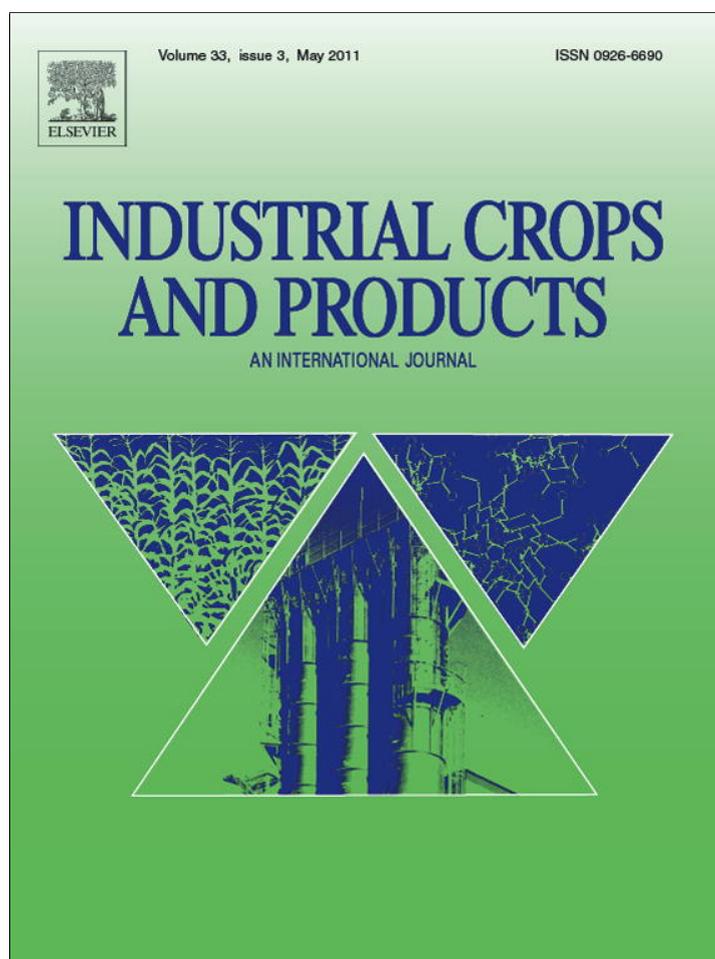


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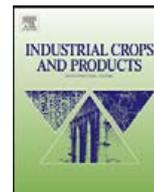
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## Effect of oxalic acid and steam pretreatment on the primary properties of UF-bonded rice straw particleboards

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### ABSTRACT

The objective is to evaluate the effect of oxalic acid (OA) and steam-pretreatment on the primary performance of rice straw particleboards. In addition, the effect of various treatment conditions on carbohydrates released from rice straw particles was investigated. The results show that steam- and short durations of OA-treatment significantly improved the mechanical properties and dimensional stability of rice straw particleboards. However, steam-treated rice straw (without OA-treatment) panels exhibited even better performance when compared with OA-treated panels. OA-pretreatment time has a negative effect on performance of panels, whereas the effect of temperature on the performance of OA-treated panels was not significant, except for the linear expansion. OA-treatment accelerated carbohydrates extraction. The sugars released from the OA-treated rice straw particles increase with increasing treatment temperature and time. Carbohydrates extracted from rice straw particles could be a potential sustainable resource for biofuel or bio-based chemicals.

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### 1. Introduction

Particleboards are widely used for construction, furniture, and interior decoration. The primary lignocellulosic material used in the particleboard panel industry is wood, but there has recently been a revival of interest in using agriculture residues to produce particleboards and other composite panels due to competition for wood raw materials and for economical and environmental considerations (Viswanathan et al., 2000; Mo et al., 2001; Yang et al., 2003; Basta et al., 2004, 2006; El-Saied and Basta, 2007; Zheng et al., 2007; Ye et al., 2007; Copur et al., 2008; Halvarsson et al., 2008; Hizioglu et al., 2008; Zou et al., 2010). Globally, wheat and rice are the most important food grains ranking second and third in terms of the total cereal production (Halvarsson et al., 2008), and appear to be the most promising agriculture residues for manufacturing composite panels.

The main chemical components of wood and straw materials both are cellulose, hemicellulose, and lignin. However, the straw materials have lower cellulose and lignin content and higher hemicellulose content compared to wood. Furthermore, the straw contains hydrophobic waxy cuticle layers and a high amount of

inorganic silica (Pan et al., 1999; Pan and Sano, 2000; Hammerr et al., 2001; Han et al., 2010; Mo et al., 2003). In addition, the curing of pH-sensitive UF resin was inhibited by the high pH and buffer capacity of the rice straw, which results in weak bonding between particles and very low internal bonding strength within urea-formaldehyde (UF) resin bonded panels (Han et al., 2001; Boquillon et al., 2004; Li et al., 2010). Many attempts have been made to improve the bondability between the straw materials and adhesives through raw material pretreatment. Han et al. (1999, 2001) investigated the effect of silane coupling agent level and ethanol–benzene treatment on board properties and found that physical properties for both reed and wheat boards were improved. They also reported that bonding performance of UF resin-bonded reed and wheat straw fiberboards could be improved if the fiber was pre-treated under different steam cooking conditions in the refining process. High temperature steam and steam explosion treatments were used to modify wheat straw for panel manufacturing by reducing the contents of pectic substance, hemicellulose, ash and silicon in straw (Lawther et al., 1996; Han et al., 2010). Enzyme pretreatment of wheat straw was also an effective way to improve the bonding performance (Zhang et al., 2003). Removal of wax-like substances, hemicellulose and other non-polar extractives from straw via raw material pretreatment could contribute to the improvement of board properties, but the use of hazardous chemical materials (such as ethanol–benzene mentioned above) during pretreatment could cause new environmental pollution or increase

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production costs. Therefore, development of UF-resin bonded straw panels with performance rivaling wood-based boards at lower production cost, negligible environment pollution is a global challenge.

With renewed interest in cellulosic ethanol production, the pulp and paper industry proposed the concept of “value prior to pulping” (VPP) to compensate for the refining process (Thorp and Raymond, 2004; Liu et al., 2006; Ragauskas et al., 2006; Van, 2006; Huang et al., 2010; Walton et al., 2010). In the technology, hemicelluloses are either partially or completely extracted from woody biomass for bio-ethanol or other chemical feedstock production through a prehydrolysis process, and the remaining solid could still be used for wood pulp or fiber products (Zhu and Pan, *in press*). Recently, the pretreatment of wood chips using a dilute oxalic acid (OA) solution prior to refining has been identified as an innovative means to reduce energy consumption during refining, extract carbohydrates for the production of cellulosic ethanol and other chemical feedstocks, and improve medium density fiberboard (MDF) water repellency properties and paper strength (Kenealy et al., 2007; Akhtar et al., 2008). The rice straw has higher hemicelluloses and extractives contents, and looser structure compared to woody biomass, the VPP pretreatment using dilute OA solution may be result in more extracted carbohydrates and improvement in physical performance of rice straw particleboard. However, the similar research on rice straw has not been reported so far.

The overall goal of this work was to investigate the feasibility of manufacturing particleboard from OA-treated rice straw particles and UF resin, and to evaluate some primary properties of particleboard as affected by various treatment conditions. In addition, we also investigated the pretreatment effect on carbohydrate released from rice straw.

## 2. Materials and methods

Rice straw was obtained from California, United States. Its variety number was Calrose M-206. It was approximately one meter tall at harvest and cut above the water line leaving the lower third as stubble in the field. This rice was also harvested with a conventional straw walker combine rather than a rotary, which optimizes straw quality by retaining much longer sections of stalks for bailing. The average moisture content of the straw was about 8.1%. After received, the rice straw was broken down into particles in a hammer mill, in which a perforated metal plate with opening size of 19.05 mm (3/4 in.) was used.

The reactor configuration and pretreatment procedure has been described previously (Winandy et al., 2008). Rice straw particles were treated with 0.33% OA solution at four temperature levels (100 °C, 120 °C, 140 °C and 160 °C). The treatment times for each temperature level were 5 and 10 min. For the control, rice straw particles were processed at the same 160 °C heating conditions for 10 min as the OA-pretreatment, but no chemical were added to the hot water. These rice straw particles would be used as the positive control (PC) or steam-treated sample, which would be compared with the eight different OA-pretreatments. After pretreatments, the pressure in the digester was released and the rice straw particles were flooded with 70 °C hot water, which was circulated for 30 min to rinse the hemicellulosic sugar from the treated rice straw. The mass ratio of added hot water to rice straw particles (oven dry weight) is 8:1 in this process. Finally, the hemicelluloses extracts and extracted rice straw particles were collected and weighted separately.

The analysis of the carbohydrates in the aqueous extracts was performed by a Dionex HPLC system (ICS-3000) equipped with integrated amperometric detector according to the method described by Shuai et al. (2010). The treated particles were then washed with water for 15 min to bring the pH of the samples near

to the level of untreated control samples. The extracted rice straw particles (OA-treated and steam-treated samples) and raw control (RC) particles without OA-or steam-pretreatment were dried at  $103 \pm 2^\circ\text{C}$  to 2–3% moisture content in a tray dryer, and then the dried particles were hammer milled again using a perforated metal plate with opening size of 6.35 mm (1/4 in.). The particles were bagged and used for later particleboard fabrication.

The commercial UF resin used in this study was acquired from Arclin Corporation (281 Wallace Road, North Bay, Ontario, Canada). The UF resin was water dispersed with a solid content of 65%. A resin content of 12% (oven-dried weight basis) was uniformly sprayed onto the particles, in a rotating drum blender. A 91.4 cm  $\times$  91.4 cm Nordberg hot press with a PressMAN control system (Alberta Research Center, Alberta, Canada) was used to press the particleboard panels at a 170 °C platen temperature. The dimensions of the boards made in this study were 508 mm  $\times$  508 mm (20 in.  $\times$  20 in.) and 12.5 mm (0.5 in.) thick. The target density was 0.70 g/cm<sup>3</sup>. A longer hot press time of 510 s (100 s closing, 320 s at the target thickness, and 90 s opening) was used to ensure complete curing of the resin. Three replicate boards were made for each condition.

Internal bonding (IB), modulus of elasticity (MOE), modulus of rupture (MOR), linear expansion (LE), water absorption (WA) and thickness swelling (TS) of the samples were prepared and tested in accordance with ASTM D 1037-06a (2006). Twelve samples from three different boards were prepared for the IB testing. Each measurement presented herein for the remaining mechanical and physical tests is the average value of six specimens cut from the three different panels. Static bending (MOE and MOR) and IB values were measured using an MTS (Material Testing System 634.11F-24) and a universal testing machine (Instron 555), respectively. The WA and TS properties were measured after 24-h immersion in distilled water at 20 °C.

## 3. Results and discussion

The quantities of carbohydrates extracted from rice straw particles are shown in Table 1. The main components of extracted solutions were glucose, mannose, and arabinose. Generally, more total carbohydrates were extracted from the OA-treated rice straw particles than from the PC sample (steam-treated without OA solution). At the same pretreatment temperature (160 °C) and time (10 min), the total carbohydrate extracted from the OA-treated rice straw particles was about 2.3 times as that from the PC samples. This result clearly demonstrates that the addition of OA in hot water accelerates the carbohydrates extraction of rice straw particles. The same result was observed in our previous studies using Loblolly Pine (*Pinus taeda* L.) wood chips as experimental lignocellulosic material. During OA-pretreatment, the amount of sugars released from rice straws was dependent upon the treatment temperature and duration. When the pretreatment temperature increased from 100 to 160 °C, the amount of the abstracted sugar increased 82% and 99% for the two pretreatment durations of 5 min and 10 min, respectively. The reason for these increases is probably due to the more degradation of hemicellulose within rice straw particles with the increase in treatment temperature and time.

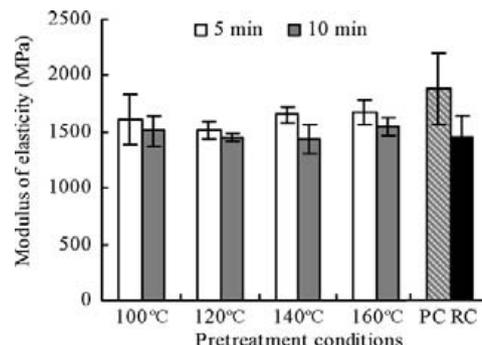
Fig. 1 shows the effect of pretreatment on the IB strength of rice straw particleboards. In comparison with the rice straw control specimens (RC), a significant improvement was observed in steam-treated rice straw boards (PC), where IB increased by 85.7%. For the OA-treated rice straw boards, the IB strength increased about 69.3% at the treatment time levels of 5 min. The increase of IB strength could be mainly due to the extraction of hemicelluloses, wax-like substances, and other non-polar extractives from rice straw in pretreatment resulting in improved compatibility between rice straw and UF resin. Unexpectedly, the IB strength of OA-treated

**Table 1**  
The content of the carbohydrates extracted from rice straw particles.

Sample ID	Content (g kg <sup>-1</sup> oven dried rice straw)					Total carbohydrate (g kg <sup>-1</sup> oven dried rice straw)
	Arabinose	Galactose	Glucose	Xylose	Mannose	
PC	0.74	0.36	4.73	0.07	3.36	9.26
100/5	0.33	0.30	5.90	0.01	3.42	9.96
120/5	1.38	0.28	5.61	0.09	2.16	9.53
140/5	3.41	0.22	5.76	1.76	3.05	14.19
160/5	2.74	0.30	8.24	0.62	6.20	18.10
100/10	0.51	0.44	5.67	1.75	2.29	10.66
120/10	2.36	0.21	4.99	1.29	2.80	11.65
140/10	2.70	0.16	6.99	1.36	4.00	15.21
160/10	2.78	0.73	10.26	0.56	6.94	21.26

panels was lower than that of PC panels, and a longer treatment time resulted in decreased IB strength of OA-treated panels at the same treatment temperature. These results clearly indicate that the addition of OA in pretreatment has a negative effect on IB strength of particleboards compared to high temperature steam-treatment, especially when using longer treatment times. The IB strength decrease in particleboards after the OA-pretreatment was probably caused by the internal and/or surface degradation of particles during OA-pretreatment. At the same time, OA-treatment maybe lower the pH-value of particles because of residual OA on the surface and interior of rice straw particles, and lead to a pre-curing of the UF resin in OA-treated panels, which can also partly explain the IB strength reduction of OA-treated panels compared to PC panels. In addition, the other possibility for the decrease is that chemical changes in the OA-pretreated rice straw particles, resulting from the extraction of hemicellulose and other substance in rice straw, might require modified resin application and processing changes to cure the resin when using UF resin (Winandy et al., 2008). Further study should be conducted to fully optimize the OA-pretreatment conditions or modify UF resin application rates and curing processes in order to produce particleboards with similar IB performance to that of particleboards made using steam-treated rice straw particles. In general, the effect of treatment temperature on IB strength of OA-treated rice straw panels is not significant. The *F*-test statistical analysis and two-tailed pair-wise *t*-test also confirms the observation at the 99% confidence level.

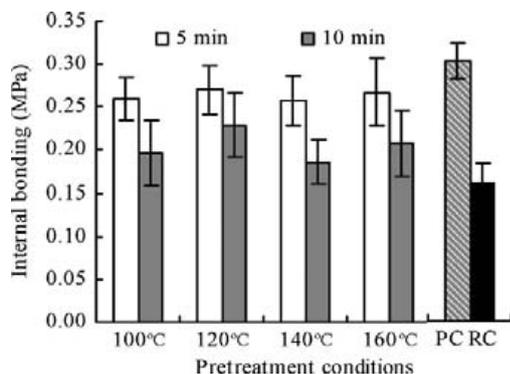
The effects of the steam and OA-treatment on the static bending properties of rice straw particleboards are shown in Figs. 2 and 3. In general, the static bending MOE and MOR of OA-treated panels were slightly higher than that of non-treated panels (RC samples), but lower than that of steam-treated panels without OA addition (PC samples). In comparison with RC samples, the MOE and MOR increase about 30% and 32% for steam-treated panels (PC samples), and 12% and 16% for OA-treated panels at a shorter treatment time (5 min), respectively. The MOE and MOR values of OA-treated panels decrease slightly when the treatment time increased from 5 to



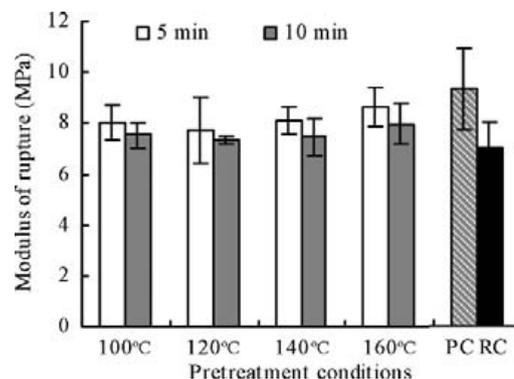
**Fig. 2.** Effects of pretreatment conditions on MOE of rice straw particleboard.

10 min at the same pretreatment temperature. The pretreatment temperature has not significant effect on the bending strength of OA-treated samples. The statistical analysis also indicates that the effect of pretreatment time and temperature on the OA-treated panel's bending performance was not significant at the 99% confidence level. The increase in the bending properties of rice straw using OA- and steam-treatment reflects a good correlation with the trend of IB strength improvement in panels. Therefore, the reason for these increases in bending properties is mainly due to improved IB strength in OA- and steam-treated panels.

LE and TS are key parameters in describing dimensional stability of wood composites. Particularly, LE is considered as the control factor in qualifying the behavior of wood panels exposed to moisture (Cai et al., 2004). The influence of various pretreatment on the dimensional stability (including in LE, TS and WA) of rice straw particleboards was examined in this study. The results presented in Figs. 4–6 show a significant decrease in LE, TS and WA of OA- and steam-treated panels. Generally, the steam-treated panels exhibit a slightly better dimensional stability than OA-treated panels. In comparison with RC panels, the LE, TS and WA decreased 32%,



**Fig. 1.** Effects of pretreatment conditions on IB strength of rice straw particleboard.



**Fig. 3.** Effects of pretreatment conditions on MOR of rice straw particleboard.

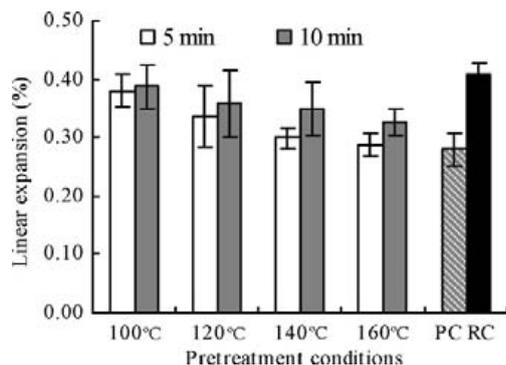


Fig. 4. Effects of pretreatment conditions on LE of rice straw particleboard.

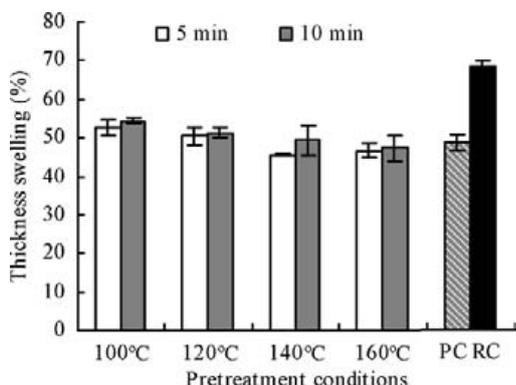


Fig. 5. Effects of pretreatment conditions on TS of rice straw particleboard.

29% and 21% for steam-treated panels, and 21%, 28% and 18% for OA-treated panels at a pretreatment level of 5 min. Increase in pretreatment time slightly increases the LE, TS and WA of OA-treated panels, and reduces their dimensional stability. The statistical analysis revealed that the effect of pretreatment time and temperature on the TS and WA of OA-treated panels was not significant at the 99% confidence level. However, the LE of OA-treated panels shows a decreasing tendency with increased pretreatment temperature at the same pretreatment time. The LE of panels decreases from 0.38 to 0.29 and 0.39 to 0.33% for 5 and 10 min OA-pretreatment when pretreatment temperature increases from 100 to 160 °C. Hemicellulose is believed to be more water-soluble and hydrophilic than cellulose and lignin. The improvement of dimensional stability in rice straw particleboards after OA- and steam-pretreatment could be mainly attributed to the partial removal of hemicellulose in rice straw particles. Another explanation for the increase is the improved IB strength in OA- and steam-treated panels.

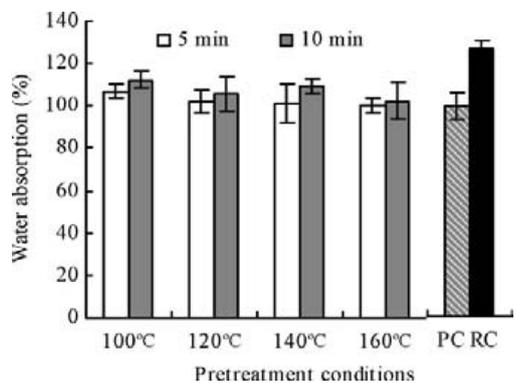


Fig. 6. Effects of pretreatment conditions on WA of rice straw particleboard.

#### 4. Conclusions

The results show that steam and short durations of OA-treatment resulted in significantly improved mechanical properties and dimensional stability of rice straw particleboards, especially for IB strength. Steam-treated panels exhibited slightly higher performance compared with OA-treated panels. Pretreatment time had a negative effect on performance of OA-treated panels, whereas the effect of temperature on the performance of OA-treated panels was not significant, except for the LE. OA-treatment accelerated carbohydrate extraction. The sugars released from the OA-treated rice straw particles increased with increasing treatment temperature and time.

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