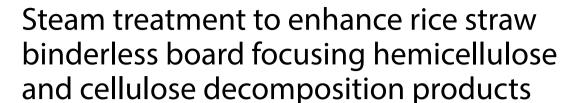


ORIGINAL ARTICLE

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Abstract

Rice straw is a troublesome biomass for an industrial application because of its high silica content and the wax-like substances covering the surface. At manufacturing binderless boards, which are considered environmentally friendly as they do not contain synthetic resins, rice straw silica contributed to water resistance, while the wax-like substances adversely affected self-bonding. This study investigated the effects of steam treatment on the self-bonding of binderless boards manufactured from rice straw. The chemical changes during steam treatment were examined by gas chromatography—mass spectrometry (GC–MS), and their influence on the bonding properties is discussed. Internal bonding strength and water resistance significantly increased by steam treatment. They also increased with increasing pressing temperature, and decreasing particle size. GC/MS analysis showed that not only hemicellulose and lignin, but also amorphous cellulose was decomposed during the steam treatment. Among the degradation products, 5-hydroxymethylfurfural was suggested to contribute to self-bonding during hot-pressing, while furfural was vaporized from the rice straw. Fine-grinding to below 150 µm after steam treatment resulted in high water resistance and an internal bonding strength of 0.6 MPa, which met the Japan Industrial Standards (JIS) requirement for Type-30 medium-density fiberboard (MDF). This mild pretreatment offers an alternative to steam explosion.

Keywords: Rice straw, Binderless, Self-bonding, Fiberboard, Steam treatment, Furfural, 5-Hydroxymethylfurfural

Introduction

Rice straw, an agricultural residue of rice production, is a potentially useful lignocellulosic material. Most rice straw is used for landfill or is burned, but this leads to methane gas production or air pollution. Manufacturing particleboard and fiberboard would be a valuable alternative, with a potentially large market. However, the characteristics of rice straw tend to lead to low-quality boards. Rice straw is not wetted or penetrated well by hydrophilic urea formaldehyde (UF) resin or phenol formaldehyde (PF) resin, due to its inorganic silica content and wax-like substances covering the surface [1–3]. Several pretreatments have been examined to improve these compatibility issues between rice straw and resins. One

was a mechanical method to break down the wax-layer, which improved the diffusion and penetration of resins into the straw fibers and increased the internal bonding of rice straw fiberboards [4]. Chemical and biological pretreatments to de-ash and de-wax also increased the bonding strength [5].

Binderless boards that contain no resin are highly desirable from an environmental perspective. Our previous studies showed that rice straw binderless boards could be successfully manufactured [6–8]. It was shown that silica contributed to water resistance, although slightly reduced bonding strength [7]. Removing the wax from the epidermis of rice straw by hexane extraction improved self-bonding [7]. The problem remains that hexane extraction is not a suitable pretreatment for a pilot scale. Steam explosion is another pretreatment to improve self-bonding, which causes hemicellulose decomposition and lignin reconstruction; these chemical

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modifications result in physical defibration [8]. However, steam explosion requires significant energy and equipment. It is thus necessary to examine new pretreatments as alternatives to hexane extraction and steam explosion.

Unlike steam explosion, steam treatment does not involve a sudden release of pressure. However, hydration of samples is accelerated, since the samples are subjected to steam at high temperature and high pressure. Steam treatment was found to induce thermo-plasticization of chemical components in wood powder, contributing to the formation of a binderless material [9]. In rice straw, steam treatment reduced the wax content [5] and improved the mechanical properties and water-resistance of particleboards [10]. Kristensen et al. [11] reported that hemicellulose decomposition and partial removal of wax occurred during steam treatment of wheat straw, similarly to steam explosion, by comparing infrared spectroscopy (IR) spectra. These changes would potentially also be effective for increasing the self-bonding of binderless board made from rice straw.

The purpose of this study was to investigate the effects of steam treatment on the properties of binderless boards made from rice straw. Rice straw was pretreated by steam at high temperature and pressure, and then hot-pressed to form boards. The mechanical and water-resistance properties of the boards were measured. In addition, gas chromatography—mass spectrometry (GC—MS) analysis was conducted to examine the chemical changes during steam treatment.

Materials and methods

Steam treatment of rice straw

Rice straw (*Oryza sativa* L. cv. Koganemochi) was obtained from the Institute for Sustainable Agro-Ecosystem Services, which is an experimental farm of The University of Tokyo, and air-dried. Whole rice straw was cut with scissors into chips shorter than 50 mm. Two-hundred grams of dry material per batch was put into a metal basket, washed with pooled tap water for 60 s, and then washed with running tap water for 60 s to rinse off

soil components. Steam treatment was carried out in an autoclave with 4 L of water; the rice straw chips were exposed to steam, but not immersed in water. The steaming conditions were at 200 °C, 1.5 MPa for 10 or 20 min. Steam-treated chips were dried in an oven at 80 °C, then stored in an air-dried state for 24 h to reduce the moisture content. The drainage was approximately 3 L, and was gathered for chemical analysis.

Manufacture of binderless boards

Table 1 shows the manufacturing conditions of each type of board. Untreated or steam-treated rice straw was ground in a Wiley mill (WT-150; Miki Seisakusho, Japan) until it passed through a 1-mm screen. Some parts were also fine-ground with a Wonder Crush/Mill (D3V-10; Osaka Chemical Corporation, Japan) until they passed through a 150- μm screen. The board size was $300\times300\times5$ mm, and the target density was 0.8 g/cm³. The pressing procedure was as described in our previous study [6]. Two boards were manufactured for each board type.

Evaluation of binderless board properties

Modulus of rupture (MOR), modulus of elasticity (MOE) and internal bonding (IB) were evaluated as the mechanical properties of boards. Eight specimens were prepared for MOR and MOE, and nine specimens were prepared for IB, for each manufacturing condition. Additionally, 24 h thickness swelling (TS) and water absorption (WA) were measured to determine the water resistance, using six specimens of each board type. These tests were carried out according to Japan Industrial Standards (JIS) A5905-2014 [12] and JIS A5908-2003 [13].

GC/MS

Rice straw samples steamed for 0, 10, or 20 min were ground smaller than 1 mm with a Wiley mill for extraction. One gram of each steamed sample was extracted with 5 mL of dichloromethane for 24 h. Extracts were filtered with absorbent cotton and silica gel. In addition, all

Table 1 Steaming and pressing conditions of each board type

Board type	Steaming condition			Pressing condition			
	Raw material size	Temperature (°C)	Time (min)	Particle size	Temperature (°C)	Pressure (MPa)	Time (min)
10-220	≦ 50 mm	200	10	≦1 mm	220	5	10
20-220	≦ 50 mm	200	20	≦1 mm	220	5	10
20-140	≦ 50 mm	200	20	≦1 mm	140	5	10
20-180	≦ 50 mm	200	20	≦1 mm	180	5	10
20-F220	≦ 50 mm	200	20	≦ 150 μm	220	5	10

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the drainage from steam treatment at 200 °C for 20 min was extracted four times with 500 mL of dichloromethane using a separating funnel. The extracts were filtered with absorbent cotton followed by removal of water with sodium sulfate. These four types of extracted sample were prepared for GC/MS analysis. The weights of extracts were measured after removing dichloromethane by rotary evaporator. 1 mL of dichloromethane was added to the extracts, and they were then trimethylsilylated with 5 μL of N, O-bis (trimethylsilyl) acetamide (BSA) and a catalyst (25 μm pyridine) for 1 h.

The analysis was carried out using GC–MS-QP5050A (Shimadzu, Japan) with a Neutral Bond-1 (30 m \times 0.25 mm) capillary column (GL Science, Japan). The oven temperature started at 60 °C and was programmed to immediately rise by 10 °C/min to 260 °C; this final temperature was kept for 25 min. Data were compared with compound libraries, such as NIST12, NIST62 and SHIM1607.

Results and discussion

Changes during steam treatment

Physical changes

Steam treatment changed rice straw chips to a dark grayish color (Fig. 1). Although the procedure did not include the sudden release pressure of steam explosion, a portion of the chips were defibrated. Figure 2 also shows that defibration occurred due to the steam treatment.

The drainage was also dark in color, including fine particles provided from the defibrated rice straw chips. This was the main reason for the weight loss of samples, which was 31.7% at the treatment for 20 min. Another reason was suggested to be chemical degradation as discussed below.

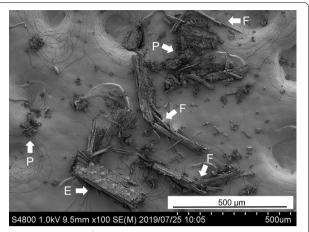


Fig. 2 SEM image of steamed and ground rice straw particles passed through a 1-mm screen. *F* fibers, *E* epidermis, *P* parenchyma cells

Wax-like substances

Table 2 shows the dichloromethane extracts of steam-treated rice straw analyzed by GC-MS. The dichloromethane extracts of untreated rice straw were all aliphatic compounds like hexadecanoic acid, *cis*-9-octadecanoic acid and octadecanoic acid. These aliphatic compounds originated from the wax-like substances covering the surface of rice straw. They were also detected in the steam-treated rice straw, the content was decreased with increasing treatment time (Table 2). This was supposed as a relative increase influenced by the increase in the other component such as hemicellulose and cellulose degradation products. If these aliphatic compounds were removed by steam treatment, they should be detected in the drainage or



Fig. 1 Rice straw chips. Left side: untreated rice straw. Right side: steam-treated rice straw after drying

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Table 2 Dichloromethane extracts of steam-treated rice straw

Compounds	Retention time GC/MS ^a (min)	Treatment time at 200 °C (%)		
		0 min	10 min	20 min
Waxes		99.03	71.65	65.52
2-Pentadecanone	18.8	2.64	1.68	1.39
Tetradecanoic acid	18.9	1.09	1.88	1.71
Octenoic acid	20.9	N/D	1.04	1.04
Hexadecanoic acid	21.1	43.32	37.91	36.61
Heptadecanoic acid	22.3	1.2	1.71	1.81
Octadecadienoic acid	23.2	15.93	9.76	6.76
cis-9-Octadecenoic acid	23.4	24.76	11.44	8.29
Octadecanoic acid	23.8	6.48	4.5	2.86
Eicosanoic acid	28	1.6	N/D	N/D
Other aliphatic com- pounds	-	2.01	1.74	5.05
Lignin degradation products		0	14.02	12.92
p-Hydroxybenzaldehyde	12.7	N/D	1.74	1.57
Vanillin	14.8	N/D	8.65	6.65
Syringaldehyde	16.9	N/D	1.34	1.15
Other aromatic compounds	-	N/D	2.28	3.55
Hemicellulose and cellulose products	degradation	0	5.3	19.09
5-Hydroxymethylfurfural	11.64	N/D	5.3	19.09
Yield ^b (%)		0.97	2.23	7.16

N/D not detected

vaporized. However, it was shown that they were not detected in the drainage actually (Table 3). The boiling points of hexadecanoic acid and octadecanoic acid are both higher than 300 °C. Vaporization should not occur at the steaming temperature, even if the boiling point is slightly increased by the elevated pressure during steam treatment. As a result, it could be concluded that the wax-like substances were not removed by the steam treatment. However, it has been reported that not only steam explosion, but also steam treatment at 195 °C for 6 min can partially remove straw waxes [11]. In their study, wheat straw was soaked in hot water at 80 °C for 30 min before the steam treatment. It is likely that this soaking procedure, rather than the steam treatment, resulted in the partial removal of wax. Indeed, it was reported that 50% of wheat straw wax was removed by extraction with hot water at 80-95 °C [14]. Steam treatment varies with the machine and treatment conditions; in our case, the raw material was not in contact with hot water, and the wax was not removed.

Table 3 Dichloromethane extracts of drainage from steam treatment (treatment time: 20 min)

Compounds	Retention time GC/MS ^a	Drainage (%)
	(min)	
Lignin degradation products		11.76
Guaiacol	8.07	1.88
o-Coumaric acid	10.12	2.58
<i>p</i> -Vinyl guaiacol	11.67	3.77
Vanillin	12.7	3.53
Hemicellulose and cellulose	88.24	
Furfural	3.94	83.46
2-Acetyl furan	5.06	0.76
5-Methylfurfural	5.84	4.02
Yield ^b (%)		1.92

 $^{^{}a}$ Column: NB-1 (30 m \times 0.25 mm); 60 °C, 0 min – 10 °C/min – 260 °C, 25 min

Hemicellulose and cellulose degradation

From Table 3, furfural was the main component of the drainage extracts, but was not detected in the steamed rice straw. Furfural is a kind of pentose produced by the decomposition of hemicellulose. The boiling point of furfural is 161.7 °C at 1 atm. As the steaming pressure was 1.5 MPa, the boiling point of furfural in this experiment was expected to be a little higher than 161.7 °C, but still lower than our steaming temperature of 200 °C. It was likely that furfural would vaporize following the degradation of hemicellulose, because the rice straw was only in contact with steam and not soaked in water. The possibility of vaporization of furfural was raised when steamexploded pulp was analyzed [15]. Our study analyzed not only the steamed samples, but also the drainage, and vaporization of furfural was confirmed.

In contrast, 5-hydroxymethylfurfural, a kind of hexose, remained in the rice straw (Table 2); its boiling point at 1 atm is 291.5 °C, so it would not vaporize under steaming conditions of 200 °C and 1.5 MPa. Although 2-acetyl furan and 5-methyl furfural are also hexoses, they vaporized and were detected in the drainage because their boiling points are lower than the steaming temperature (Table 2).

The origin of this 5-hydroxymethylfurfural should be discussed. Xylan in rice straw is a kind of arabinoglucuronoxylan, composed of the xylosyl main-chain attached partially to arabinofuranoxyl stubs and glucuronosyl stubs [16]. As glucuronic residue is a hexose, 5-hydroxymethylfurfural could be produced by the degradation of xylan. Based on the yield of the dichloromethane extracts after steaming for 20 min, the yields of furfural and 5-hydroxymethylfurfural were calculated as 1.60% and

^a Column: NB-1 (30 m \times 0.25 mm); 60 °C, 0 min - 10 °C/min - 260 °C, 25 min

^b Yield of dichloromethane extraction

^b Yield of dichloromethane extraction

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1.37%, respectively (note that the detector used in this study was not flame ionization detector (FID)). This yield of 5-hydroxymethylfurfural seems too high to have come only from hemicellulose, suggesting that a part of the cellulose decomposed and produced additional 5-hydroxymethylfurfural during the steam treatment. It has been reported that cleavage of glycosidic bonds, cleavage of some ether bonds in lignin, and cleavage of lignin—carbohydrate linkages occur during steam explosion [17]. These changes might result in the removal of very reactive amorphous cellulose on the surface. A similar reaction may have occurred in our study.

Lignin degradation

Lignin degradation products like vanillin, *p*-hydroxybenzaldehyde and syringaldehyde were detected in steamed rice straw. Their ratio to the whole dichloromethane extracts did not increase with increasing steaming time. However, it was expected that lignin degradation advanced with increasing steaming time due to the increased yield of dichloromethane extract.

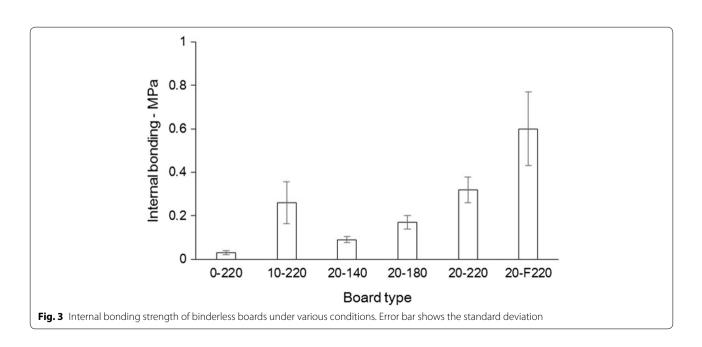
Properties of binderless boards Effect of steaming time

Comparing 0-220, 10-220 and 20-220 in Fig. 3, IB was improved by the steam treatment. The value of IB at 20-220, 0.32 MPa, was higher than 0.3 MPa, the JIS requirement for Type-15 medium-density fiberboard (MDF) [12]. This improvement was achieved by both physical and chemical changes during steam treatment as discussed below.

Rice straw has surface structures called protuberances on the epidermis. These were still observed on some particles after steam treatment (Fig. 2). Our previous study showed that these surface structures limited the bonding area and decreased IB [7]. However, in our study, most particles showed exposed inner structures, fibers and parenchyma cells (Fig. 2). With steam treatment, the negative effect of the surface structures decreased, and thus IB improved.

With longer treatment time, the higher yield of lignin degradation products were detected, indicating that lignin degradation might contribute to self-bonding. The content of hemicellulose and cellulose degradation products in steamed rice straw also increased with increasing steaming time. Although both furfural and 5-hydroxymethylfurfural can function like resins during hot-pressing, furfural which accounts for 1.60% of raw materials evaporated and could not contribute to the self-bonding, whereas 5-hydroxymethylfurfural which accounts for 1.37% remained in rice straw and had a role as bonding agent. The wax-like substances were not removed; nevertheless the disadvantage of wax-like substances in bonding was complemented with these chemical decompositions to increase self-bonding. It is common to add wax for water resistance in commercial MDF production. Therefore, this study showed one of the methods to use efficiently the wax-like substances of rice straw in binderless boards.

We previously produced a binderless board with IB of 0.46 MPa, made from rice straw pretreated by steam explosion at 205 °C, 2 MPa for 5 min, and formed at 200 °C, 7 MPa for 10 min [8]. The present study



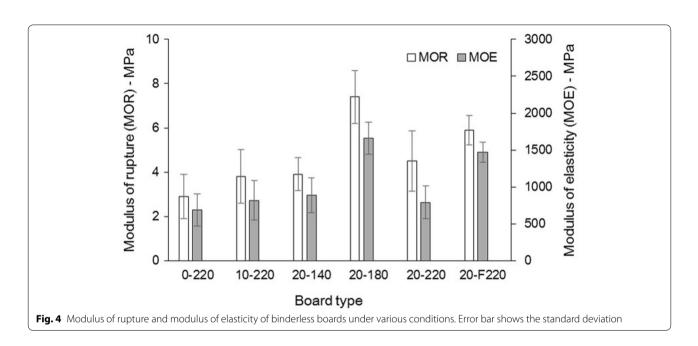
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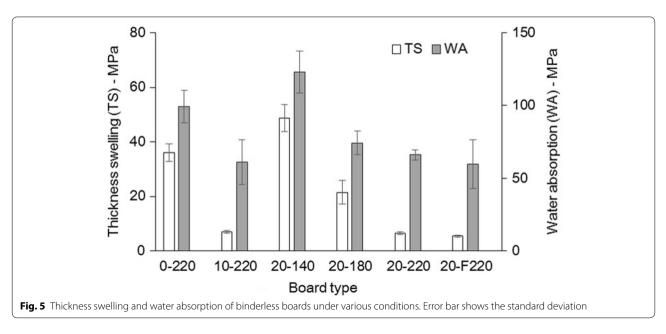
demonstrated that steam treatment was less effective for improving IB than steam explosion.

MOR and MOE did not increase significantly with steaming pretreatment (Fig. 4). We suspect that the negative effect of weakened fibers counteracted the positive effect of increased bonding area. TS and WA decreased with increasing steaming time (Fig. 5). The TS met the JIS requirement for MDF [12], indicating high water-resistance.

Effect of pressing temperature on board properties

IB increased with increasing pressing temperature (Fig. 3). The degradation products produced during not only steam pretreatment, but also hot-pressing might become thermo-plasticized and then fill the voids among particles, resulting in higher internal bonding strength. In contrast, MOR and MOE showed a different trend from IB (Fig. 4); MOR improved from 20-140 to 20-180, but decreased at 20-220. It was reported that thermal degradation might occur in the surface layer of boards [18]. In our study, the density at the surface of the board





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formed at 220 °C may thus have decreased because of the high pressing temperature, adversely affecting MOR. TS and WA improved with increasing pressing temperature (Fig. 5). Higher pressing temperatures will have induced hemicellulose degradation, resulting in a reduction in the number of hydroxyl groups easily accessible by water.

Effect of particle size on board properties

With the addition of fine-grinding, the particle size was reduced below 150 µm and the surface of the resultant board was extremely smooth. IB of 20-F220 was 0.6 MPa; approximately double that of 20-220 (Fig. 3). This satisfied 0.6 MPa, the JIS requirement for Type-30 MDF and the strictest applicable standard [12]. IB after the combined steam treatment and fine-grinding showed a value comparable to that after steam explosion. The steam treatment functioned as an alternative to the chemical changes during steam explosion, and fine-grinding as an alternative to the defibration process in steam explosion. In our previous study, we achieved IB of 0.6 MPa using dewaxing rice straw by hexane extraction [7]. The present study revealed that milder treatments than steam explosion and hexane extraction could still result in high bonding strength.

The mechanical properties MOR and MOE were also slightly increased (Fig. 4). The smooth surface of the boards made from finely ground powder might explain the positive effect on MOR. The water resistance properties TS and WA were also slightly improved, showing their lowest values (Fig. 5).

Conclusions

This study examined how the steaming time, pressing temperature and particle size affected the board properties IB, MOR, MOE, TS and WA. In addition, the chemical changes during steam treatment were investigated and discussed in relation to bonding strength. Our conclusions can be summarized as follows:

1. IB and water resistance significantly increased by steam treatment. They also increased with increasing pressing temperature. Steam treatment caused chemical changes in not only lignin and hemicellulose, but also cellulose. 5-Hydroxymethylfurfural decomposed from hemicellulose and amorphous cellulose remained in the rice straw and contributed to self-bonding. Lignin degradation products might also improve bonding strength. The negative effect of the remaining wax-like substances in bonding was complemented with these chemical decompositions to increase self-bonding. In contrast, furfural vaporized soon after its decomposition from hemicellulose, and did not remain in the steamed rice straw.

- 2. The combination of steam treatment and fine-grinding achieved a high IB of 0.6 MPa and a low TS of 5.4%, which met the JIS requirement for Type-30 MDF [12]. Even though the wax-like substances were not removed, IB showed a value comparable to that of board made from dewaxed rice straw. Moreover, this milder pretreatment achieved a higher IB than steam explosion. It seems that the steam treatment provided equivalent chemical changes to steam explosion, and fine-grinding equivalent physical changes.
- The optimal pressing temperature for MOR and MOE was much lower than that for IB. For further improvement of MOR and MOE, a milder manufacturing condition not to weaken fibers should be examined in future.

Therefore, steam treatment enhanced the self-bonding and water resistance of binderless boards made from rice straw.

Abbreviations

UF: urea formaldehyde; PF: phenol formaldehyde; MOR: modulus of rupture; MOE: modulus of elasticity; IB: internal bonding; TS: thickness swelling; WA: water absorption; JIS: Japan Industrial Standards; GC–MS: gas chromatography—mass spectrometry; FID: flame ionization detector; MDF: medium-density fiberboard

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Authors' contributions

MS made substantial contributions to the conception and design of the study and assisted in the preparation of the manuscript. YK contributed to the manufacture and testing of boards, chemical analysis and interpretation of data, and was a major contributor in writing the manuscript. Both authors critically reviewed the manuscript. Both authors approved the final version of the manuscript, and agree to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved. Both authors read and approved the final manuscript.

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Availability of data and materials

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Competing interests

The authors declare that they have no competing interests.

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