Investigation into Mechanical, Thermal, Flame-Retardant Properties of Wood Fiber Reinforced Ultra-High-Density Fiberboards

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The demand has grown in many fields for materials that are eco-friendly and sustainably developed. In this study, several formulations of novel wood fiber reinforced ultra-high-density fiberboards (UHDFs) using resol-type phenolic resin (RTPR) as binders were manufactured for application in decorative building facades. The influences of the various formulations on water resistance and the mechanical, thermal, and fire-resistant properties were systematically examined. All formulations of the UHDFs exhibited better water resistance, internal bonding, and fire resistance as the RTPR content and density increased. To better evaluate mechanical properties, the microstructure of the UHDFs was observed using scanning electron microscopy. After optimization of hot-pressing conditions, UHDFs with excellent mechanical properties of approximately 7.2 GPa, 85.9 MPa, and 5.4 MPa for bending modulus, bending strength, and internal bonding, respectively, were achieved. Good water and flame resistance were also achieved, which makes these materials competitive with other commercial products.

Keywords: Composites; Mechanical properties; Microstructure; Structure-property relations; Thermal properties

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INTRODUCTION

Currently, reducing carbon and promoting sustainable development are among the most pressing issues in high-energy-dissipation building projects, which are now making great efforts to develop green building materials. Ceramic tiles are an important material for use in decorative building facades. However, their production process creates a lot of dust and consumes a lot of electrical energy. Therefore, the authors proposed a novel, natural, fiber-reinforced biomass composite as an alternative to ceramic tiles for use in decorative facades, contributing to the sustainable development of green buildings.

Wood fibers, an environmentally friendly, natural fiber, have been widely used in indoor furniture (Saz-Orozco *et al.* 2014; Sommerhuber *et al.* 2016; Wang *et al.* 2016) and automobile interiors (Ann Gnpta *et al.* 2008; Gallo *et al.* 2013), but are not often used as structural materials in engineering due to their poor mechanical properties.

Conventionally, wood fiber reinforced composites are composed of wood fibers that have been pressed together under high temperature and pressure with a resin binder to form a homogeneous board. Studies on wood fiber reinforced composites have mainly focused on low-density fiberboard (LDF) (< 600 kg/m^3) (Chen *et al.* 2015a,b; Cai *et al.* 2016) and medium-density fiberboard (MDF) (600 kg/m^3 to 800 kg/m^3) (Sliseris *et al.*

2014; Taghiyari *et al.* 2015a,b; Liang *et al.* 2016; Taghiyari *et al.* 2016). The LDF is often referred to as insulation board because it is mainly used for thermal insulation in civil engineering. The MDF consists of 80% to 90% wood fibers and 10% to 20% resin. It is widely used to make various kinds of furniture such as shoe racks, dressing tables, and closets. It has an internal bonding strength of approximately 0.6 MPa to 1 MPa and a flexural strength of approximately 30 MPa to 40 MPa according to ISO/DIS 16895-2 (2008). To widen its application areas, significant effort has been made to improve the mechanical properties of MDF by adding reinforcing agents such as nano-wollastonite (Taghiyari *et al.* 2015a), wollastonite fibers (Taghiyari *et al.* 2016), and organo-silane derivatives (Taghiyari *et al.* 2015b). Moreover, some researchers have tried changing the types of adhesives (Sitz and Bajwa 2015; Arévalo and Peijs 2016; Wu *et al.* 2016). Sitz and Bajwa (2015) and Arévalo and Peijs (2016) prepared MDF using methylene diphenyl diisocyanate and the self-binding capability of cellulose, respectively.

However, these improvements are very limited and cannot be used in decorative facades due to its low mechanical properties according to JG/T 260 (2009). To promote the application of wood fiber reinforced composites in decorative building facades, a kind of biomass composite should be designed. Fiberboard that consists of wood fibers is a porous material on a micro-level (Lv *et al.* 2015a,b), which affects its interior mechanical properties. Therefore, the authors propose the fabrication of an ultra-high-density composite (> 1000 kg/m^3) with high pressure and high resin content (> 20% in total weight loss). Under high pressure, the vessels and lumens can be crushed. The resin can then fill the cavities of the vessels, lumens, and cracks in the cell walls, forming an ultra-high-density composite (UHDF) with fewer holes and excellent mechanical strength. Due to the ability to add colored pigments to the surface during curing, a variety of colors are possible. In addition to its use in furniture and flooring, such a material is expected to be an ideal candidate for replacing ceramic tiles in architectural decorative facades. As a novel composite, its properties are unknown. Therefore, it is necessary to systematically investigate its structure-property relationship.

EXPERIMENTAL

Materials

Resol-type phenolic resin (RTPR), a dark brown liquid, was supplied by Beijing Taier Technology Co. Ltd. (Beijing, China), with a viscosity (25 °C) of 30 mPa/s and solid content of 50%. Wood fibers (*Populus*, I-214, length <1 mm, 0.40 g/cm^3) were bought from the Chinese Academy of Forestry (Beijing, China). The wood-based panel press (DMP-30) and wood-based panel glue blend machine (QS-2) were supplied by Shanghai Wood-based Panel Machinery Co., Ltd. (Shanghai, China).

Methods

Preparation of ultra-high-density composites

The composites were manufactured using a hot press with a manually controlled hydraulic press system. Before pressing the fibers, the fibers were conditioned to a 10% to 12% moisture content by weight. Once the fibers had been sufficiently conditioned, the fibers were placed into a rotating drum (20L) with paddle mixers. The fibers were then sprayed using an atomizing air gun (2 MPa and the speed is 50 mL/min) with RTPR resin to act as a binder. After sufficient mixing (room temperature, 10 min), the fibers' moisture content was conditioned again, until a decrease to 3% to 4% by weight. The blended fibers were pressed at 155 °C for 9 min to produce 9 mm thick panels with a density of 1.05 g/cm^3. The contents of RTPR in total weight were 20%, 30%, and 40%.

Further, to investigate the effect of density on properties of UHDF, three series of UHDF with different densities (1.05 g/cm^3, 1.15 g/cm^3, 1.30 g/cm^3) were produced at 155 °C with 20% RTPR content.

Equations

The water absorption (*WA*) and thickness swelling (*TS*) properties were determined from the difference between specimen thickness and weight before and after immersing in water for 24 h according to ISO 16979:2003 and ISO 16983:2003, respectively.

The ratio of *WA* and *TS* were calculated by Eqs. (1) and (2), respectively,

$$WA\ (\%) = \frac{m_2 - m_1}{m_1} \times 100\% \tag{1}$$

$$TS(\%) = \frac{h_2 - h_1}{h_1} \times 100\% \tag{2}$$

where, m_2 and h_2 are the final weight (g) and thickness (mm) after immersing in water, respectively, and m_1 and h_1 are the initial weight (g) and thickness (mm) before immersing in water, respectively.

Test standards

The mechanical properties were characterized using a universal testing machine Zwick/Roell Z030 (Zwick/Roell, Radeberg, Germany). At least five specimens were tested for the average value.

For internal bonding strength, the samples (50 × 50 × 7 mm^3) were cut with a keyhole saw Bosh PST 900 PEL (Qingdao Sanmu Woodworking Machinery Co., Ltd., Qingdao, China). The test followed ISO 16984: 2003 with a load rate of 5 mm/min. The bending properties of composites (120 × 15 × 6 mm^3) were tested with a load rate of 10 mm/min according to ISO 16978:2003.

Scanning electron microscopy (SEM)

The microstructures of the composites was characterized *via* SEM. The samples were introduced in liquid nitrogen and fractured. The fracture surfaces were observed using a JEOL JM-6400 microscope operating at 40 kV with a gold sputtering (Mettler Toledo, Burladingen, Germany).

Thermogravimetric (TG) analysis

The TG was analyzed using a Mettler Toledo TGA/DSC1 TG (Netzsch, Bavaria, Germany). The samples were heated at 10 °C/min under a nitrogen atmosphere at a flow of 20 mL/min, and the temperature was from 30 °C to 700 °C.

Limiting oxygen index (LOI) test

The LOI tests were performed according to ASTM D2863 (2000) using a Stanton Redcraft FTA (Tarlin Scientific, London, the United Kingdom) at room temperature. The sample is 150 × 6.5 × 3 mm.

RESULTS AND DISCUSSION

WA and TS Analysis

Water absorption has an important influence on the long-term use of wood fiber reinforced composites. Thus, it is necessary to investigate the *WA* and *TS* of these composites. The *WA* and *TS* values as a function of every factor were plotted at different RTPR contents, and the results are illustrated in Fig. 1.

It was observed that the *WA* and *TS* values of UHDFs produced at 155 °C slightly decreased as RTPR content increased, with *WA* and *TS* decreasing from 11.5% to 8.9% and from 5.3% to 3.4%, respectively. This was due to the hydrophilic quality of wood fibers. As resin content increased, the hydrophilic hydroxyl content in the UHDF decreased. Thus, water permeation into the vessels was more difficult when the boards were immersed in water at room temperature, which resulted in lower *WA* and *TS* values.

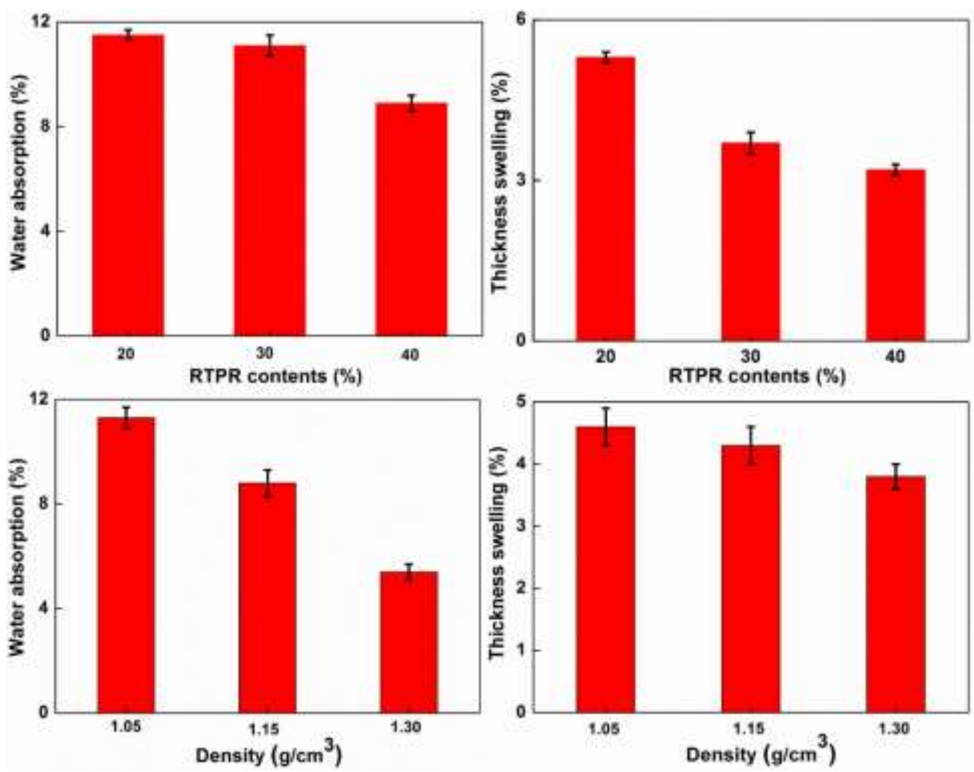

Fig. 1. *WA* and *TS* of UHDFs prepared with different RTPR contents and densities

To investigate the effect of density on water absorption, the authors examined the UHDFs produced at different densities, and the results are shown in Fig. 1. The figure shows that both *WA* and *TS* decreased as density increased. This can be explained as follows: With increased UHDF density, the internal structure became denser. Thus, more fibers were enveloped by the phenolic resin. This resulted in a more hydrophobic surface, such that water cannot permeate the panels, which contributed to water resistance. However, as the *WA* decreased the *TS* decreased. These low values of *WA* and *TS* are conducive to the use of UHDF in decorative facades.

Mechanical Property Analysis

Internal bonding strength

The effects of RTPR content on the internal bonding strength of UHDF were evaluated. Several RTPR contents (20%, 30%, and 40%) were studied for the boards with a density of 1.05 g/cm^3. The influences of these factors on the internal bonding properties of the UHDF are presented in Fig. 2.

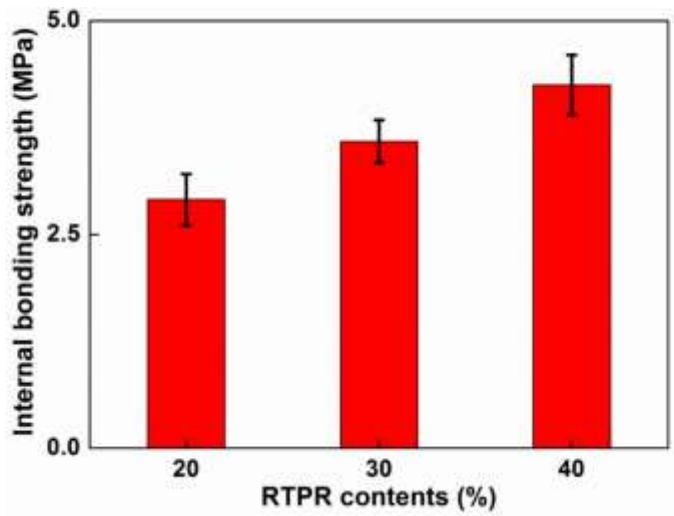

Fig. 2. The internal bonding strength of UHDFs with 1.05 g/cm^3

An increase in RTPR content led to a clear improvement in internal bonding strength (Fig. 2). Such an improvement in mechanical performance continued with resin contents of 40%, yielding a value of 4.08 MPa, which was higher than that of conventional wood reinforced fiberboard (ISO/DIS 16895-2 (2008)).

In an attempt to better understand the influence of resin content on the internal bonding properties of UHDFs, the microstructures of the internal surfaces of the boards were studied by SEM (Fig. 3). High-magnification micrographs (× 300) of the internal surfaces of samples produced using different RTPR contents at 155 °C (Fig. 3A, Fig. 3B, and Fig. 3C) showed obvious differences.

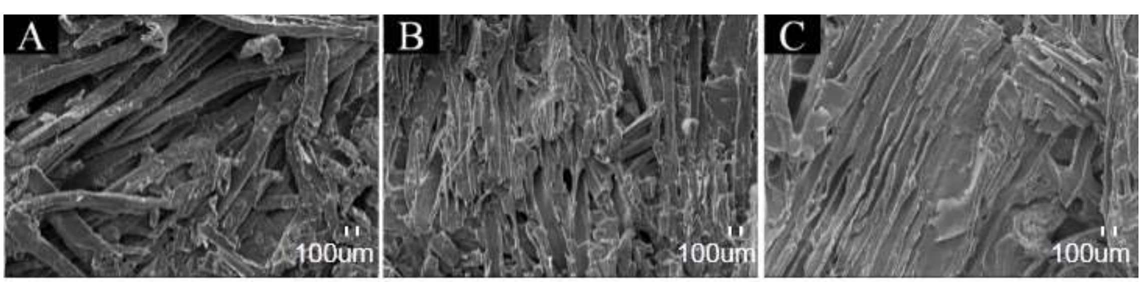

Fig. 3. High-magnification micrographs (×300) of UHDFs with a density of 1.05 g/cm^3: (A) 20% RTPR, (B) 30% RTPR, and (C) 40% RTPR

There were many visible holes in the internal structures of the samples produced with 20% RTPR content (Fig. 3A). However, the holes disappeared gradually with increased RTPR content. Especially for the samples produced with 40% resin content, the board showed very compact structures (Fig. 3C). Figure 3 shows that the wood fibers were well dispersed in the structure. The wood fibers and polymer matrix were entangled and formed a three-dimensional network structure, which resulted in physical bonding of the two components that formed a rigid framework and improved the mechanical performance of the boards. Meanwhile, these disappearing holes facilitated effective linkages between the wood fiber and the phenolic resin, thus yielding better mechanical properties.

To further illustrate the influence of the RTPR content on the mechanical properties, more details in the micrographs should be noted. In the samples produced with 20% RTPR content (Fig. 3A), the wood fibers were well dispersed in the inner structures. The broken fibers mainly occurred at the interface, with most of the fibers retaining their original structures. However, the broken fibers showed pronounced changes with increased resin content. They broke not only at the fiber–matrix interface but also along the axial and radial directions of the wood fiber, especially for samples with 40% RTPR content (Fig. 3C). Additionally, many fibers were broken in the longitudinal direction. It was inferred that stronger interfacial adhesion occurred between the fibers and the matrix as RTPR content increased, which allowed for better fiber–matrix interactions and explained the gradual increase in the internal bonding strength of the UHDFs.

To investigate the effect of density on the internal bonding strength of the UHDFs, samples with different densities (1.05 g/cm^3, 1.15 g/cm^3, 1.30 g/cm^3) were produced, and the results are shown in Fig. 4. As density increased, internal bonding strength increased from 2.9 MPa to 6.7 MPa. This may have been due to an increase in the number of linkages between the fibers and the matrix, which ensured an efficient and timely stress load transfer from the fibers to the polymer matrix.

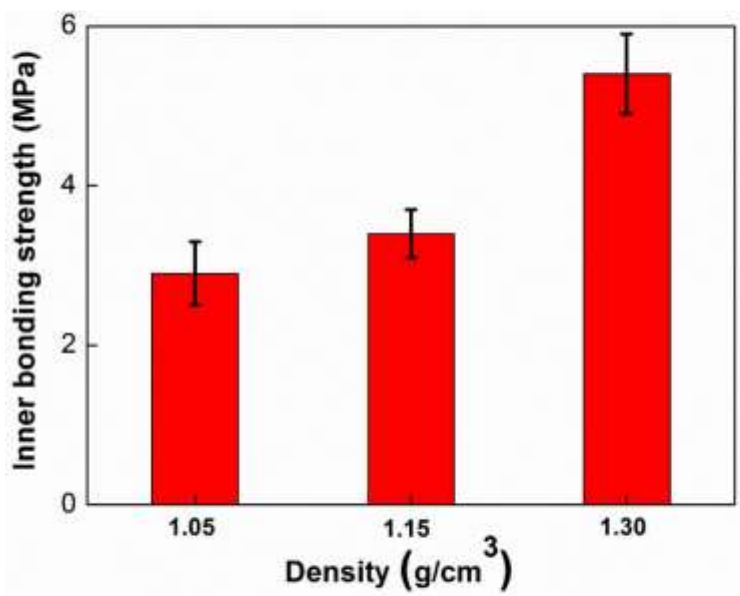

Fig. 4. The internal bonding strength of UHDFs with different densities

To assess and quantify the improved inner bonding strength, the porosity of the samples was measured using Eq. 3 (Arévalo and Peijs 2016),

$$\text{Porosity (\%)} = \frac{V_0 - V}{V_0} \times 100 \quad (3)$$

where V_0 and V are the volumes of the blended fibers and the UHDFs (cm^3), respectively.

Notably, $V_0 = V_{wf} + V_{\text{phenolic resin}}$. However, for V_0, $V_{\text{phenolic resin}}$ was too small to be negligible, thus Eq. 3 can be derived to get Eq. 4,

$$\text{Porosity (\%)} = \frac{V_{wf} - V}{V_{wf}} \times 100\% = \frac{\rho_{wf} - \rho}{\rho_{wf}} \times 100 \quad (4)$$

where ρ_{wf} and ρ are the densities of WF and UHDF (g/cm^3), respectively.

The density of dried WF was determined by their cell walls, so the authors used the density of the cell wall to simulate that of the WF to calculate the porosity. The density values ranged from 1.497 g/cm^3 to 1.517 g/cm^3 for *Populus* (Kellogg and Wangaard 1969), so the authors used 1.50 g/cm^3 as the average cell wall density. According to Eq. 4, it was inferred that a strong increase in density led to a decrease in porosity. As expected, the porosity of UHDF with a density of 1.30 g/cm^3 is 13.33%, which was much lower than the corresponding values for UHDF with a density of 1.05 g/cm^3 (42.85%) and UHDF with a density of 1.15 g/cm^3 (23.33%). The internal bonding strength of UHDF can therefore be directly related to its density and inversely related to its porosity. Microvoids cause stress concentrations and are detrimental to mechanical properties. Therefore, as expected, denser materials exhibited better mechanical properties.

Further explanations are evident in the SEM microstructures of UHDFs shown in Fig. 5. According to the micrographs, the structures became more compact, which contributed to beneficial mechanical properties.

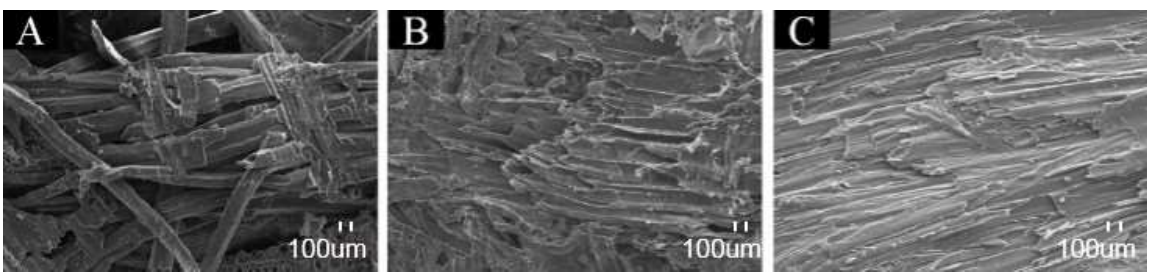

Fig. 5. The micrographic images of UHDFs with different densities: A): 1.05 g/cm^3, B): 1.15 g/cm^3, and C): 1.30 g/cm^3)

Bending strength

The bending strength is a very important index for composites; thus the effect of RTPR content on the mechanical properties of the fiberboard should be evaluated. The RTPR contents of 20%, 30%, and 40% were studied for the boards with a density of 1.05 g/cm^3. The influence of these factors on the bending properties of UHDFs are presented in Fig. 6. The results showed that an increase in RTPR content resulted in a decrease from 64.3 MPa to 58.6 MPa in the modulus of rupture (MOR). This observation conflicts with the results regarding the internal bonding strength. However, such a reduction in bending strength was reasonable with a resin content of 40%, which yielded the lowest value of 58.6 MPa.

Generally, the mechanical properties of composites are determined by several factors such as the nature of the reinforcement fiber, fiber aspect ratio, fiber–matrix interfacial adhesion, and the fiber orientation of the composites. With ultra-high-density composites, the wood fiber content decreased as resin content increased, as shown in Fig.7. When the UHDF samples were subjected to the three-point flexural test, samples with a high RTPR content resulted in fewer fibers for stress load transfer and less energy for fiber pull-out. This decrease in energy can destroy the boards. However, the modulus of elasticity (MOE) remained relatively stable at approximately 5 GPa to 6 GPa (Fig. 6). This result indicated that the RTPR content had little influence on the MOE.

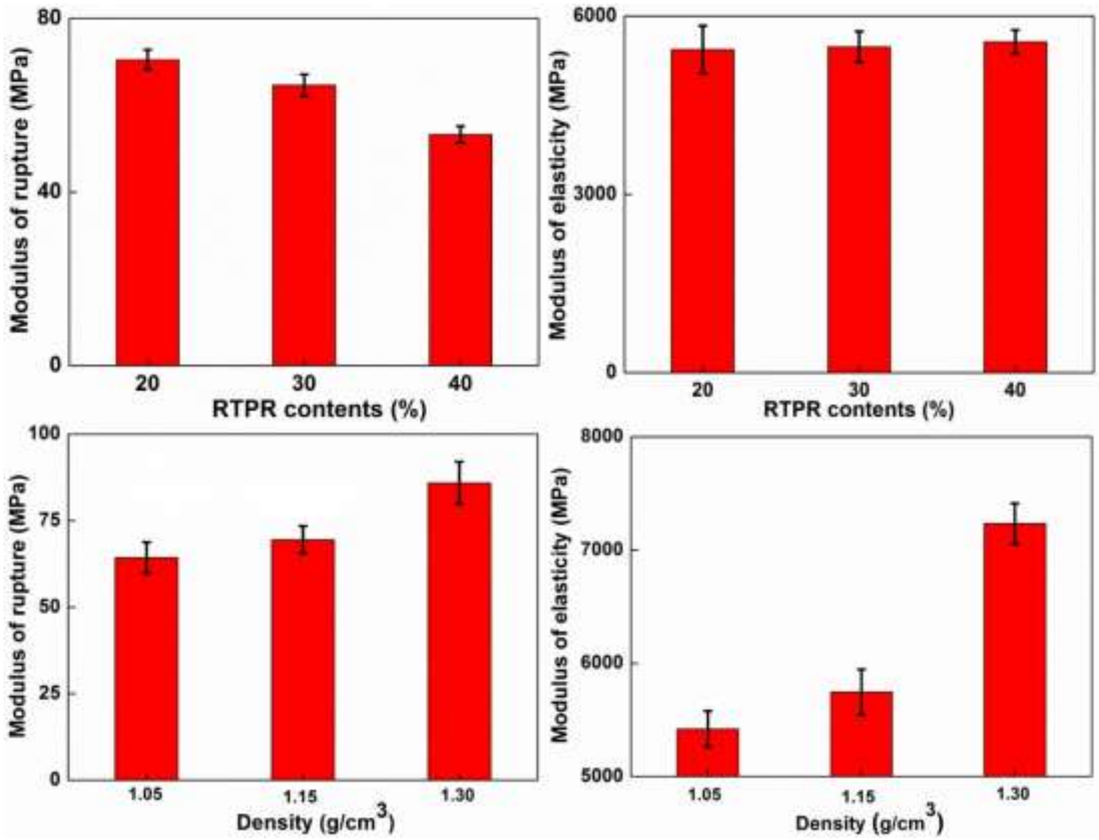

Fig. 6. The bending strength of UHDFs with different RTPR contents and densities

Fig. 7. The simulated images of UHDFs (low RTPR content (A); high RTPR content (B))

To assess whether higher density would improve flexural properties, two samples were prepared with higher densities of 1.15 g/cm^3 and 1.30 g/cm^3, respectively. As expected, this increase in density yielded further improvement of the MOR and MOE values from 64.3 MPa (1.05 g/cm^3) to 85.9 MPa (1.30 g/cm^3) and from 5420 MPa (1.05 g/cm^3) to 7230 MPa (1.30 g/cm^3), respectively (Fig. 6). According to Yildiz's (Yildiz *et al.* 2005) report, the MOR and MOE of solid wood (*Populus*, I-214) are 32.6MPa and 3.89 GPa, which are lower those of UHDFs, further, Li (Li *et al.* 2017) had also reported that composite consisted of wood fiber and polypropylene also showed low mechanical properties. The MOR and MOE are 31.8 MPa and 2.52 GPa. So we can infer that this composite exhibit good mechanical properties. The bending strength of UHDF with a density of 1.30 g/cm^3 met the requirements of JG/T 260-2009 (2009) for curtain walls in buildings. In this standards, it stipulates that the density, the bending strength and thickness swelling must more than 1.30 g/cm^3, 80MPa and 5%, respectively.

According to the porous properties described above, the number of voids decreased with increased fiberboard density, which led to a lower stress concentration in the fiberboards and contributed to improved mechanical properties. Moreover, this improvement was explained by the microstructures in the panels shown in Fig. 5. The fracture surfaces shown in Fig. 5 depicted an increasingly compact structure with increased density, especially for the UHDF sample with a density of 1.30 g/cm^3. This dense structure contributed to the transfer of stress and improvement of the flexural strength.

TG Analysis

Thermal stability is also an important index for boards used with daily frequency. Figure 8 shows the TGA and derivative thermogravimetric (DTG) profiles for the WF, RTPR cured at 155 °C (C-PR), and composites under different conditions.

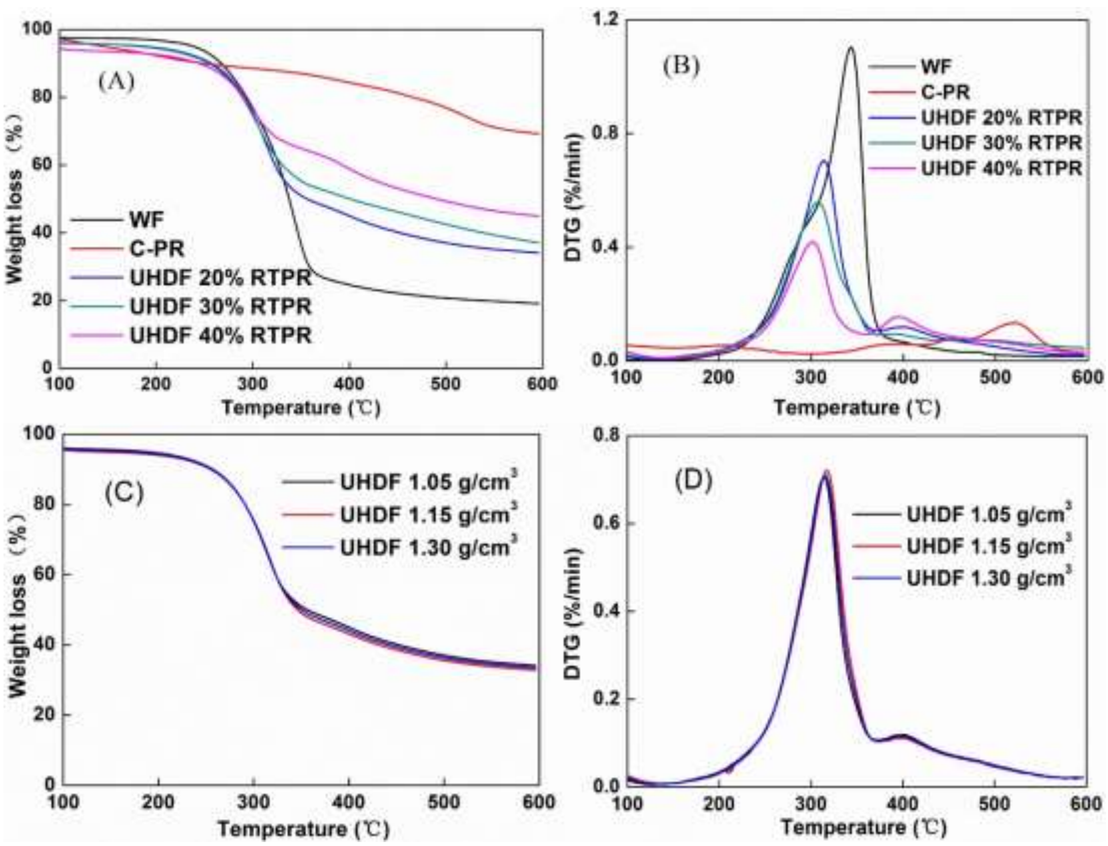

Fig. 8. TGA and DTG curves of WF, phenolic resin, and UHDFs

The WF (Fig. 8A) shows only one main step of thermal degradation. Further, the initial decomposition peak ($T_{10\%}$) occurred at 267.3 °C. In the step between 250.2 °C and 380.3 °C, polymer chains decomposed (cellulose, hemicellulose, lignin, *etc.*) and water and formaldehyde were formed and released. The cellulose fibers mainly degraded between 250 °C and 400 °C, where the cellulose decomposed through the formation of levoglucosan (Kim *et al.* 2010; Hu *et al.* 2012). Further, some weight loss occurred at temperatures lower than 100 °C, which potentially corresponded to moisture losses in the WF. The char at 600 °C was 19.4% levoglucosan, which may have been attributed to the polynuclear aromatic structures.

For C-PR, a gradual weight loss was observed. This may have been due to two reasons: first, RTPR is a mixture, as the solution contains many small molecules (water, phenol, formaldehyde, *etc*.). Thus, there should be some small molecules in C-PR after curing at high temperatures; Second, aldehydic and phenolic compounds degraded gradually from C-PR during the thermal process. However, the char amount at 600 °C was 69.0%, which indicated that phenolic resin had good char-forming ability.

The decomposition patterns of the UHDFs were similar to those of WF and C-PR. The initial degradation temperature ($T_{10\%}$) of the UHDFs was over 250 °C, which indicated that these fiberboards exhibited good thermal stability and are suitable for daily use. Moreover, $T_{10\%}$ of the UHDFs decreased with increased RTPR content due to the small molecules in the cured phenolic resin. All of the studied fiberboards showed two thermal degradation steps, one at 250.0 °C to 360.0 °C (I) and the other at 360.0 °C to 440.0 °C (II) (Fig. 8B). In step (I), the WF exhibited a high rate of weight loss. However, the rate of weight loss for C-PR was near 0. Thus, step (I) likely contributed to the degradation of the major components in the WF (cellulose, hemicellulose, and lignin). As RTPR content increased, the maximum weight loss decreased, which was attributed to the different ratios of phenolic resin with good thermal stability in the fiberboards. The opposite effect was observed in step (II) in that the maximum peak of weight loss increased as RTPR content increased (Fig. 8B). This was due to the degradation of phenolic resin in the temperature range used. As seen in Table 1, the char amounts at 600 °C increased with increased RTPR content.

To investigate the effects of density on the thermal properties of the fiberboard, a series of UHDFs were characterized and their relative data are shown in Table 1, Fig. 8C, and Fig. 8D. Moreover, UHDFs with different densities had similar thermal degradation processes. Therefore, it can be concluded that density had no effect on the thermal properties of the UHDFs.

Table 1. Thermal Properties of WF, C-PR, and UHDF

Samples Name	$T_{10\%}$[a] (°C)	T_{maxs1}[b] (°C)	T_{maxs2}[c] (°C)	Char at 600 °C (%)
WF	267.3	343.3	-	19.1
C-PR	248.7	521.3	-	69.2
UHDF 20% RTPR[d]	257.0	314.0	400.3	34.1
UHDF 20% RTPR	254.8	307.6	398.3	37.0
UHDF 20% RTPR	244.3	302.1	394.2	44.9
UHDF 1.05 g/cm^3	257.0	314.0	400.3	34.1
UHDF 1.15 g/cm^3	255.0	316.9	399.3	32.8
UHDF 1.30 g/cm^3	256.0	310.9	396.8	33.4

a: the temperature of the samples with the weight loss of 10%; b and c: the decomposition temperature of the maximum weight loss rate in the first and second step, respectively; and d: UHDF20% RTPR and UHDF1.05 g/cm^3 are the same sample in this paper

Flame Retardant Behavior

To quantitatively analyze the flammability of the UHDFs, LOI tests were performed. The LOI values of the boards produced with varying levels of RTPR content are presented in Fig. 9. The LOI values with higher concentrations of resin showed better results than those of boards with low resin content. Further, with a RTPR content of 40%, an LOI value of 28.6 was observed. This was attributed to the fire-resistant properties of phenolic resin. Increasing the RTPR content by adding more flame-retardant to the boards contributed to good LOI results. Meanwhile, the effect of the boards' densities with 20% RTPR content on the LOI values was also examined. As shown in Fig. 9, the LOI values increased from 26.1 (1.05 g/cm^3) to 30.2 (1.30 g/cm^3) with increased density. This may have been due to the compact inner structures hindering flame propagation, thus an improved flame-retardant quality. This useful flame-retardant behavior promotes the wider application of UHDFs.

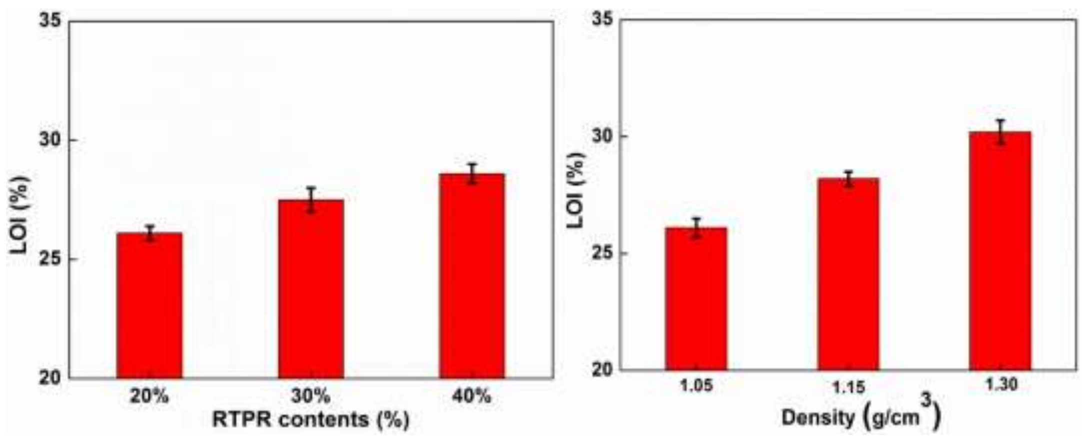

Fig. 9. The values of LOI test

CONCLUSIONS

1. This study showed the potential of UHDFs prepared with high pressure, density, and RTPR content for use as environmentally friendly panels composed of renewable resources and biodegradable wood fibers.
2. Systematic analysis revealed that the optimal conditions for achieving good mechanical properties were a RTPR content of 40% and a UHDF density of 1.30 g/cm^3.
3. The mechanical tests showed that bending and internal bonding properties of the UHDFs increased with increased UHDF density. The internal bonding strength also improved with increased RTPR content, although the bending strength was negatively affected.
4. The thermal and fire-retardant performances of the UHDFs were investigated using TGA and LOI. The results indicated that LOI had a close relationship with the RTPR content and the density of the UHDF, but thermal stability was independent of density.
5. The strong mechanical properties of the UHDFs and their excellent water resistance and fire resistance make them applicable in architectural decoration.

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