Enhanced Mechanical Strength of Polyethylene-Based Lignocellulosic-Plastic Composites by Cellulose Fibers

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Wood flour (WF) of poplar, acid hydrolysis residue (AHR) of corn cob from xylose production, and cellulose fibers (CF) from bleached eucalyptus pulp were compared as functional fillers of lignocellulosic-plastic composites (LPC) in terms of tensile strength and thermal stability. WF showed a negative effect on tensile strength of LPC. AHR-filled LPC at 10% of filling level exhibited an improvement by 8.9%, whereas higher filling level led to a decrease of tensile strength due to poor interfacial compatibility, as revealed by SEM analysis. Remarkably, tensile strength achieved a maximum of 25.8 MPa for CF-filled LPC at 2.5% of filling level, which was an approximately 76.7% improvement compared to the control. Dependence of LPC thermal stability on chemical compositions of fillers was revealed. WF-filled LPC showed a lower onset decomposition temperature compared to the control due to the presence of xylan, while thermal stability of AHR-filled LPC was enhanced due to the presence of cross-linked phenolic polymer lignin.

Keywords: Cellulose fibrils; Tensile strength; Elongation; Lignocellulose-plastic composites

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INTRODUCTION

Recent interest in building materials, gardens, packaging as well as the public's growing demand for environmentally friendly products have sparked the development of green composite materials (Pickering *et al.* 2016). Lignocellulose-plastics composite (LPC) is an environmentally progressive way of combining recycled plastics and lignocellulose. Lignocellulose is an easily available and cheap natural resource that can be added to commodity matrices in large quantities, offering economically advantageous solutions (Gurunathan *et al.* 2015). The shape stability of these materials tends to be greater than that of traditional wood products due to the additional plastic content (Fabiyi and McDonald 2010). The benefits of LPC, including its status as a raw material, widespread availability, low maintenance cost, moldability, excellent weatherability, and aesthetic appeal, have garnered interest in wood replacement applications for architectural purposes, the automotive industry, and furniture manufacturing (Wang *et al.* 2011).

Lignocellulose is chemically composed of cellulose, hemicellulose, and lignin. Cellulose fiber is commonly separated from plant cells *via* pulping and bleaching, and then used for papermaking. Celluloses are potential candidates for use as reinforcing fibers in thermoplastic materials (Hubbe *et al.* 2008, 2017). Compared with glass fibers, silica, and carbon black, the use of celluloses in composites has many advantages, such as low cost, low density, biodegradability, recyclability, and resistance to breakage during processing (Thakur and Thakur 2014). Ramesh *et al.* (2016) used the non-wood lignocellulosic fibre of kenaf (*Hibiscus cannabinus* L.) as a reinforcement filler in polymer composites. Ayrilmis *et al*. (2011) used rubberwood (*Hevea brasiliensis* Müll.Arg.) fibers in their manufacturing of wood plastic composite panels. Polypropylene was also reinforced with cellulosic fibers by Burgstaller *et al*. (2009), and showed a high elastic modulus and high tensile strength. Peltola *et al.* (2014) made a comparison of the effect of various wood fiber types in polylactic acid and polypropylene composites produced by melt processing. There have also been studies on the variations of mechanical properties of composites made of wood fibers–thermoplastic polymer as the result of exposure to white and brown-rot fungi (Ashori *et al.* 2013; Verhey *et al.* 2001). Filling level and characterizations of filler are important factors in LPC processing and property determination. Nourbakhsh *et al*. (2008) studied effects of filling levels from 10 wt.% to 40 wt.% on the mechanical properties of poplar/ polypropylene composites and found that the fiber loading levels of 30 wt.% and 40 wt.% at 190 °C provided adequate reinforcement. Valle *et al*. (2007) investigated the effect of wood content on the thermal behavior and molecular dynamics of polyvinyl chloride/wood composites. They showed that the increasing addition of wood flour caused a small but progressive improvement of the decomposition temperature of the composites, whereas the glass transition temperature remained practically unchanged. The effects of filling levels on flexural modulus (Jumadi *et al.* 2018), and water absorption ability (Feng *et al.* 2014) were also reported. Studies had showed that the aspect ratio of fillers had significant impact on mechanical properties of LCP, and lignocellulosic fillers in form of fiber with high aspect ratio had remarkable reinforcement performance (Ashori and Nourbakhsh 2010; Stark and Rowlands 2003).

Besides wood flour, agro-based fibers are viable alternative in commodity fiber-thermo-plastic composites as long as the right processing conditions are used. These renewable fibers have low densities and non-abrasive character, which permits a high volume of filing in the composite. Corncob, for example, has excellent specific properties and had been used as reinforcing fillers in plastic at 50wt% of filling level. However, it has been demonstrated that agro-residue starts to decompose at temperatures as low as 200 °C (Panthapulakkal and Sain 2007), which may be ascribed to the presence of hemicellulose. Different from cellulose, hemicelluloses are heteropolymers (matrix polysaccharides) with a random, amorphous structure; these characteristics may explain the low strength and poor thermal stability. These disadvantages may be overcome if hemicellulose can be removed prior to composite processing. Corncob is currently widely used as a raw material for xylose/xylitol production by acid hydrolysis of pentosan or for furfural production by dehydration of xylose (Mao *et al.* 2012; Zhang *et al.* 2017). Therefore, the acid hydrolysis residue (AHR) of corncob is mainly composed only by cellulose and lignin.

In the present study, AHR of corn cob was employed as lignocellulosic fillers to prepare polyethylene-based LPC. Focus was placed on mechanical properties and thermal stability of LCP. For comparison, LCP filled with wood flour (WF) and cellulose fibers (CF) from bleached pulp were also prepared and evaluated.

EXPERIMENTAL

Materials

Poplar WF was prepared from saw dust after size reduction process using a hammer mill. AHR is solid waste of xylose production from corn cob, and was provided by Longlive Biotechnology Co., Ltd., Shandong, China. Cellulose fibrils (CF) were obtained from bleached eucalyptus pulp. Particles of WF, AHR, and CF were classified using an oscillating screen. Particles passing a 45-mesh screen and having an average size of 360 μm were used for LPC preparation. WF, AHR, and CF were dried at 105 ± 5 °C until constant weight prior to blending with polyethylene for LPC preparation. Physical properties and chemical compositions of WF, AHR, and CF are summarized in Table 1. High density polyethylene was commercially purchased from Sigma Aldrich Trading Co. Ltd. China. The density of polyethylene is in the range of 0.940 to 0.976 g/cm^3, and the melting temperature is 120 to 160 °C.

Table 1. Physical Properties and Chemical Compositions (%) of Lignocellulosic Fillers

	Morphology	Size	Aspect ratio	Cellulose	Xylan	Mannan	Lignin	Ash
WF	granules	45 mesh	1:1~4:1	45.6	16.3	1.4	20.2	0.4
AHR	granules	45 mesh	1:1~7:1	63.4	1.6		27.2	4.5
CF	fibers	45 mesh	3:1~20:1	94.7	2.6			

Preparation of Composites

A conical single-screw filament extruder (Noztek Pro, West Sussex, England) with a nozzle of 1.75 mm was used to prepare LPC filament. Prior to LPC formation, lignocellulosic ingredient was mixed with polyethylene with filling levels from 5 to 30 wt%. Extrusion temperature was set at 140°C. The formed filament samples were cooled at room temperature.

Mechanical Property Determination

Prior to measurements, LPC filament was cut into specimen with 13 cm in length, and was conditioned at 25 °C and a relative humidity of 50 % for 72 h. Tensile strength was measured according to the ASTM D638 standard using a universal tensile machine (Instron 5943, Illinois Tool Works Inc. MA, USA). Stress-strain curves were recorded at the stretching speed of 1.4 mm/min at room temperature for tensile and elongation measurements. All measurements were carried out in duplicates at a minimum. The average data were reported. The standard deviations were calculated as measurement errors.

Morphology Analysis by Scanning Electron Microscopy (SEM)

The morphologies of the composites were examined with a scanning electron microscope (emplus30) supplied by COXEM (Daejeon, South Korea). The fracture surfaces of the specimens after impact testing were sputter-coated with gold before analysis. All images were taken at an accelerating voltage of 20 kV.

Thermal Property Determination

Thermogravimetric analysis (TGA) was performed on the LPCs to determine the weight loss as a function of temperature. Thermal properties were determined using a TGA-1 supplied by Mettler Toledo (Columbus, OH, USA). Samples of approximately 6 mg of each composite were analyzed and heated at a rate of 5 °C/min up to 700 °C under a 0.1 Mpa nitrogen atmosphere.

RESULTS AND DISCUSSION

Stress-Strain Curves

Figure 1 is the strain–stress curve of polyethylene filament without lignocellulosic fillers (control specimen). Like many materials, polyethylene displays linear elastic behavior at the very beginning of tensile testing, as shown in Fig. 1. Within the elastic limit point, deformations are completely recoverable upon removal of the load. Beyond this elastic region, deformation is plastic and the object does not completely return to its original size and shape when unloaded. Theoretically, the elastic limit corresponds to yield strength. If yield point is not easily defined based on the shape of strain–stress curve, an offset strain is usually arbitrarily defined, commonly using 0.2% as illustrated in Fig. 1. From the shape of the strain-apparent stress curve in Fig. 1, it is easy to identify the yield point in the engineering stress-stress curve at which the curve levels off and plastic deformation begins to occur. In this work, yield strength is determined at 'levels off point' rather than 'offset point'. Polyethylene-based LPC is a ductile material and experiences some plastic deformation and necking before rupture when subjected to tensile testing. The elongation will cause the reduction of cross-section area of specimen. Therefore, the actual stress (or true stress) is quite different from apparent (or engineering) stress as illustrated by Fig. 1. Besides necking, dislocation movements also occur with the crystal structure of polyethylene in the strain harden region in Fig. 1. Dislocations strongly influence the mechanical properties, as suggested by the fluctuation of stress. Since plastic deformation is unacceptable for many applications of LPC, engineering stress is used for calculation of yield strength and tensile strength in present work.

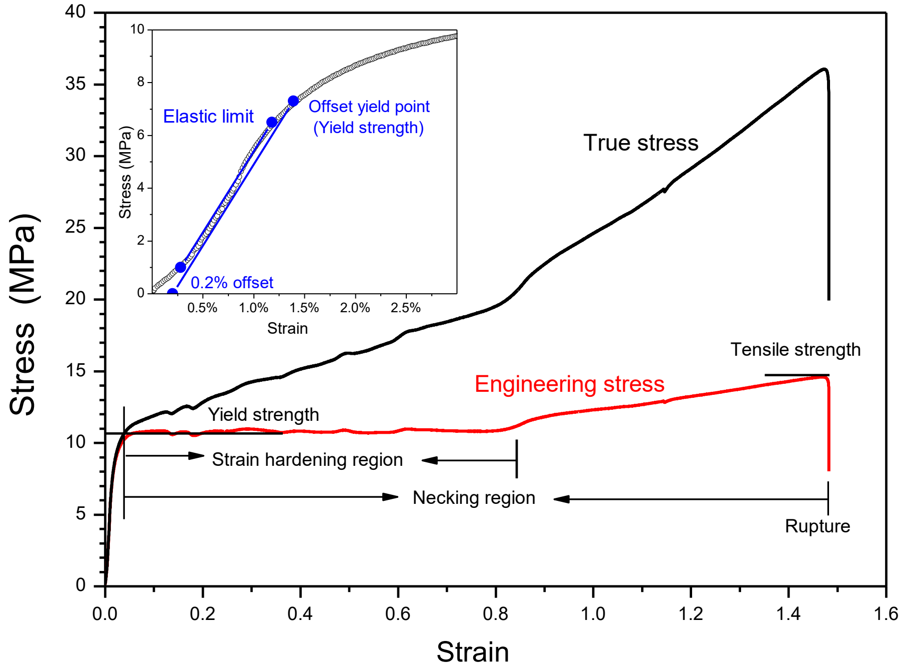

Fig. 1. Stress-strain curve of polyethylene filament (1.75 mm × 150 mm) at room temperature; Insert graph shows the definition of yield strength at a commonly used offset value of 0.2%.

LPC filaments with different fillers and filling levels were subjected to tensile testing to obtain stress-strain curve for tensile strength measurement, as shown in Fig. 2. For all LPC specimens, an elastic limit is attained within 10% stain, which suggests a short range of the elastic region. As deformation continues, the stress levels off in the range of strain hardening, where yield strength is defined and listed in Table 2. For further increasing of tensile force, tensile strength is recorded at the point of rupture. Yield strength, tensile strength, and elongation at rupture are listed in Table 2 for comparison. For WF-filled LPC, yield strength, tensile strength, and elongation showed a decline trend with increasing filling level, which is possibly attributable to the granular shape and inhomogeneous chemical constitutions. The decrease of elongation caused by addition of wood fibers was also reported by Nourbakhsh and Ashori (2008). Studies from Yeh *et al*. (2012) and Peltola *et al*. (2014) also showed that the tensile strength of LCP was lower than those without wood particles. WF, after all, is an alternative type of filler for which 20% filling level does not result in significantly compromising of mechanical properties. For AHR, positive effects on yield strength, tensile strength and elongation were observed with 10% filling level, and further increase of filling level led to a negative impact on these properties. AHR is side product from xylose or furfural production, and has economic advantages as a lignocellulosic filler. Therefore, AHR is a good choice for commercial LPC production. It is apparent from Table 2 that CF can increase mechanical properties of LPC, and achieves maximum enhancement of 56.8%, 76%, and 94% in yield strength, tensile strength and elongation, repetitively, at the 2.0% of filling level. It is assumed that these reinforcing effects come from the high crystallized structure and high aspect ratio (the length-to diameter ratio) of CF. In other words, CF-filled LPC has similar structure to reinforced concrete, and CF acts the role of steel in the thermoplastic matrix. The contribution of fiber shape to mechanical strength of LPC had been reported by Peltola *et al.* (2014), who stated that wood fibers provided clearly higher plastic reinforcement than wood flour. Corresponding results for tensile strength and length at breakage are given in Fig. S1 in the appendix.

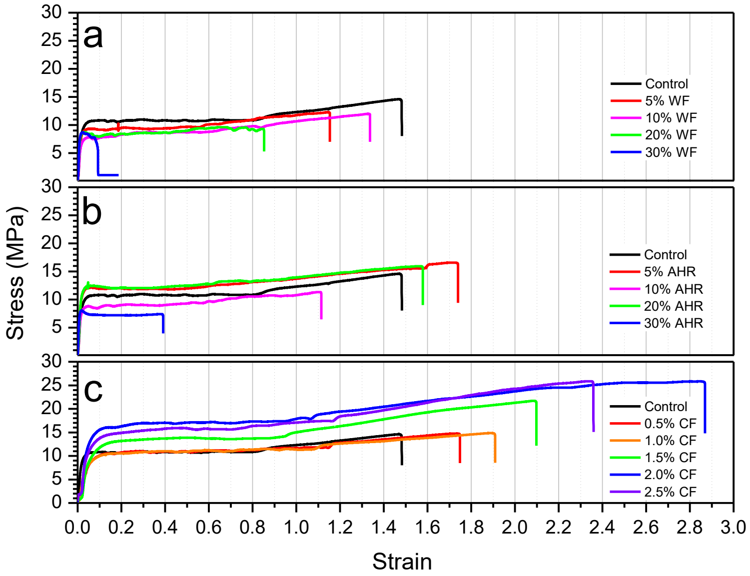

Fig. 2. Stress-strain curve of LPC at various filling levels of WF (a), AHR (b), and CF (c)

Table 2. Yield Strength and Tensile Strength for LPC with Different Filler Types at Various Filling Levels

	Filling levels (%)	Yield strength (MPa)	Tensile strength (MPa)	Elongation at rupture (%)
Control	0	10.9	14.6	147
WF	5	9.4	12.2	114
WF	10	8.5	11.9	133
WF	20	8.2	9.2	84
WF	30	7.5	8.5	9
AHR	5	11.8	16.5	174
AHR	10	11.9	15.9	156
AHR	20	9.1	11.4	112
AHR	30	7.1	7.8	38
CF	0.5	10.9	14.8	173
CF	1.0	10.9	14.8	190
CF	1.5	13.9	21.7	209
CF	2.0	17.1	25.7	286
CF	2.5	15.7	25.8	234

As already noted, polyethylene-based LPC is a ductile material and experiences necking before rupture when subjected to tensile testing. Due to significant reduction of cross-section area during specimen elongation, the true stress is quite different from apparent (or engineering) stress. The engineering tensile strength, true tensile strength, as well as elongation were calculated and plotted in Fig. 3. The true tensile strength was more than twice that of the engineering tensile strength. LCP containing 10% AHR showed improvement in both elongation and tensile strength compared to the control. The elongation and true tensile strength of LCP containing 2% CF reached 286%, and 57.5 MPa. This means that the CF reinforced LCP was stronger than unreinforced polyester resin, which has a tensile strength of 55 MPa. The results reported here also suggest significant advantages of CF-filled LPC in terms of tensile strength and elongation.

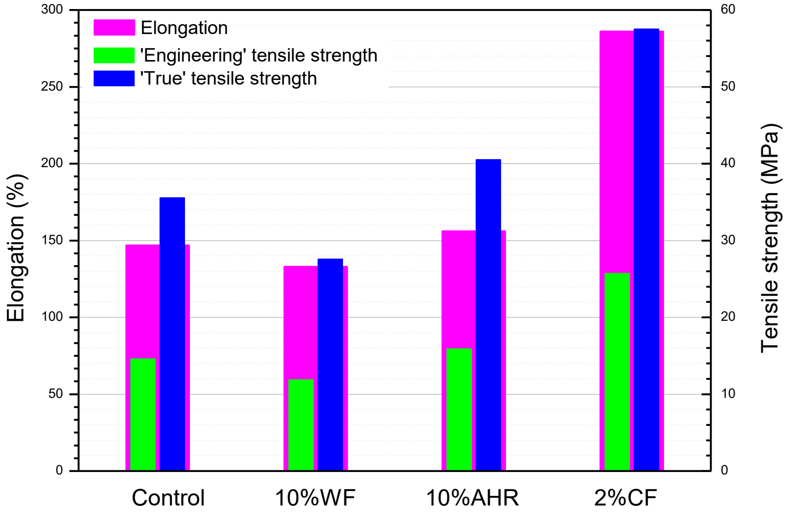

Fig. 3. Comparison of tensile strength (engineering and true tensile strength) and elongation of the polyethylene (control) and LPC containing 10%WF, 10% AHR, and 2% CF

Interfacial Analysis by SEM

Because LCP strength relies on fillers-matrix interactions at the interface, the surface chemistry also plays an important role in LCP strength development (Migneault *et al.* 2015). Microscopic methods were employed to investigate the interface compatibility. Optical microscope images show the morphologies of WF, AHR, and CF in Figs. 4(a), (b), and (c), respectively. WF, AHR, and CF are similar in size, but different in shape. SEM was used to examine the distribution and compatibility between the fillers and the thermoplastic matrix. Fractured surfaces in Fig. 4(e), (f), (g) show that fillers were randomly distributed and randomly oriented. As evident in Fig. 4(e) and Fig. 4(f), LPC filled with WF or AHR had many holes and cavities in thermoplastic matrix, which suggests poor interface compatibility. Although the addition of AHR or WF could replace a small portion of polyethylene, they could not provide an efficient stress transfer from the matrix. This causes decline of mechanical properties of LPC. For LCP filled with CF at 2% in Fig. 4 (g), no holes or cavities were observed. This means stronger adhesion between the fillers and the thermoplastic matrix, which explains the superior tensile strength and elongation. The shape of fillers is assumed to be an important factor in the way the fillers enhance the LPCs. Particle-shaped WF and AHR could not resist exterior tensile forces and therefore showed no positive effects on mechanical properties. Rod-like CF acted like steel bars in cement, helping to improve the strength of the LPCs. Chemical compositions of lignocellulosic fillers also affect mechanical properties of LPC (Migneault *et al.* 2015). CF consists mainly of crystallized cellulose, while WF and AHR consist of cellulose and lignin. Lignin is a heterogeneous biopolymer, and much weaker in mechanical strength than cellulose. Based on results from interface analysis, it has been suggested to employ a coupling agent to improve interface strength between lignocellulosic fillers and thermoplastic matrix, especially for WF, and AHR (Zhang 2014).

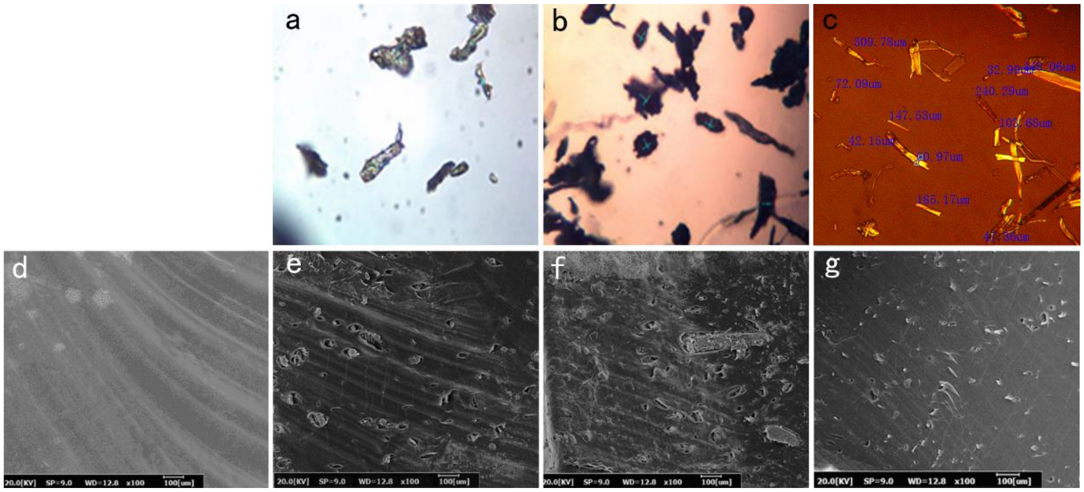

Fig. 4. Optical microscope images of WF (a), AHR (b), and CF(c); SEM cross section images of polyethylene (d, control) and LPC specimens: filled with 10% WF (e), with 10% WF (f), and with 2% CF (g)

Thermal Stability

The thermal stability of LCP containing different lignocellulosic fillers was examined by TGA in high purity of nitrogen stream from 45 ºC to 800 ºC at a heating rate of 5 ºC/min. The weight loss profiles are illustrated in Fig. 5(a). The results clearly show that the LCP samples filled with WF, AHR, and CF, respectively, had different thermal stabilities. The first derivative curves of TGA in Fig. 5(b) give information of the onset thermal decomposition temperature and the highest thermal decomposition temperature. Compared to the control sample, LCP filled with 10% WF showed a lower onset thermal temperature of 330 ºC due to the presence of hemicelluloses (mainly xylan and mannan) in WF. By contrast, LCP filled with 10% AHR showed improved thermal stability as suggested by the highest decomposition temperature of 467 ºC. The improved thermal stability can be interpreted by the chemical compositions of AHR, which is rich in thermal stable lignin, and absent in hemicelluloses. Lignin is a cross-linked phenolic polymer, and shows functions of stabilization and antioxidation from an accelerated weathering test of wood-plastic composite (Yang *et al.* 2015). CF is mainly composed of cellulose. It is well acknowledged that thermal decomposition of cellulosic materials occurs between 276 ºC and 400 ºC (Fisher *et al.* 2002; Fu *et al.* 2016). From derivative TGA curves in Fig. 5 (b), CF with 2% shows no negative effects on thermal stability of LCP, which may be interpreted by the low filling level of merely 2%.

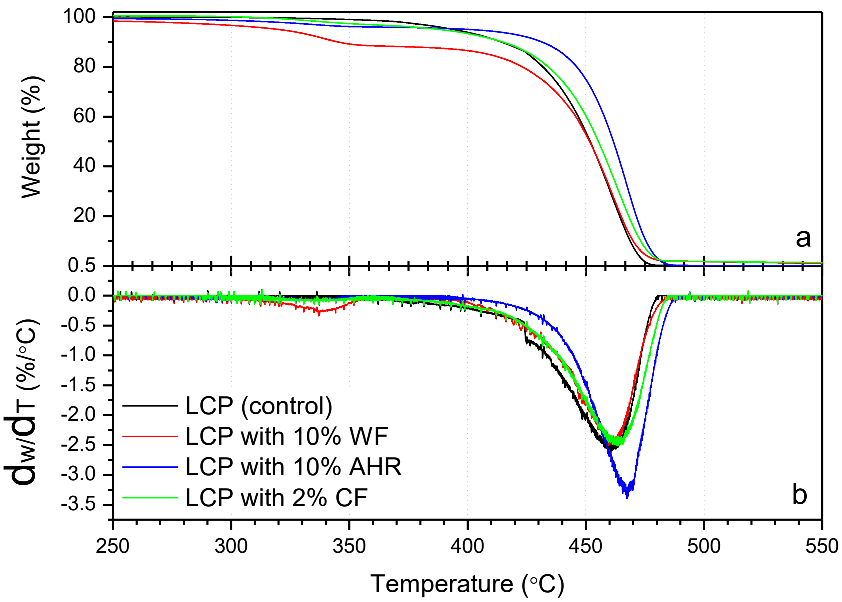

Fig. 5. Comparisons of thermal stability of different LCP samples filled with various levels of WF, AHR, or CF (a) TGA weight loss; (b) TGA temperature derivative weight loss

CONCLUSIONS

1. Mechanical properties of lignocellulosic-plastic composites (LPC) were significantly affected by the characterizations of lignocellulose fillers in terms of chemical compositions and morphology.
2. WF addition resulted in reduction of tensile strength and thermal stability due to presence of xylan.

3. Acid hydrolysis residue (AHR), which is rich in lignin and relatively free of xylan, showed a positive effect on thermal stability relative to untreated LCP, but insignificant improvement in tensile strength.
4. LCP filled with cellulose fibrils (CF) showed remarkable reinforcement in tensile strength, and elongation, which is ascribed to the crystalline structure and fiber shapes of CF.

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