Improving the Performance of Eucalyptus Wood Particle Board Panels with Low Free Formaldehyde Emission Urea-formaldehyde Resin Using Pectinase Enzyme Pre-treatments

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The potential for using the enzyme pectinase as a pre-treatment to improve the properties of eucalyptus particle-based panels was investigated at different pre-treatment times, temperatures, and particle-to-enzyme ratios. As the pre-treatment time was increased from 10 min to 30 min, the free formaldehyde emission content was reduced ($P < 0.0001$). The pectinase enzyme pre-treatment reduced the pectin content and increased the permeability of treated wood, allowing more free formaldehyde to be released from the panels. The free formaldehyde emission content of all panels was lower than 3.0 mg per 100 g, due to the kind of urea-formaldehyde (UF) resin used. When the pre-treatment time was 30 min and the temperature was 45 °C, the mechanical properties, including modulus of elasticity (MOE), modulus of rupture (MOR), and internal bonding strength (IB), of the resulting panels were the best among all the selected treatment times and temperatures. As the ratio of particles to solution was reduced from 1:100 to 1:80 or 1:60, the mechanical properties of the particle board panels were improved ($P < 0.0001$). This was attributed to the pectinase enzyme pretreatment changing the surface of the particles, resulting in a better interface between UF resin and particles.

Keywords: Particle board panel; Pectinase enzyme; UF resin; Formaldehyde emission; Mechanical properties

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INTRODUCTION

Wood-based panels are one kind of woody material that can be used to make furniture, interior decorations, wooden buildings, *etc.* due to their good performance and efficiency (Saad *et al.* 2017). Particle board panel is an important kind of wood-based composite (Huang *et al.* 2015; Rofii *et al.* 2016). It can be used to make furniture, and its global output was about 110.94 million m^3 in 2015 (FAO). Eucalyptus (*E. grandis* × *E. urophylla*) wood is an important raw material (Stackpole *et al.* 2011; Fernández-Fernández *et al.* 2014) for wood-based panels, including the particle and fiber board panels in Yunnan Province, China, due to its fast growth. Urea-Formaldehyde (UF) resin is an important kind of adhesive that has been used widely in wood industry (Li *et al.* 2017c; Zhong *et al.* 2017) due to its low cost, strength, and ease in manufacturing. One key problem with UF resin though is reducing and avoiding the hazardous free formaldehyde emissions from the panels (Jin *et al.* 2017). Thus, in this research one kind of low free formaldehyde emission UF resin was used, satisfying the E_0-grade requirement (< 3.0 mg/100g) for free formaldehyde emission (Plywood Inspection 2017). Pectinase can be used to degrade the pectin (Li *et al.* 2017a) in wood cell walls (Table 1), especially in the middle lamella and primary cell walls (Palin and Geitmann 2012; Deher and Braybrook 2015) of biomass materials, to enhance the properties of composites (Saleem *et al.* 2008; Kalia *et al.* 2013; Mamun and Bledzki 2013; George *et al.* 2016; Li *et al.* 2017b). The permeability of wood can also be increased by the pectinase enzyme treatment due to the degradation of the pectin covering the pits on the cell walls (Militz 1993; West *et al.* 2012; Durmaz *et al.* 2015). There is little information, though, about the influence of pectinase enzyme pre-treatments on the properties of the particle board panels.

Table 1. Effect of Pectinase Pre-treatments on Mass Loss and Cell Wall Polymer Content of Eucalyptus Wood Fibers [a]

Material	Treatment	Time (min)	Mass Loss (%)	Pectin (%)	Klason Lignin (%)	Holo-cellulose (%)	α-cellulose (%)	Hemi-cellulose (%)
Eucalyptus Wood	None	–	–	1.85 (0.02)	24.4 (0.05)	66.6 (0.18)	43.3 (0.10)	23.3 (0.21)
	Pectinase	30	6.60 (0.03)	1.61 (0.02)	26.0 (0.06)	67.5 (0.03)	43.6 (0.03)	23.9 (0.04)
		60	6.99 (0.03)	1.01 (0.02)	26.4 (0.02)	68.0 (0.03)	43.9 (0.02)	24.1 (0.07)
		90	7.91 (0.01)	0.72 (0.02)	26.7 (0.03)	69.3 (0.02)	44.9 (0.02)	24.4 (0.03)
		120	8.04 (0.03)	0.65 (0.02)	27.6 (0.07)	69.7 (0.03)	45.1 (0.01)	24.6 (0.03)

[a] Values represent means of 3 replicates per treatment, while figures in parentheses represent one standard deviation (Li *et al.* 2017a).

The objectives of this research were to investigate the effect of pectinase enzyme pre-treatments at different conditions on the performance of wood-based particle board panels with low free formaldehyde emission UF resin to provide information for manufacturing particle board panels with better properties.

EXPERIMENTAL

Woody Particles

Eucalyptus (*E. grandis* × *E. urophylla*) obtained from commercial sources was chipped to no more than 1 mm in thickness and oven dried at 104 °C until the moisture content was about 3%, then stored in plastic bags until use.

Urea-Formaldehyde (UF) Resin

A UF resin adhesive at a pH of 9.0, a solids content of 60 to 61%, and a low free formaldehyde content of 0.12% was used. The UF resin was provided by Xinfeilin Wood-Based Panel Co. Ltd., Yunnan, Kunming, China. The UF resin is a new low-formaldehyde-emission-type resin, and it can satisfy the E_0 grade standard (free formaldehyde emissions lower than 5 mg/100 g).

Pectinase Enzyme

The pectinase enzyme was isolated from cultures of *Bacillus mojavensis* (Roberts *et al.* 1999). The resulting enzyme had a molecular weight of 39.3 kDa and optimum pH and temperature for activity of 5 to 5.5 and 45 to 50 °C, respectively. The resulting extract had an activity of 26.98 U/mL when tested according to previously described procedures (Albersheim 1966). The enzyme was stored at 4 °C until needed.

Particles Treatment by Pectinase Enzyme

One kg of eucalyptus particles (1 kg) was weighed and placed into the enzyme solution at a ratio of 1:100 particles to solution and incubated at different times temperatures, or at different ratios of particles to solution; specifics are shown in the following: (1) Incubation for 10, 20, 30, 40, 50, or 60 min at 50 °C; (2) Incubation for 15, 30, 45, 60, 75, or 90 min at 45 °C; (3) Incubation for 30 min at 30 °C, 35 °C, 40 °C, 45 °C, 50 °C, 55 °C, or 60 °C; and (4) One kg of particles (1 kg) was weighed and placed into the enzyme solution at a ratio of 1:20, 1:40, 1:60, 1:80, and 1:100 particles to solution (enough distilled water was added to the enzyme solution to make the ratio of particles to solution the desired ratio) and incubated for 30 min at 45 °C. The enzyme solution was decanted at the end of the given time period, and then the particles were dried at 104 °C until the moisture content was about 3.0%.

Panel Production

Panels were produced by blending 90% of a given particle material with or without a given pre-treatment, 10% UF resin (solid mass), and 1% NH_4Cl as catalyst based on the UF resin solid mass. The mixture was thoroughly blended at room temperature to evenly distribute the components before being formed into 250 by 250 by 10 mm thick mats that were pressed for 5 min at 150 °C to a target density of 0.78 g/cm^3; the actual density was about 0.75 g/cm^3 to 0.80 g/cm^3. Five panels were made for each mixture.

Panel Properties

The panels from each treatment were cut into 50 by 150 by 10 mm thick samples. Fifteen samples per treatment were tested to failure at three points with a loading rate of 3 mm/min on a Universal Testing Machine (Shimadzu AG-50KNI, Kyoto, Japan) according to the procedures described in EN 310 (1993). The resulting data were used to calculate the MOE and MOR. IB was assessed on 50 by 50 by 10 mm samples according to the procedures described in EN 319 (1993); 15 samples per treatment were tested for research. Free formaldehyde emissions were assessed using the perforator method according to procedures described in EN 120 (1993); 5 iterations per sample were performed.

The data were subjected to an analysis of variance, and then the means were examined using a Tukey's pairwise comparison test ($\alpha = 0.05$).

RESULTS AND DISCUSSION

Effect of Pre-treatment Time on Panel Properties

After the pectinase enzyme pre-treatments, eucalyptus samples were observed to have significant changes in MOE, MOR, IB, and free formaldehyde emissions, although there was no consistent trend with respect to treatment time (Table 2).

Table 2. Performance of Particle Board Panels Treated by Pectinase Enzyme for Different Times [a]

Treatment Time (min)	Treatment Temperature (°C)	Free Formaldehyde Emission (mg/100 g)	MOE (MPa)	MOR (MPa)	IB (MPa)
Control	50	2.60 (0.09) abc	2999 (207) bc	15.1 (1.31) a	0.51 (0.09) c
10 min	50	2.52 (0.13) abc	3508 (397) a	15.6 (1.55) a	0.61 (0.08) ab
20 min	50	2.49 (0.08) abcd	3430 (257) ab	13.3 (0.57) b	0.58 (0.06) abc
30 min	50	2.16 (0.12) cd	3307 (307) ab	13.2 (0.63) b	0.56 (0.05) abc
40 min	50	2.62 (0.33) ab	3343 (364) ab	12.8 (0.70) b	0.53 (0.06) bc
50 min	50	2.65 (0.30) a	3059 (388) bc	12.8 (0.50) b	0.37 (0.03) d
60 min	50	2.57 (0.14) abc	2625 (299) bc	13.6 (0.85) b	0.65 (0.11) a
15 min	45	----	2600 (249) c	14.8 (1.22) bc	0.57 (0.09) ab
30 min	45	----	3242 (459) a	19.2 (2.74) a	0.61 (0.07) ab
45 min	45	----	2628 (169) bc	14.4 (1.40) bc	0.60 (0.11) ab
60 min	45	----	2666 (263) bc	14.3 (2.03) bc	0.60 (0.09) ab
75 min	45	----	2818 (289) bc	13.0 (1.42) bcd	0.64 (0.06) a
90 min	45	----	2429 (270) c	11.5 (1.38) cd	0.68 (0.07) a
Standard*	----	----	1800	11.0	0.4

[a] Values represent means of 15 replicates per treatment (5 replicates per treatment for free formaldehyde emission) while figures in parentheses represent one standard deviation. Values followed by the same letter(s) do not differ significantly from one another by Tukey's pairwise comparisons (α = 0.05); * Standard BS EN 312:2010; Requirements for boards for interior fitments (including furniture) for use in dry conditions (Type P2).

When the treatment time was 10 min at 50 °C, the free formaldehyde emission was reduced by about 3%, while the MOE and IB were both increased more than 15% compared to the control panels. When the treatment time was 30 min at 50 °C, the free formaldehyde emission was the lowest among all of the resulting panels. Due to all free formaldehyde emissions being lower than 5 mg per 100 g, the free formaldehyde emissions were not tested in later experiments. When the pectinase enzyme pre-treatment was performed at 45 °C and for 30 min, the MOE, MOR, and IB were all improved dramatically compared with the control panels, and these parameters were selected for further research. The change in properties is perhaps because the pectin is rich in the middle lamella and primary cell walls of the eucalyptus wood, and the pectinase enzyme can degrade the pectin in these regions (Li *et al.* 2017a), especially the pectin covering the surface of the pits on the cell walls, which can be seen in Fig. 1 (Militz 1993; West *et al.* 2012).

In Fig. 1 there were obvious differences between the non-treated particles (Fig. 1a) and treated particles (Fig. 1b). The surface of non-treated particles was rough; this might be because the particles were made by the chipper machine from wooden materials with a moisture content between 30 percent and 40 percent, and the cutting speed was very fast.

Therefore, many cell wall fragmentary were left on the surface of the particles and covered the pits on the fiber surface. After the particles were treated by the pectinase enzyme, the pectin that existed in the middle lamella were degraded and the cell wall fragmentary were taken off just like some fibers were taken off from the hemp chips (Li *et* al. 2017b). The surface of particles became clear and the pits were explored. Enzyme can enter the cell wall in the deeper layer of the particles from the pits and degrade more pectin in the particles. Because of this, the permeability of the wood can be increased (Durmaz *et al.* 2015) and the free formaldehyde in the panels will easily volatilize, reducing the free formaldehyde in the panels.

Besides, the clear particles surface and the larger permeability of the wood is more suitable for forming an effective bonding interface between the UF resin and the particles. This is a characteristic that can improve the mechanical properties of the particle board panels in some degree; but if the permeability is too much, the particles may “absorb” too much UF resin. Too little of the resin will remain in the bond area, adversely affecting the mechanical properties of the particle board panels. The results in this research were similar to the results in previous research; pectinase enzyme can improve the performance of fiber-based composites, such as bamboo, hemp, wood, *etc.* (Li *et al.* 2017a,b), due to the pectinase enzyme changing the fiber distribution and the pectin content of natural fibers in composite samples (Saleem *et al.* 2008).

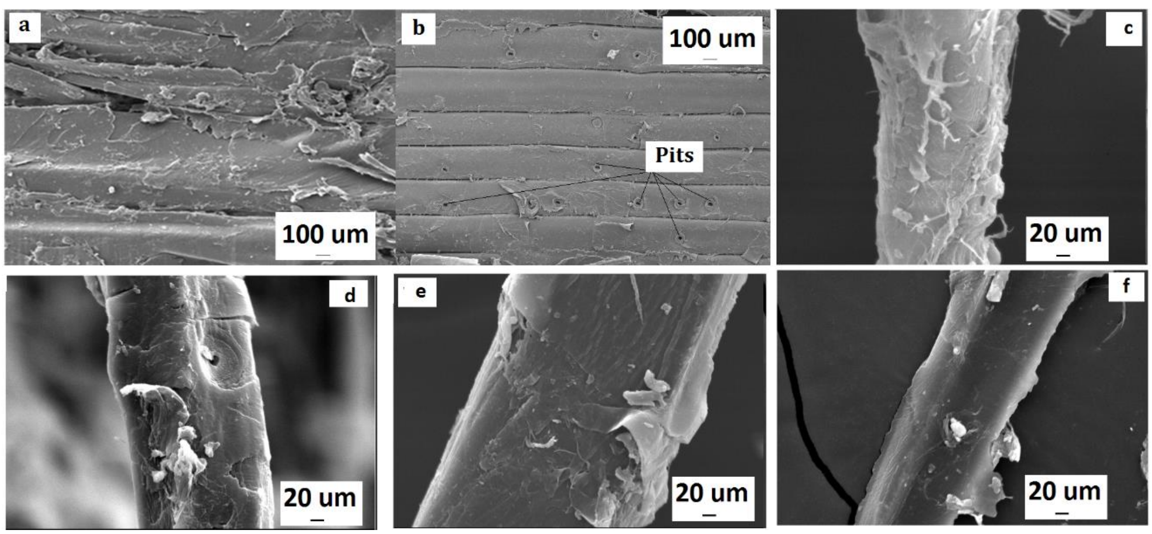

Fig. 1. The structure of eucalyptus wood particles and fibers; a: Untreated eucalyptus wood particles; b: Eucalyptus wood particles treated by pectinase enzyme at 45 °C for 30 min; Effect of pectinase pretreatment for 0, 30, 60, or 90 min (c, d, e, f, respectively) on condition of eucalyptus wood fibers samples.

Increasing the pre-treatment time from 30 min to 60 min or 90 min is disadvantageous to the properties of resulting panels for two reasons. First, the cell walls are composed of different layers including the primary cell layer and second layers (including S_1, S_2, and S_3) (Plomion *et al.* 2001). As the pectin is degraded by the pectinase enzyme, the different cell wall layers are exposed (shown in Fig.1c to Fig.1f).

The single eucalyptus wood fibers were treated by the pectinase enzyme from 30 min to 90 min and compared with the non-treated fiber (Fig.1c, 0 min), and the different cell walls layers were fallen off.

The surface structure on the different cell wall layers is different, leading to different effects on the panel properties. Secondly, a long pectinase enzyme pre-treatment may increase the permeability of woody materials, which may lead the particles to soak up more UF resin. This can have a bad influence on the properties of the resulting panels. More research is required to validate these hypotheses. In the following section, the effect of pre-treatments, temperature, and ratio of particles to enzyme solution is discussed.

Table 3. Performance of Particle Board Panels Treated by Pectinase Enzyme at Different Temperatures and Ratios of Particles to Solution for 30 min [a]

Pectinase Enzyme Treatment Temperature (°C)	Ratio of Particles to Solution	MOE (MPa)	MOR (MPa)	IB （MPa）
Control	---	2999 (207) ab	15.1 (1.31) cd	0.51 (0.09) ab
30	1:100	2831 (269) b	17.3 (2.39) abc	0.48 (0.08) bc
35	1:100	3133 (400) ab	17.6 (1.91) ab	0.47 (0.10) bc
40	1:100	3109 (352) ab	17.2 (2.00) abc	0.53 (0.14) ab
45	1:100	3242 (459) ab	19.2 (2.74) a	0.61 (0.07) a
50	1:100	3307 (307) a	13.2 (0.63) cd	0.56 (0.05) ab
55	1:100	2788 (317) b	17.1 (2.28) abc	0.52 (0.11) ab
60	1:100	3168 (390) ab	18.40 (2.40) a	0.50 (0.11) ab
45	1:80	3314 (426) a	23.0 (3.80) a	0.62 (0.12) a
45	1:60	3356 (362) a	22.6 (2.17) a	0.59 (0.14) a
45	1:40	2887 (278) abc	14.2 (1.20) d	0.56 (0.07) a
45	1:20	2936 (358) abc	19.2 (2.32) bc	0.53 (0.12) a
Standard*	----	1800	11.0	0.40

[a] Values represent means of 15 replicates per treatment while figures in parentheses represent one standard deviation. Values followed by the same letter(s) do not differ significantly from one another by Tukey's pairwise comparisons ($\alpha = 0.05$); * Standard BS EN 312:2010; Requirements for boards for interior fitments (including furniture) for use in dry conditions (Type P2).

Effect of Pre-treatment Temperature and Ratio of Particles to Solution on Panel Properties

Pectinase enzyme pre-treatments for 30 min at different temperatures or ratios of particles to solution of particle board panels were associated with significant changes in MOE, MOR, and IB (Table 3). When the ratio of particles to solution was 1:100 and when the temperature was 45 °C, the MOE, MOR, and IB of particle board panels were all significantly improved. When the ratio of particles to solution was reduced to 1:80 or to 1:60 at 45 °C for 30 min, the MOE, MOR, and IB of the resulting panels were markedly improved. Thus 45 °C and 30 min were the selected optimum parameters for this research, and the ratio of particles to solution was reduced to 1:80 or 1:60.

The resultant parameters were good for manufacturing particle board panels with better mechanical properties. This may be because the fibers are composed of several different layers including the primary cell layer and secondary cell layers (S1, S2, and S3 layers) (Plomion *et al.* 2001); cellulose, lignin, and hemicellulose are the main constituents in the different cell wall layers, but they are arrayed differently in the different cell wall layers (Khalil *et al.* 2006). The pectinase enzyme pre-treatment at different times, temperatures, or ratios of particles to solution maybe degrade the pectin or change the fiber surface by different degrees, and thus the performance of the particle board panels was different.

CONCLUSIONS

1. Pectinase enzyme pre-treatment of eucalyptus particles produced significant improvements in the MOE, MOR, and IB of the resulting particle board panels when the treatment temperature was 45 °C and treatment time was 30 min.
2. When particles were treated with an enzyme solution for 30 min at 50 °C, the free formaldehyde emission of the panels was lowest, due to the permeability of the woody materials being improved after the pectinase enzyme pre-treatments. Reducing the ratio of particles to enzyme solution was advantageous for manufacturing particle board panels with better mechanical properties. The results suggest that pectinase enzyme pre-treatment has some benefits for manufacturing particle board panels with better properties and lower free formaldehyde emissions.

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