The Effects of Processing Parameters and AC Foaming Agent on the Mechanical Properties and Morphology of Foamed Wood-Polylactic Acid (PLA) Composites

Zhenghao Ge,* Zhi Qi, Dange Si, and Min Yu

Wood flour, PLA, and other additives were mixed evenly in a high speed mixing machine. The granules were prepared by melt blending and extrusion granulating with a twin-screw extruder, and test specimens were molded by a plate curing machine. By changing the heating temperature, the molding pressure, the holding pressure time, and azodicarbonamide (AC foaming agent) contents, the influences of four factors on the apparent density, the mechanical properties, and the morphology of the biodegradable foamed WPCs were investigated. The best processing parameters and the optimum AC foaming agent content were obtained. When heating temperature was 178 °C, heating time was 10 min, holding pressure time was 25 s, and molding pressure was 7 MPa, the test specimen was lighter in color, with a smooth surface and dense, uniform cross section. The mechanical properties (flexural strength and impact strength) of the foamed WPCs were relatively good. When adding 1% AC foaming agent, the foamed WPCs showed uniformly distributed microcellular structure, and the average pore diameter was about 67 μm. The density was reduced by 18.6%, and the flexural strength and impact strength were increased by 128.6% and 40%, respectively, compared with non-foamed WPCs.

Keywords: Biodegradable; Processing parameters; Foaming agent; Mechanical properties; Morphology

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INTRODUCTION

Thermoplastic and thermosetting plastic as the matrix of wood plastic composite materials are widely used in many areas, such as the construction industry, packaging industry, and automotive interiors (Migneault *et al.* 2009; Yemele *et al.* 2010). These composites have the advantages of light weight, high strength, low maintenance, and relatively low costs.

Wood plastic composites (WPCs) consist mainly of a plastic matrix enhanced with wood flour and other additives using the appropriate processing methods. The WPCs can be easily processed into various products and recycled. As advanced composites, WPCs derive favorable properties from their components, wood and plastic. Although the wood plastic composites have so many advantages, the waste of these composite materials produces a large amount of toxic substances and damages the environment (Lee 2008). Thus, biodegradable composite materials are needed to replace the traditional composites. There has been research into degradable plastics and biodegradable composite materials (Tserki *et al.* 2006; Belhassen *et al.* 2009). Polylactic acid (PLA) is a biodegradable polymer with the good mechanical properties, thermal plasticity, and biocompatibility. When it is used alone, PLA is a comparatively brittle and stiff polymer with low

deformation at break, and the cost is relatively high. Therefore, incorporation of cheap natural fibers as reinforcement filler into biodegradable polymer is an alternative way to reduce costs. With a wide range of sources, renewable, low cost, natural plant fiber composite can improve the comprehensive performance while reducing costs. Their waste in the soil will be degraded by microorganisms into CO_2 and H_2O without leaving any toxic substances. These biodegradable composite materials are helpful to protect environment and realize the harmonious development between man and nature.

In order to manufacture fully degradable biocomposite foams with enhanced final mechanical properties, a few researchers have investigated PLA foaming behavior using several natural fibers/additives, which have included flax fiber, silk fibroin powder, wood flour, and microfibrillated cellulose (MFC). Pilla *et al*. (2009) investigated polylactide–flax fiber composites with 1, 10, and 20 wt% fiber, which were melt-compounded and subsequently molded *via* the conventional and microcellular injection-molding processes. Silane was used as a coupling agent to create a strong interface bonding between the PLA and the fibers. The foaming results showed that fiber content improved the cell morphology, static and dynamic mechanical properties, and crystallization properties. Kang *et al.* (2009) also investigated PLA bio-degradable composites that were reinforced with various silk fibroin powder contents (0, 1, 3, 5, and 7 wt%) and were prepared by solution processing technique using CH_2Cl_2 as solvent and the composites were foamed by using supercritical CO_2. The composite foams exhibited a reduction in cell size and increase in cell density at high silk content. However, the saturation temperature and pressure greatly affected cell density and foam density of composite foams. Matuana *et al.* (2010) studied poly(lactic acid) (PLA) and PLA/wood-flour composites that were microcellular foamed with CO_2 through a batch foaming process. Silane was also used as the coupling agent. They reported that an increase in the wood flour content reduced the expansion ratio from around 10-fold in neat PLA to 2-fold in PLA-40 wt% wood flour due to the matrix's high stiffness. Boissard *et al.* (2012) describes the production of ''green'' microfibrillated cellulose-reinforce polylactide cellular biocomposites using a wet mixing technique combined with supercritical carbon dioxide foaming. Similar to the previous study, the expansion ratio of the neat PLA was reduced from 6.8-fold to 3.8-fold in PLA with 5 wt% MFC. The compression modulus of these samples was also increased from 25 MPa in the neat PLA to 47 MPa in PLA with 5 wt% MFC.T. Oksman *et al.* (2003) investigated compression molded PLA–flax fiber composites and found that the tensile strength and stiffness increased with a 30% fiber loading. However, at a fiber loading of 40%, these same parameters decreased. This was attributed to poor dispersion of flax fiber within the PLA matrix.

In other studies, different blowing agents were applied during the extrusion foaming of PLA. Zhang and Sun (2007a,b) examined the extrusion behavior of PLA/starch blends using water as a foaming agent. They showed that cell sizes as large as 500 μm were induced, although an expansion ratio of over 50-fold could be achieved. Yuan *et al.* (2009) and Matuana *et al.* (2009) used a chemical blowing agent as the driving force for foaming. Both of their studies obtained PLA foams with fine cells that had average cell sizes of 10-20 μm and cell densities of between 106 and 108 cells/cm^3. However, the foam expansion ratio did not exceed 3-fold.

In this work, wood flour and biodegradable plastic PLA were used as the two main raw materials, in combination with azodicarbonamide (AC foaming agent) and various additives (Farsheh *et al.* 2011; Mengeloglu and Karakuş 2012), to make an eco-friendly biodegradable foamed WPC that was molded on a plate curing machine (Kalia and Kaith

2009). The effects of heating temperature, molding pressure, holding pressure time (Iannace *et al.* 2007), and AC foaming agent contents on the apparent quality, mechanical properties, and morphology of the biodegradable foamed WPCs were examined.

EXPERIMENTAL

Raw Materials

PLA grade 3251D from NatureWorks LLC (USA) was used as the matrix. This PLA was supplied in pellets with a MFI of 80 g/10 min (190 °C/2.16 kg) and a density of 1.40 g/cm^3. Maple WF was purchased from Shandong Dragon Wood Flour Factory (Shandong, China) and was mechanically sieved to keep only particles between 150 and 250 µm. Azodicarbonamide (Celogen 754A from Lion Copolymer, USA) was used as the exothermic chemical foaming agent (CBA). This foaming agent has a decomposition temperature range of 165 °C to 180 °C and a gas yield of 200 cm^3/g. Dioctyl phthalate (DOP) from Tianjin Jet-set Biochemical Technology Co., Ltd. (Tianjin, China) is as a colorless transparent liquid plasticizer. Zinc stearate, a lubricant and mold discharging agent, white powder, was obtained from Chemical Reagent Factory of Tianjin Development Zone. Superfine talc powder, a lubricant, white powder, was from Laizhou Shuanglong Powder Factory.

Main Equipment

The equipment employed in this work included a Plate Curing Machine, XLB-D, Huzhou CIS Rubber Machinery Co., Ltd. (Huzhou, China) ltd; high speed mixer, Zhang Jiata Shengguang Degradation Plastic Machinery Factory; High Speed Mixer, Zhejiang Ronghao Industry and Trade Co., Ltd. (Zhejiang, China); electronic scale, 202-2, Shanghai Experimental Instrument General Factory; tweezers, commercially available; drying box, 101A-1B, Experimental Instrument Factory of Shanghai City, The People's Republic of China; Flexing Testing Machine, CMT5105, Shenzhen Sans Material Detection Co Ltd; Cantilever Beam Impact Testing Machine, XJU-22, Junrui Instrument Equipment Co. Ltd. (Chengde, Country); and scanning electron microscope (SEM), FEI Q45, FEI (Hillsboro, USA)

Methods

Sample preparation

White transparent PLA particles were fully crushed into 80 mesh in a high speed, multi-function mill. According to specified proportions of raw materials, 80 mesh PLA, 80 mesh wood flour, MAPLA, AC foaming agent, DOP, stearic acid zinc, and lubricant were mixed uniformly into the high speed mixer at a speed of 1500 rpm and a temperature of 120 °C for 15 min.

On the plate curing machine, the test specimen was prepared by the hot pressing molding process. The mixed material was placed into the metal mold cavity, and the mold was placed on the plate that had been heated to make the upper and lower plate close to the mold. By setting different processing parameters, the biodegradable foamed WPCs with the best apparent quality and mechanical properties were obtained.

The composite preparation and the experimental processes are shown in Fig. 1.

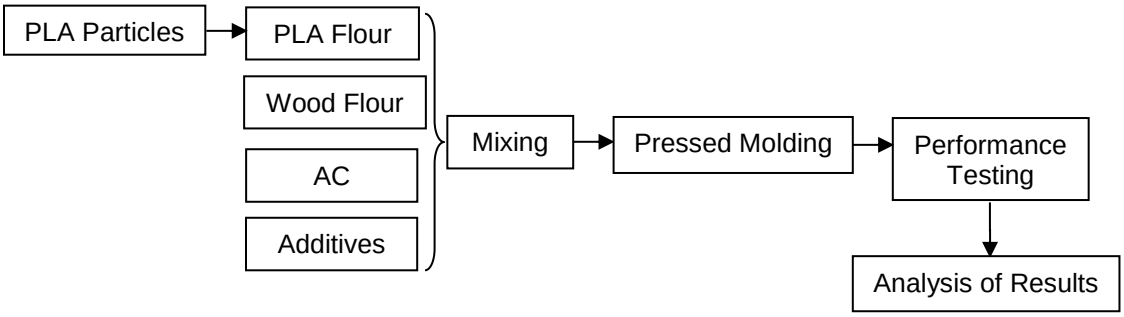


Fig. 1. The preparation process and the experimental process of the composite materials

Density measuring and calculating

In this experiment, the density of biodegradable foamed WPCs in the different factors were compared. An electronic scale with a precision of 1/1000 was used. The same group of experimental samples (three to four) without defects in quality or appearance were selected and weighed several times to obtain the average weight. The density of the material was calculated by Eq. 1,

$$\rho = M / V \quad (1)$$

where ρ is the density of materials (g/cm^3), M is the quality of materials (g), and V is the volume of materials (cm^3).

Mechanical testing

The mechanical testing was conducted according to ASTM standards. the bending strength was measured at the flexural speed of 2 mm/min according to the ASTM D790; the impact strength was measured according to the ASTM D256.

Scanning electron microscopy (SEM)

Scanning electron microscopy was used to monitor the microcellular structure of biodegradable foamed WPCs. Before using the SEM, the cross-sections of samples (1 × 1 × 1 cm) were smoothed by the sliding microtome. The samples were sputtered with a layer of gold before imaging. The microcellular structure of foamed WPCs were photographed by SEM (Ghanbar *et al.* 2014).

Evaluation of test results

Mechanical test results reflect statistical significance at the 95% confidence level. The optimum molding process conditions are obtained by using the orthogonal experiment range analysis method.

RESULTS AND DISCUSSION

The Effect of Single-factor Parameter on the Properties of Foamed WPCs

The WPCs had the following components: wood flour, 15%; PLA, 85%; MAPLA, 4%; AC foaming agent, 1%; lubricant, 2%; DOP, 2%; and zinc stearate, 1%. The heating time of the plate curing machine was 10 min.

Heating temperature

The heating temperature was used as the single factor variable (Guo *et al.* 2004), and other factors were set as invariants (molding pressure, 7 MPa; holding pressure time, 20 s; sample length, 120 mm; and sample width, 17 mm). The experimental results are shown in Table 1.

Table 1. Heating Temperature as the Single-Factor Variable and Results

Heating Temperature (°C)	Thickness (mm)	Mass (g)	Density (g/cm^3)	Bending Strength (MPa)	Impact Strength (kJ/m^2)
174	10.28	16.51	0.787	31.41	2.11
176	10.25	15.17	0.725	41.21	3.44
178	10.15	12.27	0.593	92.44	7.02
180	10.44	13.70	0.643	79.18	3.29
182	10.43	14.10	0.663	70.68	2.69

Figures 2 and 3 illustrate the trends in density, bending strength, and impact strength with increasing heating temperature. The density of foamed WPCs decreased first and then increased with the increase in the heating temperature; the bending strength and impact strength of foamed WPCs increased first and then decreased with the increase in the heating temperature.

When the heating temperature was 178 °C, the lowest density of foamed WPCs was 0.593 g/cm^3, the highest bending strength was 92.44 MPa, and the highest impact strength was 7.02 kJ/m^2. The test specimen was lighter in color, with a smooth surface and dense, uniform cross section. When the heating temperature was less than 178 °C, it did not reach the melting temperature of PLA, *i.e.*, the raw material was not completely melted. The combination of PLA and wood flour interface was so poor that the mechanical properties were reduced.

Above 178 °C, the raw material was placed in the mold for a long time, and partial PLA hydrolysis occurred. Subsequently, large bubble hole defects appeared in the test specimen, and the mechanical properties of foamed WPCs were decreased.

These phenomena can be explained as follows. The setting of the heating temperature was closely related to the melting temperature of plastic. When the heating temperature increased, the fluidity and melt viscosity of PLA increased. The distribution of wood flour in plastic was uniform, and the ability of two-phase bonding increased. At increased temperature, the AC foaming agent decomposed to ammonia gas. When the temperature was too high, partial PLA hydrolysis and wood flour carbonization occurred, and the specimen contained serious defects.

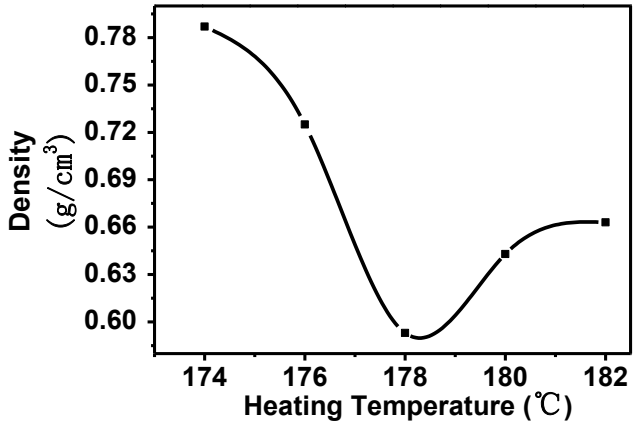


Fig. 2. The effect of heating temperature on the density of foamed WPCs

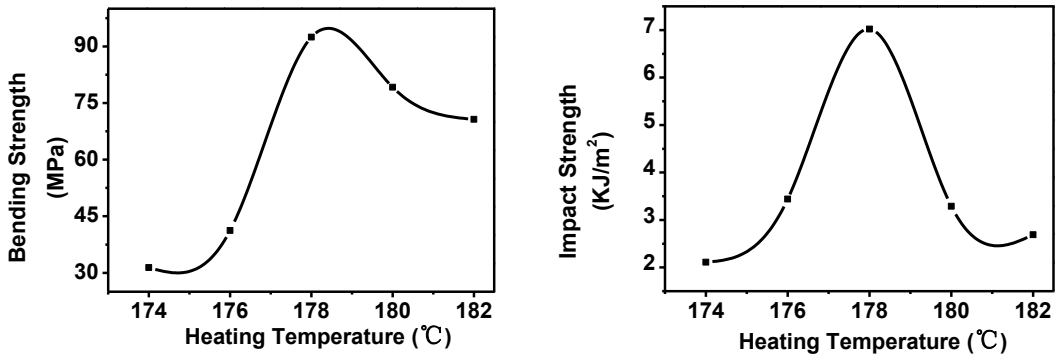


Fig. 3. The effect of heating temperature on the mechanical properties of foamed WPCs

Molding pressure

When the molding pressure was the single factor variable, the other factors were invariant (heating temperature, 178 °C; holding pressure time, 20 s; sample length, 120 mm; and sample width, 17 mm). The experimental results are shown in Table 2.

Table 2. Molding Pressure as the Single-Factor Variable and Results

Molding Pressure (MPa)	Thickness (mm)	Mass (g)	Density (g/cm^3)	Bending Strength (MPa)	Impact Strength (kJ/m^2)
6	10.31	14.74	0.701	62.31	1.51
6.5	10.25	14.37	0.687	84.05	2.78
7	10.20	13.74	0.660	92.45	7.02
7.5	10.10	13.57	0.659	87.32	2.24
8	10.43	14.10	0.663	59.92	2.07

Figures 4 and 5 show the changes in density, bending strength, and impact strength with increasing molding pressure. The density of foamed WPCs decreased first and then increased with increasing molding pressure.

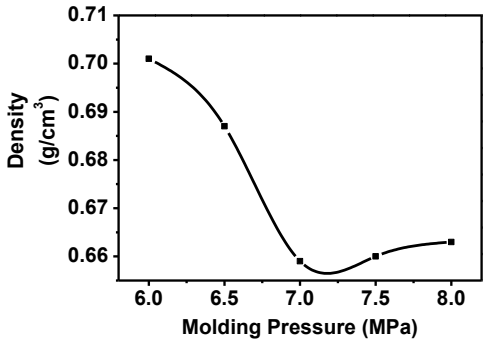


Fig. 4. Effect of molding pressure on the density of foamed WPCs

The bending strength of foamed WPCs remained steady at first and then decreased with increased molding pressure; the impact strength of foamed WPCs increased first and then decreased with increased molding pressure. When the molding pressure was 7 MPa, the lowest density of foamed WPCs was 0.660 g/cm^3. The highest bending strength of foamed WPCs was 92.45 MPa; the highest impact strength of foamed WPCs was 7.02 kJ/m2. Thus, molding pressure had a large effect on the bending strength and the impact strength of the samples. The main role of the molding pressure compacted melt material to fill the entire mold. Too much pressure compacted the samples; the internal foam hole was reduced, which led to decreased mechanical properties.

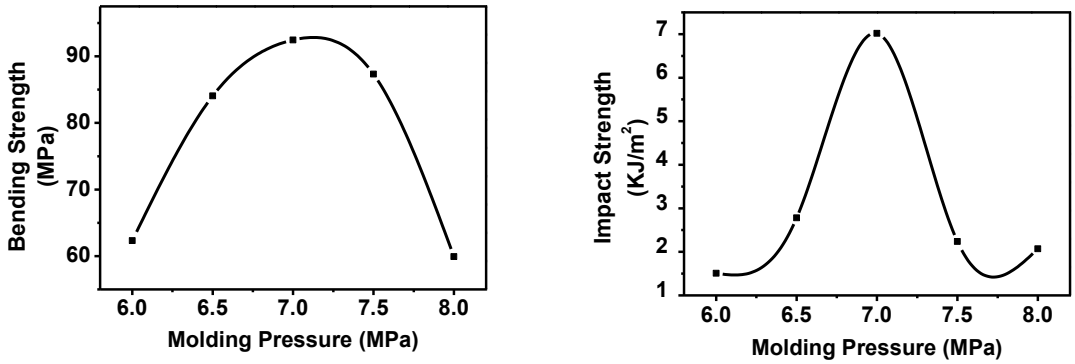


Fig. 5. Effect of molding pressure on the mechanical properties of foamed WPCs

Holding pressure time

In this set of tests the time of holding the pressure was the only factor that was varied; the other factors were invariant (heating temperature, 178 °C; molding pressure, 7 MPa; sample length, of 120 mm; and sample width, 17 mm). The experimental results are shown in Table 3.

Table 3. Holding Pressure Time as the Single-Factor Variable and Results

Holding Pressure Time (s)	Thickness (mm)	Mass (g)	Density (g/cm^3)	Bending Strength (MPa)	Impact Strength (kJ/m^2)
15	10.10	14.18	0.688	47.11	3.86
20	10.43	14.10	0.663	92.45	7.02
25	10.01	12.61	0.618	123.94	10.82
30	10.35	12.90	0.611	39.44	10.26
35	10.43	13.21	0.621	35.28	9.41

Figures 6 and 7 illustrate the trends in the density, bending strength, and impact strength with increasing holding pressure time. The density of foamed WPCs decreased with increased holding pressure time. The bending strength increased first and then decreased with the increase of the holding pressure time; the impact strength increased first and then kept steady with the increase of the holding pressure time. When the holding pressure time was 25 s, the lowest density of foamed WPCs was 0.618 g/cm^3. The highest bending strength of foamed WPCs was 123.94 MPa; the highest impact strength of foamed WPCs was 10.82 kJ/m^2.

These results can be explained as follows. The holding pressure time was also one of the important processing parameters affecting the apparent quality and mechanical properties of specimens. When the holding pressure time was less than 25 s, the raw material stayed in a short time in the mold and did not completely foam. This led to high density and low mechanical properties. When the holding pressure time was excessively long (more than 25 s), partial PLA hydrolysis occurred, and the foams collapsed. This reduced the mechanical properties of the foamed WPCs.

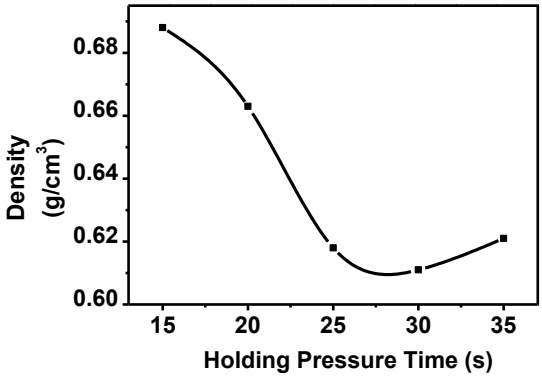


Fig. 6. The effect of holding pressure time on the density of foamed WPCs

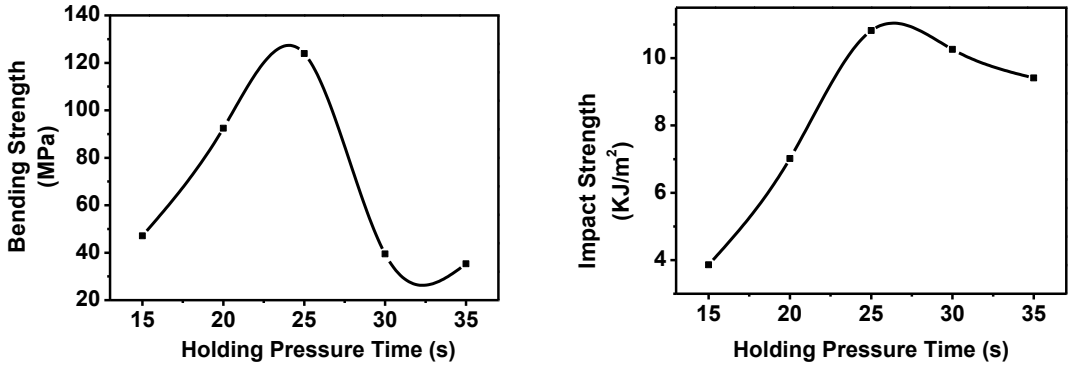


Fig. 7. The effect of holding pressure time on the mechanical properties of foamed WPCs

Performance Analysis of WPCs Based on the Orthogonal Experiment

The effects of the processing parameters on the comprehensive properties of the biodegradable foamed WPCs were studied by the orthogonal experiment. By changing the heating temperature, the molding pressure, and the holding pressure time, the optimum preparation conditions were selected. The results are shown in Table 4.

Table 4. Orthogonal Experimental Processing Parameters and Results

Number	A (°C)	B (MPa)	C (s)	Indexes		
				Density (g/cm^3)	Bending Strength (MPa)	Impact Strength (kJ/m^2)
1	178	6.5	15	0.601	55.83	3.16
2	178	7	20	0.663	42.76	2.08
3	178	7.5	25	0.708	97.31	5.41
4	180	7	15	0.672	69.91	5.22
5	180	7.5	20	0.623	50.19	3.74
6	180	6.5	25	0.598	63.51	2.65
7	182	7.5	15	0.537	92.45	1.53
8	182	6.5	20	0.513	38.67	1.99
9	182	7	25	0.500	67.65	7.02
*A is heating temperature, B is molding pressure, C is holding pressure time.						

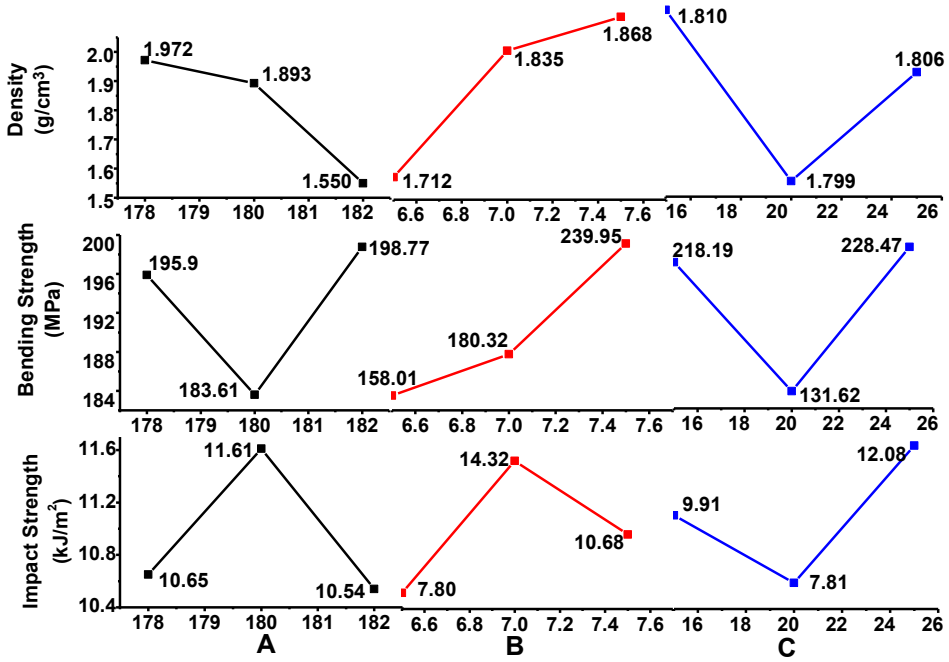


Fig. 8. Range analysis of the orthogonal experiment

Figure 8 illustrates the range analysis of the orthogonal experiment. The following interpretations can be made based on the range analysis techniques for the effects of processing parameters on the apparent density and mechanical properties of biodegradable foamed WPCs.

(1) For the density of foamed WPCs, the range value of the heating temperature was maximum, which indicates that the heating temperature was the main factor affecting the density of foamed WPCs. The best condition was $A_1B_3C_1$ for the density;

(2) For the bending strength of foamed WPCs, the range value of the holding pressure time was maximum, which indicates that the holding pressure time was the main

factor affecting the bending strength of foamed WPCs. The best condition was $C_3B_3A_3$ for the bending strength;

(3) For the impact strength of foamed WPCs, the range value of the molding pressure was maximum, which indicates that the molding pressure was the main factor affecting the impact strength of foamed WPCs. The best condition was $B_2C_3A_2$ for the impact strength .

Overall, the optimum processing conditions were a heating temperature of 178 °C, molding pressure of 7 MPa, and holding pressure time of 25 s.

The Effect of AC Content on Properties and Morphology of Foamed WPCs

The properties of foamed WPCs

In this set of tests the AC foaming agent content was the single factor variable (Bledzki and Faruk 2005), and the other factors were invariant (heating temperature, 178 °C; molding pressure, 7 MPa; holding pressure time, 25 s; sample length, 120 mm; and sample width, 17 mm). The results are shown in Table 5.

Table 5. AC Foaming Agent Contents and Results

AC Content (%)	Thickness (mm)	Mass (g)	Density (g/cm^3)	Bending Strength (MPa)	Impact Strength (kJ/m^2)
0	9.98	14.42	0.708	44.81	3.87
0.5	9.89	13.20	0.654	97.31	4.54
1	9.92	12.09	0.597	102.43	5.41
1.5	9.97	13.08	0.643	30.73	4.43

Figures 9 and 10 illustrate the changes in density, bending strength, and impact strength with increasing AC foaming agent contents. The density of foamed WPCs decreased first and then increased with increased AC foaming agent content; the bending strength and the impact strength increased first and then decreased with increased AC foaming agent content. When the AC foaming agent content was 1%, the lowest density of foamed WPCs was 0.597 g/cm^3, which was reduced by 18.6% compared with the non-foamed WPCs. The highest bending strength of foamed WPCs was 102.4 MPa, which was increased by 128.6% compared with non-foamed WPCs; the highest impact strength of the foamed WPCs was 5.41 kJ/m^2, which was increased by 40% compared with non-foamed WPCs.

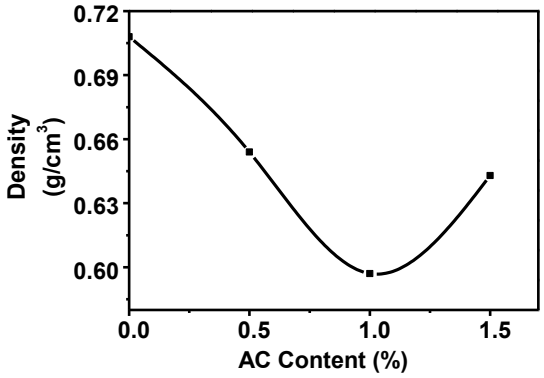


Fig. 9. The effect of AC content on the density of foamed WPCs

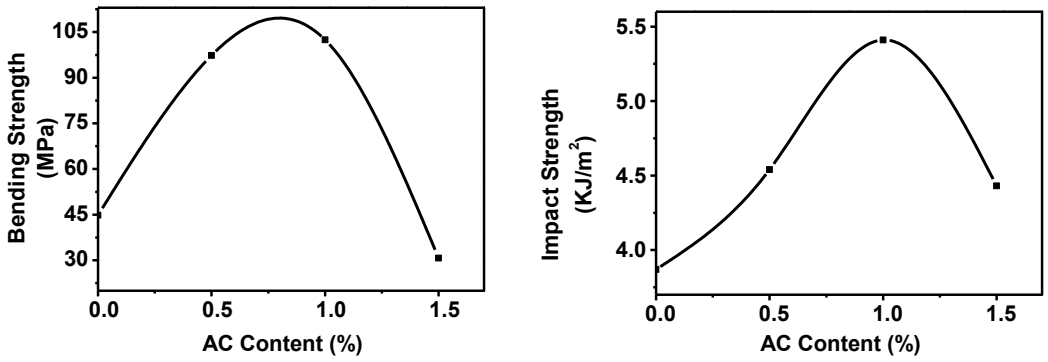
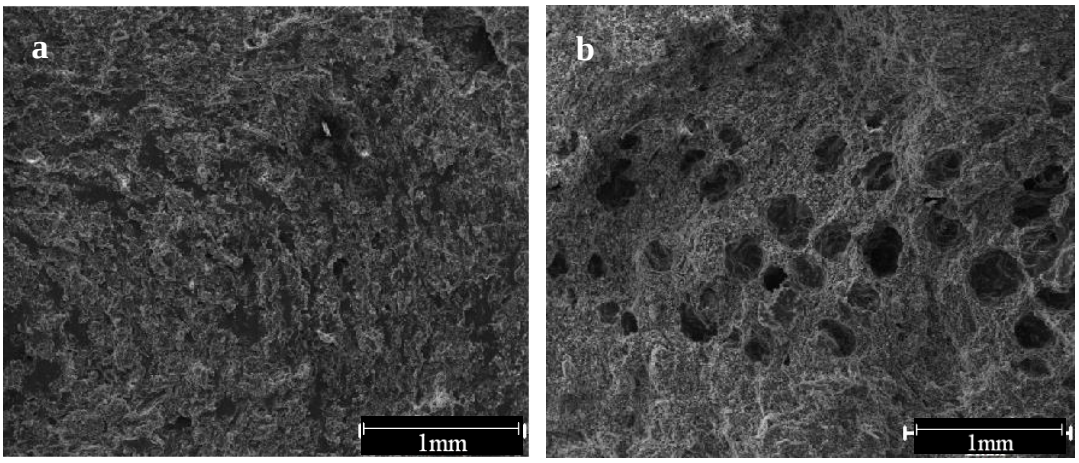


Fig. 10. The effect of AC content on the mechanical properties of foamed WPCs

The experimental process was sufficiently random that partial data was inaccurate. However, when AC foaming agent content increased from 0% to 1%, AC chemical reactions occurred that released large amounts of gas, which generated a large number of bubbles *via* nucleation. Larger cell structures led to decreased density and increased mechanical properties. When AC content exceeded 1%, too much foaming agent reached the saturation state. The melt strength could not withstand the excessive pressure, and the gas gradually escaped. This may cause collapse and burst of the foams, leading to decreased mechanical properties.

The morphology of foamed WPCs

Cell morphology, distribution, and size were observed in SEM micrographs (Fig. 11). Fracture surfaces of the foamed WPCs were examined (Xu *et al.* 2005), while also evaluating the influence of AC foaming agent contents on the foamed WPCs. Figure 11 shows fracture surfaces of samples containing 0%, 0.5%, 1%, and 1.5% of AC foaming agent.



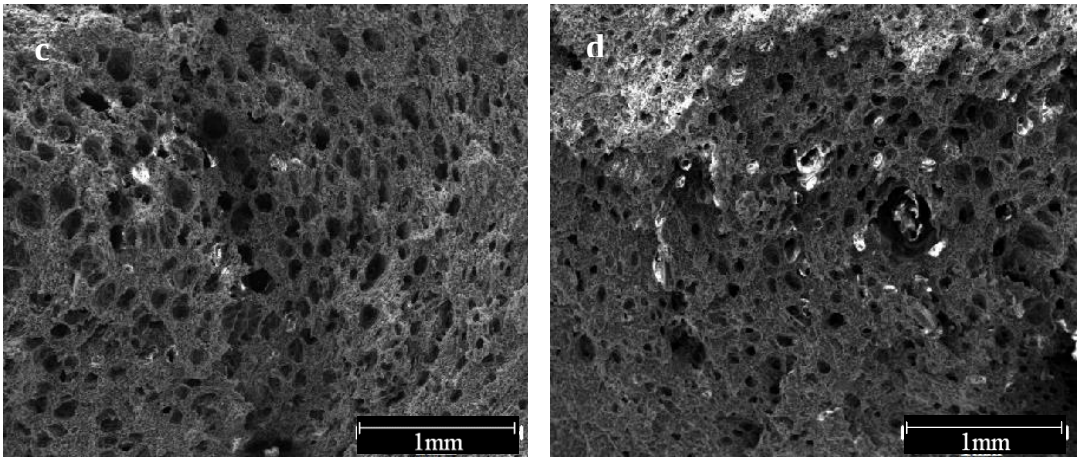


Fig. 11. The SEM images of foamed wood-PLA composites. a) 0% AC foaming agent; b) 0.5% AC foaming agent; c) 1% AC foaming agent; d) 1.5% AC foaming agent.

With 0.5% AC foaming agent, the cell distribution was not homogeneous, and the samples had larger cell structures (Fig. 11b). This result was attributed to less AC foaming agent content, resulting in inadequate foaming, which also led to higher density and lower mechanical properties. With 1.5% AC foaming agent, the cell structure was small and distributed uniformly, but large bubble holes defects appeared (Fig. 11d). This was due to the excessive AC foaming agent, which produced too much pressure over the melt pressure. This may cause collapse and burst of the foams, as well as big bubble hole defects, which reduced the mechanical properties of foamed WPCs. When adding 1% AC foaming agent, the foamed WPCs showed uniformly distributed microcellular structure, and the average pore diameter was 67 μm (Fig. 11c). Thus, the optimum AC foaming agent content was 1%.

CONCLUSIONS

In the study, the effects of processing parameters and AC foaming agent on the mechanical properties and morphology of foamed wood-polylactic acid (PLA) composites were investigated. Based on the orthogonal experimental results, the optimum conditions were a heating temperature of 178 °C, molding pressure of 7 MPa, and holding pressure time of 25 s. The test specimen was light in color, with a smooth surface and dense, uniform cross section. The mechanical properties were relatively good. When adding 1% AC foaming agent, the lowest density was 0.597 g/cm^3, which was reduced by 18.6% compared with non-foamed WPCs. The highest bending strength of foamed WPCs was 102.4 MPa, which was increased by 128.6% compared with non-foamed WPCs; the highest impact strength of the foamed WPCs was 5.41 kJ/m2, which was increased by 40% compared with non-foamed WPCs. Finally, the optimum processing parameters and content of AC foaming agent were attained, which was shown to improve the specific mechanical properties of WPCS.

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