

**PREPARATION PROCESS AND INTERFACE MODIFICATION
ON THE MECHANICAL PROPERTIES OF BAMBOO FIBER/ POLYPROPYLENE
CARBONATE COMPOSITES**

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ABSTRACT

In this study, bamboo fiber (BF) and polypropylene carbonate (PPC) were used to prepare BF/PPC composite materials. The single factor test combined with orthogonal experiment was used to investigate the effects of different hot pressing process conditions (hot pressing temperature, hot pressing pressure and hot pressing time) on the mechanical properties of BF/PPC composites. Based on the hot pressing process results, the filler nano-calcium carbonate (Nano-CaCO₃), γ -aminopropyl triethoxysilane (KH550) and maleic anhydride (MAH) were added respectively to the composites to improve the interface between BF and PPC in order to increase the mechanical properties of the composites. The results showed that the reasonable preparation conditions of BF/PPC composites with the best mechanical properties were set at 170°C, under 1.9 MPa for 10 min. Compared with PPC samples, the tensile modulus, bending modulus and impact strength of BF/PPC composites could be increased to 102%, 38.69% and 65.13%, respectively. The optimal interface modification treatments have been proved that nano-CaCO₃ with 10% content could increase the tensile modulus and impact strength to 70.53% and 65.84%, while the best result for the bending modulus of BF/PPC composites was modified with MAH with 2.5% content, which could increase to 28.46%.

KEYWORDS: Bamboo plastic composites, polypropylene carbonate, mechanical property, interface modification.

INTRODUCTION

With the rapid development of society and economy, the calls for substituting bamboo for

wood and plastic are becoming increasingly strong, because of the shortage of the forest resources and the abundant resources of bamboo in China. Compared to wood, bamboo has many advantages such as the higher bending and tensile strength properties, the shorter growth cycle, the larger growing stock, and the lower price et al (Chen et al. 2022; Li et al. 2022). In recent years, bamboo utilization technologies have been constantly progressing and innovating. Among of them, bamboo plastic composite material (BPC) is an environmentally friendly material, which is made by filling modified polymer substrates with bamboo powder or bamboo fibers. BPC not only improves the variability and the strength properties of bamboo, but also has excellent characteristics such as lower water absorption, easier regeneration and recycling, and better corrosion resistance et al (Ekawati et al., 2022).

The mechanical properties of bamboo-plastic composites would be affected by the preparation conditions, such as the raw material types, the molding methods and the corresponding treatment parameters. One of the most commonly used molding method is hot pressing, in which the hot pressing temperature, pressure and time would affect the mechanical properties of BPC significantly (Bhushan et al. 2020; Sanjita et al. 2022; Wang et al. 2018). Han et al. (2022) studied the effects of hot pressing process parameters on the mechanical properties of bamboo fibers/polypropylene composites. The experimental results showed that the mechanical properties of composites were better when the hot pressing temperature was 180°C, the hot pressing pressure was 8 MPa, and the hot pressing time was 15 min. Friendrich (2022b) had discovered the promising effects of hot pressing for the samples hot pressed at moderate temperature for a shorter duration. The other most important factor for the mechanical properties of BPC is attributed to the main components of the raw materials such as the cellulose and the resin. For the cellulose, it contains a large number of hydroxyl groups, which usually exhibits strong chemical polarity and hydrophilic and would result in the poor compatibility with non-polar and hydrophobic resin polymers. Therefore, it is difficult to form a good interface combination in BPC material, which is the main reason for the significant reduce of the mechanical properties (Wang et al. 2018). Zulfli et al. (2014) found that nano-calcium carbonate could significantly improve the bending property, impact strength and elongation at break of the composite laminates, and also significantly increased the thermal decomposition temperature of the BPC material, thus significantly improving its thermal stability. The similar results obtained by Cichosz et al. (2020) showed that after chemical grafting of maleic anhydride, the tensile strength and elongation at break of the composite were significantly improved, and the composite showed good biocompatibility with water and polar organic media. Wu and Zuo (2016) showed that R-aminopropyl triethoxysilane (KH550) could improve the interfacial bonding of composites for waste tire rubber powder (RP)/plant cellulose (PC) composites.

Polypropylene carbonate (PPC), an aliphatic polycarbonate synthesized from CO₂ and epoxide, is a non-toxic and biodegradable resin which could reduce CO₂ emissions and protect the ecological environment. However, PPC also has some obvious disadvantages such as the poor mechanical and thermal properties, which have limited its popularization and application (Chang et al. 2017; Sánchez-Safont et al. 2018; Ward et al. 2019). With the advantages of lower cost, environmentally friendly and easily processing, the composites of

plant fiber/PPC composites have been investigated widely and considered as one of the ideal alternatives to traditional petroleum-based plastics (Osorio et al. 2020; Vinod et al. 2020; Zhang and Weng 2021).

In this study, composites were prepared with the materials of BF and PPC by hot pressing process. The effects of different hot pressing parameters including hot pressing temperature, hot pressing time and hot pressing pressure, and different interface modifications including nano-CaCO₃, KH550 and MAH on the mechanical properties of BF/PPC composites were investigated through the mechanical property tests based on the single-factor analysis and orthogonal experimental analysis. The aim is to provide technical guidance for the appropriate production process of BF/PPC composites.

MATERIAL AND METHODS

Material

Moso bamboo (*Phyllostachys edulis* (Carr.) H.de Lehaie) was taken from Jiangxi Province, China, and crushed into powder, then sorted through 80 mesh sieve. Polypropylene carbonate (PPC, molecular weight: 10^4 - 12×10^4), was purchased from Tianjin Hengda Run Trading Co., Ltd. Maleic anhydride (maleic anhydride, MAH, analytically pure) and silane coupling agent (KH550, industrial grade), was purchased from Tianjin Damao Chemical Reagent Factory. Nano calcium carbonate (nano-CaCO₃, analytical pure), was purchased from Tianjin Jimi Technology Co., LTD.

Optimization of hot pressing parameters for BF/PPC composites

The dried BF was sieved into 80 mesh by standard sieves, then the BF were mixed with PPC in the ratio of 4/6. After mixing evenly, the samples were prepressed in a 250×250×4 mm mold at room temperature, and then hot pressed under different process conditions. The hot pressing parameters optimization experiment firstly used single-factor test to initially determine the variation range of hot pressing temperature, hot pressing time and hot pressing pressure (Tab.1).

Tab. 1: Single-factor experiment on optimization of hot pressing parameters.

Temperature (□)	140, 150, 160, 170, 180, 200
Time (min)	5, 10, 15, 20
Pressure (MPa)	1.3, 1.6, 1.9, 2.2

Based on the single-factor experiment results, the influence of three hot pressing parameters on the mechanical properties of the composites was evaluated by the orthogonal experiment design, which would be also obtained the optimal hot pressing process conditions for the preparation of BF/PPC. As showed in Tab. 2, the three factors of hot pressing temperature (A), hot pressing pressure (B) and hot pressing time (C) were selected as independent variables and the test results of the tensile modulus, bending modulus and impact strength orthogonal obtained from the orthogonal experiments were used to range analysis.

Range is an index that describes the range of the data distribution, which can be calculated by the difference between the maximum and minimum values. Range is often used to describe the spread of data, and the larger the range, the greater the variability of the data, it shows that different levels of this factor have greater influence on performance:

$$\text{Range} = x_{\max} - x_{\min} \quad (1)$$

where: x_{\max} is the maximum value and x_{\min} is the minimum value.

Tab. 2: Orthogonal test design of hot pressing parameters

Serial Number	A: Temperature (□)	B: Pressure (MPa)	C: Time (Min)
1	140	1.6	5
2	140	1.3	10
3	140	1.9	15
4	170	1.6	10
5	170	1.3	15
6	170	1.9	5
7	200	1.6	15
8	200	1.3	5
9	200	1.9	10

Interface modifications of BF/PPC composites

The effects of MAH, nano-CaCO₃ and KH550 on the mechanical properties of BF/PPC composites were determined by single factor test. According to the amount of addition in Tab. 3, the three modifiers were added into the material with 80 mesh of BF and 4/6 ratio of bamboo to plastic respectively, and the plate samples were prepared under the optimal hot-pressing process conditions. All additive amounts were based on the quality of BF/PPC materials.

Tab. 3: Auxiliary agent addition volume screening

Modifier type	Mass ratio (%)
MAH	1, 2, 3, 4, 5
Nano-CaCO ₃	5, 10, 15, 20
KH550	0.4, 0.8, 1.2, 1.6, 2.0

Mechanical property test

The samples with 80×10×4 mm prepared under different conditions were conditioned in RH 50%, at 20□ to reach the room temperature. In this study, the tensile modulus, bending modulus, and impact strength of the samples were selected as the main evaluation indexes of the mechanical properties and were tested by Instron 3369 universal mechanical experimental machine made in United States, and 5 repeated samples were tested for each preparation condition:

$$E_t = \frac{\sigma'' - \sigma'}{\varepsilon'' - \varepsilon'} \quad (2)$$

where: E_t is tensile modulus (MPa), σ'' is tensile stress values measured at strain $\varepsilon''=0.0025$, (MPa), σ' is tensile stress values measured at strain $\varepsilon'=0.0005$, (MPa).

$$E_f = 500(\sigma'' - \sigma') \quad (3)$$

where: E_f is bending modulus (MPa), σ'' is bending stress values measured at strain $\epsilon' = 0.0005$, (MPa), σ' is tensile stress values measured at strain $\epsilon'' = 0.0025$, (MPa).

$$P = \frac{Q \times 55}{D \times d} \quad (4)$$

where: P is the impact strength (kJ/m^2), Q is the display value (%), D is the notch thickness (mm), d is the notch width (mm).

RESULTS AND DISCUSSION

Analysis of the optimization results of hot pressing process

Analysis of the single-factor test results of hot pressing process

For the single-factor performance test, the other hot pressing variables were controlled at the same and constant variables, and the test results were shown in Fig. 1.

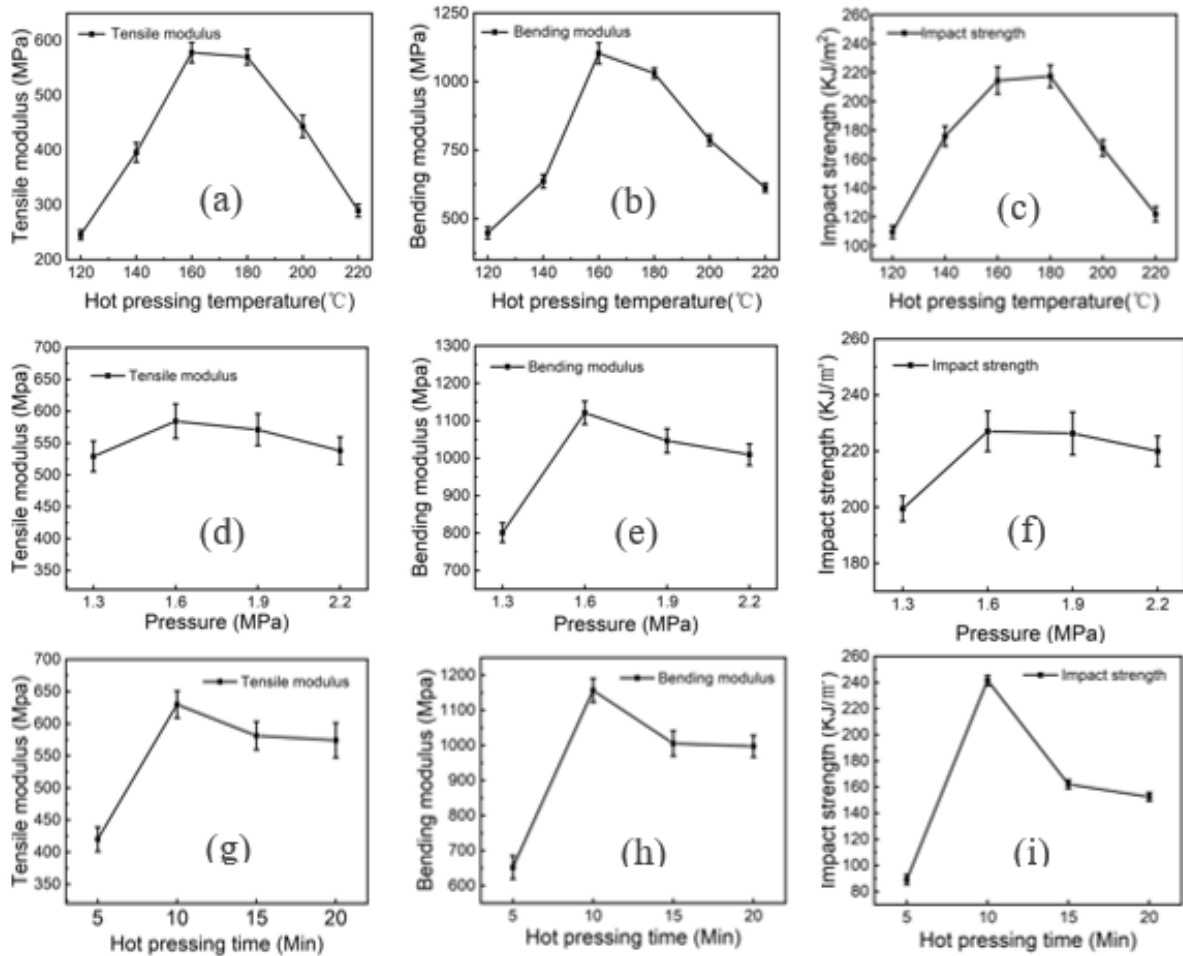


Fig. 1: Effects of hot pressing conditions on mechanical properties of BF/PPC composites (a. tensile modulus, b. bending modulus, c. impact strength).

Tensile modulus, bending modulus, and impact strength of the composites were most significantly enhanced when the hot pressing temperature, time, and pressure were 170°C, 10 min, and 1.6 MPa, respectively (Fig. 1a-i). From Fig. 1a-c, it seemed that the lower hot pressing temperature would prevent the PPC powder from completely transforming into a molten state and could not be completely coated on the BF surface for the plasticization reaction, while if the hot pressing temperature was too high, it would accelerate the thermal decomposition of the BF components, as the result, the mechanical properties would be decreased sharply (Ochi and Shinji 2022; Zhang et al. 2021). From Fig. 1d-f, it showed that shorter hot pressing time would cause the plasticization reaction between PPC and BF insufficiently, and the reason was attributed to the poor interfacial bonding ability between PPC and BF. However, if the hot pressing time was too long, it would also cause the thermal decomposition of BF (Liu et al. 2022). From Fig. 1g-i, it was found that if the hot pressing pressure was too small, the interfacial bonding ability between PPC and BF was also very poor, because the contact between PPC and BF was not sufficient to form a dense structure. However, if the hot pressing pressure was too high, it was also a negative factor the mechanical properties of the composites, because it would cause the BF/PPC composites with excessive density and hardness, which reduced the flexibility of the composites and negatively affects the tensile modulus, flexural modulus and impact strength (Daniel 2022a; Roni et al. 2021).

Analysis of the orthogonal experiments

The range analysis based on the orthogonal test is shown in Tab. 4. It can be seen that among the three factors, the three groups of temperature ranges are the largest, which indicates that temperature has the greatest impact on the three properties of bamboo-plastic composite. With the increase of temperature, the three main properties showed a trend of first strengthening and then weakening. Hot pressing pressure has great influence on elastic modulus and impact strength, and with the increase of pressure, the properties of bamboo-plastic composite show an upward trend. The hot pressing time has a certain influence on the three main properties of the plate, but the overall effect is the weakest. The influence of the factors of hot pressing on the main properties of bamboo composite is as follows: temperature > pressure > time.

Tab. 4: Range analysis of orthogonal test.

Factor	Tensile modulus (MPa)				Bending modulus (MPa)				Impact strength (kJ/m ²)			
	k1	k2	k3	Range	k1	k2	k3	Range	k1	k2	k3	Range
A: Temperature	410.84	566.10	544.64	155.26	668.17	955.72	769.40	287.54	128.00	220.36	155.54	92.36
B: Pressure	507.71	489.82	534.04	34.21	821.34	731.47	840.47	109.00	151.7	165.48	186.72	35.02
C: Time	486.90	519.49	515.19	32.58	801.92	864.19	727.18	137.01	159.04	173.53	171.33	14.50
Extent of the influence	A>B>C				A>C>B				A>B>C			
Optimal combination	A ₂ B ₃ C ₂				A ₂ B ₃ C ₂				A ₂ B ₃ C ₂			

Hot pressing process parameters validation

Through the analysis of single factor test and orthogonal experiment, according to the range results, the optimal combination of mechanical properties is obtained: hot pressing temperature 170°C, hot pressing pressure 1.9 MPa, hot pressing time 10 min. In order to verify the reliability of the parameters, the test was carried out on the automatic hot press. The hot

pressing parameters were set at 170°C, 1.9 MPa, 10 min, and 5 groups of repeated tests were carried out. As shown in Tab. 5, the statistical analysis shows that the average values of tensile modulus, bending modulus and impact modulus are obtained under the optimal parameter conditions.

Tab. 5: Verification of the test results.

Parameters	Average value	Maximum value	Minimum value
Tensile modulus (MPa)	641.33	668.21	616.68
Bending modulus (MPa)	1126.87	1176.32	1081.57
Impact strength (kJ/m ²)	234.71	245.35	227.69

Analysis of the interface modifications of BF/PPC composites

MAH modifications analysis

When the addition of MAH was increased to 3%, the tensile modulus, bending modulus and impact strength of BF/PPC composites would be increased significantly (Fig. 2). Compared with BF/PPC composites without MAH, the tensile modulus, bending modulus and impact strength of the modified samples increased by 42.91%, 28.46% and 42.94%, respectively. This was because the molecular chain of bamboo fiber contained a large number of hydroxyl groups, which presents strong hydrophilicity and polarity. However, the thermoplastic PPC powder had strong hydrophobicity and non-polarity, so the interface compatibility of composites was very poor without any modification. MAH improved the interface compatibility of the composite material by increasing the interface wettability between PPC matrix and bamboo fiber surface area, and as the result, the interface binding force between the composites had been enhanced. The results were similar with Naushad et al. (2017), who found that the damage tolerance of the sisal fiber and polypropylene composites could be improved by MAH modification. He pointed that the impact fracture morphology revealed a better interfacial bond between the fiber and the maleic anhydride grafted polypropylene. However, when the addition MAH was more than 3%, excess maleic anhydride will crystallize out and destroy the interface bonding ability between PPC and BF, and the mechanical properties of the composites decreased significantly.

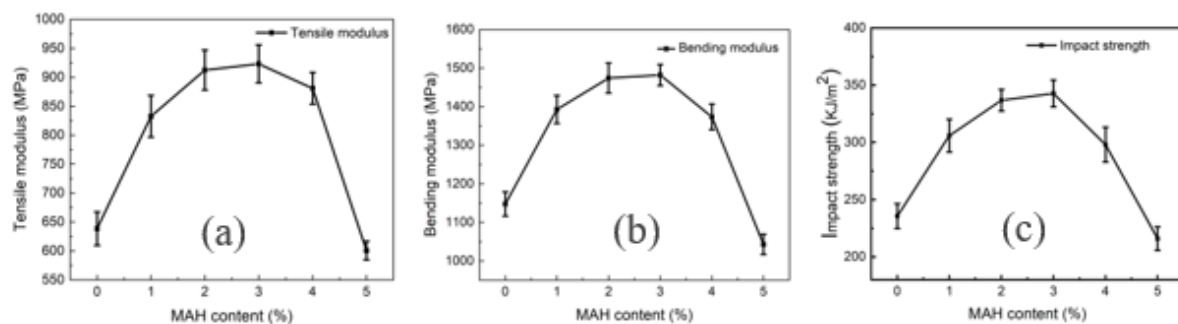


Fig. 2: Effect of MAH content on properties of BF/PPC composites: a) tensile modulus, b) bending modulus, c) impact strength).

Nano-CaCO₃ modification analysis

As shown in Fig. 3, when the addition of Nano-CaCO₃ was increased to 10%, the mechanical properties of BF/PPC composites would reach the highest level. Compared to the samples without modifications, the tensile modulus, bending modulus and impact strength of Nano-CaCO₃ modified ones would be increased by 70.53%, 7.56% and 65.84%, respectively. However, when the addition of Nano-CaCO₃ was more than 10%, the tensile modulus, bending modulus and impact strength of BF/PPC composites would be gradually decrease. This is consistent with the conclusion obtained by Zhang et al. (2019) used CaCO₃ modified PCL wood-plastic composites. Zhang et al. (2019) thought that was because excessive content of CaCO₃ reduce the activity space of molecular chain and the melt flow rate. The results indicated that within a reasonable addition amount, Nano-CaCO₃ could be dispersed evenly into the BF/PPC composites to enhance its mechanical properties because of the effect of the ultra-fine particles. Therefore, excessive addition of Nano-CaCO₃ would destroy the interface bonding ability of the composites. In addition, Nano-CaCO₃ was prone to agglomeration during the preparation of composite materials, thus impairing the mechanical properties of the composite materials (Shen et al. 2019).

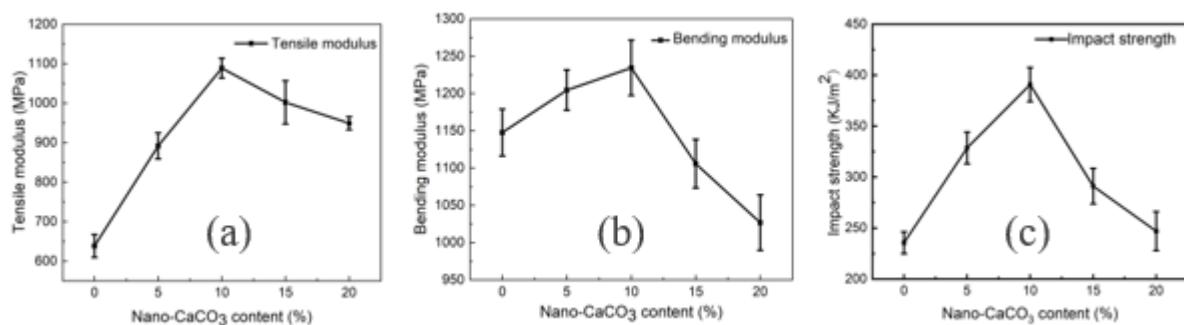


Fig. 3: Effect of Nano-CaCO₃ content on properties of BF/PPC composites: a) tensile modulus, b) bending modulus, c) impact strength).

KH550 modification analysis

As shown in Fig. 4, compared with BF/PPC composites without any modifications, when the addition of KH550 was increased to 0.8%, the tensile modulus, bending modulus and impact strength would be increased by 25.57%, 12.53% and 16.51%, respectively.

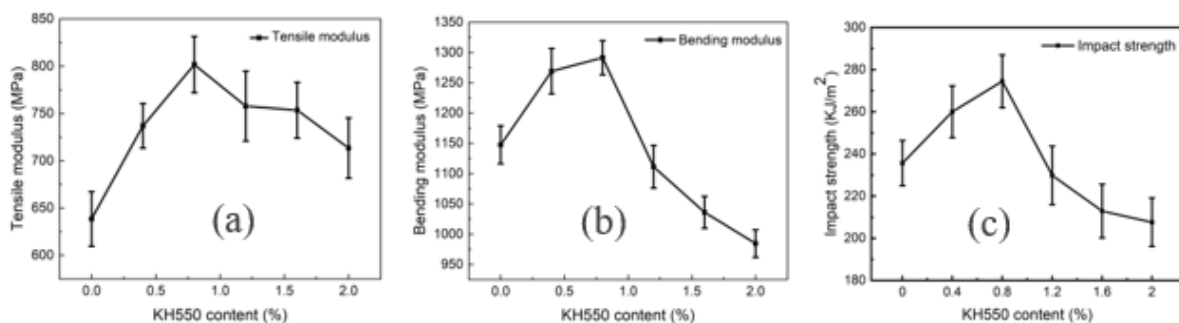


Fig. 4: Effect of KH550 content on properties of BF/PPC composites: a) tensile modulus, b) bending modulus, c) impact strength.

However, when the addition of KH550 was increased from 0.8% to 2%, the tensile modulus, bending modulus and impact strength of BF/PPC composites would be gradually decreased. The mechanical properties of BF/PPC composites modified with KH550 had the similar trend of the change with the samples modified with nano-CaCO₃ and MAH. This was because the amino and ethoxy groups in KH550 were coupled with the composites to enhance their adhesion(Wei and Wang 2018) and improve the dry and wet bending and compressive strength of the thermoplastic and thermosetting resins (Wu and Wang 2018). It could also improve the wettability and dispersion of fillers in polymers (Xu et al. 2019). However, excessive addition of KH550 would destroy the PPC matrix structure, thus reducing the mechanical properties of the composites.

CONCLUSIONS

With the shortage of wood resources, the promotion of bamboo has been put on the agenda, because bamboo was famous for its excellent mechanical properties, green environmental protection, short growth cycle and other advantages. More and more efforts had been made in the research of the bamboo/plastic composite material, which could be widely used in construction, packaging, furniture, et al. In this study, through the combination of single factor experiment and orthogonal experiment analysis, the optimum hot pressing process parameters for BF/PPC composites were determined as follows: hot pressing temperature 170□, hot pressing pressure 1.9 MPa, and hot pressing time 10 min, in which the mechanical properties of the composites could be significantly improved. Compared with the PPC material, the tensile modulus, the bending modulus and the impact strength of the composite could be increased by 102%, 38.69%, and 65.13%, respectively. On the basis, 3.0% of MAH, 10% of nano-CaCO₃, 0.8% of KH550 had been proved to strengthen the mechanical properties of the composites. Among them, nano-CaCO₃ was the top performer to improve the tensile modulus and the impact strength of the composites, and KH550 has the best performance on bending modulus of the composites.

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