Fire Performance of Si-Al Ultra-Low Density Fiberboards Evaluated by Cone Calorimetry

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To clarify how the fire resistance of ultra-low density fiberboards (ULDFs) was improved by the Si-Al compounds and to compare the effect of fire resistance between Si-Al compounds and fire retardant (chlorinated paraffin), the fire performance of ULDFs was evaluated by cone calorimetry. Comparing Si-Al compounds to chlorinated paraffin, the heat release rate (HRR), total heat release (THR), mass loss, total smoke release, and off-gases (CO and CO_2) release of ULDFs treated with Si-Al compounds significantly decreased. However, when Si-Al compounds and chlorinated paraffins were simultaneously added, the mixed fiberboards showed the best results for peak of HRR (100.76 kW m^{-2}), time to flameout (336s), THR (21.36 MJ m^{-2}), and residual mass (34.26%). These results indicated that the Si-Al compounds had a significant effect on improving the fire resistance of ULDFs, and the Si-Al compounds and chlorinated paraffins have a synergistic effect in ULDFs.

Keywords: Cone calorimetry; Fire performance; Si-Al compounds; Ultra-low density fiberboards

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INTRODUCTION

Ultra-low density fiberboard (ULDF) is made from plant fibers. In some applications, it can serve as a substitute for petroleum-based polymers and is better for human health (Xie *et al.* 2004, 2008a,b). ULDFs have many excellent properties such as ultra-low densities, low thermal conductivities, good sound absorption, *etc*. However, they also have properties which require improvement, including mechanical properties and fire resistance (Xie *et al.* 2011; Xie and Liu 2012; Lin *et al.* 2013; Niu *et al.* 2014). As a wood-based material, ULDFs are highly flammable in the absence of any retardant treatment, but such treatment could restrict their application; therefore, many efforts have to be taken to make the material more fire resistant prior to utilization.

Recently, improving the fire resistance of wood-based material by using chemical substances, including phosphorus, nitrogen, boron, silica, and their combinations, has garnered much attention (Genovese and Shanks 2008; Hagen *et al.* 2009; Sacristán *et al.* 2010; Liu 2013). To improve the chemical properties of wood, silica species, derived from sol-gel, were used in a study by Unger and colleagues (2012). The results confirmed that the dimensional stability and other properties were improved, and chemical bonding was verified between silica species and wood components. Some research looked at inorganic-wood composites using various inorganic reagents, such as SiO_2 (Saka and Ueno 1997; Fu *et al.* 2011), TiO_2 (Mahr *et al.* 2012), and Si-Al compounds (Xie and Liu 2012; Niu *et al.* 2014; Chen *et al.* 2015a). The modified specimens showed improved properties such as dimensional stability, fire resistance, and decay resistance (Saka and Ueno 1997; Fu *et al.* 2011, Mahr *et al.* 2012, Xie and Liu 2012; Niu *et al.* 2014; Chen *et al.* 2015a). These improvements were due to the stable incorporation of the inorganic components in the wood host (Unger *et al.* 2012). On the other hand, silica and aluminum compounds have a positive effect on fire resistance. This is because silicon compounds generate polysilicic acid, which can form an inorganic film on the surface of wood to insulate it against air during combustion (Unger *et al.* 2012). Aluminum compounds can also improve heat resistance of wood-based materials. When aluminum compounds are heated, they absorb a lot of heat from the dehydration reaction (Lu *et al.* 2008). Therefore, the main fire resistant agents used in this study were Si-Al compounds.

The properties of composites affected by Si-Al compounds have been widely studied. However, few studies have focused on the effects of Si-Al compounds on the fire resistance of ULDFs. Compared with previous research from Niu *et al.* (2014) on emphasizing the distribution and fire properties of ULDFs from different raw materials, the goal of this paper was mainly to clarify how the Si-Al compounds improve the fire resistance of ULDFs and to compare the effect on fire resistance between Si-Al compounds and a traditional fire retardant (chlorinated paraffin). The fire performance of the material was measured using cone calorimetry. The results of combustion behavior, mass loss, smoke release, and off-gases (CO and CO_2) release of ULDFs are all discussed.

EXPERIMENTAL

Materials

Kraft pulp (KP; Spruce-pine-fir; Tembec Inc., Canada) was utilized as a raw material to manufacture ULDF. Aluminum sulfate and sodium silicate, purchased from the Tianjin Fuchen Chemical Reagents Factory (China), were used to generate the Si-Al compounds. Chlorinated paraffins were purchased from the Changzhou Fengshuo Chemical Company, LTD (China).

Methods

Manufacture of ultra-low density fiberboards (ULDFs)

Ultra-low density fiberboards with the dimensions 200 mm × 200 mm × 50 mm ($L \times W \times H$) were manufactured separately in a demonstration line as described by Xie *et al.* (2011) and Chen *et al.* (2015b), with a target bulk density of 55 kg m^{-3}. In addition, control fiberboards (CF) were manufactured without Si-Al as described in Chen *et al.* (2015b) and fire retardant (chlorinated paraffins). The Si-Al fiberboards (SF) were manufactured only with Si-Al compounds. The fire retardant fiberboards (FF) were manufactured only with fire retardant (chlorinated paraffins). The mixed fiberboards were manufactured with and fire retardant. Additionally, the detailed parameters for fiberboards are presented in Table 1.

Table 1. Parameters for Fiberboards

Specimens	Si-Al compounds (mL)	Chlorinated paraffins (g)	Polyacrylamide resin (mL)	Alkyl ketene dimmer (mL)	Sodium dodecylbenzene sulfonate (10 wt%, mL)
CF	0	0	20	50	80
SF	500	0			
FF	0	35			
MF	500	35			

Fourier transform infrared spectroscopy

The bonding characteristics of the ULDF samples were analyzed using Fourier transform infrared (FTIR) spectroscopy. The FTIR analysis of ULDF was performed using a Nicolet 380 FTIR spectrometer (Thermo Electron Instruments, USA), employing the KBr pellet method, taking 32 scans for each sample with a resolution of 4 cm^{-1}, ranging from 4000 to 400 cm^{-1}.

Testing of fire properties

Fire properties of the ULDFs were evaluated using a cone calorimeter (FTT Co., England), which is considered to be the most significant bench scale instrument for the testing of fire properties of a new product, adopted by the International Organization for Standardization (ISO 2002) to evaluate the parameters of heat release rate (HRR), total heat released (THR), time to ignition (TTI), time to flameout (TTF), mass loss rate (MLR), effective heat combustion (EHC), total smoke rate (TSR), and off-gases (CO and CO_2) release. The size of the specimens was 100 × 100 × 30 mm ($L \times W \times H$). They were encased in aluminum foil, except for the heating surface, mounted in a stainless steel holder with a grid placed on top of the sample. The tests were performed at a heat flux level of 50 kW m^{-2}.

RESULTS AND DISCUSSION

Chemical Bonding of ULDFs

The FTIR spectra of the control fiberboard (CF), Si-Al fiberboard (SF), fire retardant fiberboard (FF), and mixed fiberboard (MF) were carried out, and results are shown in Fig. 1.

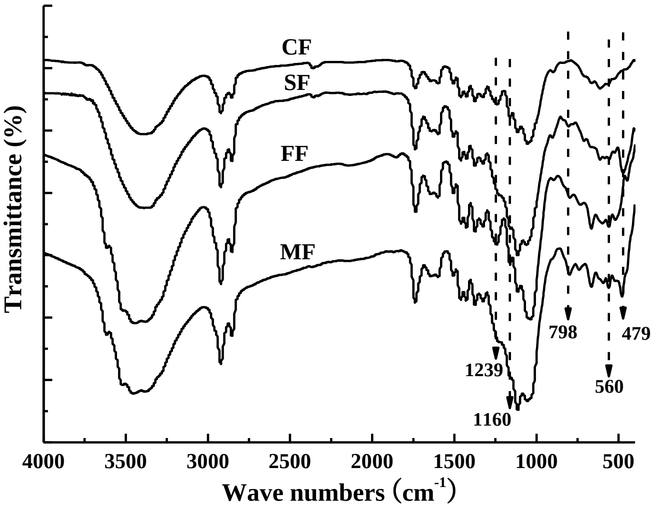

Fig. 1. FTIR profiles of the control fiberboard (CF), Si-Al fiberboard (SF), fire retardant fiberboard (FF), and mixed fiberboard (MF)

The FT-IR spectra of the four fiberboards were recorded in the range 4000 to 400 cm^{-1} (Fig. 1). They showed characteristic peaks at 1034 cm^{-1} (C-O stretching vibrations), 1381 cm^{-1} (C-H bending of CH_3 groups), 1454 cm^{-1} (C-H bending of CH_2 groups), 2921 cm^{-1} (CH_2 stretching), and 3450 cm^{-1} (-OH stretching line). The peaks at around 1650 and 1272 cm^{-1} were the characteristic peaks of lignin, and the peaks at 1088, 881, and 666 cm^{-1} were attributed to cellulose. The additional broad peaks which were not found in the spectrum of the CF at 798, 560, and 479 cm^{-1} were attributed to O-Si-O stretching and bending vibrations, Si-O-Al, or the vibration of Al-O bonds, respectively. In particular, the peak at 479 cm^{-1}, which was attributed to the vibration of Al-O bonds, also appeared in FF. This is because the industrial chlorinated paraffins, which contain Al, were added.

On the other hand, due to the similar mass of Al and Si atoms, the broad band in the range of 1200 to 1000 cm^{-1} usually corresponds to the mixed overlap of Si-O-Si, Al-O-Si, and Al-O-Al bonds (He *et al.* 2014). So, the additional peaks around 1160 and 1114 cm^{-1} in SF and MF were attributed to Si-O-Si and Si-O-C, respectively. Among these covalent bonds, Si-O-C may be formed from the reaction between Si-Al compounds and fibers (see schematic Eq. 1), which could confirm that the Si-Al compounds had been left in the fiberboards.

$$\equiv \text{Si - OH} + \text{HO - Fiber} \underset{\textit{hydrolysis / depolymerisation}}{\overset{\textit{water condensation}}{\rightleftharpoons}} \equiv \text{Si - O - Fiber} + H_2O \qquad (1)$$

Fire Resistance of the ULDFs

Combustion behavior

The fire properties of the ULDFs were evaluated using cone calorimetry (ISO 5660-1; ISO 2002). The cone calorimetric results are presented in Table 2, and the HRR and THR curves obtained using a heat flux of 50 kW m^{-2} are shown in Figs. 2 and 3. In Fig. 2, it can be seen that all the HRR curves demonstrated a two peak profile. The first peak occurred before the protective layer char could formed near the fiber’s surface. Subsequently, further decomposition and cracking of the char towards the burned ends was where the second peak occurred.

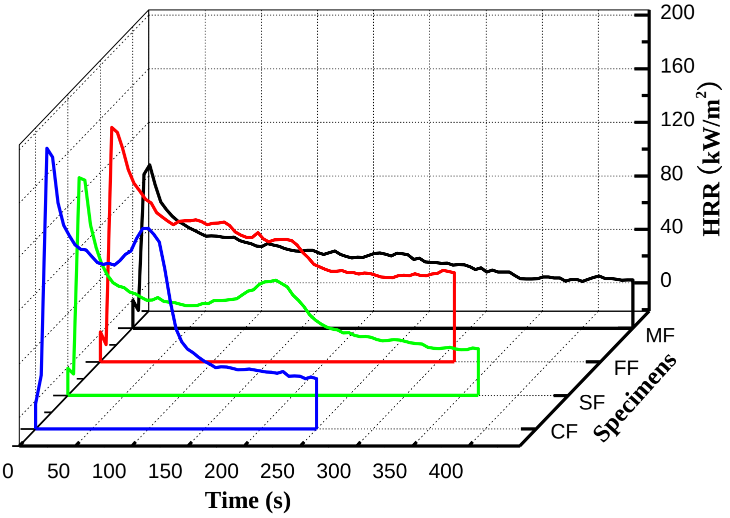

Fig. 2. HRR profiles of the control fiberboard (CF), Si-Al fiberboard (SF), fire retardant fiberboard (FF), and mixed fiberboard (MF), respectively

After flame out, all samples exhibited an after-glowing effect, which is typical for wood materials and is apparent when the black carbonaceous char turns into grey-white ash. This was because of the thermo-oxidative decomposition of the char (Hagen *et al.* 2009; Shabir Mahr *et al.* 2012). Compared to the CF, the HRR profiles of SF, FF, and MF were reduced. In particular, the second peak of each fiberboard was prolonged compared to untreated fiberboard. Similar to the tendency of HRR, the peaks of HRR (pkHRR) of SF (141.29 kW m^{-2}), FF (153.74 kW m^{-2}), and MF (100.76 kW m^{-2}) were lower than the CF (188.53 kW m^{-2}) (Table 2). It was visualized that the degree of reduction in HRR and pkHRR for SF of 500 mL Si-Al compounds (about 27.3% of dry fiber mass) was more pronounced than for FF of 35 g chlorinated paraffins (about 63.6% of dry fiber mass) at this stage. The results indicated that the Si-Al-treated ULDFs were the most successful in reducing the HRR and might be attributed to changes in the condensing phase of char production, which could reduce the rate of diffusion of pyrolysis gases. Additionally, the SF yielded fire residues that may have been caused by the presence of stable Si-Al inorganic film that effectively enhanced protection properties of the residues. It should be noted that the pkHRR of MF (100.76 kW m^{-2}) was the lowest of the four fiberboards studied; a reduction of 46.56% compared to CF. Therefore, the Si-Al compounds and chlorinated paraffins had a synergistic effect. On the other hand, the time to pkHRR was similar for all the fiberboards, which meant that the fiberboards would simultaneously reach the maximum in the energy release, unaffected by the Si-Al compounds and chlorinated paraffins.

Table 2. Fire Performance of the Control Fiberboard (CF), Si-Al Fiberboard (SF), Fire Retardant Fiberboard (FF), and Mixed Fiberboard (MF)

Specimens	pkHRR (kW m^{-2})	Time to the Peak (s)	THR (MJ m^{-2})	TTI (s)	TTF (s)	Mean EHC (MJ kg^{-1})	Mean MLR (g s^{-1})
CF	188.53	10	16.00	2	142	15.30	0.048
SF	141.29	10	15.63	3	211	13.31	0.026
FF	153.74	10	21.36	2	203	14.48	0.042
MF	100.76	15	14.77	2	336	9.46	0.031

The time to ignition is an important parameter to evaluate the ignitability of the materials. The TTIs of the four fiberboards were within the range of 2 to 3 s. Here, the Si-Al compounds and chlorinated paraffins did not play an important role in TTI, which was in accordance with the results of Niu *et al.* (2014). However, TTF (from ignition to flameout) values were affected significantly. As presented in Table 2, the TTF of SF and FF were 32.70% and 30.04% more than the CF, respectively. When the Si-Al compounds and chlorinated paraffins were simultaneously added, the combustion times for MF were extended 192 s more than the CF. Compared with the results of Niu *et al.* (2014), the time to peak and TTF of MF were prolonged nearly 5 s and 130 s, respectively. This indicated that the synergistic effect between Si-Al compounds and chlorinated paraffins plays an important role in TTF of ULDFs.

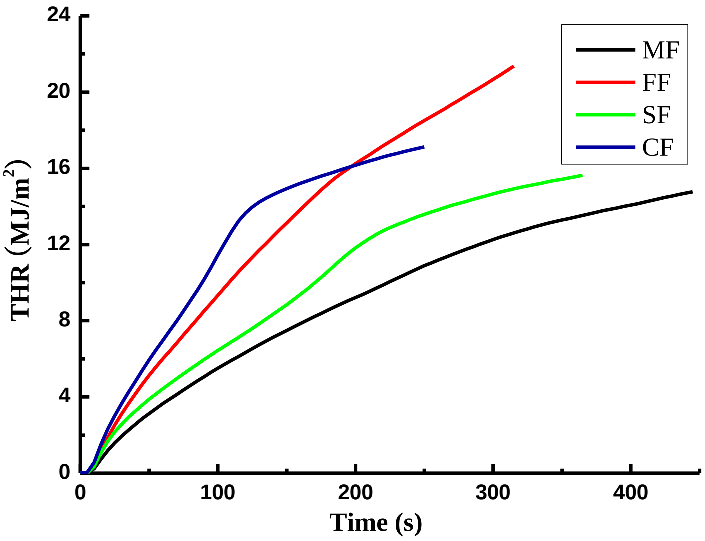

Fig. 3. THR profiles of the control fiberboard (CF), Si-Al fiberboard (SF), fire retardant fiberboard (FF), and mixed fiberboard (MF)

The total heat release (THR) profiles represented the total heat released from the four types of fiberboards as a function of time at 50 kW m^{-2} (Fig. 3). Unlike the HRR profiles, the FF had the highest THR profile (21.36 MJ m^{-2}). This indicated that the FF was combusted most effectively and the total heat release could not be decreased observably by the chlorinated paraffins in this case. And the THR profiles of the FF was higher (5.73 MJ m^{-2}) than the MF (14.77 MJ m^{-2}). This might be attributed to the synergistic effect between Si-Al compounds and chlorinated paraffins. Additionally, the THR profiles of the SF and MF were lower (0.37 MJ m^{-2} and 1.23 MJ m^{-2}) than the CF (16.00 MJ m^{-2}), respectively. It could be concluded that the Si-Al compounds played an important role in this case. On the other hand, the effective heat combustion (EHC), a measure of the combustion efficiency in the gas phase, characterizes a fiberboard with a lower EHC as having favorable fire retardant properties. As can be seen in Table 2, the average EHC of CF, SF, FF, and MF was 15.30, 13.31, 14.48, and 9.46 MJ kg^{-1}, respectively. Particularly, the average EHC of the SF was lower (1.17 MJ kg^{-1}) than the FF, which may result from the slowing of the diffusion of volatiles gaseous products entering the combustion zone caused by the thin Si-Al inorganic film. This result was corresponding to the HRR profiles.

Mass loss vs. time

Percentage mass loss curves obtained as a function of time for four fiberboards are shown in Fig. 4. The mean MLR values of fiberboards are presented in Table 2. Mass loss differences between the four fiberboards were subjected to their HRR differences. The CF (nearly 2%) which had highest mean MLR (0.048 g s^{-1}) showed a residual mass below 10% at the end of the tests. The residual mass of SF (27.37%) was higher than the FF (23.86%), whereas its mean MLR (0.026 g s^{-1}) was lower than FF (0.042 g s^{-1}). This might be ascribed to the inorganic film which could act as a barrier to fuel transport and reradiate the incident flux from the cone calorimeter heater when the fiberboard began to burn and decompose.

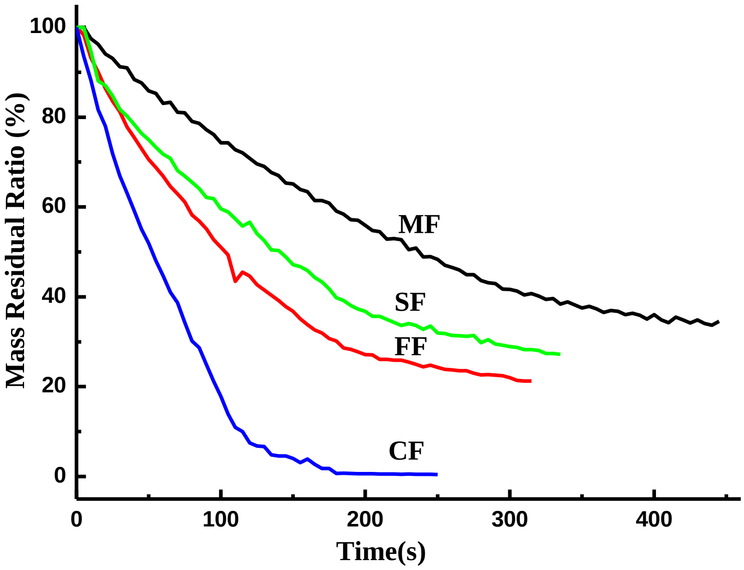

Fig. 4. Mass residual ratio of the control fiberboard (CF), Si-Al fiberboard (SF), fire retardant fiberboard (FF), and mixed fiberboard (MF)

Specially, the release of fuel from the decomposing polymer might be slowed or even be prevented to flow to the flame front (Sacristán *et al.* 2010). However, the chlorinated paraffins could not form the barrier layer to slow or prevent the release of fuel to flow to the flame front. This was in accordance with its high value of HHR, low percentage mass loss, and higher mass loss rate. On the other hand, the MF showed the largest residual mass of 34.26%, which was reported as nearly 30% in Niu *et al.* (2014). But the mean MLR (0.031 g s^{-1}) was between SF and FF. Therefore, it could be concluded that the Si-Al compounds and chlorinated paraffins showed a synergistic effect on mass residual ratio of the fiberboards, but not the mass loss rate.

Smoke and off-gases (CO and CO$_2$) release

Because the ULDFs are manufactured by plant fibers, burning this system evolves large amounts of smoke that cause death from suffocation in a fire. Thus, it is important to study the smoke release in the process of combustion (Niu *et al.* 2014). The TSR profiles of four fiberboards are presented in Fig. 5. All profiles increased rapidly during the pre-ignition period. Because of the lack of oxygen, incomplete combustion occurred while plenty of volatile matter with lower molecular weight contributed to the smoke. Later, the amount of smoke did not increase, except for the TSR profile of CF, which had one bigger climb appearing around 80s. More smoke was possibly derived from burning residue and ash. As seen in Fig. 5, the FF profile showed the highest TSR. This was because the large amount of chlorinated paraffins (about 63.6% of dry fiber mass) can be decomposed with the release of some low molecular weight gases, such as Cl_2 and HCl, during combustion. On the contrary, the fiberboards containing Si-Al compounds showed a significant decrease in the amount of smoke released, thus showing that the incorporation of Si-Al compounds covalently bonded to the fibers reduces the smoke hazard compared to the CF and FF.

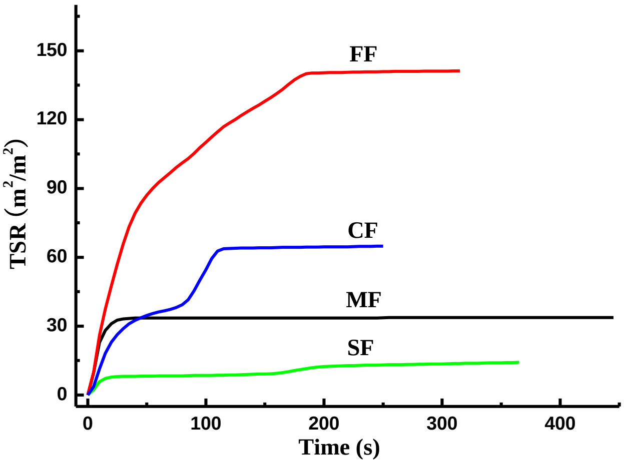

Fig. 5. TSR profiles of the control fiberboard (CF), Si-Al fiberboard (SF), fire retardant fiberboard (FF), and mixed fiberboard (MF)

The fire retardancy mechanism was indicated by CO and CO$_2$ production. Flame inhibition, which could increase charring, shows the potential decrease of the absolute CO production results in an increase in combustion products that is typical of incomplete combustion (Spontón *et al.* 2009; Sacristán *et al.* 2010). The behaviors of the CO evolution rate during these cone calorimetry experiments are shown in Fig. 6. The CO concentration profiles of all the specimens were fluctuant and gradually increased as a function of time. Compared to the control specimen, the times to the peaks of CO concentration and COY were delayed when the specimens were treated by fire retardant or Si-Al compounds. This indicated that both fire retardant and Si-Al compounds had a significant effect in the fire resistance of ULDFs. On the other hand, the CO concentration and COY of FF were larger than the SF, especially the COY. This was because the flame inhibition resulted in incomplete combustion, corresponding to the chlorinated paraffins' ability to explode a large number of Cl radicals that could combine with hydrogen and hydroxide. Nevertheless, silicon compounds can form an inorganic film on the surface of wood to insulate wood against air during combustion. Aluminum compounds could absorb a lot of heat through their dehydration reaction when heated.

Therefore, the presence of Si-Al compounds as a fire retardant agent added to ULDFs has an important effect in the condensed phase. Liu (2013) also obtained the same results by comparing the CO concentrations of ULDF with or without fire retardant treatment. And Niu *et al.* (2014) showed that the Si-Al compounds effectively reduced the total amount of smoke released, and they also decreased the CO yields. Furthermore, the MF had the lowest CO yield, whereas the time to the peaks of CO concentration and COY was also delayed. This indicated that the chlorinated paraffins and Si-Al compounds could generate the synergistic effect in ULDFs.

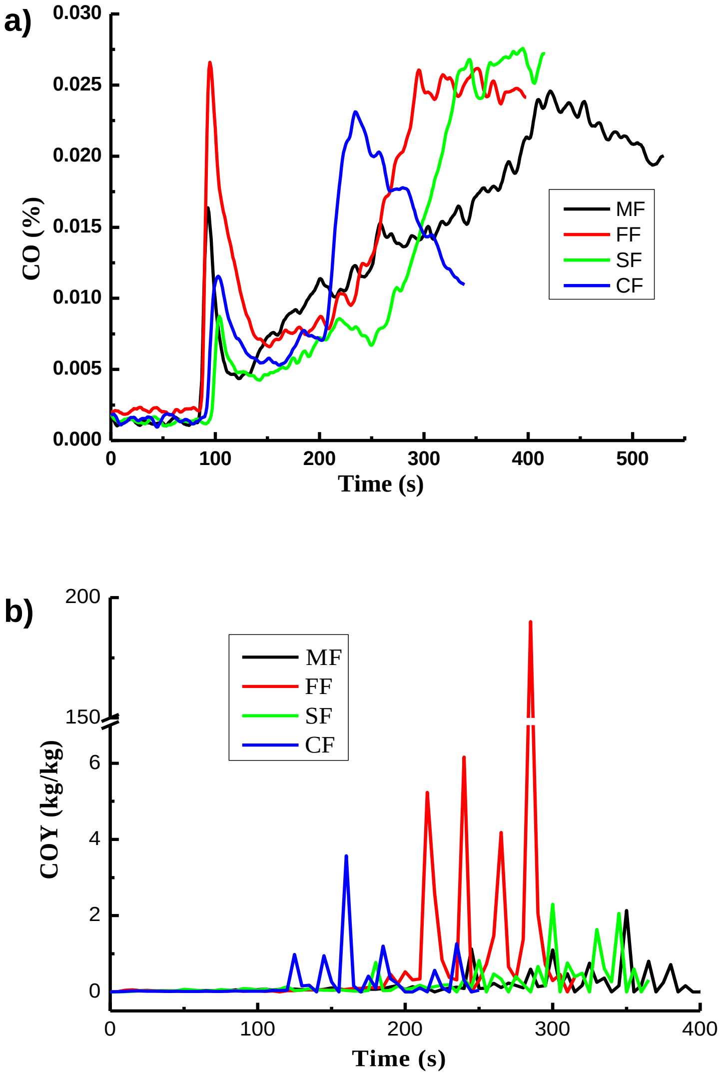

Fig. 6. CO concentration (a) and CO yield (COY, b) profiles of the control fiberboard (CF), Si-Al fiberboard (SF), fire retardant fiberboard (FF), and mixed fiberboard (MF)

The CO_2 concentration and CO_2Y profiles of all the specimens are shown in Fig. 7. By comparison, the results of CO_2 were opposite to the CO production process. CO_2 mainly came from the complete combustion of carbon at the flaming combustion stage (Niu *et al*. 2014). The CO_2 concentrations of ULDFs were higher than those in glowing combustion, and their profiles were similar to the HRR profiles. As can be seen in Fig. 7, the CO_2 concentration of CF was higher than SF, FF, and MF. Also, the time to the peaks of CO_2Y in CF was shorter than in the other specimens. This indicated that the chlorinated paraffins and Si-Al compounds had a significant effect on the fire resistance of ULDFs. Comparing FF to SF, the CO_2 concentration of FF was slightly less than in SF.

The time of the CO_2Y peaks was also prolonged. However, the peak of CO_2Y around 300s in FF was larger than in other specimens. This is dangerous, because when a large amount of CO_2 is produced instantaneously, people can suffocate. Additionally, the MF had the lowest CO_2 concentration and CO_2Y. These results also indicated that the chlorinated paraffins and Si-Al compounds had a significant effect on improving the fire resistance of ULDFs, having a synergistic effect.

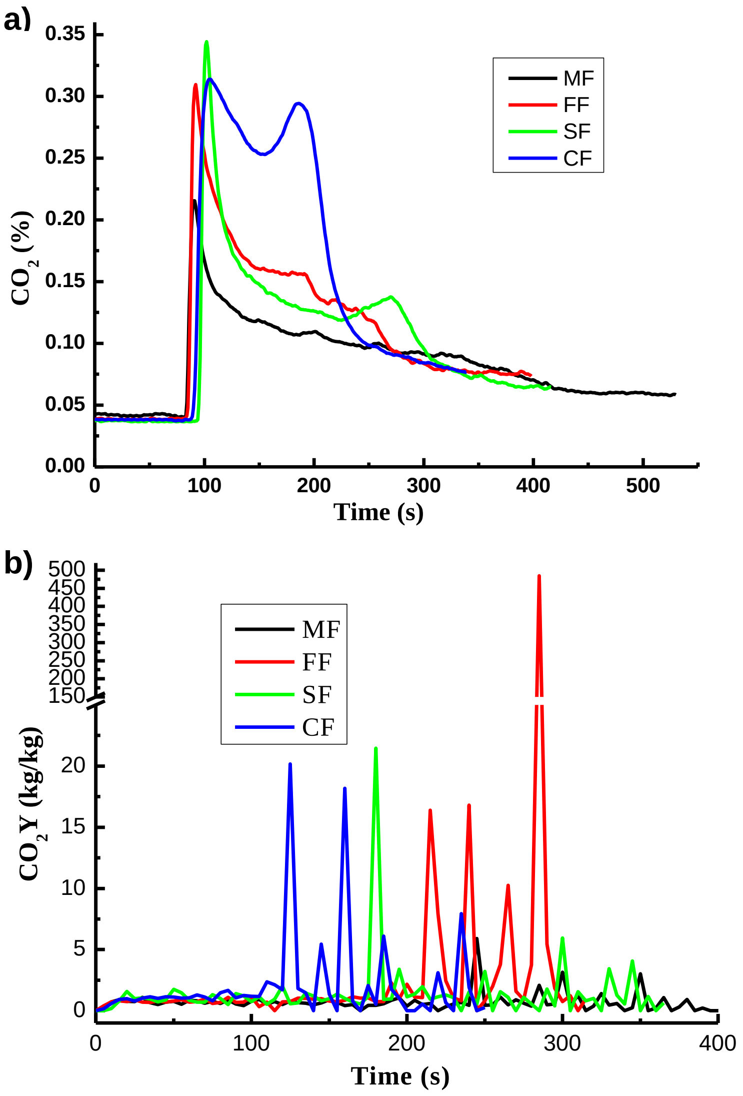

Fig. 7. CO_2 concentration (a) and CO_2 yield (CO_2Y, b) profiles of the control fiberboard (CF), Si-Al fiberboard (SF), fire retardant fiberboard (FF), and mixed fiberboard (MF)

CONCLUSIONS

1. Compared to the control fiberboards (CF), the heat release rates (HRRs) of Si-Al fiberboards (SF), fire retardant fiberboards (FF), and specimens containing both types of fire retardant component (MF) were reduced. The pkHRRs of SF (141.29 kW m^{-2}), FF (153.74 kW m^{-2}), and MF (100.76 kW m^{-2}) were lower than the CF (188.53 kW m^{-2}). The pkHRR of MF at 100.76 kW m^{-2} was a 46.56% reduction compared to CF, which was the lowest for the four fiberboards studied.
2. The results of TTF, THR, and mass loss were remarkably decreased with the addition of Si-Al compounds and chlorinated paraffin in all specimens. Especially, MF

showed the best results with TTF (336s), THR (21.36 MJ m^{-2}), and residual mass (34.26%), when the Si-Al compounds and chlorinated paraffins were simultaneously added.

3. Chlorinated paraffins and Si-Al compounds also significantly restricted the smoke and off-gases (CO and CO_2) release. Due to the fact that chlorinated paraffins can be decomposed by releasing Cl_2 and HCl gases, the FF profile showed the highest TSR. Opposingly, the fiberboards containing Si-Al compounds showed a significant decrease in the amount of smoke released. Si-Al compounds play an important role in the fire retardance of ULDFs. The synergistic effect between Si-Al compounds and chlorinated paraffins showed a better result in ULDFs.

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