, the weight gain rate of *C. lanceolata* gradually decreased, the weight gain rate after pre-freezing treatment manifested as wet specimen > oven-dried specimen> air-dried specimen. The weight gain rate of *C. lanceolata* impregnated material was the highest when the temperature was -20°C, treatment time was 8 h and under the condition of wet specimen, that is, the permeability of *C. lanceolata* was the best.

Tab. 8: Maximum analysis of variables on weight gain rate of impregnation of pre-freezing C. lanceolata.

Properties	Level	Temperature (°C)	Time (h)	Moisture content (%)
WPG（%）	Mean1	12.320	11.952	12.248
	Mean2	9.011	11.858	9.840
	Mean3	13.749	11.268	12.990
	Range	4.738	0.684	3.150

ANOVA of weight gain rate of C. lanceolata treated by freezing

Tab. 9 is the variance analysis of the weight gain rate of *C. lanceolata* impregnated materials after pre-freezing treatment. From the variance and significance analysis of factors, it can be seen that the temperature of pre-freezing treatment has a significant effect on the weight gain rate of *C. lanceolata* impregnated silica sol, and the moisture content of *C. lanceolata* also has an effect on the weight gain rate of *C. lanceolata* impregnated after pre-freezing treatment, that is, the moisture content and pre-freezing treatment temperature of *C. lanceolata* have an effect on improving the permeability of *C. lanceolata*, and the time of pre-freezing treatment had little effect on the improvement of *C. lanceolata* permeability.

Tab. 9: Variance analysis and significance test of designed parameters on WPG of impregnation of pre-freezing C. lanceolata.

Factor	Sum of squares	DF	F value	F critical value	Pr > F
Temperature	35.440	2	20.136	19	*
Time	0.825	2	0.469	19	
Water content	16.272	2	9.245	19	
Error	1.76	2			

α =0.05.

Optimization and verification of freezing treatment process of C. lanceolata

In the orthogonal test, the pre-freezing results of *C. lanceolata* under wet material condition are the best, all the specimens of *C. lanceolata* in single factor test were saturated water content state. According to the results of orthogonal test, the best pretreatment time of pre-freezing treatment was 8 hours. Since the temperature was the most significant to the increment rate of *C. lanceolata*, the treatment temperature in the test was selected as: -25°C, -30°C, -35°C, -40°C, and then the pretreated material was impregnated with silica sol at normal temperature and pressure for three days, and each group was repeated eight tests, and the average value was calculated as the final result to compare its weight gain rate.

Tab. 10 shows the change of weight gain rate of *C. lanceolata* pretreated under normal temperature and pressure after dipping silica sol. Through comparing the results of pretreated specimens at different temperatures, we can see that in the range from -40°C to -25°C, the change of weight gain rate of *C. lanceolata* decreases gradually, and the maximum value of 15.058% was obtained when the pretreated temperature was -25°C, that is, the penetration of *C. lanceolata* was the best when the pretreated temperature was -25°C, and the impregnating effect of the modifier is the best.

Based on the results of orthogonal test and single factor test of *C. lanceolata* pre-freezing treatment. The optimum parameters of pre-freezing treatment were as follows: moisture content was saturated, temperature was -25°C, time was 8 h, that is, when the *C. lanceolata* was under saturated condition, treatment temperature was -25°C and treatment time was 8 h, the best penetration and impregnation were obtained.

Tab. 10: Effect of pre-freezing temperature on weight gain rate of C. lanceolata.

No.	Temperature (°C)	WPG (%)
1	-25	15.058
2	-30	14.154
3	-35	13.649
4	-40	13.458

CONCLUSIONS

The impregnation performance of the modified *Cunninghamia lanceolata* were studied by orthogonal test by freezing and steam treatment. The following conclusions could be drawn from this work. The changes of weight gain of *C. lanceolata* under different parameters were studied by freezing and steam treatment, and the optimum steam modification technology was defined. The results from this work showed that when the water content was saturated, temperature was -25°C and time was 8 h, the optimum parameters of pre-freezing treatment of *C. lanceolata* was obtained, at this time, the three-day gain rate of silica sol impregnated at normal temperature and pressure was 15.058%. When the temperature was 120°C, treatment time was 3 h and the moisture content was air-dried specimen, the weight gain rate of *C. lanceolata* impregnated with superheated steam was the highest, at this time, the three-day gain rate of silica sol impregnated at normal temperature and pressure was 15.291%. Compared with the results of pre-freezing and superheated steam treatment of *C. lanceolata*, it was found that the permeability of *C. lanceolata* increased significantly after superheated steam pretreatment, therefore, the superheated steam treatment should be chosen.

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