

# Microstructure of Burned Ultra-Low-Density Fiberboards using Plant Fiber as the Matrix and Si-Al compounds as the Filler

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Ultra-low-density fiberboards (ULDFs) were prepared by a liquid frothing technique using plant fibers as the matrix and Si-Al compounds as the filler to be used as a versatile bio-based composite. Si-Al compounds played an important role in the fire properties of ULDFs. Fire intensity and the amount of volatiles were significantly restrained because of the Si-Al compounds. To determine the combustion mechanism of ULDFs treated by Si-Al compounds, the microstructure of burned specimens was tested by chemical analysis, X-ray diffractometer (XRD), and infrared spectrometer (IR). According to the results from gas chromatography, glucose, xylose, and mannose disappeared in the bottom ashes. After combustion, the XRD profiles of the two ashes became weaker and broader; the sharpest peaks at  $18.6^\circ$  ( $2\theta$ ) that represented Si-Al compounds remained; the obvious peaks at  $22^\circ$  ( $2\theta$ ) from cellulose were gone. The results from IR suggested the characteristic functional groups OH, CH, and C=O from carbohydrate also disappeared, and absorbance at 1200 to  $400\text{ cm}^{-1}$ , which attributed to the vibration of Si-O, Al-O, and Si-O-Si bonds, increased. In conclusion, fibers are almost completely pyrolyzed at  $780^\circ\text{C}$ . The crystalline structure of Si-Al compounds is rearranged and more amorphous silicon oxide and aluminum oxide are generated.

*Keywords:* Ultra-low density fiberboards (ULDFs); Si-Al compounds; Combustion; Microstructure

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## INTRODUCTION

To improve the energy efficiency of houses built in cold regions, extensive insulation material is applied during construction of the house. Glass fiber (Isover Co.) and rock wool (RockWool Co.) are the most popular mineral-based insulation materials in the current market of building materials. Because it can offer similar properties, bio-based insulation material as an alternative product has a potentially huge development opportunity because of the abundance of raw materials, good physical and mechanical properties, and favorable effects on the environment.

Many efforts have focused on the manufacturing method and properties of bio-based insulation materials during the past ten years. For example, this working group has applied a liquid frothing approach to develop this type of material. Through this work, fiberboards with an ultra-low density of 10 to  $90\text{ kg/m}^3$ , thickness swelling of 0.57% after 24 h of water immersion, low thermal conductivity of  $0.035\text{ W/mK}$ , high sound reduction

coefficient of 0.67, and good fire resistance and mechanical properties have been obtained (Xie *et al.* 2004, 2008a, b, 2011; Niu *et al.* 2014; Chen *et al.* 2015).

Kawai's working group (Kawai *et al.* 1998; Xu *et al.* 2004) studied the manufacture and properties of particleboard using kenaf core (*Hibiscus cannabinus* L.) as a raw material and of fiberboard using wood fiber as a raw material with density ranges from 50 to 500 kg/m<sup>3</sup> formed by steam injection. Frenette *et al.* (1996) studied cellulosic fiber insulation material using short (1 to 4 mm) cellulosic fibers as filler and bonding synthetic fibers as the matrix. Cellulosic fiber is usually composed of 5 to 8% of synthetic fibers in weight. HempFlax Co. (hempflax.com) and Dieffenbacher Co. (www.dieffenbacher.de) produce natural insulation fiberboard using a traditional fiberboard production line. Also, Cervin *et al.* (2013) studied light-weight nanofibrillated cellulose foam with a porosity of 98% and a density of 30 kg/m<sup>3</sup>.

However, the two most important parameters for the evaluation of bio-based building insulation materials are thermal conductivity and fire resistance. Therefore, the addition of fire retardants is essential to change the two parameters. Compounds such as halogens, silicate, boron, and FRW (composed of guanylurea phosphate, boric acid, and a small amount of additives), which are typically used to improve the fire resistance of wood, were employed as fire retardants for ultra-low-density fiberboards (ULDFs) in Liu's study (2013).

Si-Al compounds with a pH value of approximately 3.8, in the form of an aqueous mixture of sodium silicate and aluminum sulfate, were used in subsequent studies because of the improved high-temperature resistance and cooperation with other fire retardants (Niu *et al.* 2014). The inorganic substances were relatively evenly dispersed on the surface of fibers and filled in the spaces between the fibers. This significantly decreased fire intensity and the amounts of volatiles released in the process of combustion. In addition, silicon and aluminum compounds have also been applied to modify solid wood and obtain organic-inorganic composites with desirable fire properties (Wang *et al.* 2000; Zhou *et al.* 2000).

The microstructure of burned wood has been reported (Zicherman and Williamson 1981); however, researchers have not yet reported the structural changes of burned ULDFs with Si-Al compounds. The aim of this study is to clarify the main mechanism behind fire resistance of ULDF with Si-Al compounds by parameters and experiments.

## EXPERIMENTAL

### Materials

Recycled kraft pulp (spruce-pine-fir, Tembec Inc., Canada) and waste newspaper (collected in China) were used as raw materials in the preparation of ULDFs. An aqueous solution of sodium silicate and aluminum sulfate was applied as a reinforcing agent to improve the fire properties of the materials; the applied dosages were 500 and 900 mL. Fibers, Si-Al compounds, glue, a water-proof agent, and a surfactant were blended together to get a foaming solution with an increasing volume. The detailed manufacturing process was described in a previous study (Niu *et al.* 2014).

The ULDFs were burned in a cone calorimeter (FTT Co., England) at 780 °C, in accordance with ISO 5660-1/2002, to obtain fly ash (upper ash caused by complete combustion) and bottom ash caused by incomplete combustion. The two types of ash, along with unburned ULDFs, were used as the specimens for further testing.

## Methods

### *Carbohydrate analysis*

Carbohydrates in unburned ULDFs and bottom ash were analyzed according to the methods of Theander and Westerlund (1986) and Karlsson *et al.* (2012). The specimens (60 to 61 mg) were dissolved and dispersed in a screw-capped glass bottle (20 mL) using sulfuric acid (12 M, 0.5 mL) with magnetic stirring (0.5 h) and vacuum-pumping (1.5 h). The samples were diluted using distilled water (8.4 mL) and kept capped in hot steam from boiling water in a pressure-cooker for 2 h.

After cooling to ambient temperature, an internal standard solution of 2-deoxygalactose (5 g/L, 1 mL) was added to clearly separate the mixture into two portions by shaking. The supernatants (1 mL) were transferred into a small glass tube (5 mL), and the pH value was adjusted to 7 using ammonia hydroxide (12 M, 100-200  $\mu$ L), then reduced in a temperature-controlled water bath at 40 °C for 1 h with aqueous ammonium hydroxide (3 M, 100  $\mu$ L) solution containing potassium borohydride (150 mg).

The pH value of the solution was adjusted to 7 again by addition of glacial acetic acid (100 to 200  $\mu$ L). The supernatant (500  $\mu$ L) was reacted with 1-methylimidazole (0.5 mL) and acetic anhydride (5 mL) for 10 min in a screw-capped tube (30 mL) with gentle shaking.

Absolute ethanol (1.0 mL), distilled water (5 mL), and potassium hydroxide (7.5 M, 5 mL) were very slowly and orderly added to the tube at intervals of a few minutes. The tubes were vibromixed and left for 10 min. The upper layer was transferred into a vial for gas chromatography (GC) testing.

Gas chromatography (GC) analysis was conducted on a fused silica column SLB-5 ms (15 m  $\times$  0.25 mm  $\times$  0.10  $\mu$ m). The column temperature increased from 50 °C (hold time 1 min) to 240 °C at 10 °C/min. The temperature of the flame-ionization detector was 250 °C. The flow rate of helium, the carrier gas, was 2.54 mL/min. A split injection mode was used with a split ratio of 1:30.

### *Crystallization*

An X-ray diffractometer (XRD, PANalytical Empyrean with a PIXcel3D detector) was employed to identify crystalline substances present in unburned and burned specimens. A small amount of specimen was cut with a sharp knife and put on the sample table. The sample was moved into the diffractometer after it was pressed and labelled. The specimen was then tested in a  $2\theta$  range of 5 to 60° at a voltage of 45 kV and current of 40 mA. Cu LFF HR was used as the X-ray tube.

### *Functional groups*

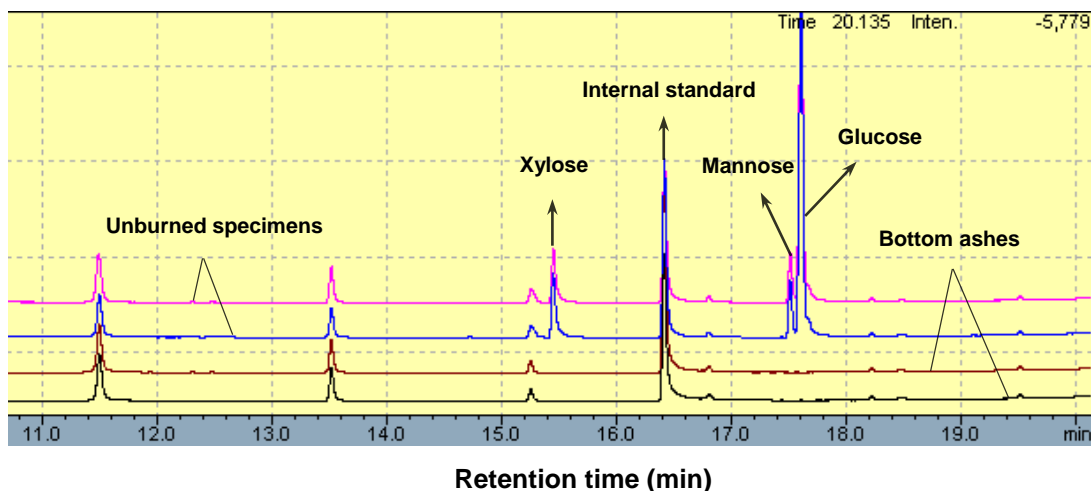
Infrared spectroscopy (IR, IFS 66V/S, Bruker) was used to determine the change in functional groups after combustion at high temperature. A small amount of powdered bottom ash and unburned specimen were put on the sample table in the spectrometer. The images were scanned and obtained at room temperature, 22 °C, over a spectral range of 400 to 4000  $\text{cm}^{-1}$ .

The data from XRD and IR were exported and changed to PRN files and DAT files, respectively. Then, they were analyzed using the program OriginPro8.

## RESULTS AND DISCUSSION

### Carbohydrate Analysis

Glucose, xylose, arabinose, galactose, and mannose were used as the standards to evaluate the location and contents of carbohydrates in unburned specimens and bottom ashes. Figure 1 presents GC images of unburned specimens and bottom ashes with 500 and 900 mL Si-Al compounds.



**Fig. 1.** Gas chromatogram (GC) images of both unburned specimens and burned specimens at 780 °C by cone calorimeter

For unburned specimens with 500 and 900 mL of Si-Al compounds, GC images showed almost the same intensity and retention time of peaks. Glucose, xylose, and mannose from holocellulose and likely corn starch (an ingredient of the glue used) were found at retention times ranging from 15 to 17 min. However, after combustion, monosaccharides disappeared in bottom ashes with 500 and 900 mL of Si-Al compounds. This suggested glucose, xylose, and mannose, which represent the elementary units of cellulose and hemicellulose, were indeed subjected to pyrolysis at high temperature, even with the protection of the Si-Al compounds.

Galactose and arabinose, which are another two types of sugar monomers from hemicellulose, are more sensitive and easier to be hydrolyzed under acidic conditions during manufacturing (Jin *et al* 2010). So they were not detected in the GC profiles of all the specimens. When retention time was about 15.25, one small peak appeared, and it could be arabinose. But the arabinose here probably resulted from the old GC analyzer, and it was not from fibers in the specimens. The contents of monosaccharides before and after combustion are listed in Table 1.

Carbohydrate content of unburned specimens ranged from 26.75% to 33.66%. Glucose and xylose were the two main monosaccharides in the fibers. The percentage of fiber in the specimens increased with decreasing dosage of Si-Al compounds when all other factors were held constant during manufacturing. Because of the higher percentage of fibers, the contents of glucose, xylose, and mannose in specimens with label “a” were higher than those with label “b”. The two types of raw materials showed a small amount of differences in carbohydrate contents. However, the effects of combustion on carbohydrate contents in two types of fibers were almost consistent. When the temperature was more than 200 °C, wood and cellulose isolated by several different fire retardants were

subjected to thermal degradation (Levan and Winandy 1990). Pyrolysis is an exothermic reaction and tends to be self-sustaining once started. Especially for the materials made from fibers with ultra-low density, the pyrolysis rate is faster than that of solid wood. Even if fire retardants such as chlorinated paraffin and the Si-Al compounds used in this study significantly restrained fire intensity and volatiles release (Niu *et al.* 2014), the pyrolysis of carbohydrate was still obvious, as can be seen in Fig. 1 and Table 1. Combustion primarily generates carbon, water, carbon oxides, and hydrocarbons.

**Table 1.** Carbohydrate Content of Both Unburned Specimens and Burned Specimens at 780 °C by Cone Calorimeter

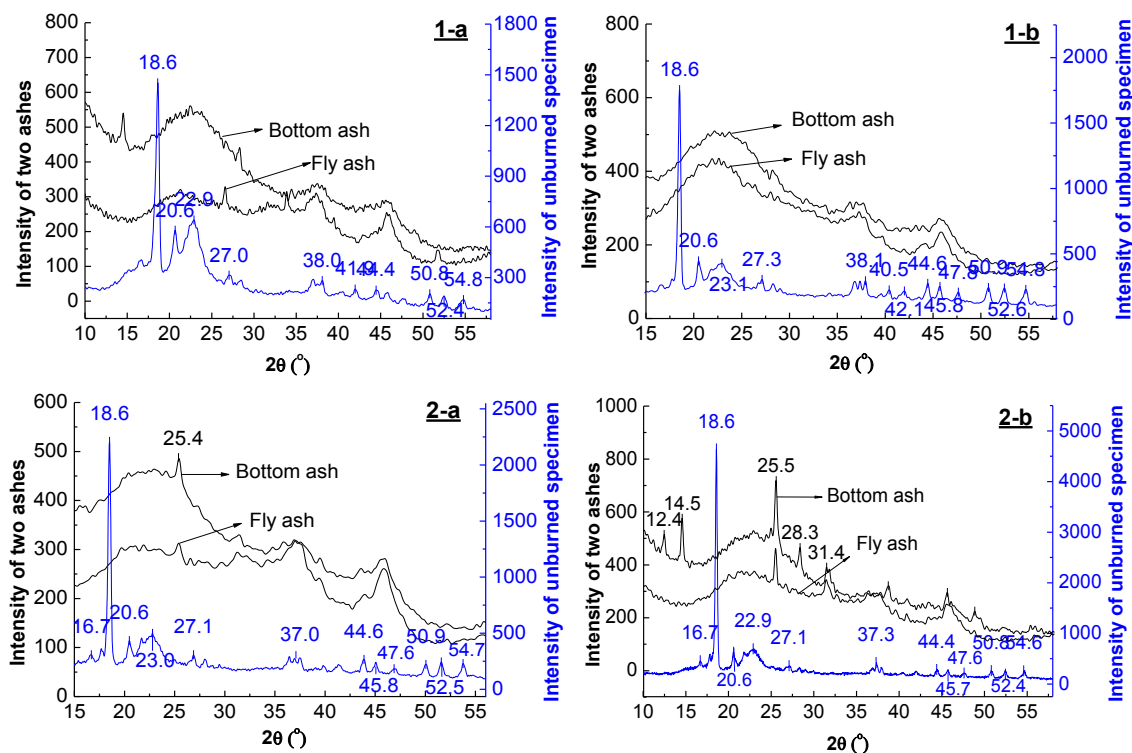
Specimens		Carbohydrate Content in the Specimens (%)			
		Glucose	Xylose	Mannose	Total
1-a	Unburned Specimen	24.12 ( $\pm 0.2577$ )	8.58 ( $\pm 0.0736$ )	0.96 ( $\pm 0.0011$ )	33.66
	Bottom Ash	—	—	—	—
1-b	Unburned Specimen	19.30 ( $\pm 0.8327$ )	6.69 ( $\pm 0.4628$ )	0.76 ( $\pm 0.0542$ )	26.75
	Bottom Ash	—	—	—	—
2-a	Unburned Specimen	26.41 ( $\pm 0.4425$ )	4.23 ( $\pm 0.2314$ )	2.88 ( $\pm 0.1154$ )	33.52
	Bottom Ash	—	—	—	—
2-b	Unburned Specimen	24.42 ( $\pm 0.5091$ )	4.11 ( $\pm 0.0453$ )	2.77 ( $\pm 0.0046$ )	31.30
	Bottom Ash	—	—	—	—

1- Kraft pulp; 2-Newspaper; a-500 mL Si-Al compounds; b-900 mL Si-Al compounds. Data in parentheses are standard deviation. “—” means no peaks found by GC.

### Crystallization

X-ray diffractometry (XRD) was applied to characterize the crystal structure, diffraction pattern, and particle size of ultra-low-density fiberboards during combustion. XRD profiles of four specimens from two types of raw materials with 500 and 900 mL of Si-Al compounds are given in Fig. 2.

Because unburned specimens contained both fibers and Si-Al compounds, their diffraction peaks in Fig. 2 were complicated and interesting. The peaks were from corporate contributions of fibers with crystalline cellulose at around  $2\theta = 22^\circ$  (Lionetto *et al.* 2012) and Si-Al compounds with a crystalline phase at  $2\theta = 18.6^\circ$  and a range from  $25^\circ$  to  $60^\circ$ , similar to crystalline zeolite (Kokotailo and Fyfe 1995; Kosanović *et al.* 2008). Furthermore, the intensity of diffraction peaks at  $18.6^\circ$  ( $2\theta$ ) and  $25$  to  $60^\circ$  ( $2\theta$ ) increased, and the intensity at  $22^\circ$  ( $2\theta$ ) decreased with a higher percentage of Si-Al compounds in the specimens, according to the comparisons between Fig. 2 (1-a) and (1-b), (2-a), and (2-b). After combustion at 780 °C by cone calorimeter, carbohydrate components were pyrolyzed and the crystalline structure of Si-Al compounds was destroyed or reorganized (Kokotailo and Fyfe 1995). Therefore, the diffraction peak from crystalline cellulose and the maximum peak at  $18.6^\circ$  ( $2\theta$ ) noticeably disappeared. Meanwhile, other sharp peaks became smaller and broader. The most significant broad diffraction pattern emerged at  $2\theta$  from  $17^\circ$  to  $30^\circ$  in the XRD profiles of fly ash and bottom ash, reflecting amorphous substances.



**Fig. 2.** Diffraction intensity of unburned and burned ultra-low-density fiberboards. 1- Kraft pulp; 2- Newspaper; a-500 mL Si-Al compounds; b-900 mL Si-Al compounds

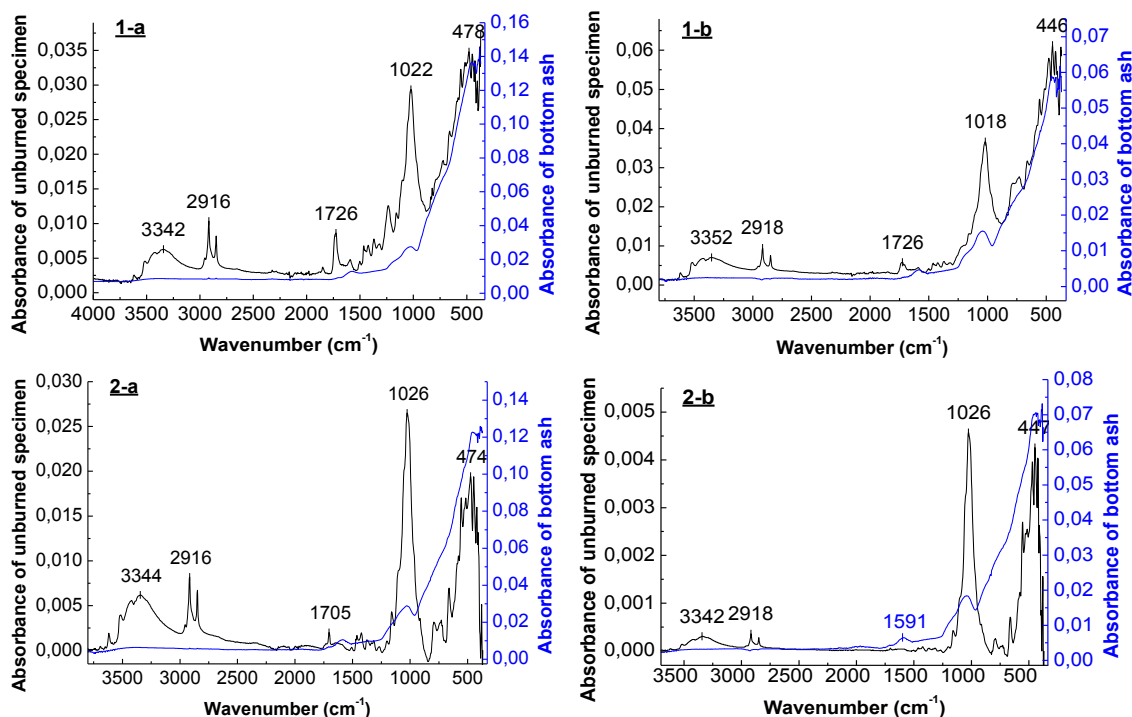
The change from a narrow and sharp crystalline phase to a broad and blunt amorphous phase was also attributed to smaller crystalline size and lower amount of crystalline substances in the specimens (Gajović *et al.* 2008) caused by pyrolysis. Amorphous carbon, amorphous silicon oxide, and amorphous aluminum oxide might remain in the ash. These substances are very important for decreasing the amount of heat released during pyrolysis of carbohydrates. At the same time, some new crystalline substances were also detected from the XRD profiles of ash, especially in Fig. 2 (2-b), *e.g.*, diffraction angles located at 12.4°, 14.5°, 25.5°, 28.3°, and 31.4°. These may have been the results of the interaction among amorphous substances or caused by re-arrangement of the crystalline structure at high temperatures. Furthermore, because the crystalline structure was subjected to less damage because of a lack of oxygen, the diffraction intensity of bottom ash was higher than that of fly ash.

## Functional Groups

Functional group changes caused by combustion were analyzed through the IR spectra shown in Fig. 3. Several main peaks, at roughly 3342, 2916, 1726, 1022, and 478 (447)  $\text{cm}^{-1}$ , can be seen for the unburned specimens. An absorbance peak between 3660 and 3060  $\text{cm}^{-1}$ , with a maximum near 3342  $\text{cm}^{-1}$ , is characteristic of a hydroxyl group typical of carbohydrates from fibers (Nishimiya *et al.* 1998). A band at 2916  $\text{cm}^{-1}$  can be attributed to the CH stretching mode of methyl and methylene groups in carbohydrates. Ketone and aldehyde groups from carbohydrates were also detected at 1726  $\text{cm}^{-1}$  or at 1705  $\text{cm}^{-1}$  (Niu *et al.* 2011). A band from 1200 to 950  $\text{cm}^{-1}$ , with a maximum near 1026  $\text{cm}^{-1}$ , resulted from the stretching vibration of Si-O and Si-O-Si (Nayak and Singh 2007; Prado

*et al.* 2010). A broad absorption from 1000 to 400  $\text{cm}^{-1}$  was due to the stretching vibration of Si-O-Al, Al-O, and Si-O (Nayak and Singh 2007).

Compared to the specimens with label a, the specimens with label b had stronger absorbance at 1000 to 400  $\text{cm}^{-1}$  because of a higher dose of Si-Al compounds. There were also differences in absorption intensity and absorption pattern for the two different raw materials. The specimens with kraft pulp as the matrix had stronger absorbance than those with newspaper in the range from 4000 to 400  $\text{cm}^{-1}$ , and their absorption pattern became broader at wavenumbers from 1200 to 400  $\text{cm}^{-1}$ . Nonetheless, kraft pulp and newspaper had similar effects on infrared absorbance.



**Fig. 3.** IR spectra of unburned and burned specimens. 1- Kraft pulp; 2-Newspaper; a-500 mL Si-Al compounds; b-900 mL Si-Al compounds

After the specimens were burned at 780  $^{\circ}\text{C}$ , their absorption peaks at 3342, 2916, and 1726  $\text{cm}^{-1}$  disappeared. Three broad peaks were detected at 1591, 1022, and 478 (474)  $\text{cm}^{-1}$ . One absorption band at 1591  $\text{cm}^{-1}$  was caused by the vibration of aromatic C-C bonds from bio-char (Părpăriță *et al.* 2014). Meanwhile, two other bands ranging from 1200 to 400  $\text{cm}^{-1}$  were induced by the vibration of the Si-Al compounds. They became broader and higher than those of unburned specimens.

These results suggest that carbohydrate components were almost completely pyrolyzed at high temperatures. Also, the percentage of Si and Al compound in burned specimens increased, corresponding with the results for carbohydrate content and crystallization. Si-O and Al-O compounds formed after the crystalline structure was rearranged, and made a noticeable contribution to the suppression of heat and volatiles released during combustion of ULDFs.

## CONCLUSIONS

1. After combustion by cone calorimeter at 780 °C, glucose, xylose, and mannose detected from the GC profiles of ultra-low-density fiberboards (ULDFs) disappeared in bottom ashes. The diffraction peak representing Si-Al compounds at 18.6° (2 $\theta$ ) and crystalline cellulose at 22° (2 $\theta$ ) disappeared, and XRD profiles of fly ash and bottom ash became smaller and broader. Characteristic functional groups in fibers, such as hydroxyl, methyl, methylene, ketone, and aldehyde, disappeared, and absorbance of Si-O, Al-O, Si-O-Si, and Si-O-Al groups from Si-Al compounds located at 1200 to 400 cm<sup>-1</sup> became stronger and broader.
2. Therefore, carbohydrates in ULDFs were almost completely pyrolyzed, even under the conditions of incomplete combustion. The crystalline structure of Si-Al compounds was rearranged and probably generated more amorphous substances (such as silicon oxide and aluminum oxide) during combustion, thus resulting in higher absorption peaks in IR profiles.
3. The release amounts of heat and volatiles decreased, attributing to generation of more amorphous Si-O and Al-O compounds.

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