Effect of steam explosion treatment on characteristics of wheat straw

Guangping Han, James Deng, Shuyin Zhang, Paul Bicho, Qinglin Wu

Abstract

Steam explosion treatments were used to modify straw fiber attributes for panel manufacturing. In particular, the effect of steam temperature and retention time on morphology, acidity, wettability, and ash and silicon contents of wheat straw was studied. After steam explosion treatments, proportion of large particles decreased, while fiber bundles increased. Higher steam temperature and longer retention time resulted in more homogeneous fiber-like material. The results showed that the pH value of the untreated wheat straw fiber was nearly 7 and the pH values and acid buffer capacities of straw were greatly reduced after steam explosion treatments. This indicated that the acidity of straw increased after steam explosion treatments. The dynamic contact angle of the straw before the treatment was nearly 90°, indicating that the straw material without treatment is more hydrophobic. After steam explosion treatments, the contact angle of straw was significantly reduced, showing that the surface wettability of the treated straw was improved. The ash and silicon contents of straw were also significantly reduced by steam explosion treatments. The improved acidity and wettability as well as decreased silicon content would contribute to the improved bondability between straw particles and water-soluble adhesive binders.

Keywords:
Wettabillity
Ash content
Silicon content

1. Introduction

In general, crop materials such as straws, sugar cane, and bast fibers (flax and hemp) have lower cellulose and lignin contents, but higher pentosan content than wood (Rowell, 1996). The ash content of all crop materials is significantly higher than that of wood (Youngquist et al., 1993, 1996). More than 90% of the ash in wheat straw was silica (Sauter, 1996). The chemical composition of crop fibers, especially high ash content, has limited the use of these crop materials as raw fiber materials for panel manufacture (Sauter, 1996). In addition, crop materials commonly contain high levels of extractives, which may influence the curing behavior of adhesives (Loxton and Hague, 1996). The pH and acid buffering capacity of aqueous extracts from the non-wood lignocellulosic materials are significantly higher than those of softwood, which increases the gel time of urea-formaldehyde (UF) resin and causes bonding difficulty (Hague et al., 1998). The presence of extractives can also influence the wettability of materials (Young, 1976; Hes and Kuo, 1988). The low wettability is related to the existence of non-polar extractives (Nguyen and Johns, 1979). Generally, there is a waxy layer on the crop material surface (Sauter, 1996; Loxton and Hague, 1996). The water-soluble UF resin is chemically incompatible with the straw material and it is probably the main factor responsible for the reduction of bond quality.

Many attempts have been made to improve the bondability between agricultural materials and adhesives through raw material pretreatment. Wax can usually be extracted by the organic solvents like ethanol/benzene (EB) (Browning, 1967; Thomas, 1959). Han et al. (1999) reported that the wettability of wheat straw surface was improved through EB treatment and the bondability of particleboards made from EB-treated wheat straw was significantly enhanced due to the removal of wax-like substances and other non-polar extractives from the straw surface. High temperature steam treatment has been used to improve dimensional stability of wood products (Giebeler, 1983; Inoue and Norimoto, 1991; Rowell et al., 1999). Lawther et al. (1996) reported that some portion of pectic substances and hemicellulose can be removed from steam-treated wheat straw. Since the pectic substances and high content of hemicellulose in non-wood lignocellulosic materials usually result in poor adhesion between adhesive and these materials, the extraction of these substances would contribute to the improvement of board properties. So far, there are limited studies on the bondability improvement through steam explosion pretreatment.

The overall goal of this work was to investigate the feasibility of improving the bondability between steam explosion pre-treated wheat straw and UF resin. The specific objective was to evaluate the
effects of different treatment conditions (i.e., steam temperature and retention time) on the morphology, acidity, wettability, and chemical properties of wheat straw.

2. Materials and methods

2.1. Raw materials and preparation

Wheat straw was collected from Quebec, Canada. The air-dried wheat straw was hammer-milled and screened to attain furnish with an average length of 25 mm. Subsequently, all the straw material was presoaked in water at 20 °C for 12 h prior to steam explosion treatment.

2.2. Steam explosion treatment

The presoaked wheat straw was treated under various steam explosion conditions shown in Table 1. Steam temperature was 190 °C and 200 °C and the retention time was 2 min and 3 min. The treatment was conducted using a specially designed steam explosion vessel at the pilot plant of the Eastern Laboratory of the FPInnovations—Forintek Division. Each batch of about 850 g of wheat straw was put into the steam chamber. The steam was adjusted to the desired treatment temperature as shown in Table 1. Counting of retention time for each run was started when steam reached to the target temperature. Steam was suddenly released at the end of each treatment to give the explosion effect. The treated straw was collected from the cyclone and pressed to remove the excess water to reach about 60% moisture content (MC).

2.3. Size characterization

The sizes of wheat straw material after various steam explosion treatments were measured in accordance with TAPPI T 233 cm-82 using a Bauer-McNett Fiber Classifier at the FPInnovations—Paprican Division in Vancouver, BC. A sample of 5-g dry straw fiber was used for each treatment condition. The sample was put into a 2000-ml cylinder with boiled water and stirred until all the straw elements were completely separated. The mixture was then diluted to 4000 ml. The sieves with different openings of 10, 12, 14, 28, and 48 meshes were set in five tanks of the classifier from various steam explosion treatments. A modified test procedure developed from FPInnovations—Forintek Division was used. Straw particles were ground using a small Wiley grinding mill (model 4) and sieved to pass a 40-mesh screen. The sieved straw samples (15 g dried weight) were refluxed in a 200 ml of boiling distilled water for 20 min. The mixture was filtered under vacuum and then diluted to 500 ml. This filtrate solution was used for pH and buffer capacity measurements. A Corning Pinnacle 530 pH meter was calibrated with pH 7.00 and 4.00 standard buffer solutions for acid buffer capacity determination, and with pH 7.00 and 10.00 for base buffer capacity determination. Each filtrate (100 ml) was titrated twice using a 25-ml burette with a standard 0.025N H2SO4 solution until reaching pH 3.00 for acid buffer capacity measurement. For base buffer capacity, each filtrate was titrated twice with a standard 0.025N NaOH solution until reaching pH 11. The initial pH was recorded prior to each titration. The buffering capacity, expressed as total milliequivalents (mEq) of acid (or base) per 100 g of oven-dried (OD) sample needed to lower the pH to 3.00 (or 11.00), was calculated as: (1).

2.4. Evaluation of pH and buffer capacity

The pH, acid and base buffer capacities were evaluated for wheat straw from various steam explosion treatments. A modified test procedure developed from FPInnovations—Forintek Division was used. Straw particles were ground using a small Wiley grinding mill (model 4) and sieved to pass a 40-mesh screen. The sieved straw samples (15 g dried weight) were refluxed in a 200 ml of boiling distilled water for 20 min. The mixture was filtered under vacuum and then diluted to 500 ml. This filtrate solution was used for pH and buffer capacity measurements. A Corning Pinnacle 530 pH meter was calibrated with pH 7.00 and 4.00 standard buffer solutions for acid buffer capacity determination, and with pH 7.00 and 10.00 for base buffer capacity determination. Each filtrate (100 ml) was titrated twice using a 25-ml burette with a standard 0.025N H2SO4 solution until reaching pH 3.00 for acid buffer capacity measurement. For base buffer capacity, each filtrate was titrated twice with a standard 0.025N NaOH solution until reaching pH 11. The initial pH was recorded prior to each titration. The buffering capacity, expressed as total milliequivalents (mEq) of acid (or base) per 100 g of oven-dried (OD) sample needed to lower the pH to 3.00 (or 11.00), was calculated as: (1).

Buffer capacity (mEq/100 g OD sample)

\[
\text{Buffer capacity} = \frac{\text{volume of titrant} \times 0.025\text{N of } \text{H}_2\text{SO}_4 \text{ or } \text{NaOH}}{500 \text{ ml \times 100 g OD sample}}
\]

Two titration replicates were performed for two samples of each treatment. The initial pH value of each type of straw was the average of eight measurements while the buffer capacity value was the average of four titrations.

2.5. Measurement of wettability

Dynamic contact angles were measured to evaluate the wettability of the straw material treated under various steam explosion conditions. The wettability of water onto the straw samples was determined using the capillary rising height method. The Kruss Laboratory Desktop K12/K14 at Laval University in Quebec City, Canada was used to perform the wetting measurements. Oven-dried sample (0.05 ± 0.005 g) was loaded into the cylindrical glass tube, which was closed at one end with a porous glass sieve. The sample glass tube was then hung on a microbalance and the weight of the samples together with the tube was tared to zero. The test liquid under the glass tube, maintained at 20 °C, was brought into the sample by penetrating through the glass sieve and wet the sample. The test liquid reservoir rose up to contact with the sample at a rate of 4.5 mm/min. The balance detected the weight increase as a function of time until steady state was reached.

The wetting of hexane on the samples was performed first to obtain the capillary constant. Subsequently, the wetting of water on the samples was performed. Six replications were performed in the measurement of each condition. The contact angle was determined with the modified Washburn equation (2) from the rate of water absorbed onto the samples (slope of the plot of mass² versus time as shown in Fig. 4):

\[
\cos \theta = \frac{m^2}{I} \times \frac{\eta}{\rho^2 \sigma c}
\]
where $\theta =$ contact angle between straw fibers and liquid, degrees, $t =$ time, $m =$ mass of the sucked liquid, $h =$ capillary rising height, $\eta =$ viscosity of the liquid, $\rho =$ density of the liquid, $\sigma =$ surface tension of the liquid, $c =$ material constant.

2.6. Ash and silicon content

The ash and silicon contents of wheat straw material treated under various steam explosion conditions were analyzed in accordance with ASTM D 1102 Standard (ASTM, 2007). Four samples of straw material with each steam explosion treatment were used for the determination of ash and silicon contents. To determine the ash content, each sample entailed an 18-h combustion at 800°C followed by an acid digestion of the total ash.

3. Results and discussion

3.1. Morphology of wheat straw under various steam explosion treatments

Fig. 1 shows the images of wheat straw particles after various steam explosion treatments. It was observed that the sizes of straw particles tended to be smaller at higher steam temperature and longer retention time. More fibers were found in the sample treated with higher temperature and longer retention time. More than 90% of fiber bundles were observed in the sample treated at 200°C for 3 min. The size distribution of the treated wheat straw is shown in Fig. 2. The largest proportion of treated straw was always coarse material, accounting for 87.4%, 75.9%, 81.0%, and 54.9% for the samples treated with 190/2, 190/3, 200/2, and 200/3, respectively. Higher steam temperature and longer treatment time resulted in more homogeneous fiber-like material. The proportion of coarse particles decreased while the small particles and fiber bundles increased with increasing severity of steam explosion treatment. This indicates that the larger straw surface with waxy and silica layer was removed, and thus more straw fibers with highly reactive hydroxyl groups were exposed. This will help improve the bonding strength between wheat straw and water-soluble urea UF resin.

3.2. Effect of steam explosion treatments on acidity properties of wheat straw

UF resin is known to be acid-catalyzed and cannot attain an optimum state of cure in a low acid environment (Johns and Niazi, 1980). Knowledge of the acidity properties of treated and untreated wheat straw material is of practical significance to better understand the interfacial bonding mechanism when the acid-setting UF resin is used as adhesive in the manufacture of straw particleboard. The results of pH and buffer capacities of wheat straw particles under various steam explosion treatments are shown in Table 2.

![Fig. 1. Images of wheat straws before and after steam explosion treatment. (a) Before treatment, (b) treated at 190°C for 2 min, (c) treated at 190°C for 3 min, (d) treated at 200°C for 2 min, (e) treated at 200°C for 3 min.](image-url)
Table 2

<table>
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<th>pH and buffer capacities of wheat straw materials under various treatments.</th>
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<td>Treatment conditions</td>
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The values in parentheses are the standard deviations.

OD: oven dry.

The pH value of untreated wheat straw material (control sample) was 6.83. This finding is in agreement with previous studies (Pan et al., 2008; Zhang et al., 2003). It was reported that the pH and acid buffering capacity of aqueous extracts from crop materials were significantly higher than those of softwoods, and in the presence of the materials, resin gel time increased greatly (Hague et al., 1998). After steam explosion treatments, the pH values and acid buffer capacities of straw decreased remarkably. The pH value of the treated straw under 200 °C for 3 min was reduced to 4.98 from 6.83 of the control sample. Acid buffer capacities were lowered from 16.25 of the control sample to 6.88 for the water-soaked sample and to 4.16 for 190 °C/2 min treatment. The results indicate that the acidity of wheat straw material was increased after steam explosion treatment. It can be predicated that the better acidity properties of the treated straw particles will help improve the bondability between straw particles and the acid-setting UF resin, and thus the performances of straw particleboards. However, the acid and base buffer capacities of wheat straw samples under different steam explosion treatments were not appreciably different.

3.3. Effect of steam explosion treatments on wettability of wheat straw

The average values of dynamic contact angles of wheat straw after various treatments are shown in Fig. 3. The contact angles were calculated from the slope of the plot of mass$^2$ versus time of hexane and water wetted onto the samples. The wetting curves of selected samples are shown in Fig. 4. The contact angle of the straw before treatment was nearly 90°, indicating that water hardly wet the straw material. This means that the raw material without treatment is more hydrophobic. Crop materials commonly contain high levels of extractives (Loxton and Hague, 1996). The low wettability...
is due to the existence of non-polar extractives (Nguyen and Johns, 1979). The hydrophobic nature of wheat straw is also related to its morphology. A straw fiber contains a relatively large number of cell elements. It includes actual fibers, parenchyma cells, vessel elements, and epidermis cells which contains a high amount of ash and silica. In a cross section of straw, the epidermis cells are the outermost surface cells, covered by a very thin wax layer (Markessini et al., 1997). The level of natural wax in crop materials is commonly between 1% and 2%, which is sufficiently high so as to lower straw wettability and inhibit bonding among particles (Hague et al., 1998). It clearly shows that the contact angles after steam explosion treatments decreased, indicating the improved surface wettability. This is probably due to the damaged waxy layer and the exposed highly reactive hydroxyl groups of the straw material resulted from steam explosion treatment. The wettability of wheat straw, which is prerequisite for the good adhesion between the straw fibers and binder, depends on many factors, such as the porosity, hygroscopicity, and chemical composition of the straw. The improved wettability of treated straw samples could also be attributed to the fact that the surface of the material becomes more porous due to explosion effect caused by the sudden release of steam at the end of treatment. The better wettability of treated straw would contribute to the improvement of the bondability between straw particles and water-soluble UF resin, thus to improved performances of the products.

3.4. Effect of steam explosion treatments on chemical properties of wheat straw

Fig. 5 shows the ash and silicon contents of the steam explosion treated wheat straw. The ash content of the straw sample before treatment was 5.8%. One of the major differences between wood fiber and agricultural fiber is their chemical properties. Previous study showed that crop materials commonly contained high percentages of ash and extractives compared with wood (Youngquist et al., 1993; Loxton and Hague, 1996). The ash content of wheat straw before treatment was 5.8% from this study, which is much higher than wood (normally less than 1%). It was reported that more than 90% of the ash in the wheat straw was silica (Sauter, 1996). The silicon content of the control sample was 5% from this study, which is in a good agreement with the previous study (Pan et al., 2008). Sawatari et al. (1996) concluded that the silicon atoms in rice and wheat straws were extremely concentrated in the surface layer. The chemical composition of straws, especially the high ash content, has a negative impact on the use of straws as raw materials for panel manufacture (Markessini et al., 1997). After steam explosion treatments, the ash and silicon contents decreased significantly. This means that part of ash was removed via steam release. As ash is hydrophobic, the removal of ash from wheat straw will contribute to the improvement in its wettability and consequently better bondability between wheat straw and water-soluble UF resin.

4. Conclusions

Steam explosion treatment can be a feasible approach to improve the bonding strength between wheat straw material and adhesive binders. After steam explosion treatments, the proportions of large particles decreased while fiber bundles increased. Higher steam temperature and longer retention time resulted in more homogeneous fiber-like material. It was found that the pH values and acid buffer capacities of straw were greatly reduced, indicating the increased acidity of the treated straw. The straw material before treatment was more hydrophobic. The dynamic contact angles after steam explosion treatments decreased significantly, indicating that the surface wettability of the treated straw was improved. The ash and silicon contents of the treated straw were significantly reduced through steam explosion. The improved acidity and wettability as well as the decreased silicon content would contribute to the improvement in bondability between straw particles and water-soluble UF resins.

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References


