Effect of PVDC on the Fire Performance of Ultra-Low Density Fiberboards (ULDFs)

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Poly vinylidene chloride-vinyl chloride emulsions (PVDC) were added as a substitute for chlorinated paraffin (CP) in the preparation of ultra-low density fiberboards (ULDFs). The micromorphology and fire performance of ULDFs were investigated using a scanning electron microscope, limiting oxygen index instrument, and cone calorimeter. The results showed that PVDC specimens were coated with a regularly smooth film, while the distribution of CP inside CP specimens was uneven. The limiting oxygen index increased with the dosage of PVDC, then reached a plateau at 50 mL and 28%, slightly higher than CP specimens (27.3%). The peak of heat release rate, mean heat release rate, mean CO, and total smoke release of PVDC specimens was reduced 43.3%, 13.5%, 38.5%, and 51.5% lower than respective CP specimens, and with nearly the same total heat release (only 0.04 MJ/m^2 higher). Thus, PVDC exhibited excellent heat-reducing and smoke-suppressing properties and could replace CP in ULDFs.

Keywords: Forming material; Plant fiber; Chlorinated paraffin, Vinylidene chloride; Vinyl chloride; Cone calorimeter; ULDFs

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INTRODUCTION

Due to the overdevelopment of fossil fuel resources, ultra-low density fiberboards that are produced by renewable, inexpensive, abundant, and environmentally benign plant fibers have attracted much attention recently (Xie *et al.* 2011; Niu *et al.* 2014; Chen *et al.* 2015a). In addition to their eco-friendly appeal, they have potential as functional materials with low densities (50 kg/m^3), low thermal conductivity coefficients (0.03 W/mK), and high sound absorption ratios (0.8%), which could be used as architectural heat preservation materials and buffering packaging materials. They are also safe and cost-effective alternatives to expandable polystyrene and styrofoam, which are based on petroleum products (Chen *et al.* 2014, 2016a; Liu *et al.* 2014).

Nevertheless, a hindrance to using ULDFs on a large scale is their high flammability. In many low density polyethylene products, aluminum trihydroxide and magnesium hydroxide are used as fire retardants (Liang *et al.* 2013; El Hage *et al.* 2014; Hoffendahl *et al.* 2015a, b) because they have high decomposition temperatures and a low smoke release (Sener and Demirhan 2008). These hydroxides release water vapor to remove heat close to flames and reduce the formation of combustible gases. To inhibit the combustion of cotton fabrics, halogenated and formaldehyde-based flame retardants have been extensively applied to cotton textiles in the past decades (Mohsin *et al.* 2013; Xie *et* *al.* 2013; Schramm *et al.* 2014; Dong *et al.* 2015). Water glass, Al-Si compounds, and chlorinated paraffin-70 (CP) are added during the manufacture of ULDFs to improve their fire performance (Niu *et al.* 2014; Chen *et al.* 2015b, 2016b). Thanks to the high content of the element Cl in CP and the synergistic action between CP and Al-Si compounds, the effects of flame retardants are significantly enhanced. There are synergetic effects hypotheses, gas and solid phase mechanisms, and discontinuous heat exchange mechanisms which could be used to explain this enhancement (Liu *et al.* 2014).

Unfortunately, CP is not an environmentally friendly flame retardant, as its continuous release is a concern due to its persistent and toxic bioaccumulation (Covaci *et al.* 2011; Kharlyngdoh *et al.* 2015). Moreover, to obtain effective flame retardants, a high loading ratio is required, up to 65% (Cavodeau *et al.* 2015).

Polyvinylidene chloride-vinyl chloride (PVDC) emulsions is a high polymer material that was initially manufactured by American Dow chemical company in 1936. It is avirulent, exhibits insipidity, does not light, can be used with water as a solvent, and it does not release odors during construction (Hess *et al.* 1995; Wessling *et al.* 1997). PVDC also has been used widely as a barrier polymer in the plastics packaging industry and architectural coatings (Howell and Zhang 2006). They exhibit low permeability to low molecule weight matter (*i.e.*, O_2, CO_2, and H_2O), which could prevent food spoilage or the loss of flavor (Howell, 1987). In many high barrier materials, PVDC is the only product approved by Food and Drug Administration that could directly contact food. It also could be the best cost-effective packaging structure to meet the requirements for odor in packaging of raw milk. PVDC coatings have been shown to be reliable sanitation properties and it is safety to be used as food and drug packaging. They are also widely used as fireproof coating in common buildings and steel-frame architecture.

Currently, there is no research in the literature concerning PVDC as a kind of flame retardant substitute for CP in ULDFs. The main objective of this study was to evaluate the effectiveness of the PVDC on the fire performance of ULDFs as tested using a cone calorimeter.

EXPERIMENTAL

Materials

Kraft pulp (KP, Spruce-pine-fir, Tembec Inc., Quebec, Canada) was used to fabricate the ULDFs. Sodium silicate, aluminum sulfate, borax, and zinc sulfate were purchased from the Tianjin Fuchen chemical reagents factory (Tianjin, China). Sodium dodecylbenzene sulfonate was purchased from the Jiangsu Qingting Washing Products Co., Ltd. (Jiangsu, China). CP and PVDC were supplied by the Changzhou Fengshuo Chemical Company, Ltd. (Changzhou, China) and the Nantong Repair-air Chemistry Bioengineering Co., Ltd. (Nantong, China), respectively.

Methods

Manufacture of ultra-low density fiberboards

The ULDFs were manufactured using a liquid foaming method (Xie *et al.* 2011) with a target bulk density of 50 to 70 kg/m^3. Adhesive (homemade), surfactant (sodium dodecyl benzene sulfate, 20 wt.%), and water repellent (alkyl ketene dimmer) were added at mixture manufacturing stages in volumes of 20 mL, 40 mL, and 50 mL, respectively.

B-Zn-Al-Si compounds were prepared as described in Wu *et al.* (2016) and used as a flocculant added to the ULDFs. Flame retardant (PVDC or chlorinated paraffin) was added during manufacturing. The detailed process is shown in Fig. 1.

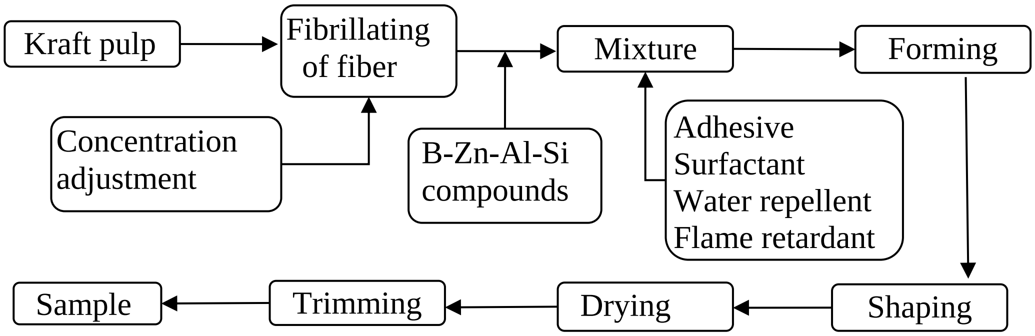

Fig. 1. The preparation of ULDFs

Microstructural characterization

The surface topography of specimens was characterized using SEM (Philips XL-30 TMP, (Eindhoven, Netherlands) with an acceleration voltage of 5 kV. Before being tested, the specimens were sputter-coated with gold using a coater.

Limiting oxygen index test (LOI)

LOI was used to test the fire performance of PVDC. Measurements were performed using a limiting oxygen index instrument (Jiang Ning County Analysis Instrument Factory, Jiang Ning, China) according to GB/T 2406.2-2009 (2009). Specimens were shaped to the dimensions of 150 × 10 × 10 mm (*L* × *W* × *H*) and placed in a holder covered by a vertical glass column. The gas flow (oxygen and nitrogen) was adjusted in order to meet test criteria.

Specimens were ignited on the top surface with a flame and burned downward, and the minimum oxygen concentration that supported combustion was recorded as a percentage. For each parameter, the experiment was repeated fifteen times. The presented results are the arithmetical average of the obtained values.

Fire properties test

A cone calorimeter (FTT Co., West Sussex, UK) was adopted to test the fire properties of the ULDFs according to ISO 5660-1 (2002), such that the parameters of heat release (HRR), total heat release (THR), effective heat of combustion (EHC), mass residual ratio (MRR), specific extinction area (SEA), and total smoke release (TSR) were measured as a function of time.

Test specimens were prepared to the dimensions of 100 × 100 × 30 mm (*L* × *W* × *H*), wrapped with aluminum foil without the heating surface, and placed in a stainless-steel frame with an asbestos board in the bottom. The tests were performed at a heat flux level of 50 kW/m^2, which could cause temperatures as high as 780 ^{o}C on the heating surface, for the purpose of simulating real fire conditions.

RESULTS AND DISCUSSION

Micromorphology of ULDFs

As shown in Fig. 2a, the fibers were straight and slipped past each other, sticking to few adhesives and flocculants. In Fig. 2b, there were many particles of CP that formed clumps, some of which clustered together with fiber and some of which did not. Raw fiber was observed, which indicated that the fire retardants were unevenly distributed. This could affect the flame resistance of CP in a fire.

A smooth and uniform film covered the fiber shown in Fig. 2c. This film could enhance the interconnection between fibers and isolate fiber from heat generated during the firing.

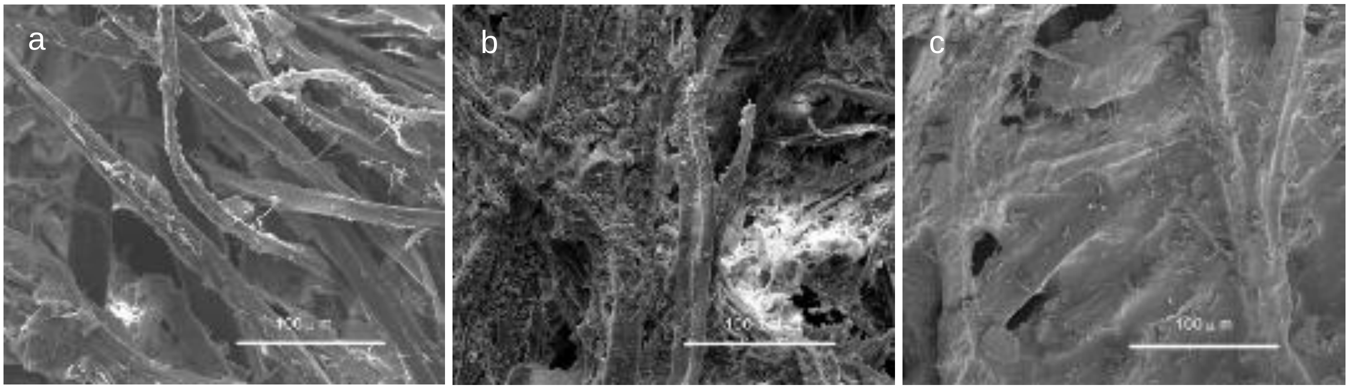

Fig. 2. SEM of (a) control specimen, (b) CP specimen, and (c) PVDC specimen

Effect of PVDC on LOI of ULDFs

The limiting oxygen index test is a reproducible test that only requires small specimens and presents a single value that can be used for a presumed flammability ranking of materials (Suzanne *et al.* 2014). The results of the LOI test with different dosages of PVDC are showed in Fig. 3.

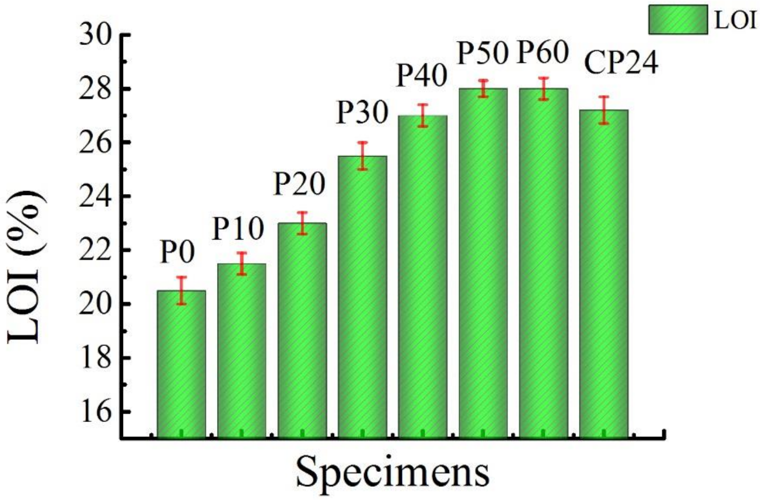

Fig. 3. Effect of addition of PVDC (0 to 60 mL) and CP (24 g) on LOI of ULDFs

The LOI increased at a smooth and constant rate before reaching 50 mL, at which point a plateau was reached with a high of 28% regardless of increases in PVDC. As mentioned in Wu *et al.* (2016), the flocculant was able to absorb fine fibers and other smaller particles (*e.g.*, PVDC, CP, and adhesives). Using flocculation, more tiny fire retardant particles would stay in the specimens. However, the flocculation ability has its ceiling. Once a hit cap was reached, the extra (additional PVDC) was drained out with water during the manufacture of ULDFs. As a result, the increasing dosage did not result in the higher LOI results. The LOI of CP was 27.3%, between 27% and 28%, with 40 mL and 60 mL of PVDC, respectively.

Fire Performance of ULDFs

According to the LOI results, four specimens, tagged by differences in dosage addition of flame retardant in Table 1, were tested using a cone calorimeter.

Table 1. Differences between Specimens

Specimens	Chlorinated Paraffin(g)	PVDC (mL, 40 wt.%)
P0	0	0
CP	24	0
P40	0	40
P60	0	60

Heat release

On the basis of the oxygen consumption principle, cone calorimeters have been extensively used in the evaluation of flammability characteristics of materials (Gallina *et al.* 1998). The HRR, as measured using a cone calorimeter, is the single most important parameter, as it expresses the intensity of a fire (Babrauskas and Peacock 1992). A highly flame retardant system normally shows a low mean-HRR value. The peak-HRR value is used to express the intensity of the fire (Zhang *et al.* 2012). The changes of HRR as a function of burning time for different specimens P0, CP, P40, and P60 are shown in Fig. 3.

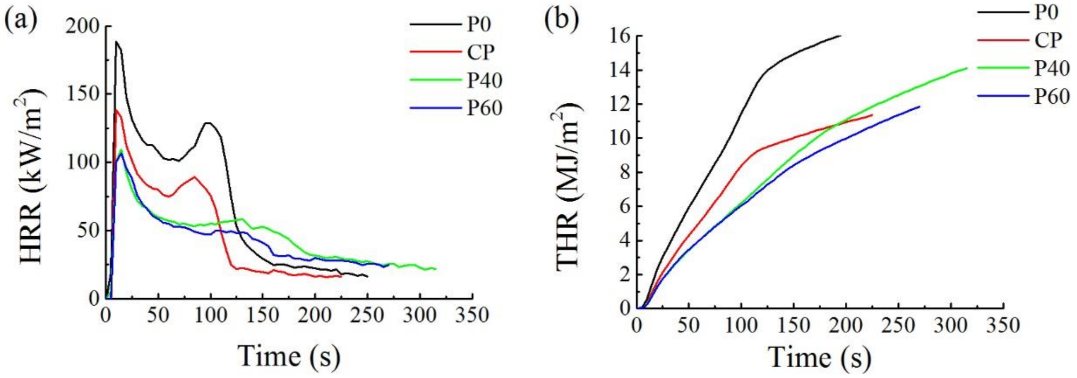

Fig. 4. HRR curves (a) and THR curves (b) of the control fiberboard (P0), fiberboard with chlorinated paraffin (CP), and fiberboard with PVDC (P40 and P60)

The cone heat flux was absorbed at the sample surface, and the degradation products were transformed into the gas phase instantly. P0 (Fig. 4a) burned very fast after ignition, and a sharp peak of HRR approached 188.53 kW/m^2 at a range of 15 to 20 s. In the cases of the treated systems, both the HRR peak and HRR mean (Table 2) were reduced remarkably with the addition of the fire retardant. The peaks of HRR were reduced by 26.6%, 41.9% and 43.3% for CP, P40, and P60 specimens relative to the control specimen, respectively. The combustion time prolonged to 315 s and 270 s for P40 and P60 from 250 s and 225 s for P0 and CP specimens, respectively. The second peak at around 100 s was generally associated with temperature increases on the unexposed surface of the specimen (Stark *et al.* 1997). However, this peak did not appear on P40 and P60 specimens. This may be the result of protection from char layers formed by PVDC. The CP and the PVDC specimens displayed remarkably different combustion behaviors. CP behaved very similarly to the control specimen, whereas the two PVDC specimens followed a fairly homogeneous path. The reason for these differences may be due to the fact that CP decomposes earlier than fiber, and that some small volatile molecules were produced from the decomposition of CP. The mean-HRR values showed a trend similar to that of peak HRR (Table 2). The lowest value of mean-HRR was P60 (only 53.4% P0), followed by P40 (54.3% P0), with CP lagging behind with 61.7% P0. The THR was reduced by 28.8%, 11.8% and 25.9% for the CP, P40, and P60 specimens, independently. The reduction of peak HRR becomes small with the increase of PVDC concentration (only 2.4% less than P40), while the reduction of peak THR was 15.9% less than P40. The reduction of peak HRR indicates that a cohesive char layer was formed during combustion which acted as an insulating barrier between the fire and plant fiber and hindered the heat and mass transport.

Table 2. Cone Data of the Control Specimen (P0), Chlorinated Paraffin Specimen (CP), and PVDC Specimen (P40 and P60)

Specimens	Peak HRR kW/m^2)	Mean HRR (kW/m^2)	THR (MJ/m^2)	Mean EHC (MJ/kg)	Mean SEA (m^2/kg)	Mean COY (kg/kg)	Total Oxygen Consumed (g)
P0	188.53	83.70	16.00	15.30	52.47	0.05	10.64
CP	138.37	51.67	11.39	12.00	65.83	0.13	7.76
P40	109.54	45.49	14.11	9.28	19.28	0.08	9.55
P60	106.86	44.72	11.86	8.27	26.45	0.11	8.27

All of the results in Table 2 illustrate that PVDC not only reduced more peak of HRR than CP, but also suppress the THR. The effect in reduction of peak of HRR is not remarkable during the increase of PVDC, which corresponds with the LOI results.

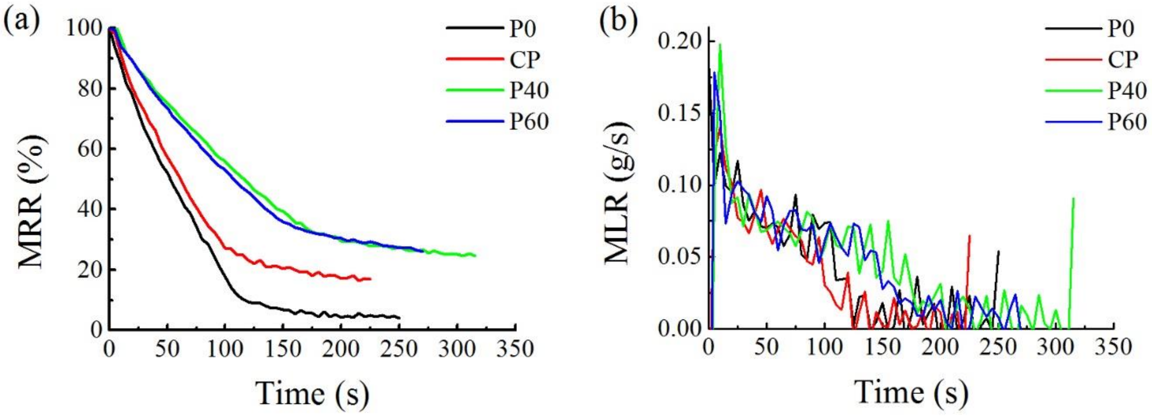

Fig. 5. MRR curves (a) and MLR curves (b) of the control specimens (P0), chlorinated paraffin (CP), and PVDC (P40 and P60)

Mass loss and thermal degradation

There was an absence of remarkable differences between the MRR curves of PVDC-treated specimens (Fig. 5a), which indicated that the combustion reaction involved the same degradation products from the fiber. Two drops were present, the highest 26.1% of P60 to 16.9% of CP and 4.1% of P0, which were subject to their HRR differences. This indicated that the film formed by PVDC (Fig. 2c) played an important role and could act as a barrier to fuel transport and reradiate the incident flux from the cone heater when the specimens began to burn and decompose.

This trend was the same for heat release rate and mass loss rate measured using the cone calorimeter (Fig. 5b). These similarities further confirmed that the reduction in heat release rate and burning mass loss rate were the result of processes in the condensed phase.

Smoke and off-gases (CO and CO_2) release

Specific extinction area indicates the release of smoke per kilogram of the specimen during combustion. As shown in Fig. 6a, a sharp spike appeared before 50 s, which manifested as a stream of smoke emitting from the specimens. The reason for this may be that the low-molecular-weight gas was coming from the decomposition of heated substances. After that, a larger peak appeared in P0 and CP specimens between 70 s and 100 s. This may have been caused by the burning of unexposed specimen surfaces when temperatures increased.

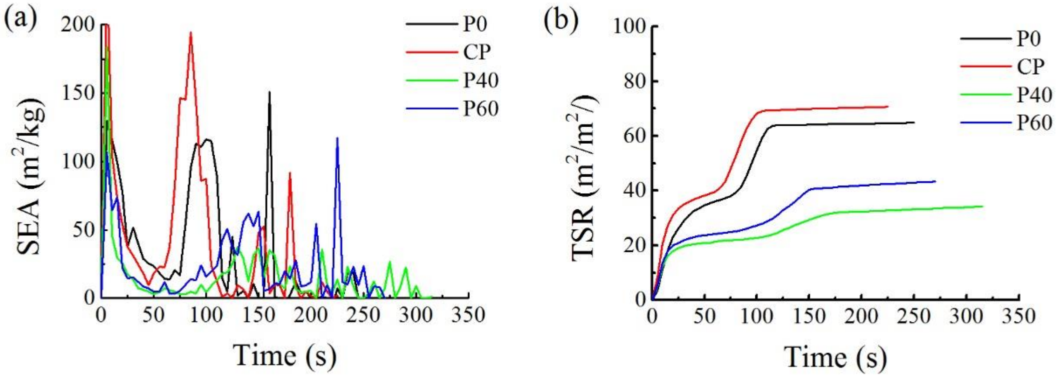

Fig. 6. SEA curves (a) and TSR curves (b) of the control specimen (P0), chlorinated paraffin (CP), and PVDC (P40 and P60)

The TSR curves are shown in Fig. 6b. The CP profile had the highest value, which was in accordance with previous results (Chen *et al.* 2015b). The reason for this may be that the chlorinated paraffin accounted for a large percentage of weight (about 48% of dry fiber mass) that could have decomposed during the release of some low molecular weight gases, such as Cl_2 and HCl, during combustion. Chlorinated paraffin could have also caused an incomplete combustion of fiber with oxygen, resulting in the production of more CO during firing. This can be seen in Fig. 7a as a series of large spikes range from 150 s to 200 s. A high dosage of PVDC decreased the peak-HRR and THR value, but produced more smoke. The lowest value of TSR was P40 at only 34.3 $m^2 \cdot m^2$. The values of P60, CP, and P0 were 43.3, 70.7, and 64.9 $m^2 \cdot m^2$, respectively. This was because as PVDC loading increased, more low-molecular-weight gases were generated during combustion, and more ash and dust was raised on the surface of the fibers. Thus, PVDC could better reduce total smoke released than chlorinated paraffin, but a smaller amount of PVDC (40 mL) was superior to a larger one.

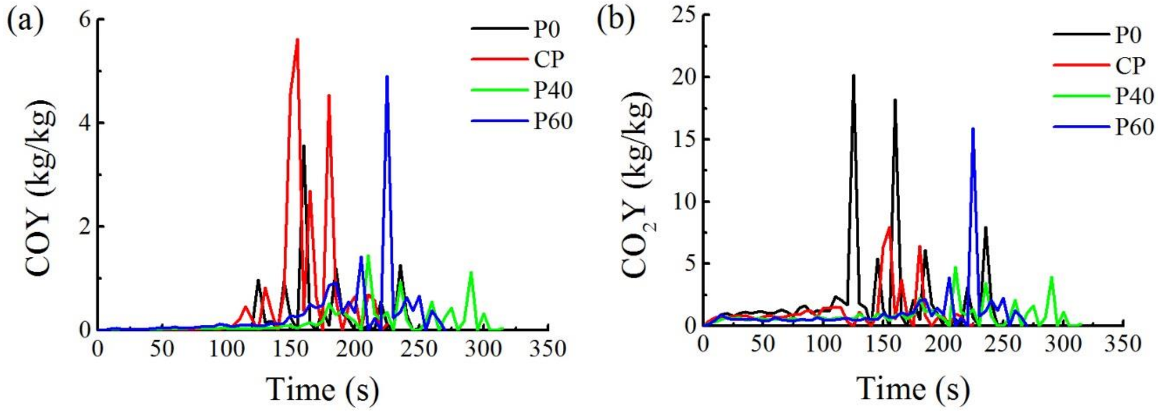

Fig. 7. COY curves (a) and CO_2Y curves (b) of the control specimen (P0), chlorinated paraffin (CP), and PVDC (P40 and P60)

Carbon monoxide is still likely to be the major toxicant in modern fires (Alarie 2002). The principal causes of death are carbon monoxide followed by carbon dioxide poisoning and/or oxygen deficiency, while the influence of heat is of minor importance (Gormsen *et al.* 1984). As shown by the yield of CO in Fig. 7a, a great amount of CO was released from P0 and CP specimens between 150 s and 200 s. However, there was a delay in CO release until between 200 s and 240 s for PVDC specimens. Compared to P0 and CP, only one sharp peak appeared for P60. The reason for this may be that PVDC formed a thin cover on the surface of fiber that could protect against heat and consequently reduced the production of CO. This also corresponded with the mean COY shown in Table 2, where P40 drops lower in value (0.08 kg/kg) than P60 (0.11 kg/kg) and CP (0.13 kg/kg). The addition of PVDC may have been unevenly distributed on the fiber surface, which could have affected the structure stability of the film. When the temperature rose, the film could have easily collapsed, resulting in low-molecular-weight substances (*e.g.*, CO) erupting at the surface of film. This corresponds with the spikes between 200 seconds to 250 seconds in Fig. 7. There was also a negative correlation between the mean COY and total oxygen consumed. The control specimen burned more completely at the flaming combustion stage than other specimens (Fig. 7). However, it was also harmful to people when major oxygen concentration is depleted (< 10%) because of efficient and complete combustion of fiber which leads to the release of massive amounts of carbon dioxide. This would make people or animals suffocate.

Based upon analysis of experiments, PVDC could not only suppress the smoke (only 52.9% of P0) but also reduce the release of toxic gases.

CONCLUSIONS

1. The loss on ignition (LOI) continuously increased before reaching 50 mL, then went to a plateau with a high of 28%.

2. The poly-(vinylidene chloride-vinyl chloride) emulsions (PVDC) not only remarkably reduced heat release, but also suppressed smoke and poisonous gases off. The peak of heat release (HRR), mean HRR, mean CO, and total smoke release (TSR) was 43.3%, 13.5%, 38.5%, and 51.5% less than chlorinated paraffin (CP), respectively.
3. Moreover, PVDC slowed thermal decomposition and improved the mass residual rate (54.4% more than CP). PVDC could substitute for chlorinated paraffin as a flame retardant in ultra-low density fiberboards (ULDFs).

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