Effect of Liquid Hot Water Pretreatment on Selected Properties of Rice Husk and Its Particleboard

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Rice husk has a water repellent wax layer on its surface and has high pH and buffering capacity, which inhibit surface adhesion and result in the poor strength and water resistance of rice husk particleboard bonded with water-borne urea formaldehyde (UF) resin. In this work, rice husks were pretreated with liquid hot water at different temperatures (130, 140, 150, and 160 °C) and residence times (10 and 20 min). Pretreated rice husks were used to produce particleboard with UF resin. The effects of liquid hot water pretreatment temperature and residence time on the wax contents and pH values were investigated. In addition, the buffering capacities of the rice husks and the mechanical properties and the water resistance of the rice husk particleboards were investigated. The results indicated that liquid hot water pretreatment remarkably reduced the wax contents, pH values, and buffering capacities of the rice husks. In addition, it improved the mechanical properties and water resistance of the rice husk particleboards. Liquid hot water pretreatment effectively improved bondability between rice husks and water-borne UF resin, and it enhanced the performance of the rice husk particleboard.

Keywords: Rice husk; Particleboard; Liquid hot water pretreatment; Urea formaldehyde resin

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INTRODUCTION

Each year about 596 million tons of rice are produced globally (Li *et al.* 2010). Rice husks, the by-product of the rice milling process, which are readily available in large quantities, have not been fully utilized (Ciannamea *et al.* 2010; Ayrilmis *et al.* 2012; Kang *et al.* 2012; Kwon *et al.* 2013; Tabata *et al.* 2017). Currently, only a small portion of rice husks are recycled for application in agriculture, livestock, and other sectors; the remaining portion are mainly burnt or used for landfilling (Kwon *et al.* 2013; Wood *et al.* 2016), which wastes precious natural resources and causes serious air pollution (Fernandez *et al.* 2001; Wood *et al*. 2016). Therefore, the development of new technologies to efficiently utilize rice husk is of great importance (Kang *et al.* 2012). Rice husk contains 25% to 35% cellulose, 8% to 21% hemicelluloses, 26% to 31% lignin, 15% to 17% silica and waxes, and 2% to 5% of other soluble substances (Ciannamea *et al.* 2010). Because the basic chemical components of rice husks are similar to those of wood and they are readily available at relatively low cost, rice husks have the potential to serve as the raw material for particleboard production (Ciannamea *et al.* 2010; Ayrilmis *et al.* 2012; Kang *et al.* 2012). Urea formaldehyde resin, which has a relatively high bonding strength and is easily applicable and inexpensive, is the most used adhesive in wood-based composite panel production (Abdolzadeh *et al.* 2011). However, UF is not suitable to produce rice husk particleboards due to its weak strength properties, the poor dimensional stability of the resultant panels, and the high resin content requirement (Ciannamea *et al.* 2010). The deficiencies are largely due to the outer surface of rice husk being covered with smooth, water-repellent wax layers, which hinder the further penetration and diffusion of UF (Ciannamea *et al.* 2010; Ayrilmis *et al.* 2012; Kwon *et al.* 2013) and results in poor adhesion between the UF and rice husk particles. In addition, the high pH and buffering capacity of the crop materials inhibit the curing of pH-sensitive UF resin, which leads to greatly increased resin gel time and weak bonding between crop materials and UF resin (Han *et al.* 2010; Li *et al.* 2010). As a result, the rice husk particleboard has poor mechanical properties and water resistance. Removal of the water-repellent wax layers can enhance the adhesion properties of crop materials to adhesives and improve the performance of the particleboard made from the crop materials (Ndazi *et al.* 2007; Han *et al.* 2009, 2010; Li *et al.* 2011). Research works showed that chemical (Han *et al.* 1999; Li *et al.*2011), steaming (Han *et al.* 2009; Li *et al.* 2011; Baskaran *et al.* 2013), and steam explosion (Han *et al.*2010) pretreatment of crop materials are capable of reducing the water-repellent wax content, which enhances the crop materials' wettability and affinity to UF.

Liquid hot water pretreatment, which uses pressure to maintain the water in a liquid state at elevated temperatures for several seconds to several minutes, is applied mainly to pre-hydrolyze and delignify lignocellulosic fiber for bioethanol production (Mosier *et al.* 2005; Wan and Li 2011; González *et al.* 2014; Rezania *et al.* 2020). Water under high pressure and temperature can penetrate the biomass and is able to separate lignocellulosic fiber components while minimizing cellulose hydrolysis (González *et al.* 2014). Acetic acid and other organic acids generated from autohydrolysis can catalyze further hydrolysis (Wan *et al.* 2011). Liquid hot water pretreatment, which is an environmentally friendly process, omits the need for neutralization and conditioning chemicals and corrosion resistant materials for the reactor because no chemicals are added (Mosier *et al.* 2005; Wan and Li 2011; González *et al.* 2014). The effects of liquid hot water pretreatment on detaching lignocellulosic fiber components strongly depend on pretreatment severity, which is determined by pretreatment temperature and residence time (Mosier *et al.* 2005; Wan and Li 2011; Rezania *et al.* 2020). Increasing temperature and/or prolonging residence time may increase the severity of liquid hot water pretreatment. Severe pretreatment conditions result in accretion of organic acids and, as a result, an acidic environment, which promotes the hydration of cellulose and removal of hemicellulose and a portion of lignin; the degradation of fermentable sugars dissolving in the liquid fraction to hydroxymethyl furfural, furfural, formic acid, and levulinic acid is also promoted (Wan and Li 2011). The liquid hot water pretreatment is thought to be capable of destroying the hydrophobic wax layer on rice husk surfaces and improving the bondability between the rice husks and UF resin. There is currently no information regarding the utilization of liquid hot water to pretreat rice husks for rice husk particleboard manufacturing. This work employed a liquid hot water process to pretreat rice husks at different temperatures and residence times. The purpose of crop material pretreatment for particleboard production is to efficiently remove the hydrophobic wax layers covering the surface of crop materials and minimize the degradation of cellulose, hemicellulose, and lignin. To avoid loss of thermal instable hemicelluloses that start to solubilize and hydrolyze at pretreatment temperatures above 150 °C (Han *et al.* 2009; Talebnia *et al.* 2010), less severe pretreatment conditions were employed in this study. The objective of this work was to investigate the effect of pretreatment conditions on the chemical properties of rice husks and rice husk particleboard bonded with UF resin.

EXPERIMENTAL

Raw Materials

The rice husks were acquired from Huiguan Village in the Xiaozhan Township of the Jinnan District (Tianjin, China). Its initial moisture content was approximately 7.16%. A screen with 0.3-mm mesh was used to manually shed undersized rice husks and dusts.

Liquid Hot Water Pretreatment

Liquid hot water pretreatment was conducted in a custom-built 15-L batch rotary stainless steel cylindrical autoclave (Model ZQS; Tongda Light Industrial Equipment Corporation, Xianyang, China) with a design pressure of 4 MPa (Fig. 1). The autoclave, connected to a rotary shaft, was equipped with an electrical heater, a motor actuator, a safe valve, a steam releasing valve, a pressure meter (0 MPa to 6 MPa), and a temperature gauge (0℃ to 300 ℃). The autoclave was loaded with 1.1731 kg of rice husks (dry weight) and tap water. The solid to water ratio inside the autoclave was 1:7 (g/mL, dry weight). The autoclave was tightly closed and simultaneously heated and rotated at 1 rpm to ensure appropriate agitation and uniform contact of water with the rice husks. The pretreatment temperatures were 130 ℃, 140 ℃, 150 ℃, and 160 ℃, and the residence times were 10 min and 20 min. The residence time for each batch was counted when the temperature gauge registered the target temperature. Heating was stopped, and steam was released gradually by opening the steam releasing valve at the end of each treatment. The autoclave was discharged when reaching atmospheric pressure. Both the pretreated rice husks and the liquid became light brown to dark brown in color depending on pretreatment severity (Fig. 2).The pulp was filtered to separate the solid residues (pretreated rice husks) from the liquid fraction. The pretreated rice husks were oven dried at 100 ± 3 ℃ to reach 2% to 3% moisture content and placed into plastic bags for future utilization.

Fig. 1. Image of the autoclave

Measurement of Chemical Composition, pH, and Buffering Capacities of the Raw and Pretreated Rice Husks

The ethanol/benzene extractives contents, the pH values, and the acid and alkali buffering capacities of the raw and pretreated rice husks were measured according to the China National Test Standards of GB/T 2677.6 (1994), GB/T 6043 (2009), and GB/T 17660 (1999), respectively.

Particleboard Preparation

Commercial UF resin (Zhengzhou Kaibang Chemical Products Co., Ltd., Zhengzhou, China) was used in this study. The UF resin was dispersed with water to reach a solid content of 55%. The UF resin was sprayed onto rice husks in a blender (Model HWJ 25;Xiaoshan Commercial Equipment Corporation, Zhejiang, China) through a pneumatic atomizing nozzle. The resin application level was 14% based on the oven-dried weight of the rice husks. To evaluate the water resistance of particleboards made of liquid hot water pretreated rice husks, no water-repelling chemicals were utilized.

The single layer particleboard mats were formed manually using a laboratory mold. The mats were prepressed at 1.5 MPa for 30 s. Then, the mats were hot-pressed in accordance with a three-phase hot press schedule at 130 °C. During the first phase, the mat was pressed at 3.5 MPa for 180 s, and during the second and third phases, the mat was pressed under 2 MPa for 120 sand 1 MPa for 60 s, respectively. The board size was 500 mm×500 mm×6 mm with a target density of 800 kg/m^3. Three panels were made for each condition. The particleboards made from untreated rice husks acted as controls.

Particleboard Evaluation

After manufacture, the rice husk particleboards were conditioned at 20 °C and 65% relative humidity for 2 weeks. Edges of the panels were trimmed to a final dimension of 450 mm×450 mm×6 mm. Then, the panels were sawed into test specimens and tested according to the China National Test Standard GB/T 17657 (1999) for density, internal bonding strength (IB), modulus of rupture (MOR), modulus of elasticity (MOE), 24 h water absorption (WA), and thickness swelling (TS). Analysis of variance (ANOVA) and Duncan's mean separation tests were used to statistically analyze the data obtained with SPSS software (SPSS Inc., Version 19, Chicago, IL, USA).

RESULTS AND DISCUSSION

Table 1. Effect of Liquid Hot Water Pretreatment on Extractives Ethanol/Toluene, pH, and Buffering Capacity of Rice Husks

Pretreatment Condition		Extractives (%) (ethanol/benzene)	pH value	Acid Buffering Capacity (mL)	Alkali Buffering Capacity (mL)
Temp. (°C)	Time (min)				
130	10	5.7	5.91	44.7	35.8
	20	5.4	5.70	43.8	34.7
140	10	5.0	5.47	43.1	33.9
	20	4.6	5.33	42.3	32.6
150	10	4.1	4.98	41.0	31.2
	20	3.9	4.76	40.3	30.5
160	10	3.8	4.62	39.9	29.8
	20	3.6	4.53	39.4	28.5
Untreated		6.2	7.67	48.2	37.1

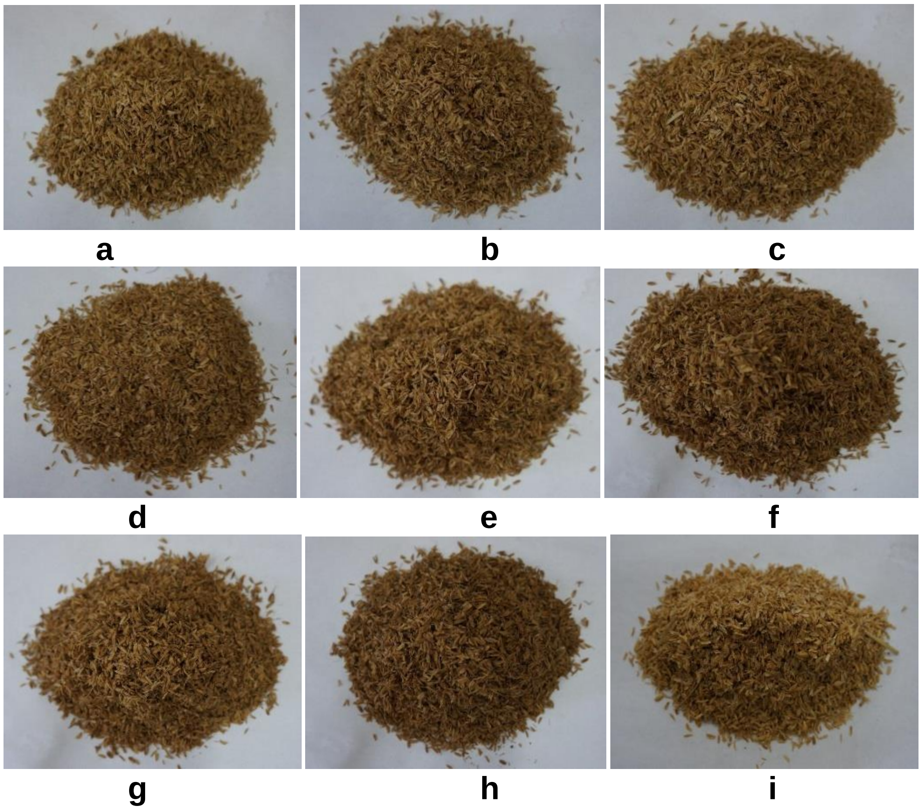

Fig. 2. Images of rice husks before and after liquid hot water pretreatment: (a) pretreated at 130 °C for 10 min; (b) pretreated at 130 °C for 20 min; (c) pretreated at 140 °C for 10 min; (d) pretreated at 140 °C for 20 min; (e) pretreated at 150 °C for 10 min; (f) pretreated at 150 °C for 20 min; (g) pretreated at 160 °C for 10 min; (h) pretreated at 160 °C for 20 min; (i) Untreated

The pH values and buffering capacities of acid and alkali from crop materials have been studied in recent years (Zhang *et al.* 2003; Han *et al.* 2010). Both the pH values and the acid and alkali buffering capacities were found to be significantly higher than those from softwood (Han *et al.* 2010). The high pH and buffer capacities from the crop materials inhibit the curing of pH-sensitive UF resin, which results in greatly prolonged resin gel time and poor bonding strength (Han *et al.* 2010; Li *et al.* 2010). Table 1 shows the effects of liquid hot water pretreatment on the pH values and buffering capacities of acid and alkali of rice husks. As shown in Table 1, the pH value of untreated rice husks was comparatively high (7.67). The pH values and buffering capacities of acid and alkali of liquid hot water pretreated rice husks were lowered significantly. The influencing parameters in the liquid hot water pretreatment of lignocellulosic biomass are pretreatment temperature and residence time. The pretreatment temperature had a remarkable impact on the rice husks. As the pretreatment temperature increased, the pH and buffering capacities of acid and alkali decreased accordingly. Similarly, the residence time had a noticeable effect on the rice husks. As the residence time was prolonged, the pH and buffering capacities of acid and alkali decreased. As UF resin cures fast under acidic conditions, the increase in acidity of rice husks shortens the curing time of UF resin, which improves the bonding strength of the rice husk particleboard.

Organic solvents such as ethanol/benzene have been found to be capable of extracting the surface wax layers of crop materials (Zhang *et al.* 2003; Han *et al.* 2009). Recent studies indicated that a reduction in surface wax content led to a significant improvement of the gluing properties of crop materials (Zhang *et al.* 2003; Han *et al.* 2010; Li *et al.* 2010). Table 1 shows that liquid hot water pretreatment effectively reduced the benzene-ethanol extract content. Increasing pretreatment temperature and/or prolonging residence time decreased the benzene-ethanol extract content. These results indicated that the liquid hot water pretreatment efficiently reduced the wax content of rice husks, which eliminated an obstacle to strong adhesion of UF resin on rice husks and enhanced board performance.

Table 2. Properties of Rice Husk Particleboards Under Different Liquid Hot Water Pretreatment Conditions

Pretreatment Condition		MOR (MPa)	MOE (MPa)	IB (MPa)	24 h TS (%)
Temp. (°C)	Time (min)				
130	10	11.82±1.03b	2037.1±196.6b	0.23±0.018 b	54.95±3.61d
	20	13.09±1.18 bc	2175.5±193.2b	0.27±0.024c	50.24±4.34cd
140	10	14.65±1.39cd	2367.1±196.1bc	0.27±0.022c	46.18±3.93abc
	20	15.72±1.26de	2584.6±221.9cd	0.29±0.028cd	44.66±3.38abc
150	10	17.13±1.57e	2758.4±212.2c	0.31±0.027cd	42.82±3.73ab
	20	15.43±1.27 d	2536.7±166.0cd	0.32±0.029d	39.46±2.74a
160	10	14.56±1.32cd	2310.0±188.6bc	0.30±0.027cd	48.92±4.51bcd
	20	12.89±1.07b	2119.5±176.3 b	0.28±0.027c	51.36±4.27cd
Untreated		7.47±0.55 a	1122.5±94.5 a	0.07±0.006a	66.90±5.18e
Data shown are mean ± standard deviation; different letters in the same column indicate significant difference at P < 0.05; IB: internal bond					

Table 2 demonstrates the effects of the liquid hot water pretreatment condition on the mechanical properties and water resistance of the rice husk particleboard. Clearly, the mechanical properties and water resistance of particleboards made from liquid hot water pretreated rice husks were enhanced remarkably. The liquid hot water pretreatment conditions had a significant influence on the mechanical properties and water resistance of particleboards. The IB values of particleboards made from liquid hot water pretreated rice husks were between 0.23 and 0.32 MPa, which indicates a noticeable improvement relative to the control. Appropriately enhancing pretreatment temperature and/or prolonging residence time improved the IB. Pretreatment at 150 °C/20 min resulted in the highest IB of 0.32 MPa, which was approximately 4.57 times that of the control. Further increasing pretreatment temperature and residence time led to a slight decrease in IB, which was still much higher than that of the control. This was attributed mainly to the removal of the water-repellent wax layer from the surfaces of rice husks and the reduced pH and reduced acid and alkali buffering capacities that resulted from the liquid hot water pretreatment, which enhanced the wettability and acidity of rice husks. As a result, the bondability of rice husks with the water-borne UF improved, and after hot pressing, the internal bonding of the panels increased. However, as pretreatment temperature and residence time increased further, both the extent of wax layer removal from the outer surfaces of rice husks and the extent of damage to the rice husks can increase. In addition, the pH and acid and alkali buffering capacities of rice husks decreased as pretreatment severity increased. This may have provoked precuring of the UF resin under acid conditions, which led to the deterioration of bond strength between rice husks and UF resin. Consequently, the IB decreased. Similar observations were also reported in previous studies by Li et al. (2011).

They stated that the particleboards produced from steam pretreated rice straws exhibited remarkably increased IB.

Fig. 3. Image of the rice husk particleboard

The MOR and MOE values of particleboards made from liquid hot water pretreated rice husks were between 11.8 and 17.1 MPa and between 2040 and 2760 MPa, respectively. Both MOR and MOE were improved remarkably compared to the control. Appropriately increasing the pretreatment temperature and/or residence time enhanced the MOR and MOE. Under the pretreatment conditions of 150 °C/10 min, the MOR and MOE of the resulting rice husk particleboards reached the peaks of 17.1 and 2760 MPa, which were 2.43 times and 2.46 times higher than the control, respectively. This was thought to be due to the removal of the water-repellent wax layers and lowered the pH and acid and alkali buffering capacities, which increased the bondability of rice husks and the internal bond after hot pressing. As a result, the MOR and MOE were significantly improved. As the pretreatment temperature and/or residence time increased further, the MOR and MOE started decreasing, though they were still much higher than those of the control. This could have been due to damage of the rice husk fibers that resulted from the increase in pretreatment severity. In addition, the increase in the acidity of rice husks with increased pretreatment severity led to precuring of the UF resin, which lowered the bonding strength between rice husks and UF. Consequently, the MOR and MOE decreased. These results were in parallel to the study by Li *et al.* (2011), who researched the effects of steam pretreatment of rice straw on the performance of the rice straw particleboards.

The effect of the liquid hot water pretreatment on the 24 h TS is presented in Table 2. Compared to the control, the TS decreased significantly for liquid hot water pretreated particleboards. With increasing pretreatment severity, the TS initially decreased to the minimum of 39.5% at 150 °C/20 min, which was just 59% of the control. Further increasing pretreatment severity caused increased TS, which was still much lower than the control. This was mainly due to increased bondability that resulted from pretreatment, which enhanced the internal bond of the panels after hot pressing. Consequently, the quantity of pores inside the panels was reduced, which reduced the access of water to the inside of the panels. Increasing the liquid hot water pretreatment temperature and residence time beyond 150 °C/20 min enhanced the elimination of hydrophobic wax and lowered pH and buffering capacities, which caused higher water absorption in rice husks and weaker internal bond of the rice husk particleboards. Consequently, the TS increased. Li *et al.* (2011) observed similar phenomena when producing particleboards with oxalic acid and steam-pretreated rice straw.

CONCLUSIONS

1. Liquid hot water pretreatment was found to be an effective approach to reduce the extractive contents of rice husks. The extractive contents decreased with the increase of pretreatment severity. The reduced extractive contents helped to absorb water-born UF resin and form a strong bond.
2. Liquid hot water pretreatment was an effective method to decrease the pH values and buffering capacity of rice husks. The pH values and buffering capacities of acid and alkali decreased with the increase of pretreatment severity. The improved acidity contributed to the fast curing of pH-sensitive UF resin and improve the bondability between rice husk particles and water-born UF resin.
3. The strength properties and water resistance of particleboards made from liquid hot water pretreated rice husks were enhanced significantly. Pretreatment at 150 °C/20 min resulted in the highest IB of 0.32 MPa, which was approximately 4.57 times that of the control. Under the pretreatment conditions of 150 °C/10 min, the MOR and MOE of the resulting rice husk particleboards reached the peaks of 17.1 and 2760 MPa, which were 2.43 times and 2.46 times higher than the control, respectively. Pretreatment at 150 °C/20 min yielded the lowest TS of 39.5%, which was just 59% of the control. The results suggested that liquid hot water pretreatment was a viable way to improve the bonding strength between rice husks and water-borne UF resin and enhance the overall performance of rice husk particleboard.

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