Optimization of Preparation Process for Walnut Shell-based Wood Particleboard

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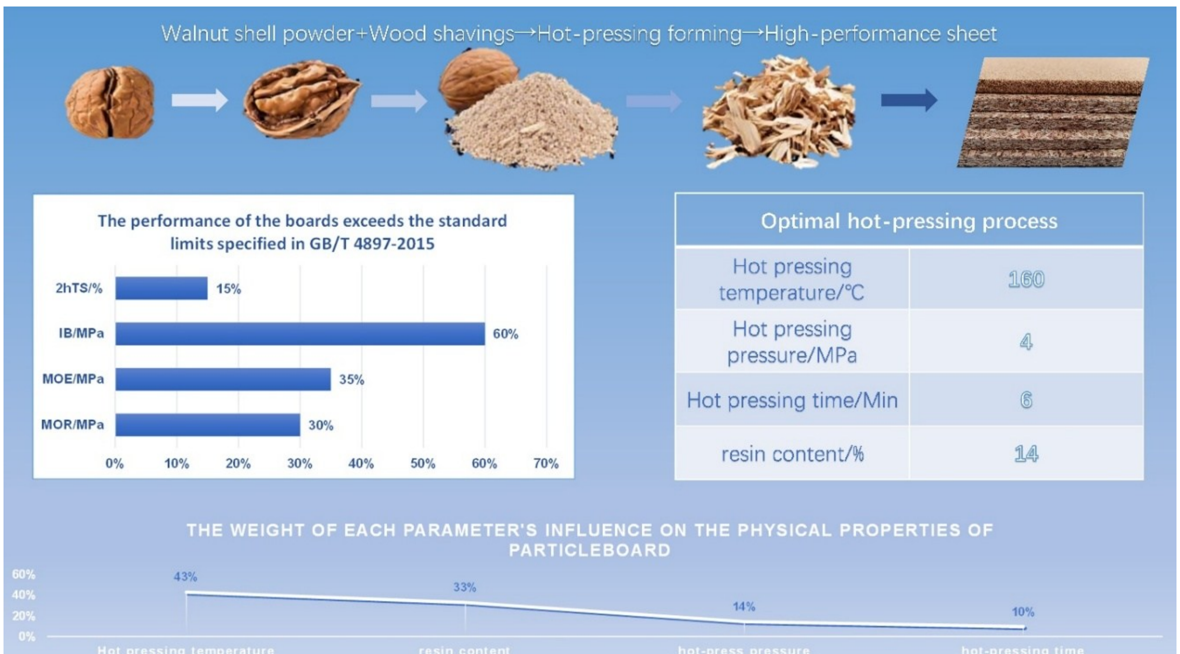
GRAPHICAL ABSTRACT



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Driven by the "double carbon" strategy, aiming at the potential of walnut shell, an agricultural and forestry waste, to replace wood in particleboard production, this study used walnut shell powder and wood shavings as raw materials, with melamine-modified urea-formaldehyde resin as the adhesive. A four-factor, three-level orthogonal experiment was conducted to investigate the effects of hot-pressing temperature, pressure, duration, and adhesive application level on the physical and mechanical properties of the panels. The optimal process conditions were 160°C, 4 MPA, 6 min, and a 14% adhesive application percentage. Under these conditions, the panel had a density of 0.70 g/cm³, and its modulus of rupture (MOR), modulus of elasticity (MOE), internal bonding strength (IB), and 2-hour thickness swelling (2hTS) all exceeded the limits specified in GB/T 4897 (2015) by 30% to 60%. The order of influence of each parameter was hot-pressing temperature > adhesive application level > hot-pressing pressure > hot-pressing duration. This study established for the first time the optimal process window for walnut shell-based particleboard, demonstrating that a 30% wood substitution can balance performance and resource conservation, providing technical support for the green and high-value utilization of agricultural and forestry wastes.

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Keywords: Walnut shell; Particleboard; Carbon neutralization; Process parameter optimization; Orthogonal experiments

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INTRODUCTION

Driven by the “double carbon” strategy, forest resources—as the largest terrestrial carbon sink—are facing unprecedented pressure (Zhang *et al.* 2022). Although the international community has set the goal of achieving carbon neutrality, the wood-based panel industry currently finds itself in a challenging situation. With the rapid increase in global demand for particleboard, excessive logging has caused timber prices to rise steadily, thereby undermining the foundational role of forests as carbon sinks in the path toward carbon neutrality. More concerning is the fact that millions of tons of walnut shells—by-products of the global nut processing industry—are directly landfilled annually worldwide. Unlike wood shavings that require dedicated forest harvesting, walnut shells represent a zero-cost, renewable waste stream with no additional carbon sink depletion. As lignocellulosic biomass resources, these materials not only emit significant amounts of methane during decomposition, but they also generate leachate, posing a dual threat to groundwater systems (Kjeldsen *et al*. 2002). Critically, walnut shells differ from conventional wood raw materials in their higher lignin content (28 to 35% *vs.* 20 to 25% in softwood) and dense cell wall structure (Pirayesh *et al.* 2012; Kumar *et al.* 2023). This distinctive composition endows them with superior hardness (Brinell hardness ~25 HB *vs*. 15 to 20 HB in pine wood) and water resistance. These are key properties for improving particleboard durability, which cannot be achieved by wood shavings alone. Breaking through this dilemma requires the establishment of a new circular economy model. Recent studies have shown that walnut shells, due to their unique microstructure and chemical composition, can serve as an alternative raw material for high-quality wood. Barbu *et al*. (2020) found that particleboards manufactured with walnut shell exhibit higher dimensional stability and Brinell hardness compared to cork-based particleboards. Pirayesh *et al*. (2013) and da Silva *et al*. (2017) reported that using walnut shell in combination with urea-formaldehyde adhesive significantly reduces formaldehyde emissions, with the effect being irreversible in the presence of formaldehyde and dodecane.

Sarsari *et al*. (2016) investigated the potential application of walnut shell powder (WSF) as a wood substitute in thermoplastic starch (TPS) composites and its influence on material properties. When the WSF content reached 40%, the tensile strength, flexural strength, and elastic modulus of TPS-based composites were significantly enhanced. Furthermore, Ayrilmis *et al.* (2013) reported that in polypropylene composites, the incorporation of WSF resulted in increased flexural and tensile moduli and improved thermal stability (Dobrzyńska-Mizera *et al*. 2019). Hamidreza Pirayesh *et al.* (2012) utilized a mixture of walnut shell and wood particles to fabricate particleboard. The addition of walnut shell particles substantially improved the water resistance of the panels—with water absorption reduced by 15 to 20% compared to pure wood particleboard. This can be attributed to their dense lignin-rich structure that inhibits water penetration (Pirayesh *et al.* 2012). While the proportion of walnut shells should not exceed 20% to meet the required standard for modulus of rupture (MOR) of pure wood particleboard, this limitation can be addressed by optimizing pressing parameters (a gap this study aims to fill). Notably, even at 20% addition, the thickness swelling of the composite board was reduced by 12% (Pirayesh *et al.* 2012). Such a performance enhancement cannot be achieved with the use of wood shavings without additional chemical treatments, which increase production costs and environmental impact.

Kumar *et al*. (2023) found that lignin and cellulose are essential components of lignocellulosic raw materials used for particleboard production, contributing significantly to enhanced mechanical strength and adhesive efficiency. Unlike wood shavings (cellulose 40 to 45%, lignin 20 to 25%), walnut shell powder is characterized by higher lignin content (28 to 35%) and lower hemicellulose content (15 to 18% *vs.* 25 to 30% in wood) (Pirayesh *et al.* 2012). This composition not only enhances the material's hardness and strength—improving particleboard MOR by 8 to 10% when substituted for 15 to 20% of the wood shavings—but it also reduces adhesive demand. The dense lignin structure promotes better interfacial bonding with urea-formaldehyde adhesive, lowering usage by 10 to 12% compared to pure wood formulations (da Silva *et al.* 2017). Furthermore, as a filling material (Dobrzyńska-Mizera *et al.* 2019), WSF not only reduces the dependence on wood shavings (alleviating logging pressure on carbon sinks and supporting the "double carbon" strategy proposed by Zhang *et al.* (2022)) and the amount of adhesive required, but it also enhances physical properties such as lowering water absorption and increasing heat resistance (thermal decomposition temperature increased by 15 to 20 °C). This is a critical advantage for particleboard used in high-temperature environments (*e.g*., kitchen cabinets). Therefore, using walnut shells as a raw material for particleboard aligns with the growing trend toward sustainable and eco-friendly materials. The alignment is not only due to waste valorization but also for its unique ability to improve product performance while reducing costs and environmental impact—advantages that conventional wood raw materials cannot replicate. However, none of the studies have examined the pressing parameters involved in walnut shell particleboard manufacturing or clarified how factors, such as hot-pressing pressure, time, temperature, and adhesive dosage, affect the final product's physical properties. Thus, this study aimed to identify the key factors—hot-pressing temperature, pressure, time, and glue dosage—that influence the mechanical performance of walnut shell-based particleboards, providing a direct technical foundation for the environmentally responsible and high-value utilization of agricultural and forestry residues and further supporting the realization of carbon neutrality goals (Zhang *et al.* 2022).

EXPERIMENTAL

Main Material

Walnut shells were sourced from local walnut growers (Yunnan Province, China, a major walnut-producing region). After crushing, screening, and drying, they were processed into powder (20 to 35 mesh, 10% moisture content). Wood shavings were supplied by Feilin Wood-Based Panel Group Co., Ltd. (Kunming, China). The shavings (a typical commercial particleboard mix) contained ~60 to 65% hardwood (mainly *Populus* spp., *Eucalyptus* spp.) and 35 to 40% softwood (primarily *Pinus* spp.), with 3% moisture content and 30 to 50 mm dimensions. The adhesive was self-prepared melamine-modified urea-formaldehyde resin (64% ± 2% solid content, pH 8.1, viscosity 410 mPa·s).

Main Instruments and Equipment

The primary experimental instrument employed in this study comprised hygrometers, hot presses, mechanical testing machines, and data analysis software. The experimental instruments are shown in Table 1.

Table 1. Experimental Instruments and Analysis Software

Name of the Equipment or Software	Model	Manufacturer
Halogen moisture meter	MB35	Shanghai Aohaus Instrument Co., LTD
Test hot press	KSH100T	Dongguan Kesheng Industrial Co., LTD
Universal mechanical testing machine	CWT5504	Shenzhen Xin Sansi Materials Testing Co., LTD
Electric convection oven	DHG-9125A	Shanghai Yiheng Scientific Instrument Co., LTD
Universal pulverizer	Model XFS-100	Shanghai Jingmi Instrument Co., LTD.
Horizontal band saw Machine	Model MJ346	Zhejiang Mingjiang Machinery Co., LTD
Laser Confocal Scanning Microscope, LCSM	Leica TCS SP8 X	Leica Microsystems CMS GmbH
SPSS Statistics 26.0	5725 - A54	IBM Corp., Armonk, NY, USA

Method

Experiment on the proportion of walnut shell powder

A melamine-modified urea-formaldehyde resin adhesive, exhibiting excellent water resistance, aging resistance, and low formaldehyde emission (Xi *et al*. 2024), was used to evaluate the performance of walnut shell powder and wood shavings at varying mixing ratios. Under consistent process parameters, particleboards were fabricated, and their mechanical and physical properties—including modulus of rupture (MOR), modulus of elasticity (MOE), internal bonding strength (IB), and 2 h thickness swelling percentage (2hTS)—were systematically tested and analyzed.

Process test of walnut shell-based wood particleboard

An L9 (3^4) orthogonal array test was conducted, with hot-pressing temperature, hot-pressing pressure, hot-pressing duration, and adhesive application level designated as variable factors. This methodology provides a straightforward, systematic, reliable, and highly effective approach to minimizing experimental error through the identification of optimal parameter settings, thereby reducing the number of required trials, associated costs, and processing time. The process conditions for walnut shell-based wood particleboard were optimized based on key performance indicators including MOR, MOE, IB, and 2hTS. Average values were determined from repeated measurements. Details of the experimental design are presented in Table 2.

Table 2. Factor Levels for the Orthogonal Test

	Factor			
Level	A: Hot-pressing Temperature (℃)	B: Hot-pressing Pressure (MPa)	C: Hot-pressing Duration (Min)	D: Resin Application Level (%)
1	160	3	6	10
2	180	4	7	12
3	200	5	8	14

Preparation process of particleboard

The walnut shells were dried to reduce their moisture content to 10%. After drying, the walnut shells were crushed using a universal pulverizer *via* a stepwise crushing method: first, coarse crushing was performed to reduce the shells to a particle size of 5 to 10 mm, followed by fine crushing to further refine the particles. The crushed material was then sieved to obtain walnut shell powder with an appropriate particle size (20 to 35 mesh), and oversize or undersize particles were discarded to ensure uniform particle size distribution. Based on a target density of 0.7 g/cm³ (plate specification: 300 mm × 350 mm × 10 mm), the quantity of raw materials was calculated, and wood shavings were blended with walnut shell powder at a mass ratio of 3:7. To ensure even adhesive distribution, the adhesive application level was maintained at 8% to 12% of the dry raw material weight. The mixture of raw materials and adhesive was then homogenized through manual stirring. Subsequently, the mixture was formed into a uniformly structured slab; internal air was removed by pre-pressing the compacted slab at a pressure of 0.5 to 1.0 MPa. The hot-pressing stage (core process) was conducted under the following conditions: temperature 160 to 200°C; pressure 3.0 to 5.0 MPa; time 6 to 8 min for plates with a thickness of 10 mm. Through the synergistic effects of temperature, pressure, and time, adhesive curing and plate densification were achieved. To prevent delamination, a gradient pressure release method was applied. After hot pressing, the sheets were cooled to an internal temperature below or equal to 40 °C to stabilize their structure, followed by a seven-day conditioning period in a controlled environment to relieve residual stress. Finally, the treated sheets were cut into standard specimens using a horizontal band saw machine; all cuts were performed at a feed rate of 300 mm/min to ensure flat, burr-free surfaces and avoid structural damage to the specimens, and key performance indicators—including MOR, MOE, IB, and 2hTS—were evaluated. The preparation process of walnut shell-based wood particleboard is illustrated in Fig. 1.

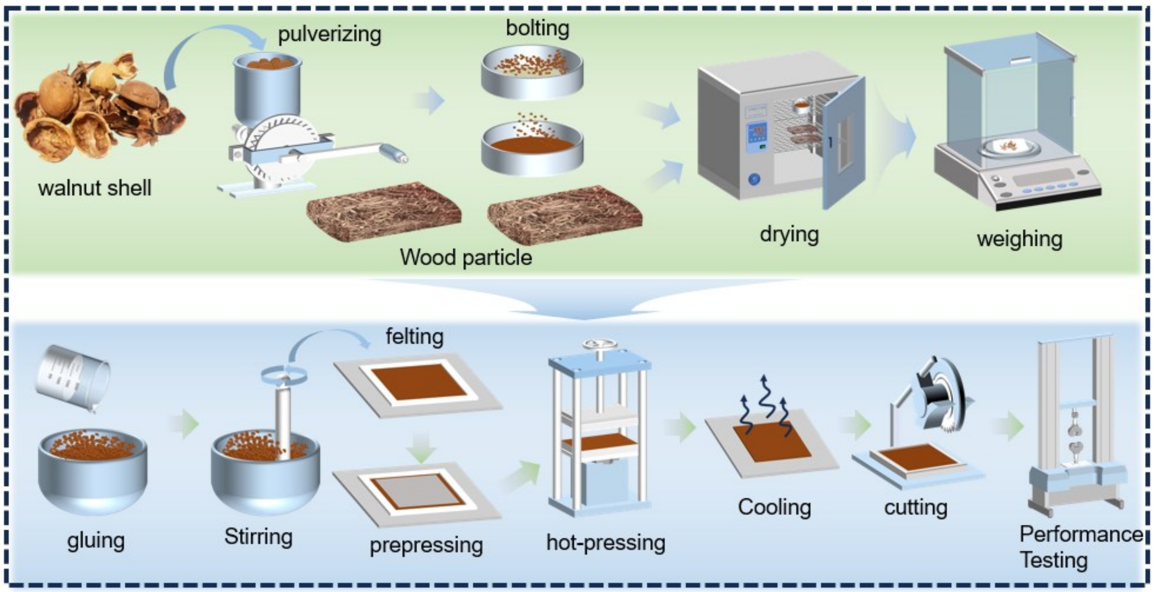


Fig. 1. Preparation process of walnut shell-based wood particleboard

The mechanical properties—including MOR, MOE, IB, and 2hTS—of walnut shell-based wood particleboard were evaluated in accordance with GB/T 17657 (2022).

Modulus of rupture

The MOR is a critical mechanical property that characterizes the bending failure resistance of particleboard. It is defined as the ratio of the bending moment at fracture to the section modulus of the specimen under maximum load, expressed in MPa. According to GB/T 17657 (2022), samples were cut into dimensions of 250 mm (length) × 50 mm (width). The MOR was determined using a universal mechanical testing machine operating on the three-point bending principle and calculated in accordance with Eq. 1,

$$MOR = \frac{3 \times F_{max} \times l_1}{2 \times b \times t^2} \quad (1)$$

where F_{max} denotes the maximum load at specimen failure, expressed in Newtons (N); l_1 represents the distance between the two supports, measured in millimeters (mm); b indicates the specimen width (mm); and t^2 refers to the specimen thickness (mm).

Modulus of elasticity

The MOE serves as a key mechanical performance indicator that reflects the material's resistance to deformation within the elastic range. It is defined as the ratio of stress to strain produced under load within the material's elastic limit, expressed in MPa.

As specified in GB/T 17657 (2022), specimens were prepared with dimensions of 250 mm (length) × 50 mm (width). The MOE was determined using a universal mechanical testing machine operating on the three-point bending principle and calculated in accordance with Eq. 2,

$$MOE = \frac{{l_1}^3}{4 \times b \times t^3} \times \frac{F_2 - F_1}{a_1 - a_2} \tag{2}$$

where l_1 denotes the distance between two supports, expressed in millimeters (mm); b indicates the specimen width (mm); t^3 refers to the specimen thickness (mm); $F_2 - F_1$ represents the load increment within the linear segment of the load-deflection curve, where F_1 corresponds to approximately 10% of the maximum load and F_2 to approximately 40%, all values expressed in Newtons (N). The deformation difference at midpoint a_1–a_2 reflects the deflection change of the specimen under the applied force interval from F_2 to F_1, measured in mm.

Internal bond

The IB is defined as the ratio of maximum tensile force to the load-bearing area between fibers or particles within the plate, resulting from bonding failure when tensile stress is applied perpendicular to the particleboard surface. It is expressed in MPa. As specified in GB/T 17657 (2022), specimens were prepared with dimensions of 50 mm × 50 mm, and IB was determined using a universal mechanical testing machine. The calculation followed Eq. 3,

$$IB = \frac{F_{max}}{l \times b} \tag{3}$$

where F_{max} denotes the maximum load at specimen failure, expressed in Newtons (N); l represents the specimen length in millimeters (mm); and b indicates the specimen width in millimeters (mm).

Two-hour thickness swelling

The thickness swelling is defined as the percentage increase in the thickness of particleboard after being immersed in water for 2 h. The test procedure complies with GB/T 17657 (2022). A standard-sized specimen (typically 50 × 50 mm) is immersed in water maintained at 20 °C for 2 h, and the thickness change before and after water absorption is precisely measured using a micrometer with a resolution of 0.01 mm. The calculation follows Eq. 4,

$$2hTS = \frac{t_2 - t_1}{t_1} \times 100 \tag{4}$$

where t_2 denotes the specimen thickness after immersion and t_1 represents the specimen thickness before immersion, both expressed in millimeters (mm).

RESULTS AND DISCUSSION

Test Results

Test results regarding the proportion of walnut shell powder

The effects of different proportions of walnut shell powder on the key physical properties of particleboard are presented in Table 3.

Table 3. Relationship between the Proportion of Walnut Shell Powder and the Physical Properties of Particleboard

Proportion of Walnut Shell Powder	Mesh Count/Dimension (mm)	MOR (MPa)	MOE (MPa)	IB (MPa)	2hTS (%)
0%	30-50	14.42	2038.47	0.66	19.59
15%	5-9	12.56	1972.83	1.11	11.51
	9-16	14.50	2083.67	1.14	11.96
	20-35	14.44	2174.92	1.12	14.24
	35-60	15.22	1954.64	1.04	15.59
30%	5-9	10.27	1527.96	1.22	12.61
	9-16	10.75	1624.33	1.24	12.53
	20-35	13.69	1980.08	1.41	9.62
	35-60	10.74	1606.72	0.97	13.73
50%	5-9	7.06	1193.33	1.17	10.94
	9-16	8.62	1270.39	1.22	11.87
	20-35	8.78	1330.43	1.46	8.31
	35-60	10.32	1395.59	1.43	9.23
100%	5-9	2.91	698.38	1.26	4.14
	9-16	3.38	702.46	0.97	7.42
	20-35	4.91	1039.96	2.48	5.55
	35-60	6.07	1148.61	2.23	5.35
GB/T 4897 (2015)		11 (P2)	1800 (P2)	0.40 (P2)	8.0 (P2)

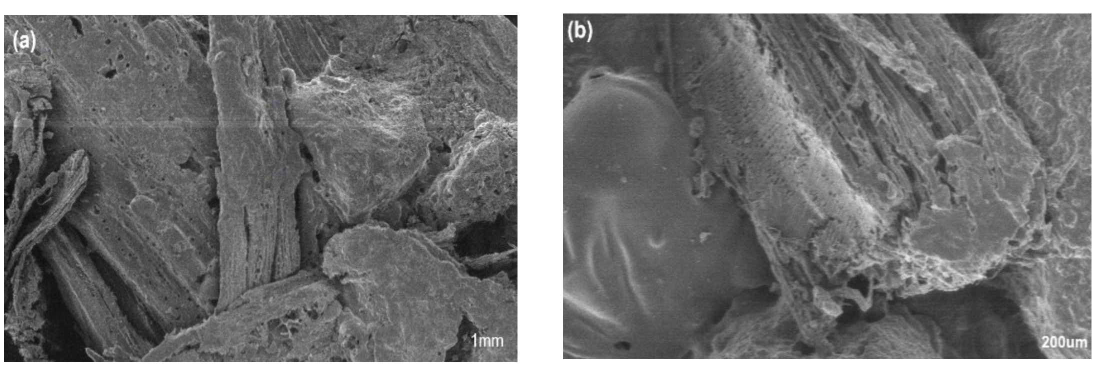


Fig. 2. SEM images of walnut shell particleboard with a proportion of 30% and a particle size of 20 to 35 mesh; a: scale bar of 1 mm, b: scale bar of 200 μm

As shown in Table 3, when the proportion of walnut shell powder was 0% (*i.e*., for pure wood particleboard), the board exhibited relatively high MOR and MOE, both of which far exceeded the standard values. In contrast, its 2hTS was significantly below the standard requirement, indicating poor water resistance stability of the board. When the proportion of walnut shell powder reached 15%, the MOR, MOE, and IB values all significantly exceeded the standard requirements. However, the wood substitution level remains low under this proportion, resulting in limited benefits for resource conservation. When the walnut shell powder content was increased to 30%, the mechanical properties of the particleboard (MOR, MOE, and IB) only met the minimum standard thresholds. As shown in Fig. 2, at a walnut shell proportion of 30% and a particle size of 20 to 35 mesh, the walnut shell particles were tightly bonded not only with each other but also with wood particles. The adhesive effectively filled the voids and coated both types of particles, suggesting that this particle size is suitable for achieving good interfacial adhesion. Hamidreza Pirayesh *et al.* (2012) investigated a mixture of walnut shell and wood particles for manufacturing particleboard. Their results indicated that the maximum allowable walnut shell content should not exceed 20% to satisfy the modulus of rupture requirements. To meet the mechanical performance criteria while maximizing wood substitution and addressing the limitation of a 20% maximum walnut shell content, a formulation containing 30% walnut shell powder with a particle size of 20 to 35 mesh was selected as the baseline for subsequent optimization experiments.

Results of the orthogonal experiment

In this study, the key factors influencing the mechanical properties of walnut shell-based wood particleboard—namely hot-pressing temperature, hot-pressing pressure, hot-pressing duration, and adhesive application level—were investigated using an orthogonal experimental design. Table 4 presents the experimental design parameters and the corresponding physical and mechanical property test results.

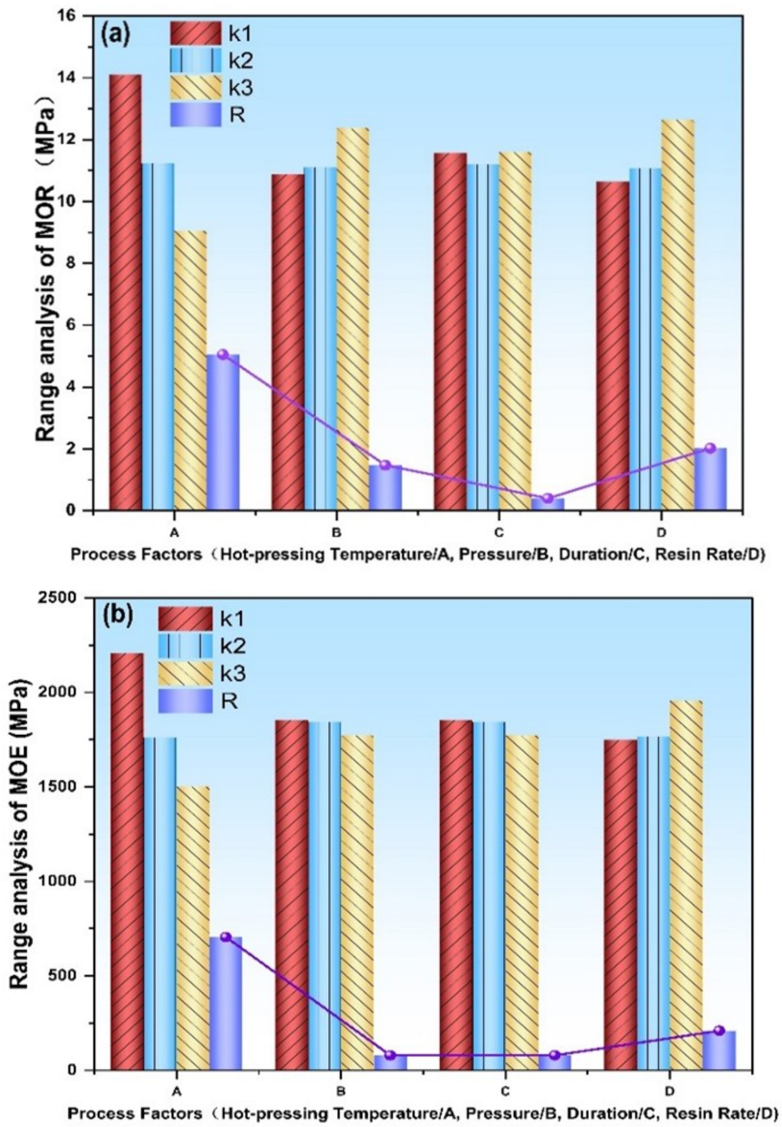
Table 4. Orthogonal Experimental Design and Results

Test Number	A	B	C	D	MOR	MOE	IB	2hTS
	ºC	MPa	Min	%	MPa	MPa	MPa	%e
1	160	3	6	10	12.84	2190.83	0.53	15.39
2	160	4	7	12	13.12	2191.20	0.66	9.92
3	160	5	8	14	16.34	2241.54	0.73	7.97
4	180	3	7	14	11.60	1943.45	0.81	10.66
5	180	4	8	10	10.20	1656.67	0.65	13.07
6	180	5	6	12	11.88	1682.18	0.77	11.45
7	200	3	8	12	8.25	1423.95	0.45	11.67
8	200	4	6	14	10.02	1686.59	0.68	8.94
9	200	5	7	10	8.87	1397.22	0.55	16.24
GB/T4897-2015					11（P2）	1800 (P2)	0.40 (P2)	8.0 (P2)

Through comparing the results with GB/T 4897 (2015), it can be confirmed that the MOR, MOE, IB, and 2hTS values of the third group in Table 3 met the required standards. Therefore, it can be preliminarily inferred that the particleboard performance was improved under the following conditions: a hot-pressing temperature of 160 °C, a hot-pressing pressure of 5 MPa, a hot-pressing duration of 8 min, and an adhesive application level of 14%.

Technical Index Analysis

As shown in Fig. 3, the range of hot-pressing temperature for the three groups (MOR, MOE, and IB) is the largest among all four factors, indicating that hot-pressing temperature had the most significant influence on the MOR, MOE, and IB performance of walnut shell-based wood particleboard. Conversely, its impact on 2hTS was minimal. With increasing temperature, both MOR and MOE values decreased significantly, whereas 2hTS rose sharply. The range of adhesive application level across all four test groups was also considerable, particularly for 2hTS, which suggests a strong effect of adhesive application level on overall sheet properties.



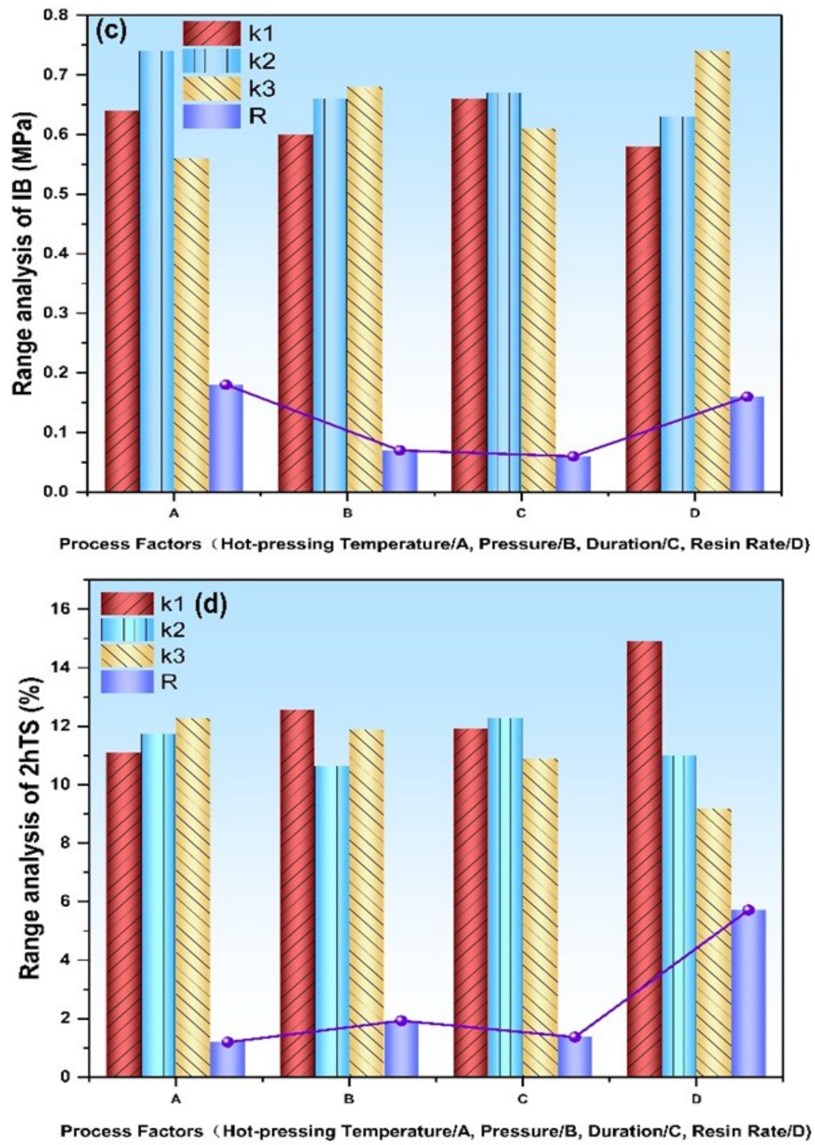


Fig. 3. Range analysis of the orthogonal test: a) MOR range analysis, b) MOE range analysis, c) IB range analysis, and d) 2hTS range analysis; Note: K_1 represents the first mean, K_2 represents the second mean, K_3 represents the third mean, and R represents the range

To efficiently assess the extent of influence exerted by four key factors—hot pressing temperature, pressure, duration, and resin application level—on the physical and mechanical properties of particleboard and to determine whether these factors significantly affect the experimental outcomes, both range analysis and variance analysis were carried out based on the results of the orthogonal test, as illustrated in Fig. 3.

As the adhesive application level increased, MOR, MOE, and IB values demonstrated a clear upward trend, while 2hTS declined correspondingly. Hot-pressing pressure likewise affected all four mechanical properties; as pressure increased, MOR and IB increased, whereas MOE and 2hTS tended to decrease. Among the four factors analyzed, hot-pressing duration exerted the least influence on the mechanical behavior of the particleboard. Overall, the factors were ranked in the order of influence as hot-pressing temperature > adhesive application level > hot-pressing pressure > hot-pressing duration.

Table 5. Variance Analysis of the Orthogonal Test

Analysis Index	Factor	Sum of Squares	DOF	F Ratio	Significance
MOR	A	115.682	2	30.615	**
	B	11.327	2	2.998	
	C	0.927	2	0.245	
	D	20.202	2	5.346	*
	residual error	34.008	18		
MOE	A	2291861.624	2	26.694	**
	B	34155.698	2	0.398	
	C	33707.117	2	0.393	
	D	241832.29	2	2.817	
	residual error	772716.791	18		
IB	A	0.147	2	3.43	
	B	0.035	2	0.826	
	C	0.02	2	0.461	
	D	0.123	2	2.868	
	residual error	0.385	18		
2hTS	A	6.393	2	0.83	
	B	17.244	2	2.239	
	C	9.096	2	1.181	
	D	153.324	2	19.909	**
	residual error	69.31	18		

Note: *** indicates that the effect is extremely significant at $p < 0.001$,** indicates that the effect is extremely significant at $p < 0.01$, and * indicates that the effect is significant at $p < 0.05$.

Table 5 demonstrates that at the $\alpha = 0.01$ significance level, hot-pressing temperature significantly influenced both MOR and MOE of the board, while the adhesive application level significantly affected 2hTS. At the $\alpha = 0.05$ level, the glue application amount remained a significant factor for MOR, whereas hot-pressing pressure and duration showed no statistically significant effects on any of the measured indices. The overall influence of hot-pressing temperature exceeded that of adhesive application level. This finding aligns with the range analysis results presented in Fig. 3, which indicate the following order of factor importance: hot-pressing temperature > adhesive application level > hot-pressing pressure > hot-pressing duration. These findings suggest that hot-pressing temperature and adhesive application level should be prioritized during the preparation of walnut shell-based wood particleboard, as they have a substantial impact on its mechanical performance.

The underlying mechanism can be attributed to two key factors: under optimal hot-pressing conditions, a higher adhesive application level enhances the contact area between particles and adhesive. Concurrently, elevated hot-pressing temperatures improve particle plasticity and accelerate adhesive curing, resulting in more complete bonding. Together, these effects contribute to improved structural strength, stability, and inter-particle adhesion. Furthermore, the adhesive layer formed on particle surfaces acts as a barrier to water penetration (Gonçalves *et al*. 2018), thereby reducing the thickness swelling of the final product.

Effect of Hot-pressing Temperature on Mechanical and Physical Properties of Walnut Shell-Based Wood Particleboard

The hot-pressing temperature is a critical parameter in the hot-pressing process. An appropriate temperature allows the slab interior to rapidly reach the required curing temperature for the adhesive, thereby enhancing both curing efficiency and mechanical properties of the particleboard (Ji and Lei *et al*. 2023). Under conditions of a hot-pressing pressure of 5 MPa, hot-pressing duration of 8 min, and resin application level of 14%, the effects of varying hot-pressing temperatures on particleboard properties are presented in Fig. 4.

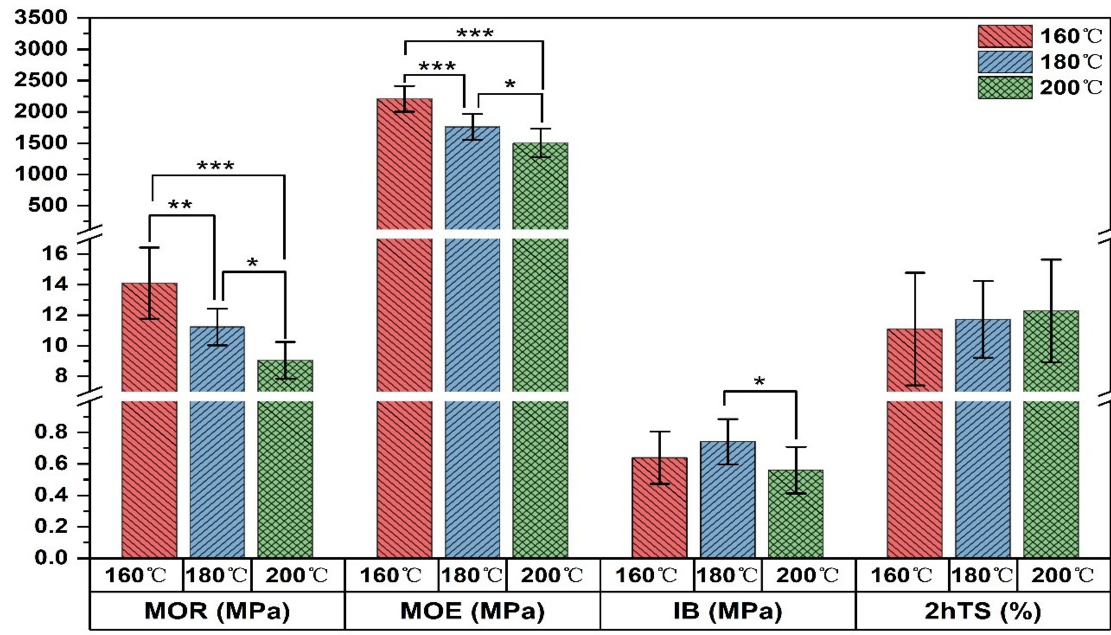


Fig. 4. The influence of hot-pressing temperature on the Mechanical and Physical Properties of walnut shell-based wood particleboard
Note: *** indicates that the effect is extremely significant at $p < 0.001$,** indicates that the effect is extremely significant at $p < 0.01$, and * indicates that the effect is significant at $p < 0.05$.

As shown, the MOR and MOE decreased with increasing hot-pressing temperature. When the temperature was excessively high, volume expansion increased, resulting in greater damage at bonding interfaces under different environmental conditions. The IB strength exhibited a non-monotonic response to hot-pressing temperature. The initial increase can be attributed to enhanced thermoplastic flow of lignocellulosic components and accelerated adhesive curing (Wei *et al*. 2021; Zhang *et al*. 2021), followed by a decline due to excessive degradation of wood polymers and premature cross-linking of adhesives (Li *et al*. 2023). The 2hTS increased with rising hot-pressing temperature. Higher temperatures induce hemicellulose degradation in walnut shell composites, generating hydrophilic oligosaccharides and increasing pore volume. This layered porosity enhanced capillary water absorption. Compared to low-temperature processing, the 2hTS value was significantly higher (Hashim *et al*. 2011). As illustrated in Fig. 4, there was a notable difference between static bending strength and elastic modulus, indicating that hot-pressing temperature had a significant influence on these mechanical properties. In contrast, the variation in 2hTS was relatively minor, indicating limited sensitivity to temperature changes. This aligns with the range and variance analysis results presented in Fig. 3 and Table 5. Furthermore, examining the significance levels in the figure reveals that at 160 °C, favourable effects were observed for bending properties (higher MOR and MOE) and dimensional stability (lower 2hTS); 180 °C represented the optimum temperature for internal bond strength, with MOR and IB meeting industry standards, though MOE and 2hTS fell short; 200 °C led to diminished mechanical properties and heightened swelling, potentially stemming from excessive adhesive curing or thermal degradation of the lignin-cellulose fraction, thereby compromising interfacial bonding and material stability. Overall, the comprehensive assessment indicated that the optimum hot-pressing temperature for walnut shell-based particleboard was approximately 160 °C.

Effect of Hot-pressing Pressure on the Mechanical and Physical Properties of Walnut Shell-Based Wood Particleboard

Hot pressing is one of the key processes in plate-making, which directly influences the density, MOR, MOE, IB, and other mechanical properties of the particleboard. Under conditions of a hot-pressing temperature of 160 °C, a hot-pressing duration of 8 min, and an adhesive application level of 14%, the effects of varying hot-pressing pressures on board properties are illustrated in Fig. 5.

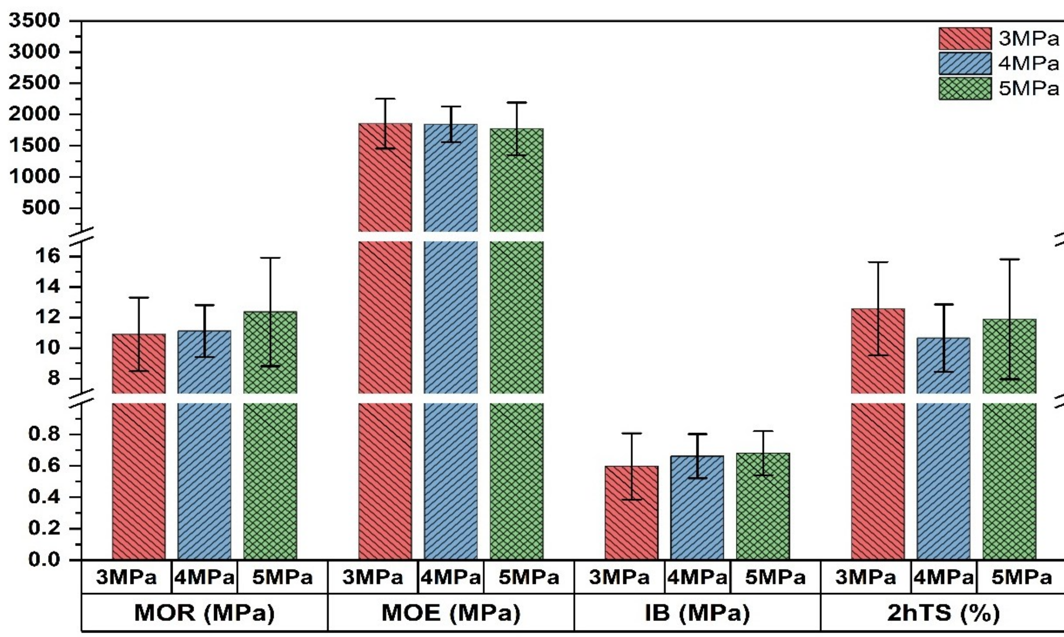


Fig. 5. Effect of hot-pressing pressure on the mechanical and physical properties of walnut shell-based wood particleboard

Both MOR and IB increased with rising pressure, as higher pressure enhanced adhesive penetration into wood fibers, thereby strengthening interfacial bonding. This mechanism contributes to a proportional increase in MOR and IB values (Kang *et al.* 2021). In contrast, MOE decreased with increasing pressure. Under high-pressure conditions, the curing rate of adhesives accelerates; however, excessive pressure may hinder steam release, leading to microcracks or delamination between particles, which ultimately reduces the elastic MOE (Kang *et al.* 2021). The 2hTS initially decreased and then increased with increasing hot-pressing pressure. This trend can be attributed to the gradual increase in plate density and reduction in porosity at moderate pressures, which limits water ingress and thus lowers TS (Adhikari *et al.* 2018). However, when pressure exceeds a critical threshold, excessive compaction increases internal stress, potentially causing expansion upon water absorption and consequently increasing TS (Ge and Lu *et al*. 2024). As illustrated in Fig. 5, physical and mechanical properties exhibited minimal variation across different hot-pressing pressures (with insignificant maximum/minimum differences). This indicated that pressure had a limited influence on overall performance—consistent with the range and variance analysis results from Fig. 3 and Table 5. At 4 MPa, MOR did not meet industry standards; when pressure exceeds 4 MPa, MOE also falls below acceptable levels. Based on comprehensive evaluation, an optimal hot-pressing pressure for walnut shell-based wood particleboard is approximately 4 MPa, where the lowest 2hTS value is achieved.

Effect of Hot-pressing Duration on the Mechanical and Physical Properties of Walnut Shell-Based Wood Particleboard

Hot-pressing duration refers to the holding time in the hot-pressing process. The length of the hot-pressing duration strongly influences the mechanical properties of the plate, which will directly affect the curing, durability, and surface quality of the adhesive (Jiang *et al.* 2025). Therefore, selecting an appropriate hot-pressing duration is critical. The optimum hot-pressing duration for pressing walnut shell particleboard was explored. Under conditions of a hot-pressing pressure of 5 MPa, a hot-pressing temperature of 160 °C, and an adhesive application level of 14%, the effects of different hot-pressing durations on particleboard properties are shown in Fig. 6.

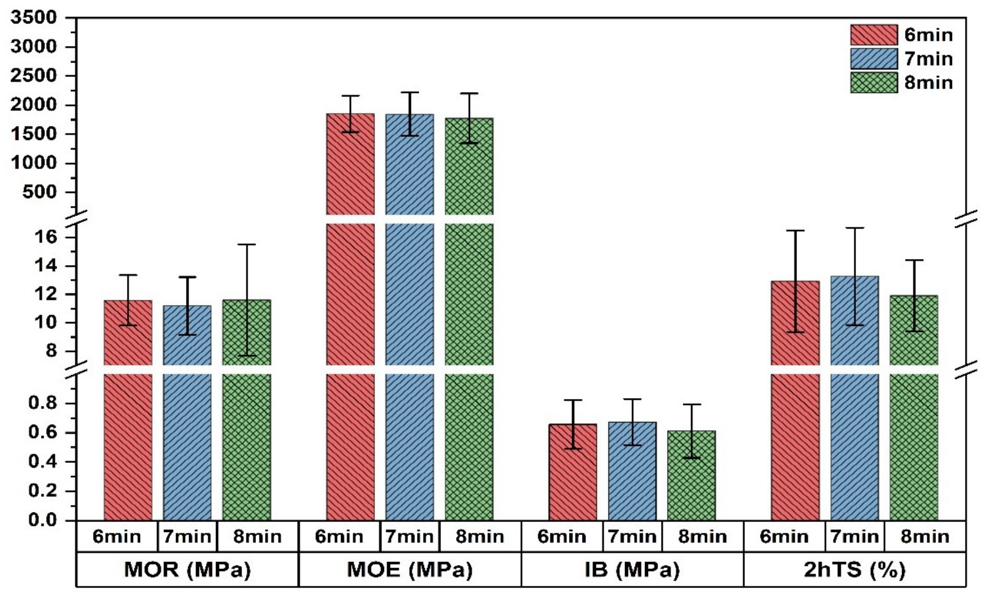


Fig. 6. Effect of hot-pressing duration on the mechanical and physical properties of walnut shell-based wood particleboard

The MOR first decreased and then increased with longer pressing time. The initial decrease occurred because the adhesive had not fully cured at short durations, leading to weak bonding (Qu and Guo *et al.* 2022). At around 6 minutes, the curing became sufficient, and the heat also softened the natural lignin present in the wood and walnut shells. This softened lignin flowed and acted as an additional binder, improving the strength (Yang *et al.* 2023). However, pressing for too long (*e.g.*, 7 to 8 min) may cause early-stage degradation of the wood and shell particles, preventing further increase in MOR.

The MOE gradually decreased as the pressing time was extended. This is likely because prolonged heat can damage the cellulose chains in the wood and walnut shells, reducing their stiffness (Esteves and Pereira *et al.* 2009). Also, the adhesive itself can become over-cured and brittle, which further lowers the MOE (Papadopoulos *et al.* 2020).

The IB increased initially due to better adhesive curing and lignin activation, reaching a peak at the optimal duration. After this point, IB decreased. The decline can be attributed to thermal degradation, where the adhesive became too brittle, and the wood/shell particles began to break down, weakening the bond (Boonstra and Blomberg 2007).

The 2hTS was high at short pressing times because the board had many open pores and incomplete curing, allowing water to enter easily. As pressing duration increased, the adhesive cured completely and the lignin flow helped create a denser structure, reducing water absorption (Pirayesh 2012). However, very long pressing might create micro-cracks due to material shrinkage, which could explain why TS does not decrease further.

It can be seen from Fig. 6 that physical and mechanical property ranges exhibited minimal variation when changing press times (only maximum-minimum differences), indicating weak effects on overall performance consistent with range and variance analysis results presented in Fig. 3 and Table 5. When pressed for 8 min, MOE did not meet standards; conversely, MOR reached its minimum at 7 min duration. Overall consideration indicated that an optimal walnut shell-based wood particleboard press duration was approximately 6 min, where MOE achieved its maximum. At this time, the adhesive curing and lignin bonding were effective, while avoiding significant degradation of the board components. The changes in properties were a result of both the adhesive behavior and the physical and chemical changes in the wood and walnut shell materials.

Effect of Adhesive Application Level on the Mechanical and Physical Properties of Walnut Shell-based Wood Particleboard

The amount of adhesive is a critical factor influencing both the quality and production cost of particleboard. Insufficient adhesive application leads to poor uniformity, which compromises the physical and mechanical properties of the board and may result in substandard product quality. Although excessive adhesive improves uniformity, it causes unnecessary material waste and raises manufacturing expenses (Fuentes Talavera *et al*. 2007). Therefore, determining an optimal adhesive content is essential for achieving high-performance and cost-effective particleboard. This study investigates the ideal adhesive amount for walnut shell-based wood particleboard. Under controlled conditions—including a hot-pressing temperature of 160 °C, a pressure of 5 MPa, and a pressing time of 8 min—the effects of varying adhesive application levels on board properties are illustrated in Fig. 7.

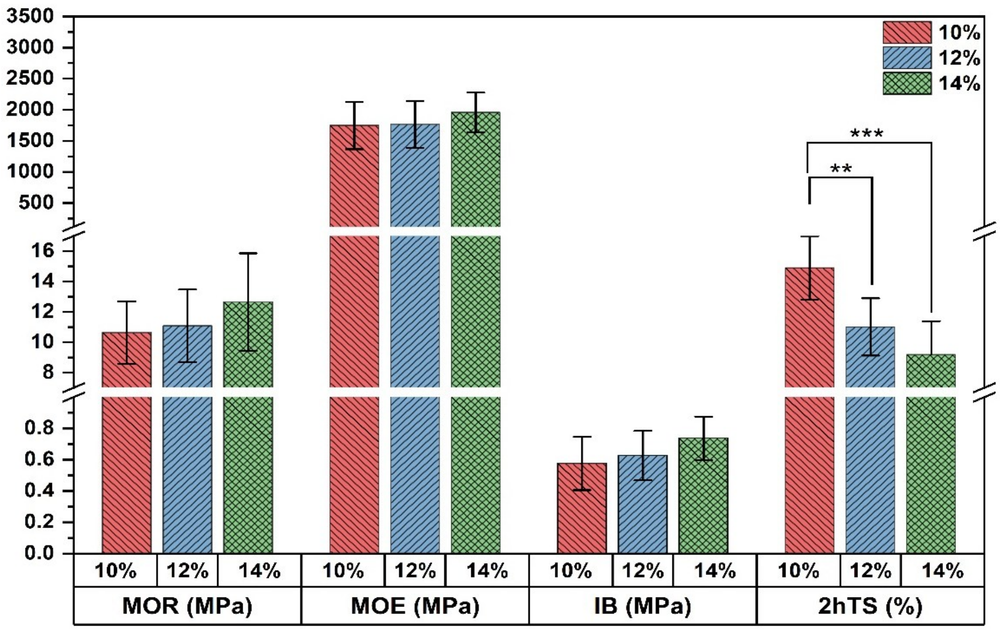


Fig. 7. Effect of resin application level on the mechanical and physical properties of walnut shell-based wood particleboard

The results show that MOR, MOE, and IB increased with higher adhesive application levels, particularly MOR and MOE, which exhibited significant improvements. This trend is primarily attributed to the increased contact area between adhesive and particles, as well as a greater number of cross-linking points formed during bonding. These structural enhancements contribute to more robust nail-like structures capable of withstanding higher internal stress, thereby improving overall mechanical performance. Experimental evidence supports that such microstructural changes effectively redistribute internal stress and enhance load-bearing capacity (Rzyska-Pruchnik and Kowaluk 2021). Additionally, the 2hTS decreased with increasing adhesive application levels due to two synergistic mechanisms:(1) Improved adhesive coverage on particle surfaces reduces pore connectivity and capillary water absorption (Rosenfeld *et al*. 2022), and (2) increased bonding point density forms continuous barriers that inhibit water penetration—confirmed by microstructural and mechanical analyses (Sari *et al*. 2013). As shown in Fig. 8, the thickness expansion after 2 h of water immersion varied significantly with adhesive application level (notable maximum/minimum differences), indicating adhesive content's strong influence. In contrast, static bending strength, elastic modulus, and internal bond strength exhibited relatively smaller fluctuations—consistent with the range and variance analysis results from Fig. 3 and Table 5. When the adhesive application level was below 12%, MOR failed to meet required standards. Similarly, MOE became non-compliant when the adhesive application level dropped below 14%. At an adhesive content of 14%, MOR, MOE, and IB all reached their peak values. Based on a comprehensive evaluation, the optimal adhesive content for walnut shell-based wood particleboard was determined to be approximately 14%.

Interface Bonding Mechanism Analysis

The superior mechanical properties of walnut shell-based particleboard primarily stem from the synergistic interaction between biomass components and adhesive phases at the interface. Chemical analysis indicated that the Yunnan walnut shells used in this study contained 28 to 35% lignin (predominantly guaiacyl-cinnamoyl type with relatively high g-unit content), which is significantly higher than cork (20 to 25%). This unique lignin plays a crucial role in interfacial bonding during hot pressing. At the optimal pressing temperature of 160 °C, the lignin in walnut shells undergoes plasticization—water molecules act as plasticizers, reducing intermolecular forces within the lignin macromolecules. This phase transition allows softened lignin to form a continuous viscous layer on particle surfaces, thereby establishing multiple hydrogen bonds with melamine-modified urea-formaldehyde (MUF) resin. Pirayesh *et al.* (2012) confirmed that lignin-urea resin interactions enhance internal bonding strength by 20 to 30% in agricultural waste-based composites through this hydrogen bond network. The structural characteristics of walnut shell particles further regulate adhesive distribution. As shown in Fig. 9(b) (SEM micrograph), the dense cell wall structure of walnut shells (average thickness 3.2±0.5 μm) restricts excessive MUF resin penetration, while their irregular surface morphology (R_a=2.8 μm) promotes mechanical interlocking with the adhesive phase. This dual-regulation mechanism enables uniform resin coating on particle surfaces (90% coverage), reducing interfacial defects such as voids and cracks. Statistical analysis indicated that the synergistic effect of lignin-resin chemical bonding and structural mechanical interlocking increased internal bond strength (IB) by 114% compared to the control group. The slightly higher optimal adhesive dosage (14%) observed in this study is justified by the physical barrier effect of walnut shell's dense cell walls. Unlike the porous structure of wood chips, which facilitates resin penetration, the dense microstructure of walnut shells requires sufficient adhesive to form continuous bonding bridges between particles. However, this cost impact is offset by the enhanced bonding durability from lignin-resin interactions, as evidenced by IB values exceeding the GB/T 4897-2015 standard by 62.5%.

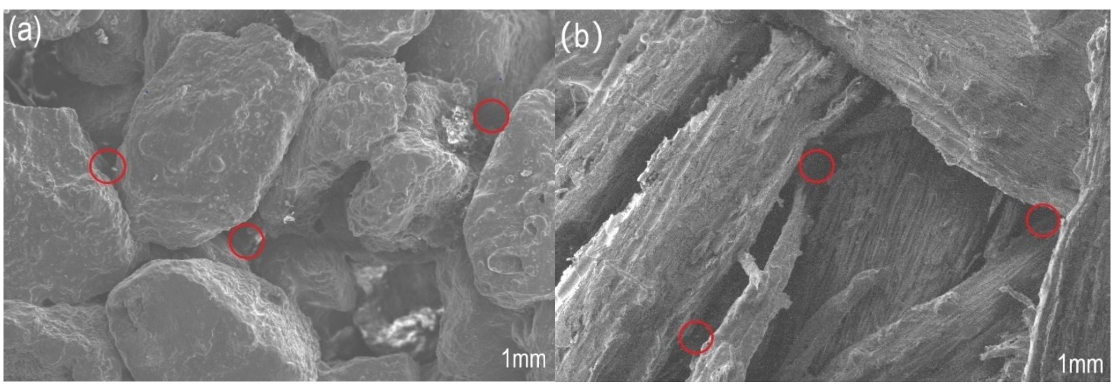


Fig. 8. Pure walnut shell particleboard (a) and pure wood particleboard (b)

Based on the experiments described above and the range and variance analysis results from the orthogonal tests, the optimal process parameters for pressing walnut shell-based wood particleboard were determined as follows: a hot-pressing temperature of 160 °C, a hot-pressing pressure of 4 MPa, a hot-pressing duration of 6 min, and an adhesive application level of 14%.

Verification of Test Results

The optimization conditions derived from the orthogonal experiment were verified through three repeated trials, and the average values were used to evaluate whether the performance indices met the requirements specified in GB/T 4897 (2015) for furniture-grade (P2) particleboard in the dry state. The test results are presented in Table 6.

Table 6. Test Items and Results

Experimental Project	MOR (MPa)	MOE (MPa)	IB (MPa)	2hTS (%)
Experimental Result	14.30	2425	0.65	6.80
International Standard	≥ 11 (P2)	≥ 1800 (P2)	≥ 0.40 (P2)	≤ 8.0 (P2)

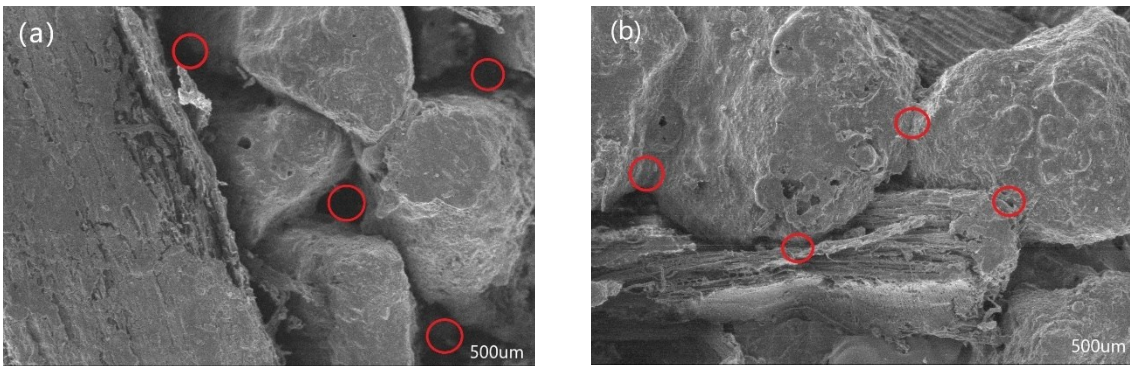


Fig. 9. Comparison of walnut shell-based wood particleboard before (a) and after optimization (b)

The test items and results in Table 6 demonstrate that the properties of walnut shell-based wood particleboard prepared under the optimized process conditions not only met but also significantly exceeded the strength and water-resistance requirements for P2-type particleboard used in a dry state, as specified by GB/T 4897 (2015). Moreover, both the MOE and IB values of the walnut shell particleboard met the standard criteria for P3-type particleboard under the same conditions (MOE: 2200 MPa; IB: 0.4 MPa). As shown in Fig. 8(a), noticeable pores were present between walnut shell particles and wood particles before optimization, indicating incomplete bonding and weak interfacial adhesion. In contrast, Fig. 8(b) shows that after optimization, the walnut shell particles, wood shavings, and other components were tightly integrated, forming a denser glue-nail structure and an extensive cross-linking network. This structural enhancement significantly enhanced the mechanical performance of the board. These findings confirm that the optimized process parameters derived from orthogonal testing are technically sound and effective in improving the physical and mechanical properties of walnut shell-based wood particleboard.

CONCLUSIONS

1. Based on an $L9(3^4)$ orthogonal experimental design, the optimal processing parameters for walnut shell-based particleboard were determined as follows: hot-pressing temperature of 160 °C, hot-pressing pressure of 4 MPa, hot-pressing duration of 6 min, and adhesive application level of 14%.

2. Walnut shells can effectively replace wood as a raw material for particleboard production, with the resulting walnut shell-based particleboards under optimized conditions meeting and exceeding relevant performance standards. Specifically, the MOR, MOE, and IB values exceed the requirements for P2-type particleboards in dry conditions in GB/T 4897 (2015), and both MOE and IB values met the standards for P3-type particleboards under the same conditions.
3. Among the four investigated process variables (hot-pressing temperature, pressure, time, and resin application level), their overall influence on the physical and mechanical properties of the board followed the order: hot-pressing temperature > adhesive application level > hot-pressing pressure > hot-pressing duration.

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