



# Corn cob residual reinforced polyethylene composites considering the biorefinery process and the enhancement of performance

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## ARTICLE INFO

### Article history:

Received 24 January 2018

Received in revised form

5 July 2018

Accepted 9 July 2018

Available online 11 July 2018

### Keywords:

Biorefinery

Corn cob residual

Corn cob plastic composite

Mechanical property

Weathering effect

## ABSTRACT

Aiming to improve the economic feasibility of the corn cob biorefinery process, fibers, including the corn cob bagasse, xylose-extracted corn cob residue and the ethanol-processing residue, were used as the reinforcing phase for the preparation of wood plastic composites (WPCs). The mechanical properties and the weathering performance of the composites were compared. The results indicated that the WPCs reinforced by the ethanol-processing residue showed balanced advantages in mechanical properties and weathering performance. The effects of the compatibilizer and the cellulose/lignin mixture on the WPC properties were further evaluated, indicating that use of 4 wt % of Polyethylene-*graft*-maleic anhydride (PE-*g*-MA) as the compatibilizer and a mixture of the EPR with lignin at a ratio of (9:1) were the optimized conditions for WPCs. The current work showed potential in making full use of the solid residue of biorefinery processes and making the waste profitable.

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## 1. Introduction

Wood plastic composites (WPCs) derived from renewable lignocellulosic biomass materials and recycled plastics (or biodegradable polymers) have attracted extensive attention due to their low cost, low environmental impact and good performance (Qiang et al., 2014; Khan et al., 2009). WPCs could be used successfully to replace the conventional wood-based products, addressing the forest degradation and accelerated deforestation (Zahedi et al., 2013; Holt et al., 2014). Among the different types of lignocellulosic fibers that were used as the reinforcing phase of WPCs, agricultural residue is considered as one of the good candidates because of its abundance and low price (Klímek et al., 2016; Nagarajan et al., 2013). In recent years, different cultivars of lignocellulosic residuals have been applied to WPC production and the product showed good performance (Nagarajan et al., 2013; Nyambo et al., 2010).

In comparison with using lignocellulosic residuals directly for WPC production, another motivation is to use the residuals from

biorefinery plants for development of value-added products. In the case, the economic competitiveness was improved and the feasibility of the agricultural residual deep processing was enhanced effectively (Wei et al., 2015). In previous studies, dried distillers grains with solubles (DDGS), the residual from the first-generation biochemical routes, was efficiently applied to reinforcement of WPC (Wei et al., 2015; Madbouly et al., 2014). However, because of the controversy of food versus biochemicals, these processes based on grains were not encouraged (Rulli et al., 2016). By contrast, the second-generation biorefinery processes that are based on lignocellulosic materials were more attractive (Losordo et al., 2016), while only a few studies focused on the WPC production from the second-generation biorefinery residuals (Yu et al., 2012; Zhong et al., 2010). In our previous study, the sweet sorghum bagasse after the bioethanol and biobutanol fermentation process was used for WPC production with acceptable mechanical properties (Yu et al., 2012). In addition, microalgae biomass was also used to produce poly (butylene adipate-co-terephthalate) (PBAT)-based composites (Torres et al., 2015).

In fact, the performances of WPC products reinforced by the biorefinery residuals might be better than the raw lignocellulosic materials due to the changes in the fiber properties and characteristics (Zhong et al., 2010). As the properties of WPC are closely related to the hydrophobic-hydrophilic interactions between the lignocellulosic fibers and the polymer matrices, the fibers

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pretreatment also leads to the changes in fiber compositions and the surface properties (Luo et al., 2017; Mohanty et al., 2005). After chemical, enzymatic and physical pretreatments, the mechanical and physical properties of WPCs would be significantly improved (Li et al., 2013, 2017). Generally, the pretreatment of fibers was cost-intensive unless the WPC production was hybrid with the biorefineries. When coupled with biorefineries, the cost for fiber pretreatment could be shared by the WPC and biochemical productions, so the economic value of the WPC products would be increased, while the process cost was reduced (Yu et al., 2012).

Except for the pretreatment of fibers, a mixture of different types of reinforcing phase also has a positive impact on the WPC performance (Mattos et al., 2014), possibly caused by the individual properties of the reinforcing phase including cellulose, hemicellulose and lignin. In previous research, additional components including nanoclays, biochar, cellulosic materials, basalt, and glass fibers were also mixed with lignocellulosic fibers for high-performance WPC production (Martelli-Tosi et al., 2017; Das et al., 2015).

Our previous work showed that the corncob was a suitable reinforcing phase for WPC (Luo et al., 2017). As a type of by-product, corncob was easily collected because it was obtained separately after harvest of the stalk (Cai et al., 2016). Moreover, because of the relatively high hemicellulose content and the unique cellulosic structure with high enzymatic hydrolysis ability (Li et al., 2016), corncob was widely used in biochemicals and biofuel production as an ideal raw material (Gu et al., 2015). Recently, a concept for corncob biorefinery with high economic feasibility has been inserted into industrialization in China. As illustrated in Fig. 1, after acid pretreatment of corncob bagasse (CCB), the acid hydrolysate was applied to xylitol and xylo-oligosaccharide (XOS) production (Cheng et al., 2014; Pu et al., 2016). The bagasse remained, xylose-extracted corncob residue (X-ER), was further delignified by alkali or other solvents. Then, the delignified corncob residue (DCCR) was further used for simultaneous saccharification and ethanol fermentation (Liu et al., 2010; Lei et al., 2014). The solid remained

after fermentation, namely, ethanol-processing residue (EPR), was finally obtained after ethanol separation.

In the present work, aiming to maximize the economic value and tightly hybridize the biorefinery process with the WPC process, the fibers at the different stages of biorefinery that illustrated in Fig. 1 were used as the reinforcing phase of WPC. The effects of X-ER and EPR on WPC production were compared by using different types of compatibilizers. CCB, the raw material of biorefinery, was managed as the control. In that protocol, DCCR was not included because it could be utilized for bioethanol production (Lei et al., 2014). The mechanical and physical properties, as well as the weathering properties of the WPC, were evaluated and compared. Then, the EPR, the final solid residual of biorefinery, was selected and was further mixed with lignin and microcrystalline cellulose for the improvement of WPC properties.

## 2. Materials and methods

### 2.1. Corncob-based fibers and the basic materials

The corncob-based fiber materials, including CCB, X-ER and EPR, used in the current work were kindly provided by Longlive Biotechnology Co., Ltd., Yucheng, Shandong, China. The corncob processing fibers were dried overnight at 105 °C and were milled to 40–60 mesh before use. Lignin was also provided by Longlive Biotechnology Co., Ltd. The high-density polyethylene (HDPE 600 has a density of 0.956 g cm<sup>-3</sup>, a melting point of 136 °C, and a melt flow index (MFI) of 1.8 g min<sup>-1</sup>) as matrix material was purchased from Jinma Plastic Co., Ltd., Beijing, China. The Polypropylene-graft-maleic anhydride (PP-g-MA) and PE-g-MA as the compatibilizers (Formela et al., 2014, 2015; Korol et al., 2015) were obtained from Nantong Rizhisheng Polymer Technology Co., Ltd., Jiangsu, China. The microcrystalline cellulose was purchased from National Medicine Group of China.

### 2.2. Component analysis and the surface features of the corncob processing fibers

The composition of the corncob processing fibers (CPF) was determined by the method of the National Renewable Energy Laboratory of USA (NREL) (Sluiter et al., 2008). The chemical groups of the fibers were analyzed by Fourier transform infrared spectroscopy (FTIR) (Nicolet 6700, Thermo Fisher, USA). A resolution of 32 scans and 4 cm<sup>-1</sup> was obtained. An X-ray diffractometer (D 8, Bruker, Germany) was used for determining the crystallinity index (CrI). Samples were scanned at the step size of 0.02°, and a speed of 1° min<sup>-1</sup> was achieved between 10° and 30°. The CrI was calculated according to the method explained in detail in the Li et al. (2016) study.

### 2.3. Preparation of the polyethylene/CPF composites

Like the method used in the Luo et al. (2017) study, the polyethylene/CPF composites were composed of 50 wt % fibers as the reinforcing phase, 40 wt % HDPE, 3 wt % compatibilizer (the loading rate was changed when testing the effect of compatibilizer loading rate), 3 wt % stearic acid and 4 wt % polyethylene wax as coupling agents. For the comparison of the compatibilizers, the effects of PE-g-MA and PP-g-MA on composite performance were evaluated. The CPFs were well mixed with lignin and microcrystalline cellulose.

For the manufacture of the composites, the mixture of fibers, plastic, compatibilizers and coupling agents was extruded by a co-rotating twin-screw extruder (Coperion ZSK series, Werner & Pfleiderer, Germany). The injection and the granulation temperatures were kept at 140–165 °C and 140–175 °C, respectively. The



Fig. 1. Flow chart of the corncob biorefinery process. Solid fractions of CCB, X-ER and EPR in different pretreatment stages were used as the reinforcing phase of the WPCs.

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