Ultra-Low Density Fibreboard with Improved Fire Retardance and Thermal Stability using a Novel Fire-Resistant Adhesive

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A novel fire-resistant adhesive made from polyvinyl alcohol, urea, phosphoric acid, and starch was demonstrated for use as a binder and fire retardant to produce ultra-low density fibreboard (ULDF) with clear environmental benefits. The results from Fourier transform infrared spectroscopy showed the presence of chemical bonding between fire-resistant adhesives and ULDFs. The limiting oxygen index (LOI), combustion behaviour, and thermal stability were characterized using a LOI text, cone calorimeter, and thermal analyzer, respectively. The results demonstrated that the LOI value of the fire-retardant ULDF can reach up to 34.2 with 300 mL of fire-resistant adhesive. It was established that the additive noticeably reduced the peak of heat release rate, total heat release, and total smoke release of ULDF. Their morphologies after combustion were elucidated using a scanning electron microscope, and a char layer in the condensed phase was observed. Thermal analysis showed that the thermal stability of ULDF improved dramatically and the residual weight increased 4-fold, to 48.32%. Therefore, such ULDFs will be tremendously attractive as renewable, sustainable, and bio-based insulating materials.

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INTRODUCTION

Ultra-low density fibreboard (ULDF) is an environmentally friendly and formaldehyde-free material made up of natural fibres. It has an ultra-low density, falling between 10 and 90 kg/m^3, high water repellency with a contact angle of 134° (Cai *et al.* 2016) and excellent performance with respect to noise reduction (noise reduction coefficient of 0.61 to 0.73) and thermal insulation (0.020 to 0.038 W/mK). It can be used as a packaging buffer material and building insulation material (Xie *et al.* 2011). However, its applications are restricted due to its content of highly flammable plant fibres. Thus, it is necessary to develop appropriate methods of inhibiting the flammability of plant fibre-based composites (Marolewski and Weil 1995; Jiang *et al.* 2010; Stark *et al.* 2010; Wang *et al.* 2015).

Fire retardants such as chlorinated paraffins and Si-Al compounds have been employed to improve the fire properties of ULDF, and results have revealed that the synergetic effect between chlorinated paraffins and Si-Al compounds can significantly restrain the fire hazard intensity and gas volatility (Xie *et al.* 2012; Cai *et al.* 2014; Niu *et*

al. 2015a; Niu *et al*. 2015b). Nevertheless, halogenated retardants are likely to generate toxic gas and corrosive smoke that is hazardous to human health when exposed to fire. Therefore, the development of an environmentally benign fire retardant capable of cross-binding fibres and concurrent inflammation retardance is required.

Phosphorus- and nitrogen-based intumescent fire retardance can improve the fire behaviour of wood-based composites (Horacek and Grabner 1996; Zhang and Horrocks 2003; Gao *et al.* 2006; Stevens *et al.* 2006; Ma and Fang 2013). The formulation of an intumescent system usually consists of three components: (i) an acid source, (ii) a carbonizing agent, and (iii) a blowing agent. Ammonium polyphosphate (APP) is part of intumescent systems, which can act as an acid source for blowing and carbonizing agents. Thus, acid species catalyse the dehydration reaction of the carbonizing agent, leading to the formation of a carbonaceous layer. It has been reported that APP releases phosphoric acid, poly-phosphoric acid, and non-flammable gases when exposed to high temperatures. The resulting acid contributes to plant fibre intermolecular dehydration and charring. The acid from the pyrolysis of APP catalyses C-O bond breaking to produce many oligomers and small molecular species at lower temperatures (Chou *et al.* 2009). However, the high polymerization degree of APP makes it hydrophobic and insoluble in water (Shumao *et al.* 2010), which is not compatible with the water-based frothing method for ULDFs. According to these facts, modification of N-P based fire retardants has emerged. Poly(vinyl alcohol) (PVOH), as a water-soluble synthetic polymer with excellent film formation, has been introduced to the N-P system. Materials based on phosphorus, nitrogen, and PVOH containing flame retardant are also versatile materials (Hoffman 2012). For example, the synthesis of ammonium polyvinyl phosphate can absorb H_2S in environmental determination (Yan *et al.* 2004). Likewise, phosphorylated polyvinyl alcohol can be obtained by selecting dimethylformamide as the reaction medium and used as paper adhesive coating (Saat *et al.* 2014; Zang *et al.* 2015). These routes open up the possibility of preparing a new ULDF with enhanced flame retardant properties.

ULDFs are manufactured using water as the liquid medium and plant fibre as the matrix during the chemical reaction. The introduction of fire-resistant adhesives including urea, phosphoric acid, and PVOH are all water-soluble and compatible with the foaming system. The hydrolysis of urea can split one of the hydrogen ions from phosphoric acid, which will in turn facilitate the esterification reaction when exposed to PVOH under certain conditions (Huheey *et al.* 2006). When they are combined with plant fibres during the mechanical frothing process, they may evenly distribute on the surface of fibres, act as an adhesive to reinforce the connection between fibres or as an intumescent flame retardant.

The combination between N-P based fire-resistant adhesives and plant fibres may play an active role in the fire properties of ULDF. However, very little attention has been paid to studying this connection, and scant information is currently available for applying N-P based fire-resistant adhesive as an independent fire retardant, other than by using one component of complex fire retardants for ULDF (Liu 2013). It is also a method of preparing an environmentally friendly and halogen-free ULDF by using N-P based fire-resistant adhesive. Therefore, the purpose of this paper was to synthesise a novel fire-resistant adhesive and to investigate its effect on fire properties and ULDF thermal behaviour. Limiting oxygen index (LOI) characterization and a cone calorimeter (CONE) were employed to test the fire behaviour of ULDFs, and the obtained residues were characterized using scanning electron microscopy (SEM). Fourier transform infrared spectroscopy (FTIR) analysis was introduced to identify the chemical bonding between fibres and N-P

based fire-resistant adhesives. Thermogravimetric analyses coupled with FTIR was also performed.

EXPERIMENTAL

Materials

Kraft pulp (KP, spruce-pine-fir, Tembec Inc., Canada) was utilized as a raw material in manufacturing ULDF. Phosphoric acid (A.R. reagent), urea (A.R. reagent), polyvinyl alcohol (PVOH-1799, polymerization degree of 1700 or more and a saponification degree of 99 mol or more), and starch purchased from the Tianjin Fuchen Chemical Reagents Factory (China) were used to generate the N-P based fire-resistant adhesives. Sodium silicate (Changzhou Fengshuo Chemical Company, LTD, China) was employed to adjust the pH value of the foaming system.

Methods

Preparation of fire-resistant adhesive

The preparation of the fire-resistant adhesive was carried out in three stages. Intermediate A was synthesised at a phosphoric acid/ urea molar ratio of 1:2 for 1 h, experiencing a stirring rate at 400 rpm. Then, a 6 wt% PVOH aqueous solution was introduced into the compounds slowly, and the mixture was stirred at a speed of 1000 rpm for 2.5 h at 90 °C (Intermediate B). A fire-resistant adhesive (4.2 wt%) was obtained when a 2.5 wt% starch solution was added to the composite. Its possible manufacture scheme is summarized in Fig. 1.
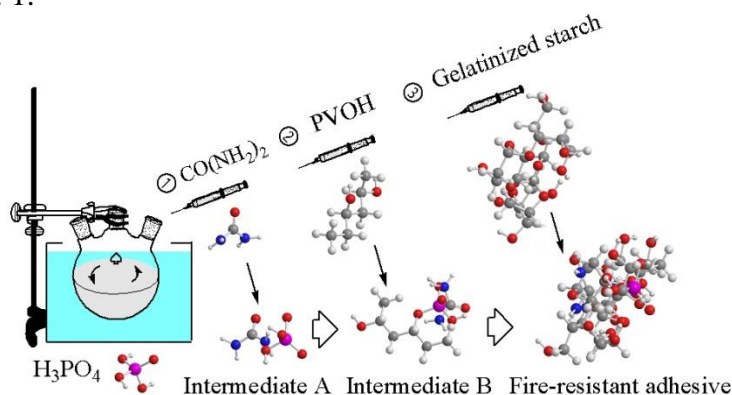

Fig. 1. Schematic illustration of the preparation of fire-resistant adhesive

Preparation of ultra-low density fibreboard

ULDFs were manufactured according to the principles of the liquid frothing method in a demonstration line as described by Xie *et al*. (2011), with a target bulk density of 55 kg/m^3. When liquid froths, a 3D-fiber network can be obtained by the "extruding" effect of bubble. The fibres form "arches" around bubbles, resulting in the orientation and bonding of fibre within the foam structure (Xie *et al*. 2008).

The detailed preparation process of the panels is presented in Fig. 2. The specific amount of absolute dried pulp was set at 46 g. The amount of fire-resistant adhesive was set at five levels, consisting of 0, 100, 200, 300, and 400 mL, respectively. The total volume of water and fire-resistant adhesive was 1000 mL. The mixture solvent was transferred into an open foaming tank attached to a pulp disintegrator (AB Lorentzen & Wettre, Sweden)

at ambient temperature for 5 min. After foaming with sodium dodecylbenzene sulfonate surfactant (10% of concentration, 50 mL, foaming agent), the pH value of the solution was controlled at 6 to 7 by adding sodium silicate solution of 33 wt%. Additionally, 50 mL of alkyl ketene dimer (AKD) water-repellent was added before the resultant mixture was quickly foamed to obtain ULDF, which was then transferred into a cubic mould (200 mm $\times$ 20 mm $\times$ 5 mm, $L \times W \times H$). Finally, the resulting fibre-based foam was kept in a drying oven at 103 °C until reaching a constant moisture content.

Fig. 2. Preparation process for ultra-low density fibreboard

Characterization

Limiting oxygen index (LOI)

The limiting oxygen index (LOI: Standard Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-like Combustion of Plastics) was measured using a HC-2 oxygen index meter (Jiangning Factory, China) at room temperature. The specimens used for the test were cut into dimensions of 120 mm $\times$ 10 mm $\times$ 10 mm according to GB/T 2406.2 (2009). All reported results are the average of 10 replications.

Cone calorimeter test (CONE)

Cone calorimeter apparatuses (FTT Company, UK) were used to obtain different combustion parameters under an incident heat flux of 50 kW/m^2 (corresponding to 780 °C) according to the ISO 5660-1 (2002) procedure. The samples of untreated ULDF and treated ULDF (300 mL fire-resistant adhesives) were cut into sizes of 100 mm $\times$ 100 mm $\times$ 30 mm for the testing.

Scanning electron microscopy (SEM) analysis

Scanning electron microscopy (SEM) studies were performed on the char residue after the cone calorimeter combustion test using a Nova Nano SEM 230 (FEI Company) without gold sputtering. Char samples were examined at magnifications of 500 and 20,000 times.

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Fourier transform infrared (FTIR) spectroscopy analysis

Fourier transform infrared spectroscopy (FTIR) spectra were recorded on a Nicolet 380 spectrometer (Thermo Fisher Scientific Inc., Waltham, MA, USA) for the characterization of fire-resistant adhesives and ULDFs using a compressed disk technique with the ratio of sample to KBr at 1:100 by mass. All of the samples except for the original one were placed in a muffle furnace until they reached temperatures of 200, 300, 400, 500, or 600 °C.

Thermal analysis

Thermal analyses of fire-resistant adhesives and ULDFs (8 ± 1 mg) were conducted using a Netzsch STA449F3 thermo-analyser instrument (Netzsch Company, Germany). The thermograms were obtained between 25 and 600 °C at a heating rate of 10 °C/min in a nitrogen atmosphere (at a flow rate of 10 mL/min). The temperature is reproducible to ±1 °C and ± 0.2% mass.

RESULTS AND DISCUSSION

Limiting Oxygen Index (LOI) Analysis

The limiting oxygen index (LOI) is commonly used as a quantitative analytical method to measure the flammability of a material. Figure 3 shows the LOI values of ULDFs with various amounts of fire-resistant adhesive.
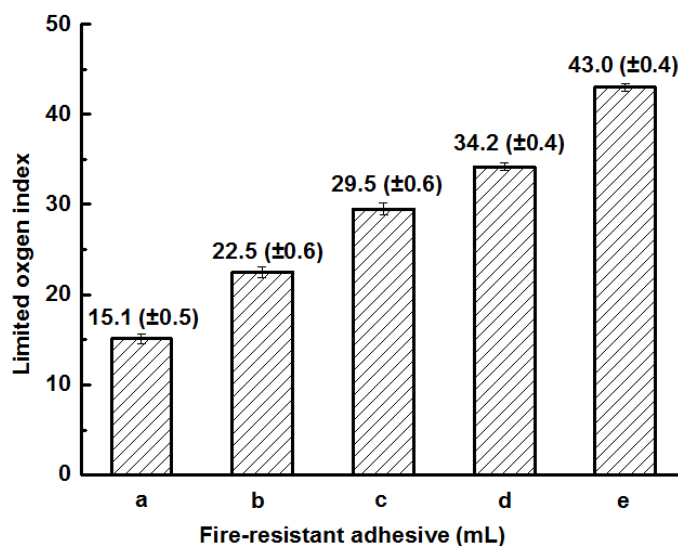

Fig. 3. LOI of (a) untreated ULDF and ULDFs treated with (b) 100 mL, (c) 200 mL, (d) 300 mL, or (e) 400 mL of fire-resistant adhesive

As can be seen in Fig. 3, the LOI of ULDFs could be improved significantly using a specific loading of fire-resistant adhesive. The untreated ULDF (specimens without fire-resistant adhesive) was an easily flammable polymeric material with the lowest LOI value of 15, while the LOI of the panels containing 100 mL of fire-resistant adhesive increased to 22.5. The LOI value of ULDF can reach up to 43, especially when the content of fire-resistant adhesive in ULDF reaches 400 mL. That is to say, the LOI values of ULDFs increased dramatically with the concentration of fire-resistant adhesive in the range of 0 to 400 mL. Generally speaking, an LOI value of at least 32 is necessary for a realistic degree

of flame retardance for building materials, and cost performance, as well as compatibility with fibres, also needs to be taken into consideration before industrialization (Chapple and Anandjiwala 2010). As such, ULDF containing 300 mL of fire-resistant adhesive (LOI of 34.2) is suitable for commercial use.

Fire Resistance of ULDFs Determined by Cone Calorimeter (CONE)

The flammability properties of the previous materials (0 and 300 mL) were further analysed at the bench-scale with a cone calorimeter (Fig. 4). A cone calorimeter is one of the most used fire-like methods for studying the flammability properties of materials. Particularly, the heat release rate (HRR) and peak of HRR (peak-HRR) have been found to be the most important parameters for evaluating combustion behaviour. The heat release rate (HRR) curves are shown in Fig.4, and the corresponding parameters from cone calorimetry are listed in Table 1.
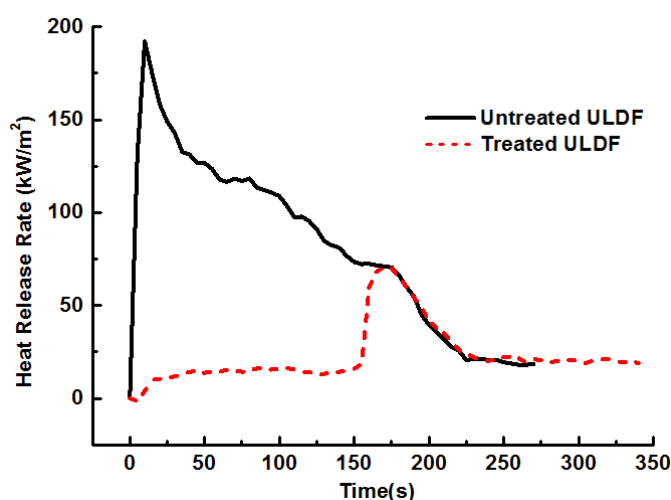

Fig. 4. Heat release rate (HRR) curves of the specimens at 30 mm thickness from untreated (specimens without fire-resistant adhesive) and treated ULDF (at a 300 mL level of fire-resistant adhesive) at 50 k/m^2

Table 1. Parameters Derived from Cone Calorimeter Test (50 kW/m^2)

Samples	Peak-HRR (kW/m^2)	TIP (s)	TTI (s)	TTF (s)	THR (MJ/m^2)	TSR (m^2/m^2)	Residual mass (%)
Untreated ULDF	192	10	0	273	22	275	2.34
Treated ULDF	70	175	10	340	6	11	46.92
Liu[1]	75	15	3	570	589	24	34.12
Niu[2]	5	10	1	250	10	18	30.45
Chen[3]	101	15	2	336	15	33	34.26
1) ULDF with multiplex flame retardant including N, P, Si and halogen (Liu *et al.* 2014).							
2) ULDF with 500 mL Si-Al compounds and chlorinated paraffins (Niu *et al.* 2014).							
3) ULDF with 500 mL modified Si-Al compounds and chlorinated paraffins (Chen *et al.* 2015).							

Note: TTP, TTI, and TTF represented time to peak, time to ignition, and time to flame out, respectively.

Both HRR curves had a sharp peak during cone combustion. However, it is noticeable that the HRR profiles of untreated ULDF occurred across a broader time range (0 to 230 s) and always with a higher HRR value than that of the treated one (150 to

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230 s). Furthermore, burning occurred immediately after ignition with a piercing peak (a maximum of approximately 192 kW/m^2 after 10 s), while the latter showed different combustion behaviour, with a much lower peak-HRR (70 kW/m^2 after 175 s) and a longer ignition time. The significant reduction in both HRR and peak-HRR indicates that 300 mL of fire-resistant adhesive in ULDF does play an active role in restricting the burning of ULDFs. The reason for this may be as follows: when the treated ULDF is exposed to fire, it forms a compact and intumescent charred layer on the surface of the fibre. This charred layer provides an efficient shield and insulation which can prohibit the underlying material from coming in contact with heat, oxygen, and fire directly. Thus, the structure and formation of the char layer were significant factors for flame retardancy on the treated ULDF, which is discussed in detail below.

Table 1 lists the times to ignition (TTIs) for those two samples and also confirms the expected reductions in TTI for corresponding materials. Specifically, the rapid TTI of the untreated ULDF was attributed to the inflammable plant fibres. It was also concluded that the low density of ULDFs affected their own ignition process, and the space truss structure induced the fibre-based materials to easily ignite with only one peak profile. This was definitely different from wood, as wood generally holds two peak profiles (Devi and Maji 2012). On the other hand, the TTI of fire-resistant adhesive-treated ULDF was considerably prolonged to 10 s, while previous studies showed a much more rapid ignition of the ULDF at 3 s (Liu *et al*. 2014), 1 s (Niu *et al*. 2014), and 2 s (Chen *et al*. 2015). The increased TTI indicated that the fire-resistant adhesive can effectively prevent the treated ULDF from ignition.

The formulation involving fire-resistant adhesive presented a different scenario, which reduced the THR by 16 MJ/m^2, and their main contributions to the THR was 6 MJ/m^2 in total. Moreover, significant reduction in TSR (up to 264 m^2/m^2) was observed with a comparatively higher charring yield than expected. The mass residue significantly increased by 44.58%, from 2.34% to 46.92%. The substantial increase of mass residue and considerable delay in the duration of decomposition demonstrated that fire-resistant adhesive plays an important role in char formation or intumescence of ULDF composites. This was also consistent with the LOI analysis discussed above.

However, employment of fire-resistant adhesives did not play an important role in time to flameout (TTF). TTF of the treated ULDF was delayed by 67 s, when compared to the untreated ULDF. It was also comparatively lower to the recently research (Liu *et al*. 2014; Chen *et al*. 2015), but higher than the research provided by Niu (Niu *et al*. 2014). This may indicate that fire-resistant adhesive could not inhibit the smouldering combustion of ULDF.

Morphology of ULDFs Residues after CONE Combustion

The appearance of untreated and treated ULDFs after cone burning is presented in Fig. 5. The untreated ULDF had large volume shrinkage with totally grey ash, while the treated ULDF formed a compact char layer with little ash. This suggested that fire-resistant adhesives have a great char-forming effect on ULDFs.

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Fig. 5. Macro-morphology of untreated (left) and treated (right) ULDFs viewed using a common digital camera

To further investigate how the structure of intumescent charred layers affected by the fire-resistant adhesives, morphologies of the charred layers obtained after the cone test were examined using SEM, and are shown in Fig. 6.
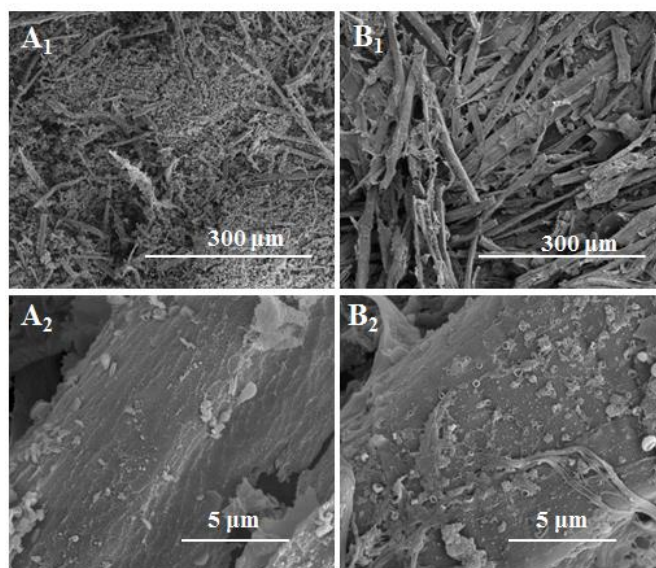

Fig. 6. SEM images of residue chars at 50 kW/m^2: A_1 and A_2 are untreated ULDF; B_1 and B_2 are treated ULDF.

Figure 6 (A_1 and A_2) corresponds to a ULDF without fire-resistant adhesive and shows very fragmented char, which was heterogeneous and lacking a complete structure. However, Fig. 6 (B_1 and B_2) depicts a ULDF with fire-resistant adhesive as a flame retardant and show a more compact and homogeneous structure of the charring layer. There were some bubbles but no cracks and gas holes in the surface. The improved structure suggests that fire-resistant adhesives have a large influence on the fire behaviour of ULDFs in binding fibres to a spatial structure, and providing a good barrier for the transfer of heat, mass, and flammable gases during a fire. Such protective barriers successfully inhibit oxygen diffusion and the escape of flammable volatiles, and thermally shield the underlying material against fire. To cut off oxygen from the degraded volatiles more efficiently, the outer surface of the charred layer should be compact enough to prevent the penetration of gases (Marolewski and Weil 1995). In other words, fire-resistant adhesive may play a very important role in catalysing the polyphosphate or the urea to crosslink with

ULDF during combustion in order to form an expansion charred layer with a close outer surface, which is of great significance in protecting the underlying materials from further pyrolysis and burning.

The improvement of performance of fire-resistant adhesive relative to the overall fire resistance of ULDF suggested that it may have influence on the internal structure of ULDF. Therefore, ULDF without fire-resistant adhesive and with 300 mL of fire-resistant adhesive were further analysed to show the changes on the structure and thermal stability of ULDF.

Chemical Bonding between Fire-Resistant Adhesive and ULDF

To check the chemical reaction between fire-resistant adhesive and ULDFs, FTIR spectroscopy was performed. Figure 7 shows the FTIR spectra of fire-resistant adhesive, untreated ULDF, and treated ULDF (with the amount of fire-resistant adhesive at 300 mL).
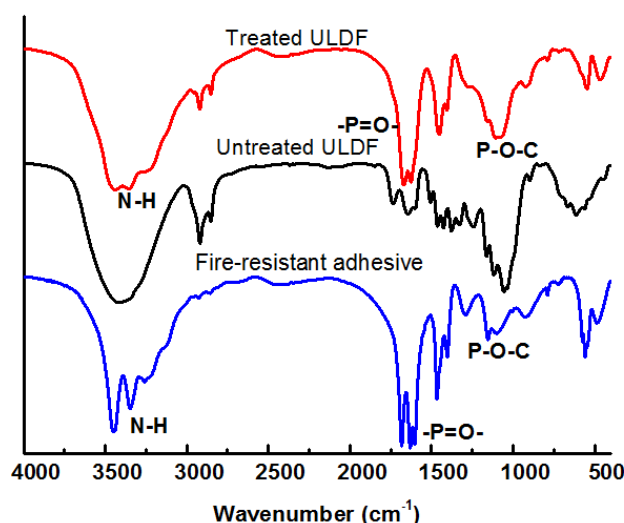

Fig. 7. FTIR profiles of fire-resistant adhesive, untreated and treated ULDFs

The FTIR spectrum of the control specimens (Fig.7) showed typical characteristics of plant fibre. The absorbance peak between 3700 and 3050 cm^{-1}, with a maximum near 3340 cm^{-1}, was attributed to the hydroxyl group of carbohydrates in fibres. The peaks at 1638 and 1272 cm^{-1} were the characteristic peaks of lignin, and the peaks at 1065, 893, and 667 cm^{-1} resulted from cellulose. FTIR spectra of fire-resistant adhesive showed characteristic bands of N-H stretching vibrations at 3500 to 3400 cm^{-1}, and its N-H bend vibrations at 1650 to 1580 cm^{-1}. The peak corresponding to the -P=O- stretching vibration was present at 1670 cm^{-1}. The presence of the P-O-C was detected at 1056 cm^{-1}. Additionally, the strong band at 1168 cm^{-1} was assigned to P-O-C and C=O groups (Schmitt and Flemming 1998). As expected, the N-H, -P=O-, and P-O-C free group bands can be found in the treated ULDF with peaks at 3500, 1670, and 1168 cm^{-1}, respectively. This was due to the fact that the hydroxyl groups were able to react with active urea linked to phosphate groups, which act as a fire retardant in the ULDF discussed above. A hypothesis reaction model between fire-resistant adhesive and ULDF is shown in Fig. 8.
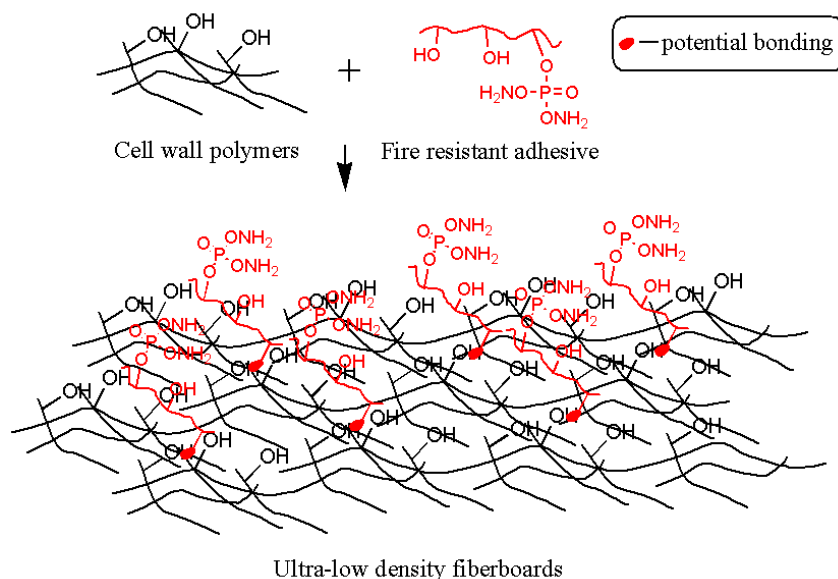
Fig. 8. Potential reaction model for fire-resistant adhesive and ULDF

Based on the FTIR analysis, the enhanced fire behaviours of ULDF may be attributed to three aspects. Firstly, -OH groups in both plant fibres and fire-resistant adhesives facilitate the physical bonding of ULDF. The hydrogen bonds and van der Waals' forces played a positive role in the binding of fire-resistant adhesive to plant fibre. Thus, the functional groups such as -P=O- and -P-O-C- connecting urea could be arranged outward, and may actively act as a fire retardant when undergoing combustion, as mentioned above. More importantly, the esterification crosslinking reaction between cellulose and fire-resistant adhesive, or cellulose itself, may have resulted in a compact coating that wrapped the fibres. Connecting is also consolidated as the water evaporates during the drying process (Neimo and Yhdistys 1999). This coating is especially important, as it prevents ULDF from burning during the CONE test. Also, as a binder, fire-resistant adhesive may fill in the voids between space truss structures, increase the bonding of disorderly fibres, or may even leach into the internal portions of the cell wall. In other words, the fire-resistant adhesive on the surface, or even in the fibres, may affect pyrolysis processes such as the LOI index, HRR, and TSR.

Thermal Pyrolysis Analysis of ULDFs

Thermogravimetric analysis (TGA) was used to investigate the effect of fire-resistant adhesive on the thermal stability of ULDFs. TG and DTG (differential thermal analysis) curves of fire-resistant adhesive, untreated, and treated ULDFs in nitrogen atmospheres are shown Fig. 9a and b, respectively.
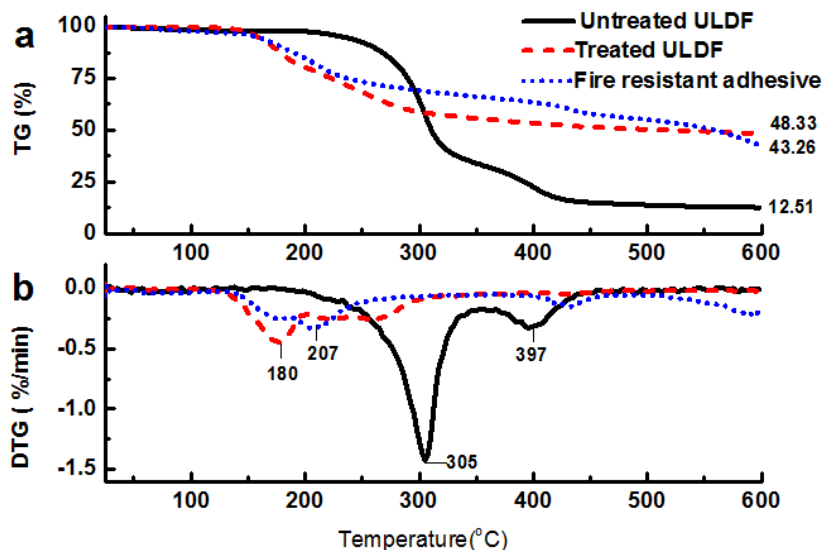
Fig. 9. (a) TG and (b) DTG curve of fire-resistant adhesive, untreated, and treated ULDFs

As shown in Fig. 9, TG curves of untreated and treated ULDFs were totally different shapes. The untreated ULDF underwent three decomposition steps while the treated ULDF experienced two. Both of the initial decompositions were attributed to water evaporation. Concerning the untreated ULDF, typical degradation stages corresponded to those of hemicellulose (approximately 200 to 300 °C) and cellulose (approximately 300 to 400 °C). Lignin degradation appears as a shoulder at the end of the sample degradation (approximately 400 to 600 °C) (Sebio-Punal *et al.* 2012) . For the treated sample, TG curves in Fig. 9a clearly show that the degradation of ULDF during the first stage was accelerated by the presence of the fire-resistant adhesive. This acceleration may be due to the fact that the P-O-C bond is less stable (usually decomposes at 180 °C) than the usual C-C bond (Xing *et al.* 2010). Although the addition of fire-resistant adhesive leads to small initial thermal stability during the first step, it considerably changes the further thermal degradation pattern which takes place during the following step.

Increasing char formation is an important means of improving thermal stability and flammability performance of ULDFs. Figure 9a shows that the residue of untreated ULDF was 12.48 % at 600 °C, indicating that the decomposition of fibre was almost completed, leaving some inorganic materials behind. However, the fire-resistant adhesive in ULDF produced an increase in residual mass (increased 4-fold, to 48.32%). This fact may be explained by the fact that the fire-resistant adhesive in the sample could decompose to form poly-phosphoric acid, which catalyses the ester group to form char residue (Yunchu *et al.* 2000).

According to Fig. 9b, the DTG curves of all the tested specimens (Fig. 9b) show narrow peaks of high intensity and display a rapid degradation process between 180 and 430 °C. It is evident that the degradation process of the treated ULDF occurs on a broader temperature range and that the degradation rate was not as significant for the untreated ULDF. For example, the degradation rate (-1.5%/min) of the untreated ULDF reached a maximum at 305 °C. However, the treated ULDF indicated a much lower maximum degradation rate (-0.5%/min), and a lower temperature (177 °C). In comparison with untreated ULDF, the treated ULDF curve shape shifted towards a lower temperature (around 128°C), implying a catalytic action on cellulose and on hemicellulose degradation.

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On the other hand, the DTG curves of the untreated and treated ULDF were well superimposed after 400 °C, which is mainly attribute to the decomposition of lignin (Li 2003).

The FTIR spectra of untreated and treated ULDFs at different pyrolysis temperature points were employed to elucidate the details of the thermo-pyrolysis behaviour (Fig. 10.).
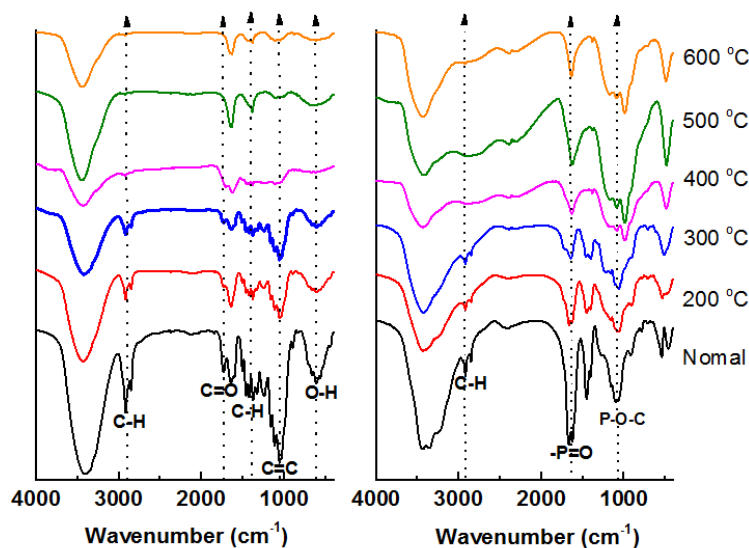

Fig. 10. FTIR spectra at various pyrolysis temperatures for untreated and treated ULDFs

The FTIR spectra of untreated ULDF at each temperature point indicates the degradation of fibre composition. The absorption intensity of untreated ULDF was weakened progressively when the pyrolysis temperatures ranged from 25 to 600 °C. For example, the bands in the 1733 and 1042 cm^{-1}, which were assigned to hemicellulose, disappeared above 300 °C. As the temperature rose to 400 °C, cellulose degraded completely. The FTIR spectra at 600 °C indicated the presence of lignin, with 3430 cm^{-1} assigned to benzene hydroxyl stretching vibration and 1600 cm^{-1} ascribed to a weakened benzene ring vibration. In contrast, the spectrum of treated ULDF at 400 °C suggested the formation of nitrogen-phosphate-carbon complexes, which is a great benefit in enhancing the stability of the char. As estimated by TGA, the UDLF treated with fire-resistant adhesive showed relatively more thermal stability than the untreated ULDF. Therefore, the adhesive may have acted as an acidic catalyst, accelerating the carbonization of the degradation products. This catalytic charring process at low temperatures is advantageous, and enhances the thermal stability and flame retardancy of the panels.

CONCLUSIONS

1. Fire-resistant adhesives can act as good fire barriers for ultra-low density fibreboard (ULDF).

2. Overall, increasing fire-resistant adhesive induces a significant increase in the limiting oxygen index (LOI). Cone calorimetry results were encouraging and showed an increase in the time to ignition and residual mass, and a decrease in peak-HRR, THR,

as well as TSR. Additionally, the condensed phase effect was evidenced by compact char observed using SEM.

3. Fourier transform infrared spectroscopy showed the presence of chemical bonding between fire-resistant adhesives and ULDFs. The results highlighted the importance of fire-resistant adhesive, and its role on the thermal degradation of ULDFs under nitrogen atmospheres. Therefore, these ULDFs will have the impressive potential for use in insulating materials.

ACKNOWLEDGMENTS

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