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OPTIMIZATION OF THE MANUFACTURING OF *METASEQUOIA*-BASED THREE-LAYER STRUCTURE PARQUET FLOORING BY A RESPONSE SURFACE METHODOLOGY

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ABSTRACT

On the basis of a single-factor experiment, a mathematical model was established by the response surface analysis method based on the Box-Behnken experimental design principle. The effects of three factors, including hot-pressing temperature, hot-pressing time, and hot-pressing pressure, and their interactions on the modulus of rupture (MOR) of *Metasequoia*-based three-layered structure parquet flooring were studied. The results show that the quadratic polynomial model in the regression equation is significant, and the correlation between the value predicted by the model and the experimental value is 91.17%. The optimized best hot-pressing process parameters are determined to be as follows: hot-pressing temperature of 96.03°C, hot-pressing time of 6.70 min, and hot-pressing pressure of 8 kg·cm^{-2}. Under these conditions, the best MOR are obtained, reaching a value of 102.05 MPa. The theoretically predicted value is in good agreement with the experimental results.

KEYWORDS: Dawn redwood, Box–Behnken design, hot-pressing technology, modulus of rupture.

INTRODUCTION

Metasequoia glyptostroboides Hu et Cheng (dawn redwood) is a deciduous tree and a member of the Sequoioideae subfamily and is often described as a living fossil (Bartholomew et al. 1983, Ma and Shao 2003). Since the 1940s, *Metasequoia glyptostroboides* has been discovered and widely introduced around the world. In many areas, *Metasequoia* has become forests and is used for timber and has a wide range of adaptability (Ma 2007, Li et al. 2012). At present, *Metasequoia glyptostroboides* has become an important timber tree species, coastal and farmland shelter forest tree species, urban greening and landscape tree species in China (Sanmartín 2012). In many areas, *Metasequoias* have become forests and are used for timber and have a wide range of adaptability (Li et al. 2005). Conservation, development and utilization are the three most important themes for research into *Metasequoia glyptostroboides* today. However, the development and application of *Metasequoia glyptostroboides* is restricted due to it having the characteristics of a light and soft material, loose structure and low strength (Nelson 2004). There have been many studies on the high-quality utilization of *Metasequoia glyptostroboides* for the processing and utilization of wood boards, sheets for furniture, packing box material, forestry chemicals, activated carbon, liquefaction of wood and other aspects (Honglu and Tiejun 2006, Xie and Shi 2010, Wang et al. 2019). Various parts of *Metasequoia glyptostroboides* have extremely high medicinal value (Juvik et al. 2016), and some studies have shown that the extract of *Metasequoia glyptostroboides* subjected to a column chromatographic analysis can result in the isolation of an abietane-type diterpenoid, terpenoids, flavonoids, and taxodone (Dong et al. 2011, Tu et al. 2019). Furthermore, taxodone can show potential antibacterial effects as diameters of zones of inhibition against foodborne pathogenic bacteria (Bajpai and Kang 2010). Research has investigated the anticancer effects of organic extracts derived from the floral cones of *Metasequoia glyptostroboides*. Clarified *Metasequoia glyptostroboides* potentiates anticancer effects against cervical cancer via the intrinsic apoptosis pathway (Lee et al. 2021). Scholars have studied the high-value utilization of *Metasequoia glyptostroboides* from various aspects.

With the increasing expansion of China's wood flooring market and the relative scarcity of wood resources, it is necessary to develop low-quality wood as much as possible to alleviate the shortage of wood resources and achieve the purpose of high-quality utilization of low-quality wood (Hu et al. 2018, Li et al. 2018). Parquet flooring is a kind of floor covering that consists of small rectangular blocks of wood arranged in a pattern, which has high dimensional stability because it overcomes the shortcomings of veneer, warping and cracking in solid wood flooring (Blanchet et al. 2003, Blumer et al. 2009).

In this study, *Metasequoia glyptostroboides* was used as a material for the core part of a parquet block. The manufacturing of a *Metasequoia*-based three-layer structure parquet flooring (MPF) was optimized by response surface methodology. In a previous study, multilayer plywood was used as the core layer end to meet the performance requirements of the floor groove and tenon. *Xylosma racemosa* and *Populus tomentosa* was used as the top and bottom veneers, respectively. Finally, several veneers were laid up and hot-pressed to prepare MPF (Zhang et al. 2021a). Compared with the *Populus*-based multilayer parquet flooring and fir-based three-layer parquet flooring available on the market, the MPF designed with such a structure has the advantages of relatively low cost, low density, strong sound absorption (Wu et al. 2016), and comfortable foot feel. However, the problem of low strength also needs to be addressed. Therefore, to improve the mechanical properties of the product, response surface methodology was used to optimize the hot pressing process for MPF, and MPF was prepared with an excellent modulus of rupture (MOR) performance.

MATERIALS AND METHODS

Experimental material and equipment

The adhesive was composed of modified urea-formaldehyde resin glue with a solid content of 70% and a viscosity of 230 mPa·s. The surface veneer was *Xylosma racemosa*, with a thickness of 4 mm and a moisture content of 8 - 9%; the core veneer was *Metasequoia glyptostroboides*, with a thickness of 9 mm and a moisture content of 9 - 10%; the bottom veneer was *Populus tomentosa* Carr., with a thickness of 2 mm and a moisture content of approximately 7%. All three types of wood were purchased from the forest products market in Suqian City, Jiangsu Province, China.

The hot-pressing equipment (model: BY214×8/60(8) ZRC, total pressure: 6000 kN, hot press veneer format: 1370 mm × 2700 mm) was manufactured by Shanghai Qiulin Machinery Co., Ltd. A universal mechanical testing machine (model: MTS/CMT) produced by Meister Testing Machine Co., Ltd. was used, together with a Vernier calliper, a thickness gauge (12 mm), glue-making equipment, and an electronic balance (accurate to 0.01 g).

Technological process

Urea-formaldehyde resin glue was evenly applied to the surface of the veneer with a gluing roller. The amount of glue applied to one side of the veneer was approximately 200 $g \cdot m^{-2}$ (painted twice).

The length, width and thickness of the *Metasequoia glyptostroboides* core veneer used in this test was 800 mm, 90 mm and 9 mm, respectively. A 4 mm thick layer of *Xylosma racemosa* was used for the surface veneer and a 2 mm thick layer of *Populus tomentosa* was used for the bottom veneer with plywood used to form part of the floor tongue and groove joint structure. The above veneers were prepared by lay-up and hot pressing to obtain MPF with product specifications of length (900 mm), width (125 mm), and thickness (12 mm), which were stored for 7 days before testing.

Single factor experiment

Taking the three factors of hot-pressing temperature, hot-pressing time, and hot-pressing pressure as variables, six gradient changes were set, and the MOR for MPF was used as the evaluation index. The single-factor test level is shown in Tab. 1.

Tab. 1: Factors and levels in the single-factor experiment.

No.	1	2	3	4	5	6
Hot pressing temperature *A* (°C)	90	95	100	105	110	115
Hot pressing time *B* (min)	5.5	6	6.5	7	7.5	8
Hot pressing pressure *C* ($kg \cdot cm^{-2}$)	7	8	9	10	11	1.2

Response surface analysis test

According to the single factor test results, the response surface method Box-Behnken model test design and data analysis were carried out (Wang et al. 2017). The experimental design used the following parameters: MOR as the response value, design hot-pressing temperature *A*, hot-pressing time *B*, and hot-pressing pressure *C*, with the low, medium, and high levels of the independent variable represented by the values -1, 0, and 1, respectively. The experimental design factors and levels are shown in Tab. 2.

Tab. 2: Factors and levels used in the response surface design.

Factors	Level		
	-1	0	1
Hot pressing temperature *A* (°C)	95	100	105
Hot pressing time *B* (min)	6.0	6.5	7.0
Hot pressing pressure *C* ($kg \cdot cm^{-2}$)	8	9	10

Test method

The detection and analysis of the size deviation and physical and chemical indicators for the MPF were based on Chinese national standards GB/T 18103: 2013 (Parquet), GB 18580: 2017 (Indoor decorating and refurbishment materials, limit for formaldehyde emission from wood-based panels and finishing products) and GB/T 17657: 2013 (Test methods for evaluating the properties of wood-based panels and surface decorated wood-based panels).

RESULTS AND DISCUSSION

Effect of hot pressing temperature on the MOR

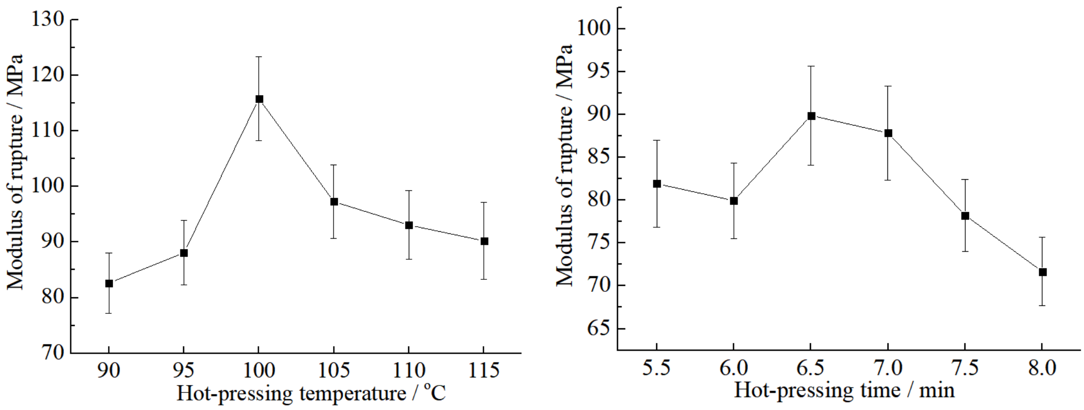
The proper hot pressing process could ensure that the thickness, adhesive curing and performance of the parquet meet the design requirements and minimize the cost (Thoemen and Humphrey 2006). The hot-pressing conditions were set as follows: hot-pressing time of 6.5 min, and hot-pressing pressure of 9 $kg \cdot cm^{-2}$. The effect of different hot-pressing temperatures on the MOR is shown in Fig. 1a. In the temperature range of 90 - 100°C, the MOR of the product gradually increases with increasing hot pressing temperature. The main reason for this is that after the combined veneer enters the hot press, the press transfers heat to the *Xylosma racemosa* of the surface veneer. Then, the surface veneer further transfers the heat to the core layer, resulting in an increase in the overall temperature, and the heat transfer of the veneer is enhanced, which promotes the full curing of the modified urea-formaldehyde resin adhesive (Okuda et al. 2006, Kumar et al. 2015). However, the temperature should not be too high. When the hot-pressing temperature ranges from 100°C to 115°C, the MOR for the product gradually decreases. This is because with a further increase in the hot pressing temperature, the adhesive becomes brittle due to excessive curing, and the physical and mechanical properties of the product decrease. After comprehensive consideration, a hot-pressing temperature of 100°C was chosen to obtain a product with better density and strength.

Effect of hot pressing time on the MOR

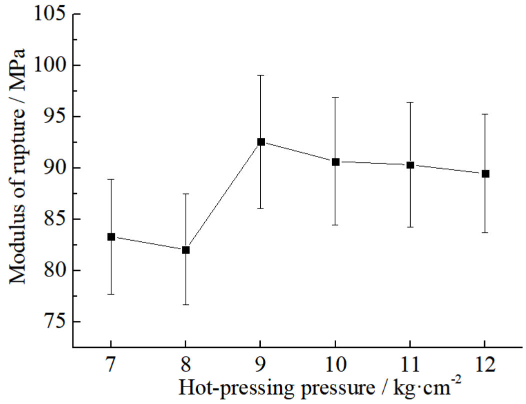
According to the experimental data, the MOR line chart for the pressed products obtained using different hot-pressing times was drawn. Fig. 1b shows that for a hot pressing time of 5.5 - 6.5 min, the MOR of the floor first is decreased and then increased. For a hot-pressing time of 6.5 - 7.5 min, the MOR is decreased with increasing hot-pressing time. The main reason for this is that when the hot-pressing time of the combined veneer is 5.5 - 6.5 min, the modified urea-formaldehyde resin glue is gradually cured completely, and the MOR of the product reaches a maximum value at 6.5 min. However, the hot-pressing time should not be too long. With an increase in the hot-pressing time to longer than 6.5 min, the compression rate of the veneer can increase, the surface layer of the combined veneer will be carbonized, and the curing degree of the urea-formaldehyde resin glue will be too high, resulting in a decrease in the strength of the surface layer. The brittleness of the modified urea-formaldehyde resin adhesive layer increases, which is prone to cracking. The MOR of the product mainly depends on the strength of the surface layer, which can lead to a decline in the mechanical properties of the product due to the brittleness of the adhesive layer (Hoong and Paridah 2013, Li et al. 2019). For a hot-pressing time of 6.5 min, the MOR performance index of the product exceeds the requirements of GB/T 18103: 2013 (Parquet). To shorten the hot-pressing cycle and improve the production efficiency, an optimum hot-pressing time of 6.5 min was chosen under the test conditions.

Effect of hot-pressing pressure on the MOR

Fig. 1c shows that under the action of a certain hot-pressing pressure, the combined veneer removes the internal air, the contact between the layers increases, and the gap in the adhesive layer and the thickness of the combined veneer decrease. The migration of glue layer molecules under the action of an external force enables the adhesive to more effectively impregnate the surface of *Metasequoia glyptostroboides* cells and fill the holes and depressions inside the combined veneer. Finally, the bonding strength inside the combined veneer is improved, and the MOR performance of the floor is improved (Pablo et al. 2001). However, when the hot-pressing pressure exceeds 9 kg·cm^{-2}, the MOR of the floor gradually decreases. This can be attributed to the migration of glue between the layers with the continuous increase in the hot-pressing pressure, resulting in a local lack of glue between the surface and core veneers and between the core and bottom veneers. This is not conducive to gluing and the discharge of water vapour in the combined veneer, resulting in a decline in the MOR performance of the product. At the same time, excessive hot-pressing pressure will not only result in a large load on the press but also increase the compression rate and glue penetration rate of the combined veneer and eventually lead to warping and deformation of the product, so the hot-pressing pressure should be properly selected. According to the single factor test results, the hot pressing pressure that can be used in the production of MPF should be set to 9 kg·cm^{-2}.



(a) (b)



(c)

Fig. 1: MOR for products prepared using different hot-pressing factors: a) hot-pressing temperature, b) hot-pressing time, c) hot-pressing pressure.

Response surface design results

In this experiment, the response surface test method was used to conduct the test (Islam et al. 2012). The hot-pressing process parameters for the MPF are discussed with the hot-pressing temperature, hot-pressing time and hot-pressing pressure as influencing factors. The specific test arrangement is shown in Tab. 3:

Tab. 3: Box–Behnken experimental design and results.

No.	Hot-pressing temperature *A* (°C)	Hot-pressing time *B* (min)	Hot-pressing pressure *C* ($kg·cm^{-2}$)	Response value Y_1	
				Test value (MPa)	Predictive value (MPa)
1	95	6.0	9	85.1	82.74
2	95	6.5	8	87.8	93.24
3	100	6.5	9	102.4	89.36
4	100	6.5	9	99.3	92.06
5	100	7.0	10	91.7	87.79
6	100	6.0	10	88.2	100.69
7	100	6.5	9	100.6	91.11
8	95	6.5	10	87.8	91.41
9	100	6.0	8	87.2	89.58
10	100	6.5	9	100.7	92.95
11	105	6.0	9	92.3	87.25
12	95	7.0	9	90.3	89.33
13	105	6.5	8	104.0	101.28
14	100	6.5	9	103.4	101.28
15	105	6.5	10	91.4	101.28
16	105	7.0	9	89.7	101.28
17	100	7.0	8	92.0	101.28

Regression model analysis

The regression equation obtained after fitting and analysing the variance of the data given in Tab. 3 using Design-Expert software is expressed as follows: $Y_1 = 101.28 + 3.30A + 1.36B - 1.49C - 1.95AB - 3.15AC - 0.32BC - 4.48A^2 - 7.45B^2 - 4.05C^2$ (Myers et al. 2004, Zhang et al. 2021b). The results from variance analysis show that the Model F value is 8.029877, and the model significance level P value is 0.0059, indicating that the quadratic polynomial model has good significance. Lack of fit analysis shows that the P value for the lack of fit is equal to 0.0565 (>0.05), and the model is not significant, indicating that the quadratic equation shows a better fit to the true level and that the experimental error is small. This model can be used to analyse and predict the MOR for MPF. The correlation between the predictive value and test value for the model is 0.9117, and the R^2 correction value is 0.7982. This model can explain 91.17% of the changes observed in the response value in the experiment. This shows that the model has a good degree of fit, the experimental error is small, and that the model has a good degree of fit. This equation can be used to well predict and explain the law for the change in the product's MOR for each parameter.

Tab. 4: Variance analysis using the regression model.

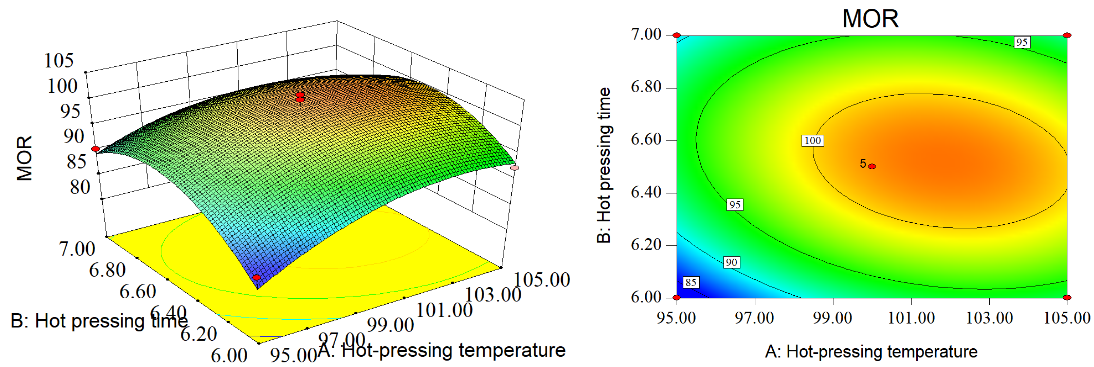
Source	Sum of squares	Degree of freed. of var.	Mean square	F value	Prob > F	Source
Model	603.1407	9	67.01563	8.029877	0.0059	**
A	87.12	1	87.12	10.4388	0.0144	*
B	14.85125	1	14.85125	1.779491	0.224	
C	17.70125	1	17.70125	2.120981	0.1886	
AB	15.21	1	15.21	1.822477	0.219	
AC	39.69	1	39.69	4.755694	0.0656	
BC	0.4225	1	0.4225	0.050624	0.8284	
A^2	84.41266	1	84.41266	10.11441	0.0155	*
B^2	233.8516	1	233.8516	28.02032	0.0011	**
C^2	69.14845	1	69.14845	8.285433	0.0237	*
Residual	58.4205	7	8.345786			
Lack of fit	47.9525	3	15.98417	6.107821	0.0565	
Pure error	10.468	4	2.617			
Cor to al	661.5612	16				
R^2	0.9117					

Note: *Means significant at $P < 0.05$, ** means extremely significant at $P < 0.01$.

B^2 in Tab. 4 reaches a very significant level ($P < 0.01$). Factors A, A^2 and C^2 reach a significant level ($P < 0.05$). Factors B, C, AB, AC, and BC are not significant. Within the scope of the test parameters, the influence of each factor on the results obtained from large to small follows the order of temperature > pressure > time.

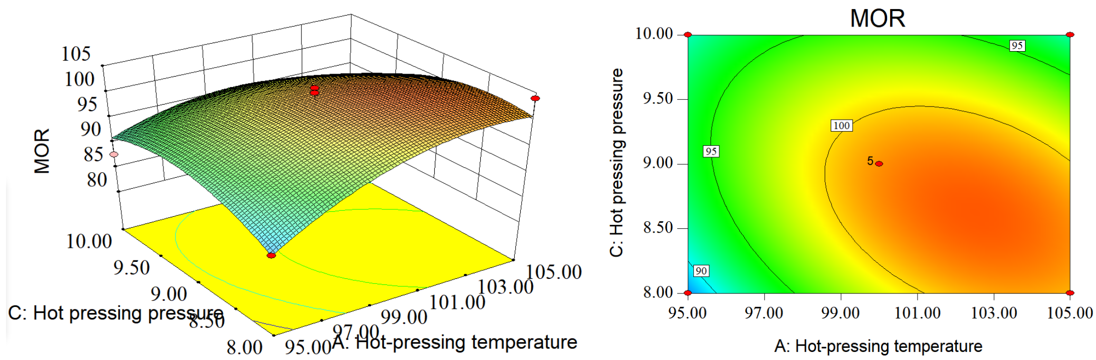
Response surface interaction analysis

To visually express the influence of the interaction of the two factors on the MOR of the product, the response surface and contour lines for the interaction of the hot-pressing temperature *A* and the hot-pressing time *B* on the MOR of MPF were drawn (Fig. 2). In the same way, the response surface and contour lines for the interaction between the hot-pressing temperature *A* and hot-pressing pressure *C* and the interaction of the hot-pressing time *B* with the hot-pressing pressure *C* on the MOR were drawn (Fig. 3, Fig. 4). Gul et al (2017) investigated the performance of MDF with respect to hot-pressing temperature and time. Hot pressing temperature was determined according to the performance of boards, type of glue, and production efficiency of hot press. As a general rule, a higher hot-pressing temperature difference will cause difficulty in production of thick boards and low hot-pressing temperature will make the adhesive difficult to cure. This research also follows this rule.



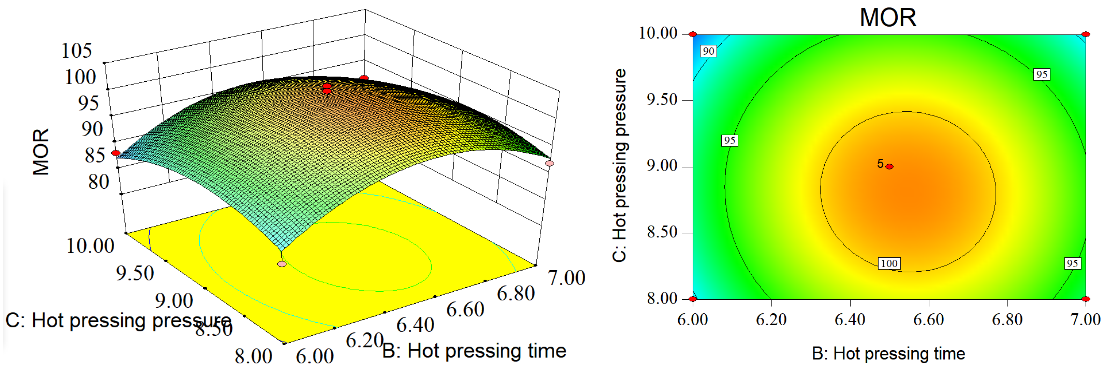
(a) (b)

Fig. 2: Response surface (a) and contours (b) for the effects of hot-pressing temperature and time.



(a) (b)

Fig. 3: Response surface (a) and contours (b) for the effects of hot-pressing temperature and pressure.



(a) (b)

Fig. 4: Response surface (a) and contours (b) for the effects of hot-pressing time and pressure.

Fig. 2 shows that the interaction of hot-pressing temperature and time has no significant effect on the MOR of MPF. When the hot-pressing pressure is a constant, with increasing hot-pressing temperature, the MOR first increases and then decreases. Under a certain hot-pressing time condition, the MOR first increases and then decreases with increasing hot-pressing pressure. Compared with the hot pressing time, the hot pressing temperature has a greater influence on the MOR. Fig. 3 shows that the interaction between the hot-pressing temperature and the hot-pressing pressure is not significant. The rate of change in the MOR with hot-pressing temperature is greater than the rate of change in the hot-pressing pressure (Song et al. 2018). The interaction of hot pressing time and hot pressing pressure does not have a significant effect on the MOR (Fig. 4), which results in circular contour lines. Under the same hot-pressing temperature conditions, with increasing hot-pressing pressure, the MOR is increased, reaching a maximum value for a hot-pressing pressure of 9 kg·cm^{-2}, and then shows a downwards trend.

Verification of the optimal conditions

Within the range of the test parameters, the optimal hot-pressing process conditions for predicting the MOR by solving the equation with Design Expert 11 software were as follows: hot-pressing temperature of 96.03°C, hot-pressing time of 6.70 min, and hot-pressing pressure of 8 kg·cm^{-2}. Under this condition, the MOR is determined to be 102.053 MPa. To verify the validity of the model, the optimal conditions were revised as follows: hot-pressing temperature of 96°C, hot-pressing time of 6.7 min, and hot-pressing pressure of 8 kg·cm^{-2}. The average value for the MOR for MPF obtained from three parallel verification tests is 100.6 MPa, which is close to the theoretically predicted value.

Dimensional deviation, physical and chemical performance testing

The dimensional deviation and physical and chemical properties of the MPF prepared under the optimal process conditions were as follows: right angle with $q_{max} \leq 0.2$ mm and edge straightness of ≤ 0.3 mm·m^{-1}; for the warpage, a width and length direction of $f_w \leq 0.20\%$ and $f_t \leq 1.00\%$, respectively; for the thickness deviation, an absolute value for the difference between the nominal thickness t_n and the average thickness t_a of ≤ 0.5 mm, and a difference between the maximum thickness t_{max} and minimum thickness t_{min} of ≤ 0.5 mm; average and maximum values for the open jointing of $\sigma_a \leq 0.15$ mm and $\sigma_{max} \leq 0.2$ mm; a modulus of elasticity (MOE) of 6544 MPa, and a free aldehyde emission of 0.112 mg·m^{-3}, all of which meet the requirements of relevant national standards. The physical performance of *Metasequoia*-based three-layered structure parquet flooring was similar to cork oak wood flooding (Knapic et al 2012).

CONCLUSIONS

The developed MPF not only realizes the goal of high-quality utilization of low-quality wood *Metasequoia glyptostroboides* but can also reduce production costs. The influence of the three elements of hot pressing on the MOR of MPF was investigated through a single factor test and response surface test, and the mathematical model of the response to each factor level was obtained by analysing the data. Then, the interaction between the response value and the factors was drawn out graphically to give a relationship diagram. The predicted and experimental values for the MOR model show a correlation of 91.17%, indicating a good fit to the model. The hot-pressing process for MPF was optimized by Box–Behnken experimental design, and the optimal process conditions are determined to be as follows: hot-pressing temperature of 96 °C, hot-pressing time of 6.7 min, and hot-pressing pressure of 8 kg·cm^{-2}. Three parallel verification tests were conducted, and the average MOR for the product is determined to be 100.6 MPa, which is similar to the theoretically predicted value.

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