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Optimization of the Hot Pressing Process for Preparing Flax Fiber/PE Thermoplastic Composite

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Abstract:
The hot pressing process parameters were optimized to prepare flax fiber reinforced polyethylene (PE) thermoplastic composite by the Taguchi method. The optimal hot pressing process parameters were determined to increase the tensile strength of the composite. The optimal parameters of the design include the following sections: hot pressing temperature, pressure, hot pressing time and coupling agent modification time. An L_9 (3*4) orthogonal matrix based on the Taguchi method was created. By means of analysis of signal-to-noise ratio and analysis of variance, the optimal hot pressing process parameters combination was found, compared to the average tensile strength in the nine design experiments, and the tensile strength was improved nearly 10%.

Keywords: *flax fiber; hot pressing process; optimization; Taguchi method; ANOVA*

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1. Introduction

In recent years, with global energy shortages and environmental pollution, fiber-reinforced composite materials are gradually developing towards a green ecology [1-10]. Natural fiber reinforced composites with the characteristics of low cost and recyclability are used to replace other synthesized fiber reinforced composites [1–3]. The good mechanical properties is showed by composite reinforced with natural fibers [5]. As a kind of natural fiber, flax fibers have several advantages: low density [6], high Young's modulus [2], biodegradability relatively high tensile and flexural modulus [7], and it shows the same outstanding performance as glass fiber reinforced plastics [8–10]. At the same time, thermoplastic resin is superior to thermosetting compound in terms of its excellent characteristics. The thermoplastic resin is used widely as the matrix material for environmental protection. Because the polymerization reaction has been completed before the impregnation, the hot pressing process is completely a physical process, and compared with the thermosetting resin, there is no environmental pollution problem, and it is called a green material of the 21st century [11].

By reading a wide range of literature to study green composite or reinforced biocomposite [5-9]. With the help of hot pressing process, flax fiber and polypropylene (PP) were used as raw materials to prepare composite materials. The influence of hot pressing parameters (temperature and hot pressing time) on the mechanical properties of the composite plate was analyzed by orthogonal design. The optimal solution was obtained by the range analysis and variance analysis. It found that the mechanical properties of flax fiber/PP composite were the best with the hot pressing temperature of 180°C and hot pressing time of 40min [12]. When the flax fiber has a content of 30 and 40wt.%, the strength of the flax/PLA (polylactic acid) composite is 50% better than flax/PP composites [13]. The Flax fiber/PLA Through the analysis of peeling test and tensile test, the optimum hot pressing parameters of the flax fiber/PP composite were that pressing temperature was 180°C, hot pressing time was 5min and hot pressing pressure was 6MPa [14]. For bamboo fiber/PP composite, by analysis of tensile strength and flexural strength. 170**°C, 3**MPa and 140s are optimum hot pressing process parameters [15]. In addition, the optimum hot pressing parameters of other plant fiber composite are temperature 179°C and pressure 178bar [16].

Besides the basic hot pressing parameters, in order to obtain better mechanical properties, flax fibers need to be modified during hot pressing. Coupling agents are also important for enhancing the mechanical properties of natural fiber thermoplastic composite. Some studies showed that the cellulose/PP composite which the fiber modified with the maleic anhydride-polypropylene copolymer can increase the tensile strength by 80% [17]. Mo [18] studied the effect of ramie short fibers surface KH-550 modification on the interlaminar fracture toughness of laminates. Besides the silane coupling agent was used to modify aramid. The contact angle test shows that the contact angle of the modified aramid becomes smaller, indicating that the KH550 silane coupling agent can be improved the hydrophilicity of aramid [19]. It found that alkali modification improved the compatibility between jute fiber and matrix, and their interface bonding strength. The mechanical properties of jute fiber reinforced composites after alkali modification are better than unmodified composite [20]. Tensile tests have been performed for evaluated the tensile strength of flax fiber reinforced composites. The Max. tensile strengths for linear low density polyethylene and its related composites reinforced by flax fibers were about 13.40MPa and the 14.30MPa [21], which were about 19.50MPa and the 20.50MPa for the cases of high density polyethylene and its related composites reinforced by flax fibers [22].

The flax fiber and PE that has low cost and can be decomposed are chosen as the raw material. Besides, the flax fiber/PE thermoplastic composite have a wide range of applications.
Many attempts have been made to study the hot pressing process of natural fiber composite materials. The most important of these is the optimal design of the pressing parameters. In addition to the traditional hot pressing parameters, fiber modification is also considered to enhance the mechanical properties of the composite plate. The selection of hot pressing parameters involves four parts: hot pressing temperature, hot pressing pressure, hot pressing time and coupling agent modification time. Reasonable adjustment of the size of the four parameters is critical to the quality of the specimen. In order to find suitable hot pressing parameters, the hot pressing experiment was carried out using the Taguchi method.

The experimental factors include four hot pressing parameters, and there are three levels in the each parameter. The L_9 (3*4) orthogonal experimental table was designed. Signal-to-noise (S/N) ratio analysis and analysis of variance (ANOVA) were carried out to determine the influence of different hot pressing parameters on the tensile strength of the specimen and the optimal experimental parameters.

2. Experimental procedure

2.1 Experimental details

As shown in Figure 1, there are flow chart of hot pressing process and material property characterization. The flax fiber and PE film were used as raw material to prepare fiber/PE thermoplastic composite, the hot pressing process was applied with the help of hot pressing machine to prepare composite plate. After that, the composite specimens were prepared with the size of 100mm×10mm×3mm, the tensile test was performed following by ASTM. The cross section of morphology of composite specimens were observed by SEM (scanning electron microscope).

The Taguchi method was originally established to reduce the number of experiments, and the design of experiments (DOE) were designed to find the best process parameters [23]. The DOE was realized by the matrix tool. The S/N ratio and the ANOVA are two important tools for Taguchi method analysis. They can be used to determine the optimal parameters and the influence of various experimental factors on the optimal parameters [24-25].

The hot pressing process of the material is completed by using a hot press machine. The setting of the hot pressing parameters was designed by the Taguchi method. The mechanical properties of the specimens were tested by a universal tensile machine with a tensile rate of 1 mm/min.

The main parameters in the hot pressing process include hot pressing temperature (A), hot pressing pressure (B) and hot pressing time (C). Because the flax fiber composite itself has poor adhesion to fibers and resins, it is necessary to modify the flax fiber with a silane coupling agent to enhance the cross-sectional bonding property. Thus, another parameter modification time (D) by silicon coupling agent is studied. As shown in the Table 1, there are four hot pressing parameters and three levels for each hot pressing parameter.

Table 1. Hot pressing process parameters and corresponding levels

Parameters	Unit	Level 1	Level 2	Level 3
A (hot pressing temperature)	°C	120	130	140
B (hot pressing time)	h	0.25	0.50	0.75
C (pressure)	MPa	1.00	1.50	2.00
D (modification time)	h	0.50	1.00	2.00

According to the Taguchi method, an orthogonal matrix of L_9 (3*4) was used as shown in Table 2, it contains nine hot pressing experiments.

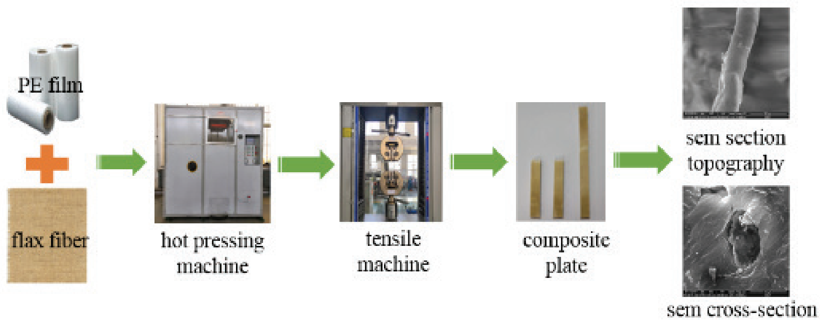

Figure 1. The flow chart of hot pressing process and material property characterization

Table 2. An L_9 (3*4) orthogonal array of the Taguchi method

Number of experiments	Parameters				
	A	B	C	D	Tensile Strength /MPa
No. 1	120	0.25	1.00	0.50	23.83
No. 2	120	0.50	1.50	1.00	23.45
No. 3	120	0.75	2.00	2.00	26.42
No. 4	130	0.25	1.50	2.00	24.38
No. 5	130	0.50	2.00	0.50	25.18
No. 6	130	0.75	1.00	1.00	22.03
No. 7	140	0.25	2.00	1.00	25.64
No. 8	140	0.50	1.00	2.00	22.33
No. 9	140	0.75	1.50	0.50	22.11

3. Results and discussion

3.1 Analysis of the S/N ratio

Through the S/N ratio, the influence of each experimental factor on the response value can be analyzed and the optimal experimental parameters can be predicted. Besides, smaller-is-better (Eq. (1)), larger-is-better (Eq. (2)) and nominal-is-the best (Eq. (3)) are used to calculate the S/N ratio.

$$\eta = -10\log\frac{1}{n}\left(\sum_{i=1}^{n} y_i^2\right) \tag{1}$$

$$\eta = -10\log\frac{1}{n}\left(\sum_{i=1}^{n} 1/y_i^2\right) \tag{2}$$

$$\eta = 10\log\frac{1}{n}\left(\sum_{i=1}^{n} y_i^2/s^2\right) \tag{3}$$

and η is mean the S/N ratio, n represents the number of experiments, y_i represents the response value of i^{th} experiment, and s is square error. In this study, tensile strength was used as an optimized response. The greater the tensile strength, the better the mechanical properties of the plate, so the second formula is chosen.

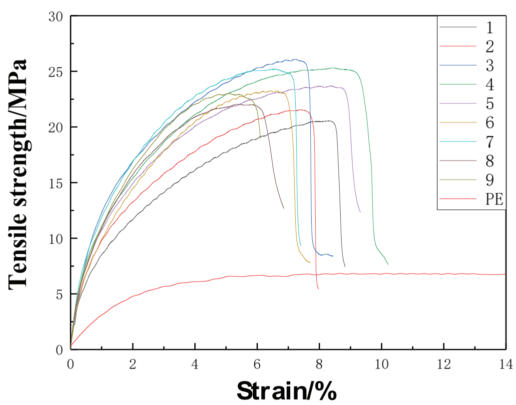

Figure 2. Stress-strain curves for flax fiber/PE composite specimens and pure resin specimens

As shown in the Figure 2, the average of the results of the three tensile specimens under each hot pressing parameter and the average of the tensile results of the pure PE resin specimens were used for comparative analysis. The flax fiber reinforced thermoplastic composite specimens has higher tensile strength than pure PE resin specimens, the average stress is 23.93MPa, and the tensile strength of the pure PE resin specimen is 7.87MPa. However, the pure PE resin specimens have larger strain than flax fiber/PE composite tensile specimens.

The results of the signal to noise ratio are shown in Table 3. According to Table 3, for tensile strength, the best combination of parameters: the hot pressing temperature is 120 °C, the hot pressing time is 0.25 h, and the hot pressing pressure is 2.00MPa, silane coupling agent modified the flax fiber for 2.00 h.

Table 3. The S/N response table (larger-is-better) for tensile strength

Level	Parameters			
	A	B	C	D
1	27.79	27.82	27.13	27.49
2	27.54	27.47	27.35	27.48
3	27.35	27.40	28.21	27.72
Delta (Δ)	0.45	0.42	1.09	0.24
Rank	2	3	1	4

3.2 Analysis of variance (ANOVA)

As shown in the Table 4, the ANOVA are also used to analysis the influence of each experimental factor on the response value. Besides, the percentage of contribution (P%) of variance and p-value are the important for the ANOVA to analyze experiment. The analysis and calculation of Taguchi experiment can refer to these two books [26, 27]. The sequential sums of squares (*Seq SS*) and adjusted sums of squares (*Adj SS*) of experiment factor k are given by

$$Seq\,SSk = Adj\,SSk = \sum_{i=1}^{n} 3\left[(mk)l - m\right]_l^2 \tag{4}$$

The formula of the adjusted mean square (Adj *MS*) and the F-statistic (F) are given by

$$F = \frac{Adj\,SS_k}{DF_k} \div \frac{Seq\,SS}{DF_E} \tag{5}$$

$$Adj\,MS_k = \frac{Adj\,SS_k}{DF_k} \tag{6}$$

and E is mean error, SS_T represents the total sum of squares given by

$$SS_T = \sum_{i=1}^{n} (\eta_i - m)^2 \tag{7}$$

The formula of the percentage contribution of the different factors to the response value is given by

$$P = \frac{SS_k}{SS_T} \times 100 \tag{8}$$

From the analysis of variance in Table 4, it was found that, there are differences in the magnitudes of the four P (%) of A, B, C, and D. According to the magnitude of the P (%), it can be inferred that the contribution of the four hot pressing parameters to the tensile strength is that: C>A>B>D. Combined with the above analysis, the optimal hot pressing process parameters are A1B1C3D3.

3.3 Microstructure

As shown in the Figure 3, there are flax fiber on the section morphology of the specimens after tensile, three microscopic pictures of tensile sections with different modification times were used as controls. It can found that more matrix attached to the surface of flax fiber with the extension of the modification time with the coupling agent. Because the coupling agent modification changes the functional group on the surface of the fiber, which made the fiber and matrix have better bonding.

In addition to modification time with the coupling agent, hot pressing temperature and pressure also have a great influence on microstructure, as shown in the Figure 4. No. 3 has more pressure than No. 1, and the gap between the fiber and matrix is smaller, so the combination is closer. No. 8 has a higher temperature than No. 1, and the gap between the fiber and matrix is smaller, so the combination is closer. From the above observation, it found that the PE matrix can be more easily filled into the fiber bundle in the higher hot pressing temperature and pressure. Because the higher temperature reduces the viscosity of the matrix, it has better mobility, and under the higher pressure, it is easier to immerse into the inside of the fiber bundle and reduce the internal gap of the fiber bundle.

Table 4. Summary of ANOVA results for tensile strength

	DF	Seq SS	Adj SS	Adj MS	F	P (%)	p-Value
A	1	2.1814	2.1814	2.1841	109.20	10.58	0.061
B	2	2.1485	2.1485	1.0742	53.71	10.42	0.096
C	2	15.3617	15.3617	7.6808	384.04	74.54	0.036
D	2	0.8978	0.8978	0.4489	22.45	4.36	0.148
Error	1	0.02	0.02	0.02		0.10	
Total	8	20.6120	20.6120			100	

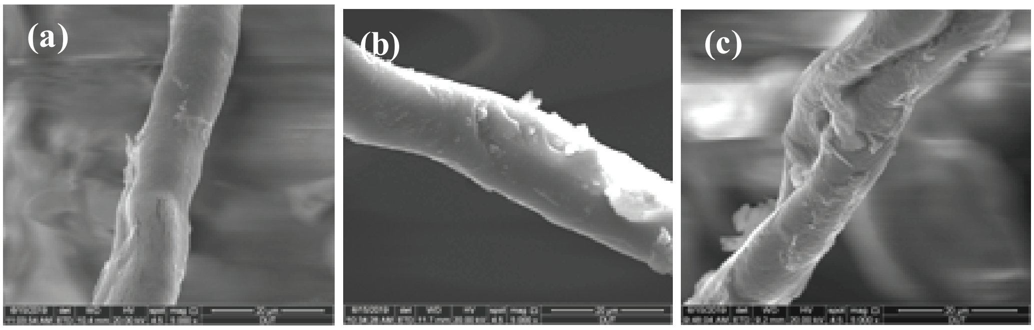

Figure 3. SEM images of flax fiber in composite (a) no modification, (b) with 0.50 h modification, and (c) with 2.00 h modification by the silane coupling agent

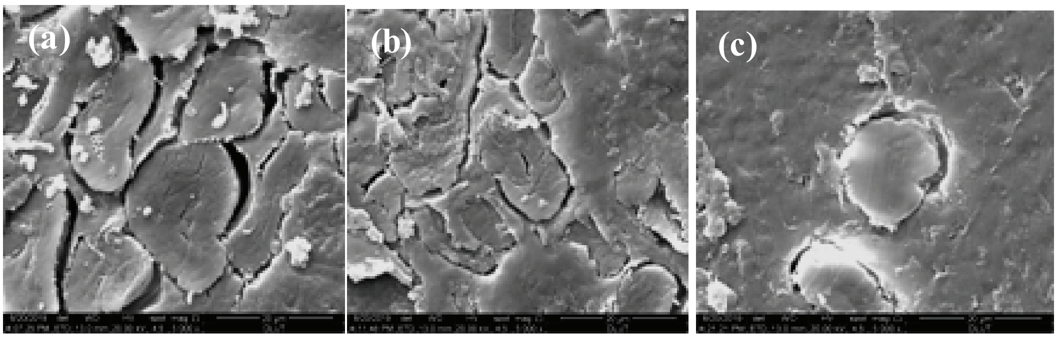

Figure 4. SEM images of composite cross section prepared at different processing conditions: (a) No. 1, (b) No. 3, and (c) No. 8

3.4 Verification experiment

Verification experiments are very important in engineering analysis. The optimization results can be verified by the four hot pressing parameters. The verification experiment was carried out by using the best hot pressing process A1B1C3D3. To obtain the maximum tensile strength produced by the optimization process. Base on the systematical and scientific study, the tensile strength of the flax/PE thermoplastic composite material made by other researchers were about 20MPa [21-22], and it was improved to 27MPa, which was increased more than 30% by our optimization method. Results show that the hot pressing process parameters can be optimized effectively by the Taguchi method. It can effectively improve the tensile strength of flax fiber thermoplastic composite.

4. Conclusion

The Taguchi method was proposed to determine the optimal hot pressing process parameters which improved the tensile strength of the test composite specimen. In total, nine experiments were performed using an L_9 (3*4) orthogonal matrix, four hot pressing parameters were chosen as design parameters, and each hot pressing parameter had three levels. Besides, the S/N ratio and ANOVA were calculated for the optimization. The validation experiment was performed to verify the best parameters which can improve tensile strength. Through the analysis of the optimization of the hot pressing process, the following conclusions can be drawn:

(1) Through the analysis of the S/N ratio, the optimal hot-pressure parameters are suggested as: the hot pressing temperature is 120 °C, the hot pressing time is 0.25 h, and the hot pressing pressure is 2.00MPa. The silane coupling agent modified the flax fiber for 2.00 h. Analysis of variance results showed that, the contribution of the four factors to the tensile strength of the panel is: C>A>B>D, combined noise ratio analysis, the final hot pressing optimization parameter is: A1B1C3D3.

(2) The optimal hot pressing process parameter verification results show that, compared to the average tensile strength, the tensile strength under the optimal parameters is improved about 10%. The Taguchi method effectively increase the tensile strength of flax fiber thermoplastic composite.

(3) Combined with hot pressing parameters and microstructure pictures, as can be seen, coupling agent modification can enhance the degree of fiber and matrix bonding, so it make the fiber surface have more matrix adhesion. The temperature and pressure work together to fill the PE matrix into the fiber bundle, high temperature reduces the viscosity of the matrix, so that it has better mobility, and under the larger pressure, it is easier to immerse into the inside of the fiber bundle. The hot pressing time has little effect on the performance of the specimen.

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