JOURNAL OF PURE AND APPLIED MICROBIOLOGY, Sept. 2015. Vol. **9**(3), p. 1913-1917

Effect of Weakly Alkaline Salt Pretreatment on Bio-Boards for Medicine Safety

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(Received: 29 April 2015; accepted: 16 June 2015)

To improve the self-bonding reactivity, *Eucalyptus* wood fibers were pretreated, processed and analyzed. The results showed that the bio-boards was 1.40 MPa and 1.16 MPa by pretreatment in 0.3% and 1.5% Na_2SiO_4 solution, respectively. FT-IR and ^{13}C-NMR spectra showed that the chemical structure and reactivity of *Eucalyptus* wood fibers had different changes after pretreated in sodium silicate solution. It suggested that cellulose, hemicellulose and lignin participated in united chemical reaction and let the pretreated fiber be self-bonded and resistance to water.

Key words: Groups; *Eucalyptus urophydis*; Na_2SiO_4 pretreat;
Wood fibers; ^{13}C-NMR; FT-IR; Self-bond.

Medicines were used to treat manage symptoms of chronic diseases, infectious diseases, and helped relieve suffering and pain. Medicines were generally safe if they were correctly used and safely stored[1]. However, medicines have both benefits and risks, and there were all risks in taking any medicine[2]. Especially, Patients couldn't be helped reduce the risk of harm if medicines were unintentionally polluted and took, resulting that over 700,000 visits to hospital emergency departments only in the United States each year[3,4]. Though the guidance document had either a short-term or long-term medical need and put into place effective management systems to support them in the setting, medicine poisoning continued to occur on a regular basis[5,6]. In remote rural areas and desolate mountains, medicines were polluted and took very much because storage apparatuses had the release amount of formaldehyde[7-9].

Since wood-based panels were invented, the people demanded the self-bonded bio-boards which would have been growing eagerly without formaldehyde emission[10-12]. The bio-boards were a kind of wood-based panels bonded together without resin under pressure and heat. Now wood-based panels were almost manufactured by urea formaldehyde adhesive and phenol formaldehyde adhesive[13,14]. This was mainly because wood fiber could not be self-bonded without enough active bonds[15]. And the self-bonding strength was improved by activating the chemical components of wood fibers before hot press[10]. What's more, it was the urgent task to study the bio-boards with the eager market demand. Therefore, *Eucalyptus* wood fibers were pretreated in the Na_2SiO_4 solution, processed by hot press, and analyzed by FT-IR and ^{13}C-NMR in order to improve the self-bonding reactivity.

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MATERIALS AND METHODS

The 4.5-years-old *Eucalyptus urophydis* was collected from Yangjiang Forestry Bureau of Guangdong Province owned forest farmforest bureau forest of Eucalyptus test. Eucalyptus urophydis wood was processed into wood chips, dried to oven dry, and mechanically disintegrated to 40-60 mu short fiber. Na_2SiO_4 which was prepared for the subsequent experiments, was analytical reagent.

Experiment Methods

Weakly Alkaline Salt Pretreatment. The above short fiber was treated in Na_2SiO_4 solution for 6h. The mass percentage concentration of Na_2SiO_4 solution was 0.3%, 0.7%, 0.9% and 1.5%, respectively. After treatment, the fiber was filtered, dried at room temperature, done to oven dry at 103ºC. The pretreated fiber was named G11, G12, G13, and G14, respectively.

Bio-board process. The pretreated fiber was pressed to self-bond under the conditions of 20 min, 160ºC and gauge pressure 15MPa. The obtained bio-boards, which thickness were all 4mm, were named G21, G22, G23, and G24, respectively. Then the internal bond strength of bio-boards was determinated based on China National Standard GB/T 11718-2009.

Goup Characterization. FT-IR spectra of the above samples were obtained using a Thermo Scientific Nicolet iN10 FT-IR microscope as previously[16-18]. CP-MAS ^{13}C-NMR spectra of the above samples were determined on solid state and obtained using a Bruker AVIII 400.M spectrometer (Germany) as previously[16-21].

RESULTS

The bond strength of bio-boards was measured and obtained. Wood fiber was pretreated in Na_2SiO_4 solution, and bio-boards were obtained. Their FT-IR spectra and ^{13}C-NMR spectra were depicted in Fig.1 and Fig.2, respectively.

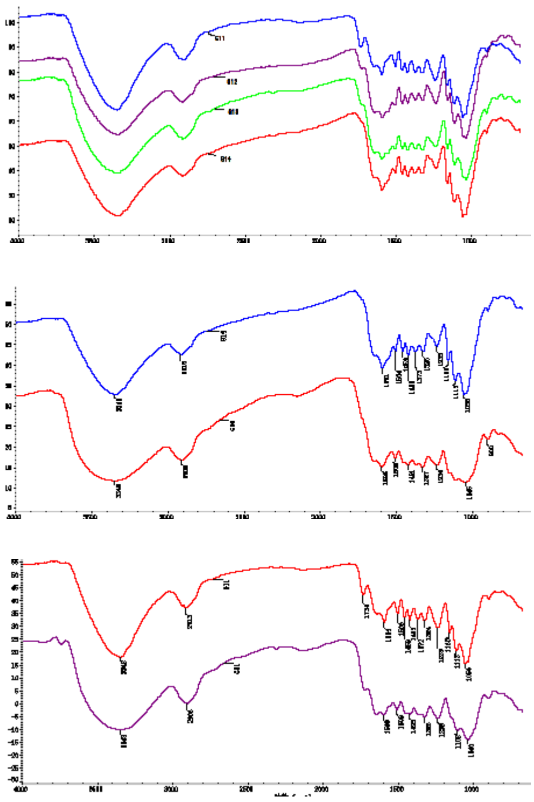

Fig. 1. FTIR spectra of some samples

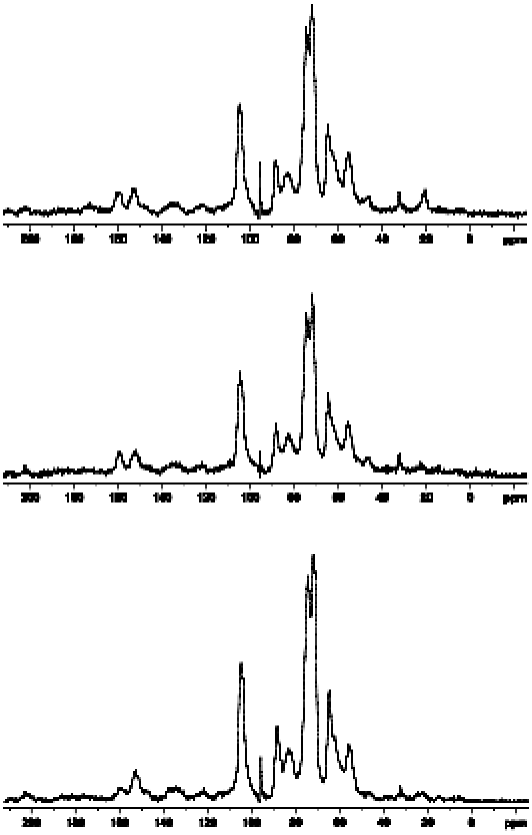

Fig. 2. ^{13}C-NMR spectra of some samples

DISCUSSION

Analysis on internal bond strength

The bond strength of bio-boards was measured. The short fiber, which was pretreated in 0.3% and 1.5% Na$_2$SiO$_4$ solution, and the internal bond strength of tis bio-boards was 1.40 MPa and 1.16 MPa, respectively. The strength numerically meeted the requirements of national standards GB/T11718-2009 which the internal bond strength should be more than 0.6 MPa with the boards' thickness of 3.5-6.0 mm. Na$_2$SiO$_4$ was a weak acid salt, and its solution presented alkaline. Na$_2$SiO$_4$ solution might dissolve part extractives from the short fiber, and expanse cell wall of the short fiber. What's more, after the short fiber was pretreated in Na$_2$SiO$_4$ solution, the binding activity of its Cellulose, hemicellulose and lignin were all activated. It resulted that the bond strength became higher.

FT-IR spectra

FT-IR spectra could be used to investigate the structural groups of the bio-boards. For comparison, the spectra of the bio-boards were plotted in supporting information Figure 1. The spectra of all samples showed the 3345cm^{-1} (O-H stretch), 2916cm^{-1} (-CH$_2$ stretch), 1735cm^{-1}(unconjugated C=O stretch), 1459cm^{-1} (C-H deformation vibration), 1372cm^{-1} (C-H bending vibration), 1235cm^{-1} (C-C, C-O plus C=O stretch), 1160cm^{-1} (C-O-C stretch), 1113cm^{-1} (C-O or C-C stretch), 1032cm^{-1} (C-O-C stretch), 898cm^{-1} (cellulose beta glycosidic bond stretching vibration). Particularly, the conjugated units in lignin were probably related to oxidation at ±-position of side-chain of lignin during hot press. The similar spectra patterns of aromatic skeletal vibrations in the lignin samples (1591cm^{-1}, 1504cm^{-1}, and 1423cm^{-1}) suggested that the basic aromatic structures of lignin were not changed during these processes. The syringyl lignin stretching vibration was at 1325cm^{-1}.

As can be seen from the Fig.1, after the fiber was pretreated in Na$_2$SiO$_4$ solution, -OH association degree increase for the group at 3345cm^{-1} strengthened. The absorption of the group at 1737cm^{-1} decreased in 0.3% and 0.7% Na$_2$SiO$_4$ solution, and it resulted that hemicellulose partly removed. The characteristic peaks at 1591cm^{-1}, 1504cm^{-1}, and 1423cm^{-1} slightly reduced, and it shown that effect pretreated in Na$_2$SiO$_4$ solution on lignin was not obvious. Hemicellulose and part of lignin was dissived for C-H bending vibration enhanced. The peaks at 1227cm^{-1} and 1030cm^{-1} attenuated. Some –OH was destructed for the peak at 899cm^{-1} slightly reduced.

After hot press, -OH stretching vibration reduced, resulting that Hydroxyl formed hydrogen bond association. The characteristic absorption peaks at 1592cm^{-1}, 1505cm^{-1}, and 1420cm^{-1} reduced, and it shown lignin participated in the self-bonding reaction. The characteristic absorption peaks at 1157cm^{-1} and 1371 cm^{-1} reduced, and it shown cellulose also participated in the self-bonding reaction. And the peaks at 1107cm^{-1} and 1032 cm^{-1} reduced.

The characteristic groups of –OH, -CH$_3$, -CH$_2$, C=O and Ar have all the obvious change after the hot press, suggesting that Cellulose, hemicellulose and lignin participated in united chemical reaction and let the pretreated fiber be self-bonded. The number of hydroxyl group decreasd, it showed that the interaction of hydrogen bond formed hydroxyl, and reduced the number of free hydroxyl hydrophilic so as to make the board with a good water resistance and the higher bonding strength.

Analysis on ^{13}C-NMR goups

^{13}C-NMR spectroscopy is a reliable method for investigating the structural features of samples and it also provided a more comprehensive view of the entire lignin macromolecule. The ^{13}C-NMR spectra of the three samples are shown in Fig. 2. As can be seen from the Fig.2, C=O of ester bond presented 171.4 ppm, its content was relatively small and almost disappeared with the increase of the concentration of sodium silicate. The groups of lignin appeared 110-160 ppm, C3/C5 of etherification S-lignin at 152-153ppm, C-1 of unetherification S-lignin and G-lignin at 132.8 ppm and 132.4 ppm, Ar of lignin at 121.4 ppm. Cellulose peak decreased such as C-1 of cellulose at 104.6 ppm, C-4 (crystalline region) of cellulose at 88.4 ppm, C-4 (amorphous regions) of cellulose at 83.1 ppm, C-3/C-2/C-5 of cellulose at 74.6 ppm, C-6 of cellulose at 64.5 ppm, showing that alkalescent salt and cellulose reacted and its crystalline structure destructed. C-γ of lignin side chain at 62.1 ppm, -OCH$_3$ of ligin at 55.3 ppm, and -

CH_2 of fatty hydrocarbon at 32.1 ppm decreased. And ester bond between hemicellulose and lignin was broked.

After hot press, the peaks weakened such as the groups of lignin at 110-160 ppm, C3/C5 of etherification S-lignin at 153.0 ppm and 152.3 ppm, Ar of lignin at 122.0 ppm. Cellulose peak decreased such as C-1 of cellulose at 104 ppm, C-4 (crystalline region) of cellulose at 88.6 ppm, C-4 (amorphous regions) of cellulose at 82.9 ppm, C-3/C-2/C-5 of cellulose at 74.7 ppm, C-6 of cellulose at 64.6 ppm, C-γ of lignin side chain at 71.9 ppm, -OCH_3 of ligin at 55.6 ppm, and -CH_2 of fatty hydrocarbon at 32.1 ppm.

CONCLUSION

FT-IR and ^{13}C-NMR spectra showed that the chemical structure and reactivity of *Eucalyptus* wood fibers had different changes after pretreated in sodium silicate solution. While *Eucalyptus* wood fibers were pretreated in Na_2SiO_4 solution, hemicellulose all removed with the higher concentration, and hemicellulose partly removed with the lower concentration,

The characteristic groups of –OH, -CH_3, -CH_2, C=O and Ar have all the obvious change after the hot press, suggesting that Cellulose, hemicellulose and lignin participated in united chemical reaction and let the pretreated fiber be self-bonded. The number of hydroxyl group decreasd, it showed that the interaction of hydrogen bond formed hydroxyl, and reduced the number of free hydroxyl hydrophilic so as to make the board with a good water resistance and the higher bonding strength.

ACKNOWLEDGMENTS

This work was financially supported by the National Natural Science Foundation of China (31170532), and the National 948 Plan (2014-4-38), and Invitation Fellowship Programs for Research in Japan of Japan Society for the Promotion of Science (ID No. S14748).

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