

Chemical Characteristics of Thermo-Hydrolytically Recycled Particles

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Particles from waste laminated particleboards were recovered using various thermo-hydrolytic treatments. The size distribution and three main chemical properties, such as pH, buffer capacity, and nitrogen content of the control and resulting particles were determined. The effects of treatment temperature and duration on the chemical properties of recycled particles were investigated. The effects of the resulting particles on the gel time of urea-formaldehyde (UF) resin were also measured. The test results suggested that the pH of particles after hot water soaking pre-treatment and the different thermo-hydrolytic treatments increased to different extents, with the exception of the particles treated at 140 °C for 50 min and 160 °C for 20 min. The acid buffer capacity and base buffer capacity of particles treated at 140 °C for 50 min and 160 °C for 20 min had no statistical difference, but they were much higher than those of other types of treated particles. A high treatment temperature facilitated the decomposition of wood polymers and UF resin. Both temperature and treatment duration had significant effects on pH, acid buffer capacity, and base buffer capacity of wood particles. Wood particles recycled at a high temperature had a negative effect on the gel time of UF resin.

Keywords: Particleboard; Recycle; Particle size distribution; pH; Buffer capacity; Nitrogen content

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INTRODUCTION

According to the North American Particleboard Summary Report provided by Resources Information System Inc. (RISI), the total capacity of particleboard in North America is 4.510 billion square feet (BSF) on a ¾-inch basis in 2019, and the particleboard capacity in Canada is approximately 1.354 BSF on a ¾-inch basis. The total capacity of particleboard in North America has shown an increasing tendency over seven years (5.080 BSF in 2025) based on the data published by RISI (2019). However, this increasing demand for panel products may cause two important environmental issues: one is the shortage of raw material supply. Today, in Canada, to produce particleboards the manufacturers typically use wood residues from sawmills, furniture, and flooring mills, but these wood residues may not be enough to meet the increasing market demand for the corresponding particleboards. The other problem is to find a suitable usage for the wood waste accumulated after the particleboard panels complete their service life.

Due to the increasing prices of particleboard and the shortage of raw material supply, the recycling of wood wastes, such as discharged furniture, production remainders, and rejections, is nevertheless recognized as an economically attractive alternative source of raw material. Moreover, reusing the material brings ecological benefits (Deák 2013). For the recycling of wood composites, these materials need to be disintegrated to recover particles or fibers. Three different principles are commonly applied for disintegrating panels: mechanical, thermo-hydrolytic and chemical, or combinations thereof (Karazipour and Kües 2007). The target of any recycling process is to recover the amount of high quality particles or fibers as much as possible for subsequent utilization as raw materials in the wood-based composite panels industry.

In a thermo-hydrolytic recycling treatment, steam and pressure are generally applied to cleave existing bonds in wood composite panels that were glued by hydrolysable adhesives. Myers (1983) had revealed that the thermo-hydrolytic degradation of urea-formaldehyde (UF) resin may be catalysed by acids originating from wood (such as formic acid and acetic acid) as well as other acids obtained from frequently used hardeners such as ammonium sulphate and ammonium nitrate. These acids may lower the activation energy needed to disrupt the structure of cross-linked UF resins, leading to the formation of UF pre-polymers. Lubis *et al.* (2017) used five aqueous solutions (including water, two acids, and two alkalis) to hydrolyze medium-density fiberboard (MDF) panels for removing the UF resin. They reported that an optimum condition for the removal of cured resins from MDF was 80 °C for 2 h with 1% oxalic acid solution. Lykidis and Grigoriou (2008) reported that most of the mechanical properties, except for the modulus of elasticity (MOE) of the particleboards manufactured with recycled particles, decreased in comparison to those of the particleboards manufactured with virgin wood particles. The degree of quality degradation of recycled particleboards can be significantly reduced by using milder conditions during hydrothermal treatments for particle recovery.

The knowledge of the acidity properties of particles is of practical importance for better understanding the interfacial bonding mechanism when the acid-setting UF resin is used as adhesive in the manufacture of particleboard. Several researchers have revealed that the pH and acid buffer capacity of particles or fibers have important effects on gel time and bonding quality of UF resin (Johns and Niazi 1980; Wang and Wan 2004; Xing *et al.* 2004; Pedieu *et al.* 2008). An acidic condition is preferable for this type of amino resin to cure, so measuring the acidity properties such as pH and buffer capacity of particles will be useful to predict their potential influence on the curing behavior of UF resin in the particleboard manufacturing.

Internal bond (IB) strength has been found to be strongly related to the pH value of fibers, *i.e.*, the IB strength increased with the pH value of fibers. In addition, the panel mechanical properties of IB, MOE, and modulus of rupture (MOR) increased with alkaline buffer capacities of fibers (Xing *et al.* 2006). However, limited information on chemical properties of recycled wood particles could be found in the literature. Therefore, the aim of this study was to effectively recover particles from waste laminated particleboard using various thermo-hydrolytic treatments and to investigate the effect of treatment temperature and duration on the chemical properties of recycled particles.

EXPERIMENTAL

Particles Recycling Process

Hot water (100 °C) soaking pre-treatment to remove the overlay of boards

Used UF resin bonded paper-laminated particleboards collected from waste furniture available at FPInnovations Laboratory in Québec City (Canada) were cut into small pieces (2 in × 2 in). Subsequently these small pieces were soaked in hot water at 100 °C for 30 min to remove their overlaid paper.

Thermo-hydrolytic Treatment

Approximately 429 g (moisture content of 9.5%) of pre-treated particles with 2.5 L of tap water were transferred into a 5-L volumetric pressure reactor (Parr Series 4580 Bench Top Reactor, Parr Instrument Company, Moline, IL, USA). Seven different thermo-hydrolytic treatments were performed with various temperature and duration conditions (Table 1). A pressure regulator on the bench top reactor was used to control the maximum pressure of the reactor at 1 bar, 2 bars, 3 bars, and 5 bars, corresponding to the treatment temperatures of 100 °C, 120 °C, 140 °C, and 160 °C, respectively. For all of the treatments, the treatment time started when the target temperature was reached. Virgin wood residues obtained from a local particleboard plant were refined into particles (P0) using a Thomas Wiley lab-scale mill (Model 4; Thomas Scientific, Swedesboro, NJ, USA). Laminated particleboards without any treatment were polished to remove the overlaid paper using a Rockwell/DELTA 18" planer (Rockwell Manufacturing Company, Pittsburgh, PA, USA), cut into small pieces, and refined into particles (coded P1) using a Thomas Wiley lab-scale mill (Model 4; Thomas Scientific, Swedesboro, NJ, USA). All resulting particles were oven-dried and stored in Ziploc plastic bags prior to their properties determination.

Table 1. Thermo-hydrolytic Treatment Design and Resulting Particles Identification

Particle ID	Thermo-hydrolytic Treatments
P0	Virgin wood particles
P1	Laminated particleboard particles without any treatment
P2	Laminated particleboard particles After hot water soaking pre-treatment
P3	Thermo-hydrolytically recycled particles treated at 100 °C for 60 min
P4	Thermo-hydrolytically recycled particles treated at 100 °C for 100 min
P5	Thermo-hydrolytically recycled particles treated at 120 °C for 30 min
P6	Thermo-hydrolytically recycled particles treated at 120 °C for 60 min
P7	Thermo-hydrolytically recycled particles treated at 140 °C for 20 min
P8	Thermo-hydrolytically recycled particles treated at 140 °C for 50 min
P9	Thermo-hydrolytically recycled particles treated at 160 °C for 20 min

Measurements of Particle Properties

Particles size distribution

The size distribution of the resulting particles was evaluated using the Tyler Standard Screen Scales (W.S. Tyler Company, Mentor, OH, USA). Approximately 60 g of dried particles per sample were classified using 12-, 20-, 32-, and 48-mesh screens and a pan. The screens and pan were used in stacks and equipped with a Tyler portable sieve

shaker (Model RX-24; Gilson Company, Inc. Lewis Center, OH, USA) by shaking 15 min for each sample to divide particles into five size fractions (> 1.4 mm, 0.84 to 1.4 mm, 0.5 to 0.84 mm, 0.3 to 0.5 mm, and < 0.3 mm). The particle size distribution was based on weight percentage of each size fraction.

pH and buffer capacity

In this study, a two-point-calibration was performed for calibration of the pH meter. The pH meter (Corning Inc., Corning, NY, USA) was calibrated with pH 7.00 and 4.00 standard buffer solutions.

Methods

Extraction procedure

Approximately 25 g (oven-dry weight) of particle sample and 200 mL of distilled water were placed in a 500-mL flat bottom flask. The flask was installed on a heating plate and equipped with a condenser to reflux the sample for 20 min (refluxing began when distilled water started boiling). The mixture was filtered under vacuum with an 11-cm Whatman *Grade 4 filter paper (Whatman Inc., Piscataway, NJ, USA), and the flask and sample were rinsed twice with distilled water. Then, the solution was transferred into a 500-mL volumetric flask and diluted to 500 mL. The extraction procedure of each sample was repeated twice. The 500-mL volumetric flask with the extracting solution was sealed prior to pH and buffer capacity determination.

Titration procedure for measuring pH and acid/base buffer capacities

The 100 mL of extracting solution was pipetted into a 200-mL beaker and constantly stirred, then titrated with a standard 0.0125 mol/L or 0.025 N H₂SO₄ solution until reaching pH 3.00 for the acid buffer capacity measurement. For the base buffer capacity, 100 mL of extracting solution was titrated with a standard 0.025 mol/L or 0.025 N NaOH solution until reaching pH 11.00. Both acid and base titration procedures of each extracting solution were repeated twice.

The acid or base buffer capacity, expressed as total milliequivalents (mEq) or millimole (mmol), can be calculated as follows:

$$\text{Acid or base buffer capacity (mEq/100 g dried sample)} = \frac{\text{Volume of titrant} \times 0.025 \text{ N H}_2\text{SO}_4 \text{ (or NaOH)} \times 500 \text{ mL} \times 100 \text{ g of dried sample}}{\text{Volume of titrated sample} \times \text{weight of dried sample}}$$

$$\text{Acid buffer capacity (mmol)} = 0.0125 \text{ mol/L of H}_2\text{SO}_4 \times \text{volume of titrant (mL)} \quad (1)$$

$$\text{Base buffer capacity (mmol)} = 0.025 \text{ mol/L of NaOH} \times \text{volume of titrant (mL)} \quad (2)$$

The initial pH value before titration was noted as the pH of the extracting solution. The initial pH value of each type of particle sample was an average of eight measurements (2 extractions \times 2 acid buffer capacities \times 2 base buffer capacities), while the acid or base buffer capacity value was an average of four titrations.

Nitrogen content

All of the resulting particles and virgin particles were sent to Université Laval (Laboratoire Daishowa, Centre de Recherche en Horticulture, Québec, Canada) to perform the nitrogen content analysis using the digestion Kjeldahl method.

The resin content can be calculated as follows,

$$RC = N_p / N_u \quad (3)$$

where RC is the measured resin content (%), N_p is the nitrogen content (%) in the particle, and N_u is the nitrogen content (%) in the pure UF resin. Resin removal (%) from the sample was calculated as,

$$RR (\%) = (RC_b - RC_a) / RC_b \quad (4)$$

where RR is the resin removal, RC_b is the resin content (%) of particleboard particles before treatment, and RC_a is the resin content (%) of the particleboard particles after treatment.

Gel Time of UF Resin in the Presence of Wood Particles

A commercial UF resin designed for particleboard manufacturing was obtained from an adhesive supplier with the following typical characteristics: the viscosity was 0.355 Pa·s, the solid content was 67.2%, pH at 25 °C was 8.2, the buffer capacity was 11.49 mL, and the refractive index was 1.4772. The effects of wood particles and catalyst dosage on the curing process of UF resin was evaluated by determining the gel time at 100 °C. The experimental design for the gel time test is shown in Table 2.

Table 2. Experimental Design for the Gel Time of UF Resins

No.	Particle Size (mm)	Mass of Resin (g)	Mass of Wood Flour (g)	Catalyst Dosage (%)	20% NH ₄ Cl Solution (g)
P0	< 0.3	25	3.25	1	0.84
	< 0.3	25	3.25	2	1.68
	0.5 to 0.84	25	3.25	1	0.84
P1	< 0.3	25	3.25	1	0.84
	< 0.3	25	3.25	2	1.68
	0.5 to 0.84	25	3.25	1	0.84
P2	< 0.3	25	3.25	1	0.84
	< 0.3	25	3.25	2	1.68
	0.5 to 0.84	25	3.25	1	0.84
P4	< 0.3	25	3.25	1	0.84
	< 0.3	25	3.25	2	1.68
	0.5 to 0.84	25	3.25	1	0.84
P6	< 0.3	25	3.25	1	0.84
	< 0.3	25	3.25	2	1.68
	0.5 to 0.84	25	3.25	1	0.84
P7	< 0.3	25	3.25	1	0.84
	< 0.3	25	3.25	2	1.68
	0.5 to 0.84	25	3.25	1	0.84

A total of 25 g UF resin sample, 3.25 g oven-dried wood particles, 1% or 2% catalyst of 20% NH₄Cl were added in a beaker and were mixed well *via* stirring with a wood stick (2 × 5 × 250 mm³). Approximately 5 g of the mixture was weighed and added

into a 150-mm glass tube (15 mm in diameter). The tube with a holder was then placed into the boiling water, and the time count was started. The wood stick was rotated until the resin hardened. The gel time was then recorded. The gel time test was repeated twice for each mixture.

Statistical Analysis

An analysis of variance (ANOVA) was performed to investigate the effects of temperature and duration on the chemical properties of particles using SAS 9.4 software (SAS Institute Inc., Cary, NC, USA) at the significance level $\alpha = 0.05$. Duncan's test was conducted for multiple comparisons between the average values obtained under different treatments.

RESULTS AND DISCUSSIONS

Particle Size Distribution

Size distribution is an important physical property of wood particles. Li *et al.* (2010) reported that particle sizes had an important effect on IB strength of particleboard. The smaller the particles size, the higher was the IB strength of particleboard. Their results also revealed that particle sizes had a significant effect on MOR and MOE and a positive impact on dimensional stability of particleboard. An increase in particle size resulted in less water or moisture adsorption of particleboard. The particles size also had an effect on the gel time of UF resins. A shorter gel time was observed with finer particles (Medved and Resnik 2004).

Table 3 presents the size distribution of different wood particles. For particles P0 and P1, most of the particles were larger than 0.84 mm and smaller than 1.4 mm. For particles from P2 to P9, the highest percentage of particles (40.1 to 61.1%) was found for those being retained on a 12-mesh (size > 1.4 mm). The lowest percentage of particles (0.3 to 3.7%) was found for those passing through a 48-mesh (size < 0.3 mm). The particles from P2 to P9 with similar size distributions would have a limited impact on wood chemical properties measured in this study. This result also indicated that the different thermo-hydrolytic treatments did not have significant influence on particles size in comparison to that of the particles treated by hot water soaking pre-treatment (P2).

Table 3. Size Distribution of Different Wood Particles

Size (mm)	P0 (%)	P1 (%)	P2 (%)	P3 (%)	P4 (%)	P5 (%)	P6 (%)	P7 (%)	P8 (%)	P9 (%)
> 1.4	18.2	20.0	55.2	61.1	40.9	42.7	45.2	48.3	40.1	45.4
0.84 to 1.4	45.0	43.7	25.2	25.2	29.3	29.1	31.0	29.9	29.6	30.1
0.5 to 0.84	23.1	22.2	14.5	11.1	17.8	18.9	17.5	14.8	20.2	17.0
0.3 to 0.5	8.4	4.8	4.3	2.3	10.2	5.7	2.7	5.2	7.0	4.2
< 0.3	4.6	9.2	0.8	0.3	1.8	3.7	3.7	1.8	3.2	3.3

pH and Buffer Capacity

The pH of wood particles is an important property to consider when UF resin is used in the manufacture of particleboard, because this type of resin is an acid-curing

adhesive. In general, a low pH facilitates the curing of UF resin. Normally, low pH particles result in shorter curing time of UF resin (Pedieu *et al.* 2008). Thus, the pH of the wood particles would reveal important information on the curing time of UF resin during hot-pressing.

Table 4 presents the pH and buffer capacities of different wood particles. The pH of the virgin particles (P0) was 4.66. The pH of laminated particleboard particles without any treatment (P1) was 5.37. In the current study, the pH of P1 was used as one control to evaluate the effect of the treatment on the pH value of the particles. Compared to the pH of P1, the pH of the particles after hot water soaking pre-treatment and after different thermo-hydrolytic treatments increased to a different extent, with the exception of the particles treated at 140 °C for 50 min (P8) and 160 °C for 20 min (P9). This might have been due to the fact that more acid that was released during the hydrolysis of wood polymers for samples treated at relatively high temperature and long treatment time. The pH of particles after the pre-treatment (P2) and treated at 140 °C for 20 min (P7) had shown no statistical difference. The highest pH (6.57) was observed for the particles treated at 120 °C for 60 min (P6). At the treatment temperature of 100 °C (P3, P4) and 120 °C (P5, P6), the pH of wood particles increased with an extension of the treatment time. However, the opposite results were observed for the particles treated at 140 °C (P7 and P8).

During the thermo-hydrolytic treatment, both the hydrolysis of UF resin and the hydrolysis of wood components occurred. It is known that UF resin is mainly composed of urea and formaldehyde. Urea can decompose into ammonia and carbon dioxide under acid or alkaline aqueous solution. Ammonia will be converted into ammonium hydroxide (NH₄OH) in water solution. This alkaline substance will result in an increase of the pH of wood particles. However, the hydrolysis of wood components decreases the pH of wood particles, due to the release of formic and acetic acids caused by wood polymer degradation at high temperature and pressure. The final pH of wood particles depends on the equilibrium of the two hydrolyses aforementioned. High temperature and long treatment time would accelerate the hydrolysis of wood components, and this may explain why the pH of P8 and P9 was not increased after the thermo-hydrolytic treatment. Neutralization reactions occurred continuously between the acids and NH₄OH. The NH₄OH reacts preferably with formic acid due to the higher acidity (pKa 3.7) of formic acid compared to acetic acid (pKa 4.8) (Roffael and Hüster 2012).

According to the above analyses, it may be concluded that under the treatments of 140 °C for 50 min and 160 °C for 20 min, some advanced degradation of wood components occurred, releasing more acids in the wood particles solution and resulting in a decrease of the pH value of the wood particles. The pH of P2 was higher than that of P1. This indicated that the hot water soaking pre-treatment was helpful to decompose the UF resin in the particleboard.

The acid buffer capacities of particles P0, P3, P4, and P5 were not statistically different. This meant that the acid buffer capacity of P3, P4, and P5 was close to that of the virgin wood particles. At the treatment temperature of 120 °C (P5, P6) and 140 °C (P7, P8), the acid buffer capacity of wood particles increased notably when the treatment time was extended. The acid buffer capacities of P8 and P9 had no statistical difference, but were much higher than that of other types of particles. The higher value of acid buffer capacity implies that more acid catalyst was required for the proper curing of UF resin to overcome the impact of increased acid buffer capacity (Maloney 1977).

The base buffer capacities of P0, P1, P2, P4, and P6 were not statistically different. At the treatment temperatures of 120 °C (P5, P6) and 140 °C (P7, P8), the base buffer capacity of wood particles increased notably when the treatment time was extended. The base buffer capacities of P8 and P9 had no statistical difference, but were much higher than that of other types of particles.

In terms of both acid and base capacity points of view, the wood particles treated at 100 °C for 100 min (P4) had the acid and base buffer capacity values close to those of the virgin wood particles among all of the thermo-hydrolytic treatments.

Table 4. Summary of Chemical Characteristics of Virgin and Recycled Particles

Particle ID	Initial pH (n = 8)	Acid Buffer Capacity (n = 4)			Base Buffer Capacity (n = 4)		
		mL 0.025 N H ₂ SO ₄	mEq/ 100 g OD Wood	mmol	mL 0.025 N NaOH	mEq/ 100 g OD Wood	mmol
P0	4.66 ^h (0.02)	9.18 (0.15)	4.59 (0.08)	0.115 ^{de} (0.002)	15.68 (0.45)	7.84 (0.23)	0.392 ^{bc} (0.011)
P1	5.37 ^f (0.04)	9.50 (0.08)	4.75 (0.04)	0.119 ^{cd} (0.001)	16.15 (0.29)	8.08 (0.14)	0.404 ^{bc} (0.007)
P2	5.58 ^d (0.03)	8.08 (0.31)	4.04 (0.16)	0.101 ^e (0.004)	13.38 (0.97)	6.69 (0.49)	0.334 ^{bc} (0.02)
P3	5.48 ^e (0.05)	8.53 (1.86)	4.26 (0.93)	0.107 ^{de} (0.023)	11.45 (4.17)	5.73 (2.08)	0.287 ^c (0.104)
P4	5.97 ^b (0.09)	8.58 (0.35)	4.29 (0.18)	0.107 ^{de} (0.004)	13.9 (1.75)	6.95 (0.87)	0.348 ^{bc} (0.044)
P5	5.77 ^c (0.09)	9.10 (0.64)	4.55 (0.32)	0.114 ^{de} (0.008)	12.6 (1.03)	6.3 (0.52)	0.315 ^c (0.026)
P6	6.57 ^a (0.20)	11.5 (0.89)	5.75 (0.45)	0.144 ^b (0.01)	15.43 (0.56)	7.71 (0.28)	0.386 ^{bc} (0.01)
P7	5.61 ^d (0.03)	10.63 (1.03)	5.31 (0.52)	0.133 ^{bc} (0.013)	18.03 (0.94)	9.01 (0.47)	0.451 ^b (0.024)
P8	5.36 ^f (0.03)	14.8 (0.36)	7.4 (0.18)	0.185 ^a (0.004)	25.98 (7.26)	12.99 (3.63)	0.649 ^a (0.181)
P9	5.07 ^g (0.02)	15.53 (0.53)	7.76 (0.27)	0.194 ^a (0.007)	26.1 (3.90)	13.05 (1.95)	0.653 ^a (0.097)

Values in parenthesis are standard deviations; the superscript letter shows Duncan's multiple-comparison test result of the average values, the average values with the same letter indicate no significant difference at $\alpha = 0.05$ in Duncan's multiple-comparison test

Effect of Treatment Temperature and Duration on Wood Chemical Properties

Both the treatment temperature and duration had significant effects on pH, acid buffer capacity and base buffer capacity of wood particles. This indicated that treatment temperature and duration were two important factors during the thermo-hydrolytic treatments.

Table 5. Analysis of Variance of Wood Chemical Properties *versus* Temperature and Duration

Dependent Properties	Factors	DF	Sum of Squares	Mean Squares	F Value	p
pH	Temperature	3	7.349	2.449	30.51	< 0.0001
	Duration	4	5.217	1.304	10.55	< 0.0001
Acid buffer capacity (mmol)	Temperature	3	0.0243	0.008	19.11	< 0.0001
	Duration	4	0.021	0.005	9.28	< 0.0001
Base buffer capacity (mmol)	Temperature	3	0.463	0.154	13.91	< 0.0001
	Duration	4	0.458	0.114	9.72	< 0.0001

DF: degrees of freedom

Nitrogen Content Analysis

The UF resin contains elemental nitrogen (N) from urea, so the nitrogen content in UF-bonded panels can be used to estimate the quantity of UF resin in the panel (Grigsby *et al.* 2014; Lubis *et al.* 2017). The nitrogen contents of the control and resulting particles are presented in Fig. 1. The nitrogen content of virgin wood particles (P0) was 0.19%. This value is in good agreement with Cowling and Merrill (1966), who found that most of the wood species examined contained approximately 0.1% of nitrogen. The nitrogen content of P1 was 2.3%, much higher than that of P0. This was attributed to the UF resin and catalyst NH_4Cl added during the manufacturing of the particleboards. Therefore, for any treatment of UF bonded particles, the change of nitrogen content in wood particles was likely attributable to the change of UF resin content and NH_4Cl content. The nitrogen content was normally applied to evaluate the ratio of the resin residue and the resin being removed from wood fibers recycled from UF-bonded MDF undergoing different treatments (Lubis *et al.* 2017). In the current study, the nitrogen content of the wood particles was also used to evaluate the extent of UF resin decomposition under different thermo-hydrolytic treatments.

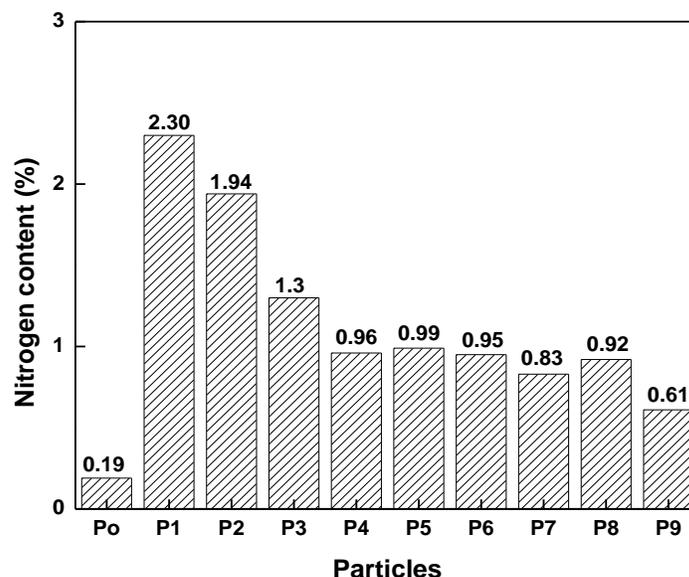


Fig. 1. Typical nitrogen content of particles under different treatments

After pre-treatment, the nitrogen content of P2 decreased 15.7% compared to that of P1 (the control particles without any treatment). This indicated that the UF resin started to decompose during the pre-treatment process. The nitrogen contents of P4, P5, P6, P7, and P8 were not notably different, but much lower than those of P1 and P2. At the treatment temperature of 100 °C (P3, P4), the nitrogen content of wood particles decreased when the treatment time was extended. Among all the thermo-hydrolytic treatments, the lowest nitrogen content was 0.61%, which was obtained for the wood particles treated at 160 °C for 20 min (P9). This implied that 73.5% of nitrogen was removed from the sample treated at 160 °C for 20 min. Figure 1 also shows that 63.9% of the nitrogen was removed when the sample was treated at 140 °C for 20 min (P7 in Fig. 1). Because the amount of nitrogen removed corresponds to the degree of UF resin decomposed, this study revealed that an elevated temperature can facilitate the decomposition of UF resins.

Effect of Wood Particles Type and Catalyst Dosage on the Gel Time of UF Resin

Table 6 shows the results of gel time of UF resin in the presence of wood particles. The effect of wood particles on gel time of UF resin is illustrated in Fig. 2. The catalyst dosage had a significant effect on the gel time of UF resin. For all kinds of particles, the gel time of UF resin decreased notably with the increase of catalyst dosage. The effect of particle size on gel time of UF resin was not as significant as that of catalyst dosage. Figure 2 reveals that the gel time of UF resin in the presence of P0 (the virgin particle) and P4 (treated at 100 °C for 100 min) was not significantly different. This was probably due to a similar level of acid buffer capacity despite of a big difference in pH value. Pedieu *et al* (2008) reported that UF resin mixed with low pH particles needed shorter curing time. However, the results of this study did not show a similar relationship between wood particles pH and gel time of UF resin. This is probably due to the fact that all particle samples measured in this study had pH values falling in the range of pH 4 to 5.5 for the majority of wood species. It was also observed that both the acid buffer capacity and base buffer capacity of P0 and P4 had no statistical difference. Thus, this study indicated that the gel time of UF resin in the presence of wood particles was influenced by the wood buffer capacity instead of the wood pH. Particularly, the gel time of UF resin in the presence of particles P7 was much longer than that of the control particles. This indicated that wood particles recycled at an elevated temperature had a negative effect on the curing rate of UF resin. Therefore, it implied that more catalyst dosage or longer curing time may be needed for the manufacturing of particleboards with particles recycled at high temperature (140 °C). The reasons for UF resin in the presence of particles P7 required a longer gel time are not clear. Figure 1 shows the nitrogen content of particles under different treatments. P7 contained 0.83% of nitrogen content which was lower than 0.95%-2.30% observed for P1-P6. The lower nitrogen content means that more UF resin was removed from the wood after the treatment due to the more severe treatment condition for P7, as indicated in Table 1. Such a severe hydrothermal treatment could alter wood composition due to the loss of some extractives and composition degradation during the treatment. It is anticipated that these changes in wood chemical property might lead to increase the swelling of the wood particles during the gel time test and consequently result in a change in the wood rheology to slow down the curing of UF resin.

Table 6. Effect of Wood Particle and Catalyst Dosage on Gel Time of UF Resins

Particle ID	pH	Acid Buffer Capacity (mEq/100 g OD Wood)	Particle Size (mm)	Catalyst Dosage (%)	20% NH ₄ Cl Solution (g)	Mean Value of Gel Time (s) n = 2
P0	4.66	4.59	< 0.3	1	0.84	67 ^{cde}
			< 0.3	2	1.68	62.5 ^f
			0.5 to 0.84	1	0.84	71.5 ^b
P1	5.37	4.75	< 0.3	1	0.84	60.5 ^f
			< 0.3	2	1.68	55 ^g
			0.5 to 0.84	1	0.84	62 ^f
P2	5.58	4.04	< 0.3	1	0.84	61.5 ^f
			< 0.3	2	1.68	56 ^g
			0.5 to 0.84	1	0.84	63.5 ^{ef}
P4	5.97	4.29	< 0.3	1	0.84	67.5 ^{cd}
			< 0.3	2	1.68	62.5 ^f
			0.5 to 0.84	1	0.84	69 ^{bc}
P6	6.57	5.75	< 0.3	1	0.84	64 ^{edf}
			< 0.3	2	1.68	56 ^g
			0.5 to 0.84	1	0.84	62 ^f
P7	5.61	5.31	< 0.3	1	0.84	78 ^a
			< 0.3	2	1.68	62 ^f
			0.5 to 0.84	1	0.84	76.5 ^a

The superscript letter shows Duncan's multiple-comparison test result of the average values; the average values with the same letter indicate no significant difference at $\alpha = 0.05$ in Duncan's multiple-comparison test.

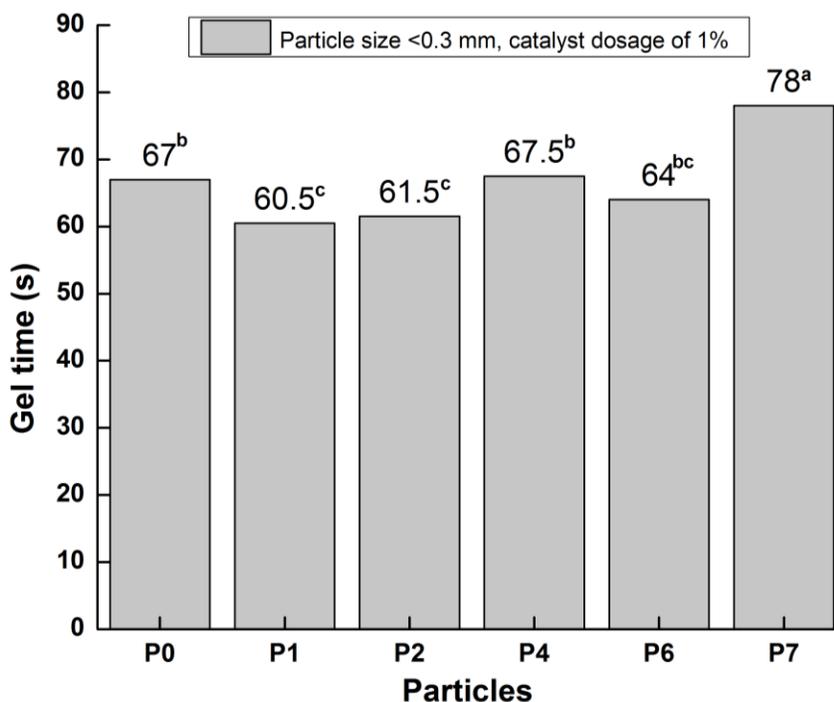


Fig. 2. Effect of wood particles on the gel time of UF resins (the superscript letter shows Duncan's multiple-comparison test result of the average values; the average values with the same letter indicate no significant difference at $\alpha = 0.05$ in Duncan's multiple-comparison test).

CONCLUSIONS

1. Compared to the pH of laminated particleboard particles without any treatment (P1), the pH of particles after hot water soaking pre-treatment and after different thermo-hydrolytic treatments increased to different extents, with the exception of the particles treated at 140 °C for 50 min and 160 °C for 20 min.
2. Treatment temperature and duration were two important factors during the thermo-hydrolytic treatments. High temperature and long treatment time caused the hydrolysis of wood polymers. This resulted in the decrease of pH of recycled particles.
3. The acid buffer capacity and base buffer capacity of particles treated at 140 °C for 50 min and 160 °C for 20 min were not statistically different, but they were much higher than those of other types of particles treated at 100 to 120 °C for 30 to 100 min.
4. The nitrogen content of laminated particleboard particles (P2) decreased 15.7% after hot water soaking pre-treatment and 73.5% after thermo-hydrolytical treatment at 160 °C for 20 min compared to that of the virgin particles (P1). This indicated that the UF resin started to decompose during the pre-treatment process. High treatment temperatures facilitated the decomposition of wood polymers and UF resins.
5. Wood particles recycled at temperatures higher than 140 °C increased the gel time of UF resin.

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