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THERMOPRESSED BINDERLESS FIBERBOARDS FROM WHEAT STRAW BY ADDING BLACK LIQUOR

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ABSTRACT

For the shortage of timber resources and the sake of the formaldehyde emissions, people desire to use non-adhesive bonding technology. This paper studies the chemical composites of black liquor, at different contents ranging from 20 to 40 wt%, into fiberboards made from wheat straw pulp. Adding a little black liquor has positive effect on qualities of boards, contributing to presence of proteins and lignin in black liquor, but adding too much liquor would decrease properties of them for the ash content. The FT-IR measurements indicated that there are more low-molecular substance and hydrogen bonds producing after fining and thermopressing processes. The thermo analysis were conducted to better understand these results. The physical and mechanical properties of the resulting fiberboard were evaluated. The results showed that binderless fiberboards by adding 30 wt% have good mechanical and water resistance properties which can partly satisfy the requirements of the relevant standards specifications.

KEYWORDS: Wheat straw, pulp, black liquor, self-bonding, fiberboards.

INTRODUCTION

Using wood for pulp cause the large-scale felling of trees and replantation, which poses risk of negative ecological balance and makes for the climate - changer. Wheat straw fiber is an agriculture residue regularly discarded in the land on farming and harvesting. The cellulose content in water straw is marginally less (43%) than hardwood (50%-64%) (Wang and Zhou 2009). However, it can fill the shortage of wood materials given the probability of gaining cellulosic pulp with cellulose components, and hence has potential applications in papermaking or board with different features, in particular regions without rich forest resources.

Bio-composite fiberboards are usually bonded with petroleum-based adhesive from non-renewable resources (Galperin et al. 1995). For instance, phenol-formaldehyde (PF) and urea-formaldehyde (UF) resins can be used as binders, taking about approximate 60% of the total cost during fiberboard manufactures (Abdul Khalil et al. 2010, Hashim et al., 2010). Though this kind of boards have satisfactory mechanical properties (ANSI A 208.2 - 1994), the emission of formaldehyde from the resins in the composites has toxic effect on human involving multisystem organ. California Secretary of State had put formaldehyde, benzene and a variety of other substances in the list of the potential carcinogenicity of compound (Broder et al. 1998). With the concerning of environment and regimen, some researchers focus on investigation of producing self-bonding boards using different raw materials since the 1980s (Jain and Handda, 1982). Woody raw materials, such as spruce, pine (Anglès et al. 2001, Anglès et al. 1999), *P. radiata* (Riquelme et al. 2008), spruce's fiber (Widsten et al. 2003), beech and rubber's fiber (Felby et al. 1997a 2004, Nasir et al. 2013), and non-woody raw materials, like coconuts (Van Dam et al. 2004), plantain (Álvarez et al. 2011), cotton stalks (Fahmy and Mobarak 2013), bamboo (Shao et al. 2009) and kenaf core (Okuda et al. 2006a) used in production of binderless fiberboard by hot pressing or steam injection (Xu et al. 2006). Halvarsson et al. (2008) attempted to characterize the wheat straw fibers as a low-cost raw material for binderless fiberboard. In order to reinforce fiber interaction proving desirable mechanical performance, Feton's reagent (Mejía et al. 2014) and enzymatic (Kharazipour and Euring 2010, 2013, Euring et al. 2011a, b; Kudanga et al. 2011) and addition other composition (Mansouriet al. 2007, Bouajila et al. 2005, Anglès et al. 2001) were used for treating the raw materials. However, the fabrication of binderless boards are confined in the laboratory because the complex process. Simplifying the fabrication process is a key of promoting the industrialization production.

Black liquor, the pulping effluent in fiber and paper making, is a complex mixture of inorganic and organic component which mainly containing lignin, polysaccharide, and some other ingredients used in the pulping process (Rastegarfar et al. 2015). These wastewater could cause water quality pollution, for instance, lignin and phenolic in black liquor are given rise to chemical oxygen demand (COD) and high color (Ghatak et al. 2002). But it also has some components which contributing to fibers' adhesive (Geng et al. 2007b, Zerhouni et al. 2009). Utilizing the bonding characterize of black liquor and manufacturing fiberboards could save water and pursue the worldwide aimed zero waste strategy. Nowadays, the exploitation of black liquor is trivial, complex and low efficiency (Dyna et al. 2017) which is difficult to adapt industrial production.

Under this state of affairs, the current work was projected to achieve two purposes. The first is to obtain the binderless fiberboards by adding black liquor directly with zero waster goal and the influence of adding different content black liquor on the product was investigated through performance testing. The second longer objective is to assess each component of the raw materials does.

MATERIALS AND METHODS

Materials

Wheat straws, black liquor and wheat straw pulps were supplied by Rong Yeda Fiber Technology Co. in Dongping Country of Shandong Province, China. The pulp were passed through a defibrator by alkaline. Before testing, straws and pulp were air-dried for 3 days at room temperature. Black liquor was dried by lyophilization (the type of LJC-10C) for 48 h.

Binderless fiberboards producing

The pulp fibers were brought together with the black liquor in different proportions (0, 20, 30 and 40 wt%). And then, the plate blanks were dried to 30% of moisture content. Panel target density was 800 kg·m^{-3} with a target of 1.50 mm, pressing temperature was 160°C, and pressing cycle was 460 s (including the of closing and opening). Fig. 1 shows the pressing curve.

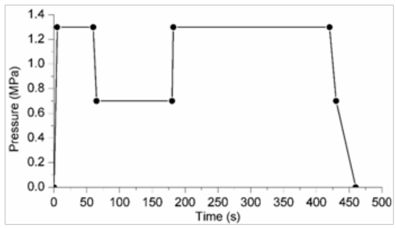

Fig. 1: Pressing curse.

The analytical method of the main chemical components

Wheat straw and pulp fibers were ground to pass through a 425 mm sieve but not to pass a 250 mm sieve by plant mill. The cellulose content of samples was determined by the traditional method as it cannot be dissolved in alkali solution (Wang 2012). Ash content was determined on ignition at 600°C for 4h, according to the GB/T742 (2008). Extractive content with different liquid and acid-insoluble lignin was determined by filtering the soluble matter (GB/T2677.4 1993, GB/T 2677.5 1993, GB/T10741 2008). The Klason lignin component of all lignin was verified by the conventional approach as the segment left insoluble after two-step sulfuric acid hydrolysis (GB/T747 2003). The pH of black liquor was measured by PHS-3C and use ultraviolet spectrophotometric to determine the content of protein by Waddel (1956).

The analytical method of physical and mechanical characterization

The modulus of elasticity (MOE), modulus of rupture (MOR), water absorption (WA), thickness swell (TS) and internal bonding strength of the boards were obtained for each samples, as the Chinese standards (GB/T17657, 2013).

The analytical method of fourier transform infrared (FT-IR)

FT-IR analyses were measured by KBr method on Nicolet IS10. All spectra was collected in the wave number range from 3000 to 400 cm^{-1} with a dpi of 4 cm^{-1} and at least 32 scans each specimen.

The analytical method of thermo gravimetric analyses (TGA)

TGA were processed on raw materials and boards. Thermal transitions were measured by TA Instruments Q100 TGA at a heat-up speed of 10°C·min^{-1} under the air atmosphere. The samples were heated from the ambient temperature to 550°C.

RESULTS AND DISCUSSION

Tab. 1 shows the consequences of chemical constituents of wheat straw and pulp. Comparative results revealed that wheat straw had been changed during the refining process. The lignin content in pulp did not varied much before treated, which is parallel to that got in milled wood lignin by Migneault et al. (2011).

Tab. 1: Chemical composition of wheat straw and pulp.

Characteristics		Wheat traw	Wheat straw pulp	Spruce[a]	*Robinia pseudoacacia*[a]
Ash (%)		8.32	2.17	0.26	1.30
α-cellulose (%)		55.68	32.27	44.04	21.80
Holocellulose (%)		60.00	33.97	-	-
Lignin (%)		12.80	12.61	26.20	21.80
Extractives (%)	Cold water	14.22	47.59	0.26	5.65
	Hot water	15.84	53.12	1.19	9.17
	1%NaOH	37.84	58.49	11.09	20.68
	Alcohol-benzene	1.49	4.71	3.51	8.16

[a] Wang. (2002)

The α-cellulose and holocellulose content in pulp were much lower than wheat straw. Also, the ash content in straw was higher than the pulp and 73.92% of it had been moved away. Because silicates and mineral components which are the major components in the ash had been removed by its treatment in the presence of sodium hydroxide (Mancera et al. 2011). Comparing with wood resources, straws have moderate cellulose and low lignin percentages that recommended it suitability as alternative wealth of wood fibers used in boards. There are a variety of secondary components in straws, like the extractives, apart from cellulose, hemicellulose and lignin. Similar values were reported in the references for some other agriculture residues and materials that came into being fine fiberboards without supplement, such as the fibers and pith of coconut husk (α-cellulose: 36.3%, 21.0%; klason lignin: 31.9%, 24.1%) (Van et al. 2004). The extractives all increased a lot after refining, especially through hot water and 1% sodium hydroxide solution. This revealed that a lot of tannin, oligosaccharide and aromatics were produced and it may be positive to form covalent bonds which can be formed between lignin and carbohydrates, the lignin-carbohydrate complexes (LCCs), use as fixing points where the lignin and carbohydrates produce covalent bond between each other. The bonds could enhance the mechanical properties of binderless boards by mechanical entanglement though the softened lignin molecules under high heat and pressure. Tab. 2 shows the properties of black liquor. Resin, fat and wax can be extracted by alcohol- benzene, very little in black liquor. There was a little lignin in the liquor and its addition can facilitate the mechanical properties of boards (Zhu et al. 2014). Protein-rich liquor was expected to play a positive role for internal bond strength, as mentioned by the application of proteins in wood adhesive formula (Pizzi and Mittal 2003, Rowell 2005).

Tab. 2: Chemical composition of black liquor.

Characteristics	Values
pH	9.17
Density ($g \cdot ml^{-1}$)	1.00
Dissolved solids concentration ($g \cdot l^{-1}$)	17.50
Extractive (alcohol-benzene) (%)	0.09
Ash ($g \cdot ml^{-1}$)	752
Lignin ($g \cdot ml^{-1}$)	190
Protein ($g \cdot ml^{-1}$)	2.95

FT-IR analyses was performed to understand bonding properties of fibers. Raw materials and boards spectra are given for comparison in Fig. 2.

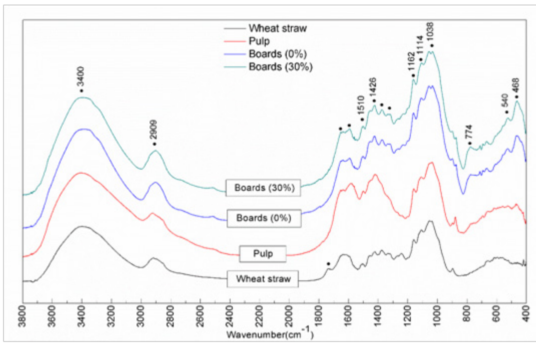

Fig. 2: FT-IR spectra of wheat straw, pulp and binderless boards made with 0 wt% and 30 wt% black liquor.

According to literature on biomass materials and pulp (Pandey 1999). The board band centered at around 3400 cm^{-1} was assigned to O-H and N-H stretching vibration. From straws to pulp, and then to boards with different content of black liquor, the peak shape of the absorption peak about O-H bonds became broaden. This demonstrates pulping, pressing and adding black liquor produced more hydrogen bonding. Cellulose and xylan had characteristic band ant around 1000-1200 cm^{-1} ascribed to alcoholic C-O stretching vibration. This indicates C-O-C bond angle vibration contributing to hemiacetal shows that there were more hemicellulose hydrolyzing and generating more hydrolysis of aldehyde compounds, like hydroxymethylfurfural and furfural, after pulping and adding black liquor. The band at 1730 cm^{-1} disappeared in the sample of pulp and boards, this result suggests that hemicellulose hydrolyze and produce furfural, furfuryl alcohol, organic acid and other organic matter. Lignin has a characteristic absorption and distinguishable from cellulose and xylan at around 1500 cm^{-1} contributed to aromatic ring framework vibration of polyphenol moieties. Lignin were existent in all samples. These monomers were supposed to generate lignin–furfural linkages or spontaneous polymerization during hot-pressing process, which can improve the main self-bonding strength of binderless fiberboards. As previously reported (Fahmy and Mobarak 2013, Sun et al. 2014, Suzuki et al., 1998, Tshabalala et al. 2012) and discussed, potential self-adhering mechanism occurred in the fabrication of fiberboards without adhesive. The main bond vibration bands which represent protein are 330, 3100, 4690-1600, 1575-1480, 1301-1229, 767-625, 800-640, 606-537 and 200 cm^{-1} (Haris 1994).

However, these bands were overlapped by those of straws. The aliphatic N-H out-of-plane bending can only find in spectra of pulp and boards. Si-O at 1100 cm^{-1} occurred in all spectra and that shows there are SiO_2 in the samples which has bad effect on mechanical properties.

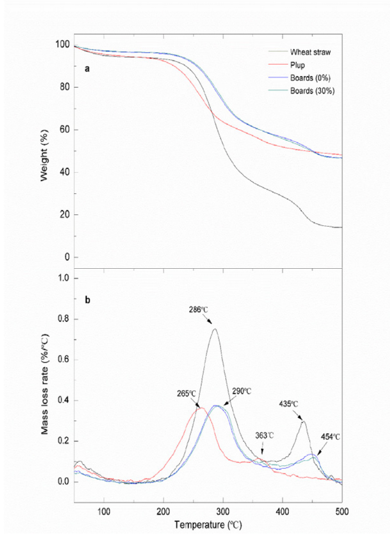

Fig. 3: Pyrolysis curves of wheat straw, pulp and binderless boards made with 0 wt% and 30 wt% black liquor.

Fig. 3 shows the episodes of the sample weight dropping and the rate of the sample weight loss versus temperature. Plenty loss occurs in three steps, described by three weight loss rate peaks. They are for water evaporation (at ~100°C), thermal degradation of water soluble components, hemicellulose and potion of cellulose (at 260-290°C), thermal degradation of remaining cellulose, part of lignin plus the oxidation of the breakdown products from the precious stage (at ~430°C). There are no significant thermal changes without water loses because the moisture contents of this four materials which have distinct water retention capacity are different for a period of time in certain environment. The initial decomposition temperature can defined as the temperature with 10wt% mass loss (T_{ds}), listed in Tab. 3.

Tab. 3: 10% weight loss temperatures of wheat straw, pulp and binderless boards made with 0 wt% and 30 wt% black liquor.

Samples	**T_{ds} (°C)**
Wheat straw	237.43
Pulp	217.69
Boards (0%)	255.73
Boards (30%)	257.90

The Tds of pulp is the lowest. Comparing to the two kinds of boards' samples, they are near but much higher than wheat straw and pulp. This is because the pulp have more extractives and low-molecular substance like sugar, hemicellulose and cellulose. They can easily thermal decompose in lower temperature. And, new covalent bonds and more hydrogen bonds may be generated under high temperature and moderate moisture so boards has higher T_{ds}, indicating better thermal stability at high temperature for the boards sample than the raw materials. The same results of DTG in Fig. 3, T_{p2} (the second peak) of this four samples in the order of the easiest to the most difficult to degrade are pulp > wheat straw > boards without black liquor > boards with 30 wt% black liquor. T_{p3} (the third peak) of wheat straw was highest and emerged at ~435°C which is higher than T_{p3} of pulp and lower than T_{p3} of boards. There is a possibility that the thermal-pressing process might contribute to the weight loss of biomass in the two temperature range (210-320°C and 350-480°C).

According to Fig. 4 to Fig. 8, hot-pressing of straw fibers in the presence of black liquor generated materials that appear to have interesting properties. The black liquor were diluted to different concentration and added to straw fibers. The weight percentage of black liquor were respectively 20 wt%, 30 wt% and 40 wt%, comparing with the straw binderless fiberboards without black liquor. Mechanical properties of fiberboard samples in terms of MOR, MOE, TS and WA were significantly influenced by the percentage of black liquor. MOR and MOE of the panels was improved when black liquor was added.

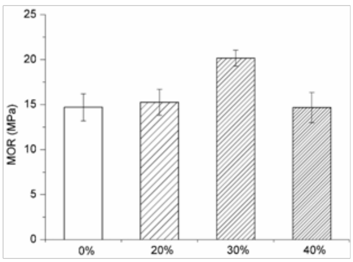

Fig. 4: Mean modulus of rupture (MOR) of fiberboards.

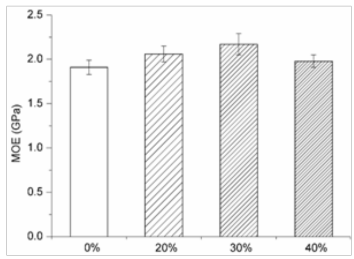

Fig. 5: Mean modulus of elasticity (MOE) of fiberboards.

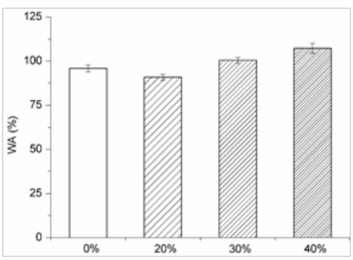

Fig. 6: Mean water absorption (WA) of fiberboards.

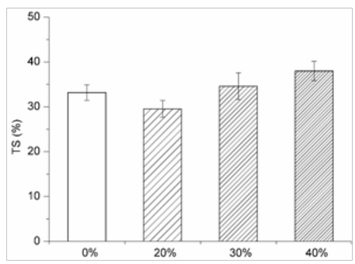

Fig. 7: Mean thickness swelling (TS) of fiberboards

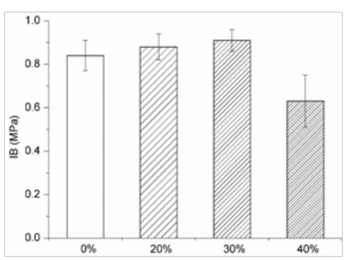

Fig. 8: Mean internal bond strength (IB) of fiberboards

The boards made with 30wt% content of liquor yielded the highest MOR values (20.16 MPa). TS and WA is physical properties relevant to the dimension stability of the boards. This property can tell us a thought of how the products will behave when applied under the humid environment and is a vital assessment method to think over boards where can be used. Fig. 7 shows that TS decreased with increasing the content of black liquor and became a minimum value (29.57%) at 20 wt% content. There was no immediate change of IB in different content of black liquor and the maximum value was 0.91MPa. On the whole, black liquor has active effect on the mechanical properties of them. However, if adding too much liquor, the properties would become worse. The mechanical properties of this kind of binderless fiberboards displayed a convinced improvement, especially the MOR and TS. The parameters of the boards were up to the standards of GB/T21723, except for MOR and MOE (2008) (MOR: 27 Mpa; MOE: 0.27 GPa; IB: 0.6 MPa; TS: 45%). Nonetheless, the samples were just up to the standards and they still need further process optimization to obtain straw-based binderless fiberboards with excellent performances or functions. It is proverbial that covalent bonds bring about intermolecular forces which are much more powerful than those of hydrogen bonds. In addition, fibers with lignin-rich and protein-rich surfaces improve the mechanical performance of boards through the mechanical twist of the melted lignin molecules (Mancera et al. 2012) and high polymer between protein and cellulose (Han et al. 2015) under pressure and temperature. However, the ash in black liquor had a bad effect on mechanical properties of the boards so that the boards with 40wt% black liquor behaved bad performance in all samples.

CONCLUSIONS

The finding of this work indication how the properties of wheat straw after fining. The chemical composition of pulp revealed moderate lignin and low ash content, which supposed to use as an alternative source with low cost to manufacture fiberboards. The binderless fiberboards were prepared by the hot-pressing of mixtures of pulp and black liquor at 160°C. The black liquor content was from 0 wt% to 40 wt%. The boards' mechanical properties, which have different percentages of black liquor, were determined. The FT-IR results suggested that the proteins and lignin in the black liquor can help create new covalent bands under thermal-pressing so that have better MOR, TS and IB. Nonetheless there is a little ash content in the liquor and it has negative influence. The formulation with 30 wt% black liquor nearly conformed to GB/T requirements for medium density fiberboards. Optimizing the hot pressing parameters is the fur further work. By utilizing pulping wastewater, black liquor, can not only enhance the qualities of boards, but also obtain cleaner production and cost saving. "Zero drainage" is the ultimate goal.

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