The Properties of Particleboard Made from Alkaline-treated Wheat Straw and Methylene Diphenyl Diisocyanate Binder

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Properties were evaluated for particleboard made from polymethylene polyphenyl polyisocyanate (pMDI) binder and wheat straw treated with sodium hydroxide (NaOH) and ammonia hydroxide ($NH_3 \cdot H_2O$) solutions. The crystallinity (X_c) of wheat straw and board properties including internal bond strength (IB), thickness swelling after 24 h (24 hTS), modulus of rupture (MOR), and modulus of elasticity (MOE) were investigated. XRD indicated that the crystallinity of wheat straw was increased with the mass fraction range from 1% to 3%, and the bending properties of board were also improved. MOR and MOE were significantly ($P < 0.05$) improved, and a positive correlation with solution mass fraction was shown. Bending properties, especially MOE, were superior to the requirements in GB/T 4897-2015. IB of NaOH solution treated board was also significantly higher ($P < 0.05$), but results for IB of ammonia solution treated and 24 hTS for board treated with both solutions failed to meet the standard. Effects of the two solutions were not the same according to a t-test, and the effects of NaOH were better.

Keywords: Particleboard; Wheat straw; Alkaline treatment; Ammonia; Polymethylene polyphenyl polyisocyanate (pMDI)

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INTRODUCTION

Wheat (*Triticum aestivum* L.) is the second most widely cultivated crop in the world. A large amount of wheat straw is produced and needs to be reused. In China, about 112 million tons, representing 21.6% of the total production of crop straw, was burned in fields (Li and Wang 2013). The open burning of straw is not only a waste of resources, but also causes air pollution. About 20% of all the agriculture straw is wheat straw. Thus, its industrialized utilization in pulp, bioethanol, pellet fuels, or particleboard manufacturing could be very important.

Particleboard is a very important wood composite product. Unlike wood, the bond strength of straw board is poor when using UF resin or PF resin because of the waxy layer on the surface of straw (Cheng *et al*. 2004) and a higher silica content (Halvarsson *et al*. 2010). An alternative high-performance binder, methylene diphenyl diisocyanate (MDI), has been adopted. The bond strength is mostly formed by urethane linkages generated by the reaction between –N=C=O group in MDI and –OH groups in lignocellulosic materials (Johns 1982). This is usually sold and used as PMDI (polymeric MDI) (Papadopoulos 2006) in wood industry. The bond properties and water resistance are excellent, and the resin content is much lower than UF resin or PF resin. Furthermore, the formaldehyde emission of board is almost zero, making it an environmentally friendly binder.

MDI is the preferred binder in manufacturing boards with agriculture residues (Sellers 2001). However, MDI is expensive and mostly used as a structural binder. The properties of wood-MDI particleboard are very stable after being exposed to outdoor conditions for 5 years, as the IB still exceeds standard requirements (Korai *et al*. 2015). Compared with the boards made of MDI and wood particles, the properties of straw board are poor, and the straw should be pretreated if a higher quality straw board is required.

There are several methods of pretreatment for wheat or rice straw before particleboard manufacturing. These methods include enzymes (Zhang *et al*. 2003), oxalic acid and steam (Li *et al*. 2011), acid, and alkaline treatments (Liu *et al*. 2004; Zhang and Hu 2014). Alkaline treatment, or NaOH treatment, is a very effective method for the pretreatment of wheat straw for bio-ethanol manufacturing because it dissolves lignin and hemicellulose (Wan *et al*. 2011). Additionally, the NaOH treatment breaks ester linkages between lignocellulose and the wax layer on the exterior surface of straw (Binod *et al*. 2010). Liu finds that the surface wax layer becomes rough as a consequence of NaOH solution treatment and calls this phenomenon "chemically etched"; the wettability of binders is also improved by such treatment (Liu *et al*. 2004). The removal of the wax layer increases the bonding and does not decrease the water resistance (Kurokochi and Sato 2015); thus, it is very important in improving the properties of straw particleboard. $NH_3 \cdot H_2O$ is easily recycled and also breaks the ester linkages of wax on the surface of straw (Yang *et al*. 2012), so this method also would be effective.

The NaOH treatment of particles has been shown to improve the properties of wheat straw particleboard. Research involving $NH_3 \cdot H_2O$ pretreatment in board manufacturing has been uncommon, though in theory such treatment would be feasible. This research evaluated the properties of boards made from wheat straw after two different alkaline treatments and also compared the difference between the two alkaline solutions.

EXPERIMENTAL

Material Preparation

The wheat straw (*Triticum aestivum* L.) was collected in Henan Province, Central China, stored for more than 3 months, and then processed to particles using a hammer mill. The particle length was mostly between 5 mm and 10 mm. Particles were screened by standard sieves to a size between 0.70 mm (24 mesh) and 1.40 mm (12 mesh) to manufacture particleboard.

PMDI (WANHUA WANNTE® PM-200, Wanhua Chemical Group Co., China) was used as a binder; the solid content was considered 100%. NaOH powder (analytical reagent (AR), Beijing Chemical Works, Beijing, China) and 25 wt.% ammonia (AR, Beijing Chemical Works) were dissolved into 1 wt.%, 2 wt.%, and 3 wt.% solutions in distilled water separately.

The straw was sprayed with an alkali aqueous solution at a ratio of 1 mL to 1 g (oven dried weight), for each group 800 g wheat straw (original moisture content = 9%) was selected, and the consumption was 720 mL. The treated straw was stored at room temperature (25 °C) for 12 h and then dried in an air-dry box (DF82-8, Beijing Tongxian

Experiment Instrument Works, China) at 90 °C to reduce the moisture content to 5% prior to processing.

Particleboard Manufacturing

MDI binder was dissolved in acetone (1:1 by weight) and mixed with straw particles in a blender (FTR 250, Draiswerke GmbH, Mannheim, Germany) using a spray gun at a pressure of 0.40 MPa. The resin content was 4%, as calculated by the ratio of resin consumption and the oven-dried weight of the particles.

The mat was formed in a paving mold on steel plates coated with Teflon film, and then it was pressed at 3.5 MPa for 7 min in a hot-presser (BY302 × 2/15 150T, Xinxieli group, Suzhou, China). The temperature was 180 °C, and the thickness of the board was conditioned to 10 mm iron bars. To reduce the error, two boards were made for each formulation. After hot-pressing, the board was cut into 300 by 300 mm pieces and conditioned at room temperature for more than 24 h until a constant weight was reached.

Property Testing

The board properties, including internal bond strength (IB), thickness swelling after 24 hours (24hTS), modulus of rupture (MOR), and modulus of elasticity (MOE) were tested according to GB/T 17657-2013 (2013). 24hTS was measured by using a 0 to 25 mm micrometer; IB, MOR, and MOE were tested by a mechanical testing machine (MWW-50, Tianhua Test Device Co., Jinan, China). The standards for boards are described in GB/T 4897-2015, this standard is equivalent to ISO 16893-2: 2010.

Internal Bond Strength

To test IB, the specimens were cut into 50 × 50 mm pieces, and 5 specimens were selected from each board (Fig. 1A). The load speed was 2 mm/min and IB was calculated by Eq. 1,

$$\mathrm{IB} = \frac{F_{max}}{bl} \quad (1)$$

where IB represents the internal bond strength (MPa), $F_{\max}$ represents the maximum load of tensile failure (N), l is the length of specimen (mm), and b is the width of specimen (mm).

Thickness Swelling

To test 24hTS, the specimens were cut into 50 × 50 mm pieces; 5 specimens were selected from each board and soaked in distilled water (pH 7 ± 1, temperature 20 ± 1 °C) for 24 h. The thickness was measured by a micrometer and was finished within 10 min. The 24hTS was calculated by Eq. 2,

$$\mathrm{24hTS} = 100\frac{t_2 - t_1}{t_1} \quad (2)$$

where t_1 represents the initial thickness of specimen (mm) and t_2 represents the thickness of the specimen soaked after 24 h (mm).

Static Bending Properties

The MOR and MOE were tested in a 3-point bending test. The specimens were cut to 250 by 50 mm pieces, and for each board, 3 specimens were chosen. The test is displayed in Fig. 1B . MOR and MOE was tested with a load speed of 10 mm/min and calculated by Eqs. 3 and 4 respectively,

$$\sigma_b = \frac{3F_{max}\, l_1}{2bt^2} \tag{3}$$

$$E_b = \frac{l_1^3}{4bt^3}\left(\frac{\Delta F}{\Delta x}\right) \tag{4}$$

where σ_b represents MOR (MPa), E_b represents MOE (MPa), F_{max} represents the maximum load of failure (N), b represents the width of the specimen (mm), l_1 is the length of the span (mm), t is the thickness of the specimen (mm), and ΔF (N) and Δx (mm) represent the variation of load and deformation of the straight-line portion of the load-deflection curve, respectively.

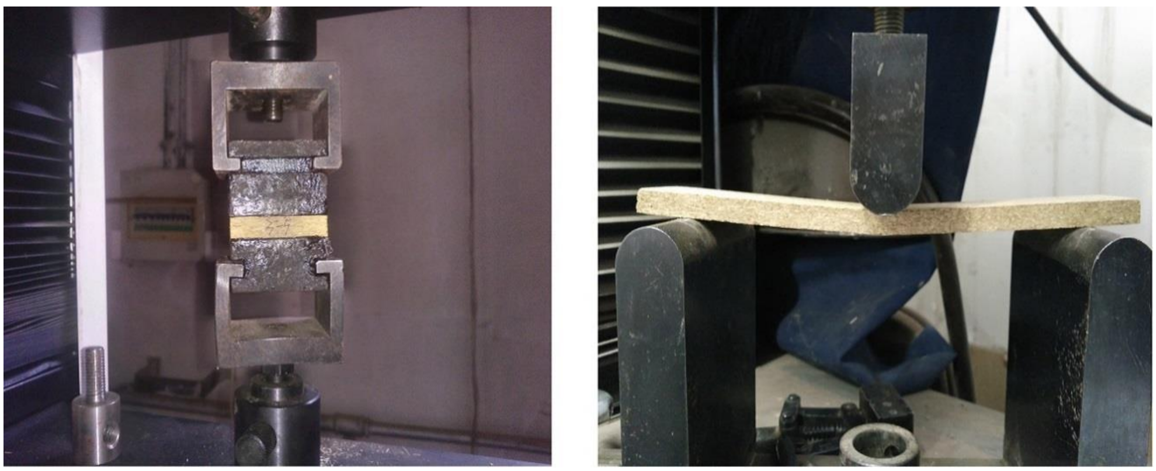

Fig. 1. Test of internal bond strength (L) and static bending properties (R)

Crystallinity of Wheat Straw

Wheat straw particles are ground to 0.250 mm (60 mesh) and scanned by X-Ray Diffractomer (D8 Advance, Bruker AXS GmbH, Karlsruhe, Germany). The crystallinity of cellulose (X_c) was calculated through software DiffracPlus EVA (Bruker AXS GmbH, Karlsruhe, Germany) by Eq.5,

$$X_c = \frac{A_c}{A_c + A_{am}} \times 100 \tag{5}$$

where A_c represents the area of crystalline phrase and A_{am} represents the area of amorphous phrase.

Statistical Analysis

One-way analysis of variance (ANOVA) was adopted to compare the relationship between each property and the mass fraction of the alkaline solution. SPSS Statistics 23 software (IBM, Armonk, United States) was used for the analysis with a confidence level of 95% ($\alpha = 0.05$). $p < 0.05$ was considered a significant difference in the mass fraction of alkaline solution.

Requirements in GB/T 4897-2015

The general requirements of particleboard used as load-bearing board in dry or humid conditions are shown in Table 1. Besides, optional tests inducing IB were carried out after the boiling test or MOR after 70 °C the dipping test if the board could be used in humid conditions.

Table 1. Requirements of Particleboard (thickness =10 mm) Used as Load-Bearing Boards in Dry Conditions in GB/T 4897-2015

	IB	MOR	MOE	24hTS
Dry	> 0.40 MPa	> 15 MPa	> 2200 MPa	< 19%
Humid	> 0.45 MPa	> 17 MPa	> 2450 MPa	< 13%

RESULTS AND DISCUSSION

Static Bending Properties: MOR and MOE

The results of MOR and MOE are shown in Table 2 and illustrated in Figs. 2 and 3. The MOR of NaOH-treated straw particleboard increased from 18.29 MPa to 26.62 MPa and 22.65 MPa, respectively, when the alkaline mass fraction of NaOH and $NH_3{\cdot}H_2O$ increased from 0% to 3%.

Table 2. Properties of Boards Manufactured in this Research

Sample	Density (g/cm^3)	IB (MPa)	MOR (MPa)	MOE (MPa)	24 hTS (%)
Untreated	0.723 (4.15)	0.30 (16.29)	18.29 (13.30)	2070 (10.81)	22.75 (10.06)
1% NaOH	0.714 (6.00)	0.48 (10.42)	20.62 (8.77)	2280 (7.51)	22.41 (13.78)
2% NaOH	0.718 (4.96)	0.53 (12.09)	25.25 (5.90)	2570 (10.06)	22.11 (13.96)
3% NaOH	0.724 (3.01)	0.55 (9.62)	26.62 (8.04)	2880 (5.76)	20.81 (11.15)
1% $NH_3{\cdot}H_2O$	0.694 (5.79)	0.25 (14.05)	18.84 (9.92)	2430 (11.80)	24.61 (8.53)
2% $NH_3{\cdot}H_2O$	0.710 (6.06)	0.26 (19.49)	19.01 (6.05)	2890 (10.17)	25.63 (15.18)
3% $NH_3{\cdot}H_2O$	0.721 (6.12)	0.31 (12.57)	22.65 (8.31)	3070 (9.16)	24.19 (11.29)
Note: Numbers in the parentheses denote coefficient of variation (C.V. %).					

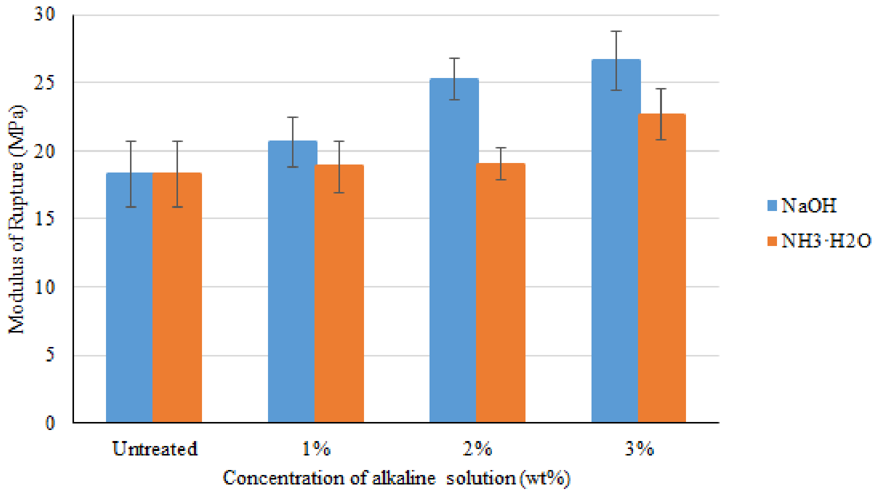

Fig. 2. Modulus of rupture and the comparison of two pretreatment methods

A statistical difference was found for NaOH ($p = 0.000 < 0.05$) and $NH_3{\cdot}H_2O$ ($p = 0.019 < 0.05$). Thus, the mass fraction of alkaline had significant impact on MOR, which is also reflected in the bar chart. The MOE increased from 2070 MPa to 2880 MPa and 2070 MPa to 3070 MPa for NaOH and $NH_3{\cdot}H_2O$, respectively, when the alkaline mass fraction increased from 0% to 3%. The mass fraction of NaOH ($p = 0.000 < 0.05$) and $NH_3{\cdot}H_2O$ ($p = 0.000 < 0.05$) had significant impact on MOE. MOE showed a positive correlation with the mass fraction and it was obviously that the effect of $NH_3{\cdot}H_2O$ was better than NaOH.

The strength of particleboard is formed mainly between MDI and cellulose hydroxyls. The improvement of MOR and MOE and of alkaline-treated boards may be because of increased crystallinity. Generally speaking, the MOE of materials such as plastic, timber, and wood-based composites are improved with increased crystallinity. Increased cellulose crystallinity could increase the MOR and MOE of boards (Wu *et al*. 2011). NaOH solution treatment destroys the amorphous region of the cellulose first (Zheng *et al*. 2012). Kim also found the ammonia treatment would remove the amorphous part of biomass (Kim *et al.* 2003). Thus, the crystallinity of cellulose in the straw may increase and the bending properties may be improved. This phenomenon could also certified by XRD spectrum in Fig. 4. The crystallinity of untreated wheat straw was 32.80% and the crystallinity (X_c) raised to 35.90% and 36.90% by NaOH and ammonia solutions treated.

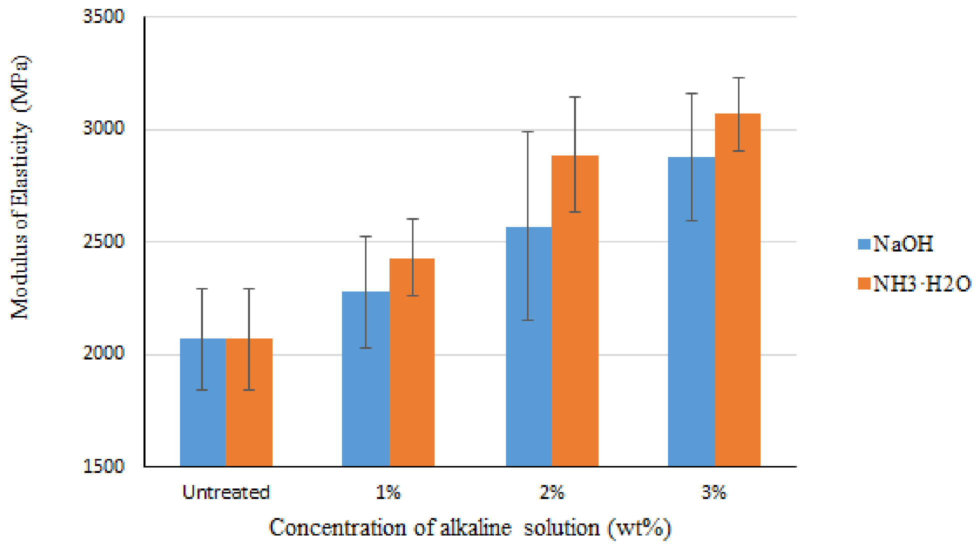

Fig. 3. Modulus of elasticity and the comparison of two pretreatment methods

The MOR of NaOH-treated board was significantly improved, and MOE of $NH_3{\cdot}H_2O$ treated board was better at the same alkaline mass fraction. Alkaline solution treatment could improve the bending properties of particleboard, and the properties were superior to the requirements for use as a load-bearing board in dry (MOR > 15 MPa, MOE > 2200 MPa) or humid conditions (MOR > 17 MPa, MOE > 2450 MPa) as specified in GB/T 4897-2015.

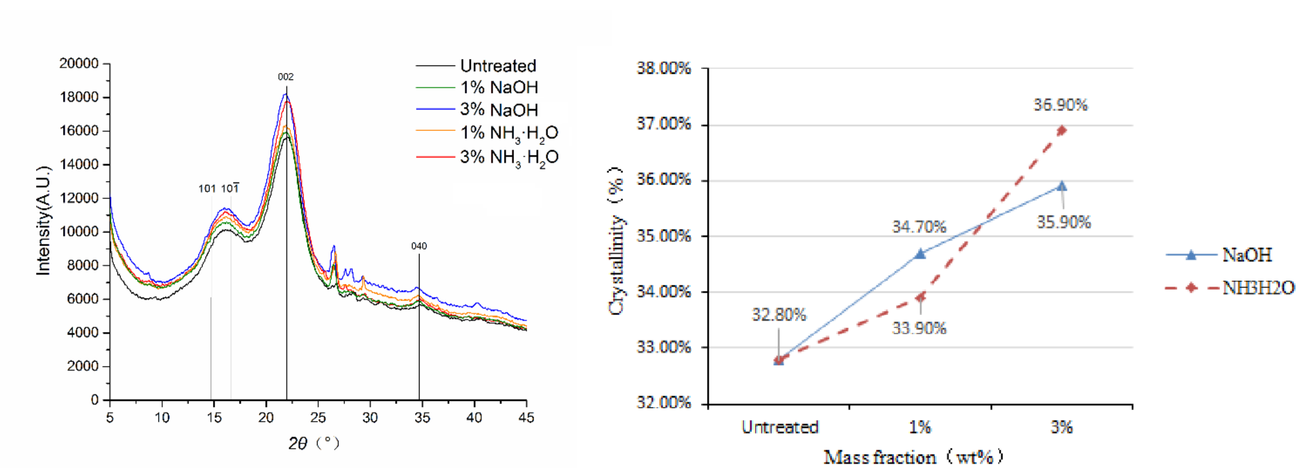

Fig. 4. XRD of untreated and alkaline solution treated wheat straw particle

Internal Bond Strength

The results are shown in Table 2 and illustrated in Fig. 5. For the NaOH treatment, the average IB increased from 0.30 MPa to 0.55 MPa when the mass fraction increased from 0% to 3%. For $NH_3{\cdot}H_2O$ treated boards, the value was 0.30 MPa, 0.25 MPa, 0.26 MPa, and 0.31 MPa when the mass fraction increased from 0% to 3%. A statistically significant difference (p =0.000 < 0.05) was found between mass fraction of NaOH and the IB of 3 wt.% treated board is 83% higher than the untreated. The results are also reflected in Fig. 2. Alkaline treatment by NaOH could increase the specific surface of particle by reducing the content of silica and waxy layer (Han *et al.* 1998) and resulting in more chemical linkages formed with binder and increased bonding quality. Additionally, the alkaline solution treatment could also improve the wettability on the surface of straw, which could improve the penetration ability of binder (Shen *et al.* 2011) and achieve higher bond strength.

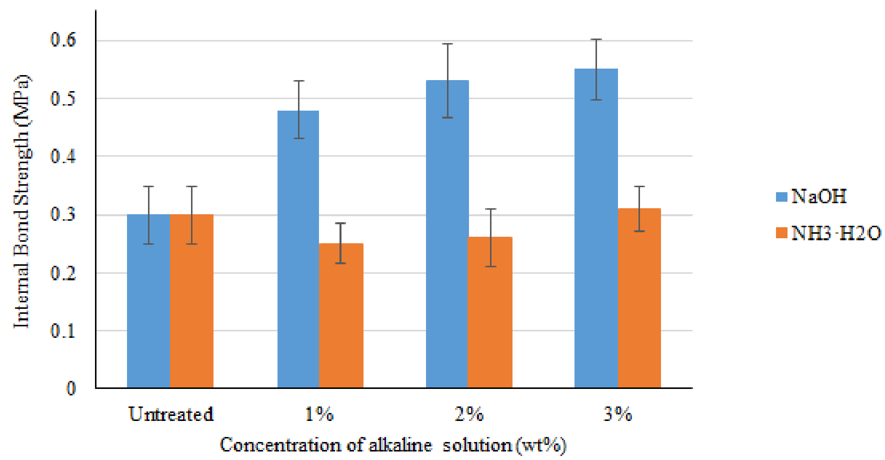

Fig. 5. Internal bond strength and the comparison of two pretreatment methods

Unlike NaOH, $NH_3 \cdot H_2O$ could not improve the IB of board. Besides, ANOVA indicated that no statistically significant difference ($p = 0.615 > 0.05$) was found between IB and mass fraction of $NH_3 \cdot H_2O$.The reason could be the reaction between NH_3 and MDI binder and the impact of hydrogen bond.

MDI binder could react with the residual NH_3 or $-NH_2$ groups and form urea and biuret keys (Johns 1982), and the related reaction could partly substitute the reaction between binder and lignocellulosic materials and so the bonding efficiency would be decreased. Besides, NH_3 could partly form $OH \cdots NH_3$ keys instead of $OH \cdots OH$ keys between cellulose molecules (Yang *et al.* 2012). Therefore, the existence of $H \cdots N$ hydrogen bond would impact the bond between cellulose, lignin, and hemicellulose and result in a weaker bond strength.

IB of NaOH solution treated board was significantly improved and superior to the requirement in the related standard, but the $NH_3 \cdot H_2O$ solution treatment could not improve the IB.

Thickness Swelling

The thickness of swelling after 24 h is shown in Table 2 and illustrated in Fig. 6. The 24 hTS for the NaOH-treated board decreased from 22.75% to 20.81% when the mass fraction increased from 0% to 3%. There was a slightly negative correlation with mass fraction, but no significant difference was found ($p = 0.579 > 0.05$). Thus, the influence of mass fraction in 24 hTS was not significant.

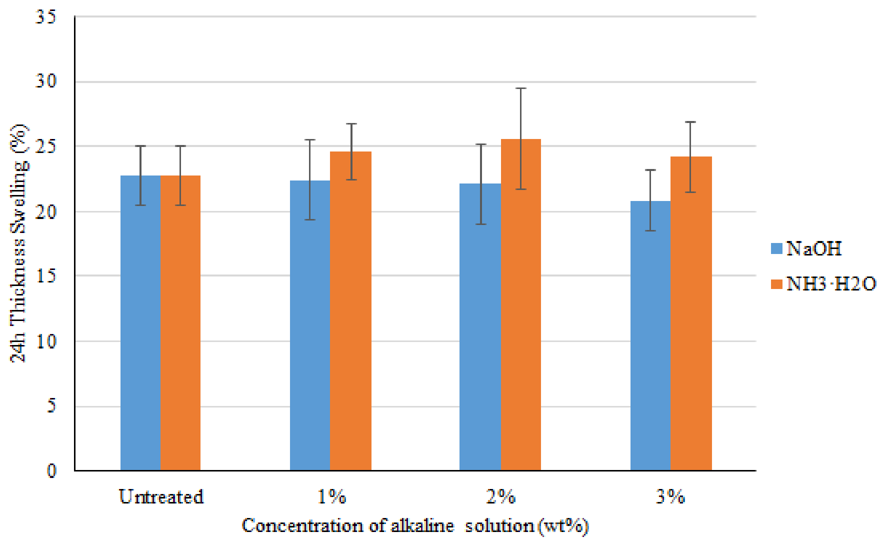

Fig. 6. 24-h thickness swelling and the comparison of two pretreatment methods

$NH_3 \cdot H_2O$ solution treatment slightly increased the 24 hTS of board, but the correlation between mass fraction and 24 hTS was the same as NaOH-treated board and the impact of mass fraction on 24 hTS was also not significant ($p = 0.388 > 0.05$).

Alkaline treatment could remove hydrophobic lignin and hydrophilic hemicellulose partly and the change of crystallinity could also affect the dimensional stability of board. It was difficult to explain the change of 24 hTS with the above factors. But it was known that thickness swelling was affected by bond quality and adhesive properties (Mo *et al.* 2003), which could inhibit the 'spring-back' of board (Okino *et al.* 2004). IB and 24 hTS also showed a negative correlation in this experiment, as illustrated in Fig. 7. Thus, the change of 24 hTS could be related to the change in IB. Besides, the change in crystallinity could also impact the dimensional stability of board, but the impact seemed to be not significant in this experiment.

The thickness swelling of particleboard could not meet the requirement of board used as load-bearing board in dry conditions (24 hTS < 19%)

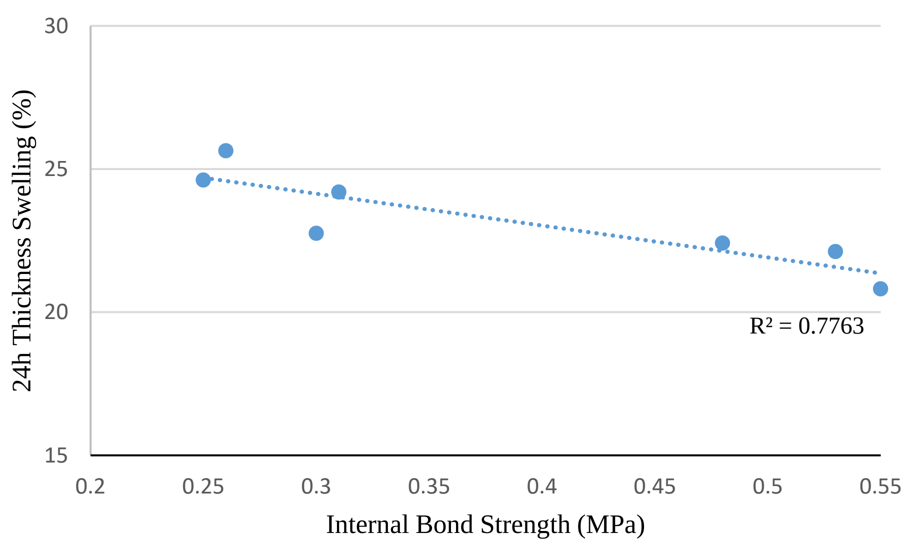

Fig. 7. Correlation between 24 hTS and IB in this experiment

Difference between NaOH and $NH_3 \cdot H_2O$ solution on properties

T-test of samples was given in Table 2 to evaluate the difference between the two alkaline solutions. The differences of IB, 24 hTS and MOR were significant ($P < 0.05$), which meant that the results of NaOH and $NH_3 \cdot H_2O$ were different. The difference of MOE was not significant ($P > 0.05$), so the impact of NaOH and $NH_3 \cdot H_2O$ solution on MOE was the same. According to the t-test, there was a significant difference between the NaOH and $NH_3 \cdot H_2O$ solution. Based on the results, the effect of NaOH solution treatment on improving the properties of board was better than $NH_3 \cdot H_2O$ solution.

Table 2. Independent Samples T-test of Properties

	t-test for equality of means						
	t	df	P-value	Mean difference	Std.Error difference	95% confidence interval	
						Lower	Upper
IB	13.498	32.240	0.000	0.2478	0.018	0.21040	0.28516
24hTS	-2.230	32.896	0.033	-2.1272	0.954	-4.06779	-0.18653
MOR	3.656	26.368	0.001	3.999	1.094	1.75258	6.24671
MOE	-1.648	26.422	0.111	-219.458	133.133	-492.90515	53.98894

CONCLUSIONS

1. XRD indicated that the crystallinity was increased by alkaline treatment and showed a positive correlation with mass fraction in this experiment. Statistical differences were found between bending properties (MOR and MOE) and mass fraction. MOR and MOE were both significantly improved, and the highest values were all found at a mass fraction of 3%.
2. The internal bond (IB) strength of NaOH-treated board met the requirements of load-bearing board in dry or condition. Both solutions could increase MOR and MOE and the values were superior to the requirements but IB of $NH_3 \cdot H_2O$ solution treated and the 24 hTS of all the specimens in this experiment were fail to meet the requirement.
3. NaOH or $NH_3 \cdot H_2O$ solution treatment could be used to improve some properties of wheat straw particleboard. Compared with $NH_3 \cdot H_2O$, the effect of NaOH was better at the same mass fraction. A mass fraction of 3% showed the best results in this experiment.
4. Statistical difference was found between the effect of NaOH and $NH_3 \cdot H_2O$ solution treatment on the properties of board. The effect of NaOH treatment was better.

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