



# Effects of environmental-friendly modified rubber seed shell on the comprehensive properties of high density polyethylene/rubber seed shell composites



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## ABSTRACT

This study aimed to evaluate the effects of rubber seed shell modified by high temperature superheated vapor on the comprehensive properties of high density polyethylene/rubber seed shell (HDPE/RSS) composites with two superheated vapor temperatures (200 and 220 °C) and various particle sizes of RSS (60–80, 80–100 and 100–120 mesh). The target properties of the composites were analyzed using electronic universal mechanical instrument, thermogravimetric (TG) analyzer, differential scanning calorimeter (DSC), scanning electron microscopy (SEM) and dynamic mechanical analysis (DMA). The results indicated that the composites with 100–120 mesh RSS particles modified at the optimum superheated vapor temperature of 200 °C (HDPE/RSS200-3) appeared the strongest interfacial bonding ability with the excellent comprehensive analysis data. Compared to unmodified samples at the same particle size, the flexural and tensile strength of HDPE/RSS200-3 increased by 21.27% and 12.92%, respectively,  $T_{\max 1}$  and  $T_{\max 2}$  shifted toward the higher temperature value from 349.5 °C and 475.3 °C to 351.9 °C and 478.9 °C, there was a marked increase for  $E'$  from 3370 to 3819 MPa but decrease for  $\tan \delta$  from 0.355 to 0.326. In addition, the HDPE/RSS200-3 also exhibited the relatively outstanding water resistance with the maximum water absorption and thickness swelling rates of 4.01% and 1.02%.

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## 1. Introduction

Today natural renewable fiber/polymer composites are receiving considerable attention for use in wood, bamboo, flax, hemp, kenaf, palm, pineapple leaf, cotton and other agricultural biomasses in place of partial synthetic fibers (glass, nylon and carbon fibers) due to the great concerns about environment protection and sustainable development (La Mantia and Morreale, 2011; Zini and Scandola, 2011; Dicker et al., 2014; Holt et al., 2014; Bulota and Budtova, 2015; Xia et al., 2016). Compared to synthetic fibers, natural fiber has the attractive advantages of enormous supply, non-toxicity, low energy consumption, low cost, high specific strength and biodegradability (Chen et al., 2014). Therefore the global production of natural fiber-reinforced bio-composites is expected to increase from 12% in 2010 to 18% in 2020 and 25%

in 2030 (Gurunathan et al., 2015). Application for bio-composites has also gradually extended from original non-structural or decoration components to essential elements of buildings, automotive, aircraft, package and other functional products that will withstand large, complicated external loads (Rana et al., 2003).

It is true that natural fiber components have negative characteristics, because natural fibers will strongly absorb moisture resulting in a special anisotropy that results from strong hygroscopic hydroxyl groups in the cellulose and hemicelluloses in the material. Consequently, the physical and mechanical properties of natural fiber materials are directional and uneven. (Jawaid and Abdul Khalil, 2011). Furthermore the resident hydroxyl groups could react with air borne water molecules, causing hydrogen bonding that will lead to serious damage in the interfacial bonding of biocomposites with emerging micro-voids (Hosseinaei et al., 2012). This poor interfacial character in biocomposites that results from inadequate bonding between the hydrophilic natural fiber and the hydrophobic polymer has been a long-standing challenge. It can result in weak static and dynamic mechanical properties, ther-

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mal instability and poor water resistance. Until now, a great many strategies have been tried to overcome the interfacial adhesion defect of biocomposites, many of which have focused on chemical modifications, including alkali treatment (Ichazo et al., 2001; Alam et al., 2012), grafting copolymerization (Gao et al., 2012; Yemele et al., 2013), silanization, acetylation, esterification and isocyanates (Lee and Wang, 2006; Hosseinaei et al., 2012). These chemical agents promote reactions with the natural fiber surface and the polymer producing a bridge between the fiber and the polymer matrix.

In addition, novel non-chemical modification approaches have been explored for improving the overall properties of biocomposites, such as the addition of nano-particles (Devi and Maji, 2011; Deka and Maji, 2012), plasma treatment and biological treatments using enzymes, fungi, and bacteria as well as high temperature superheated vapor treatment (Park et al., 2004; Kaboorani et al., 2008; Mukhopadhyay and Fanguero, 2009; Theapsak et al., 2012). High temperature superheated vapor treatment has been considered to be one of most effective and environmental-friendly methods. During the treatment process, no external chemical reactions occurred that can result in potential chemical pollution on the treated natural fiber. Some inner chemical transformations of natural fiber with some cellulose, hemicellulose and lignin usually result from autocatalytic reactions of the cell-wall constituents. Hemicelluloses are the most reactive chemical component among the three natural cellulose substances, which can be hydrolysed into oligomeric or monomeric structures. Under high temperature conditions, hydroxyl groups in the cellulose can be partially broken and chemical bonds within the lignin complex can be cleaved, forming phenolic groups. These changes imposed by high temperature superheated vapor treatment can reduce the polarity of natural fiber and make them more compatible with the non-polar thermoplastic matrix, which leads to an effective enhancement of mechanical strength, dimensional stability and durability of the composites (Pétrissans et al., 2003; Tjeerdsma and Militz, 2005; Kaboorani et al., 2008).

The rubber crop is a traditional economic agricultural plant, which is distributed throughout low latitude tropical regions such as Indonesia, Thailand, Malaysia and in south and southwest in China. (Homkhiew et al., 2014). In China, rubber crop plantations have experienced considerable growth recently with the plantation exceeding 1 million ha over the past decade. (Sun and Jiang, 2010; Ng et al., 2014; Xiao et al., 2014). Rubber seed is one of common products of the rubber crop besides rubber wood, rubber latex and rubber honey. It has been discovered to be a potential animal feedstock. Some studies show that rubber seed cake can feed up to 10% of animals' diet meal without affecting the growth of animals (Madubuike et al., 2006). Rubber seed shell (RSS), is a primary by-product of rubber seed and was once considered to be agricultural waste. As such, RSS was usually disposed of or burned resulting in serious environmental contamination. However, in recent years, the RSS has begun to attract more attention for use in producing activated carbon and biocomposites. (Ioannidou and Zabaniotou, 2007; Nor et al., 2013; Chin et al., 2014; Ng et al., 2014). Sun et al. demonstrated that high-quality activated carbon can be prepared from RSS by physical activation with steam (Sun and Jiang, 2010). Ismail et al. were the first to prepare and study the properties of RSS/polypropylene and polyethylene composites, which generally exhibited poor interfacial bonding between the RSS particles and the resins. This resulted in relatively poor physical, mechanical and morphological properties (Ismail and Shafiq, 2014).

Therefore, research on developing RSS biocomposites shifted focus to improving the interfacial bonding between the RSS and the polymer resin. To date, there have been few reports about how modification of rubber seed shell affects the comprehensive properties of RSS-reinforced biocomposites. In this reported

study, the goal was to evaluate how high temperature superheated vapor treatment of rubber seed shell (RSS) affects the properties of HDPE/RSS composites. In particular, the static-dynamic mechanical properties and the melting temperature, thermal stability, water absorption and micro-morphology of the composites as a function of various superheated vapor temperatures and RSS particle sizes. The properties of unmodified and modified composites were analyzed using an electronic universal mechanical test instrument, differential scanning calorimeter (DSC), thermogravimetric (TG) analyzer, scanning electron microscopy (SEM) and dynamic mechanical analysis (DMA).

## 2. Materials and methods

### 2.1. Materials

High density polyethylene (HDPE) pellets with the density of  $0.954\text{ g/cm}^3$  and melt flow index of  $0.70\text{ g/10 min}$  were used in the study as the resin matrix, they were bought from Daqing Petrochemical Co., China. The rubber seed shells (RSS) were obtained from Jinghong, Xishuangbanna, Southwest China, the main components of RSS consists of cellulose (71.98%), hemicellulose (24.25%), lignin (2.92%) and ash (0.85%). The RSS were smashed by a high speed disintegrator, then sieved to different particle sizes using the vibrating screen equipping before mixing with the other components. Silane coupling agent (KH-570) was purchased from Guangzhou Jingke Chemical Technology Ltd. Co., China.

### 2.2. Treatment of rubber seed shell

The rubber seed shells were treated in high temperature superheated vapor environment with a three-step process. At the first step, the natural RSS with various particle sizes (60–80, 80–100, 100–120 mesh) were exposed to a laboratory-scale superheated vapor oven ( $950 \times 950 \times 900\text{ m}^3$ , Jiangsu, China) with both dry-bulb and wet-bulb temperatures of  $100^\circ\text{C}$  for 30 min. Secondly the dry-bulb temperature increased to  $200^\circ\text{C}$  and  $220^\circ\text{C}$  respectively for different experiments at a heating rate of  $20^\circ\text{C/h}$  under the condition of constant wet-bulb temperature of  $100^\circ\text{C}$  through continuous steam spray, subsequently the dry-bulb temperature maintained for 3 h. In the third process, continuous steam supplying would not suspend until the dry-bulb temperature decreased to below  $100^\circ\text{C}$ . Finally the whole oven system was closed and the RSS particles cooled down to room temperature for use.

### 2.3. Preparation of HDPE/RSS composites

The RSS particles were dried at  $80^\circ\text{C}$  for 6 h in an oven to make sure the moisture content less than 3%. The RSS particles and the HDPE resin were blended with 1600 rpm at  $80^\circ\text{C}$  for 10 min to produce a homogenous mixture in a high-speed mixer (SHR-10A, Zhangjiagang, China). The mixtures were mixed in a two roller mill (BP-6175-A, Dongguang, China) at  $160^\circ\text{C}$  for 10 min. Then a hot press instrument (BY302  $\times$  2/2 150T, Suzhou, China) was used to prepare the HDPE/RSS composites by compressing the mixture with a pressure of 1.5 MPa for 6 min at  $170^\circ\text{C}$ . Polytetrafluoroethylene films were used to avoid direct contact of mixtures with the metal platens during hot pressing process. After hot pressing, the composites panel was cooled down to room temperature in a cold press. The dimension of formed panel was  $300\text{ mm} \times 300\text{ mm} \times 4.5\text{ mm}$  with an average density of about  $1.0\text{ g/cm}^3$ . Subsequently the composites panels were placed in the climate room with relative humidity of 65% and temperature of  $20^\circ\text{C}$  for two days before they were cut into test samples for further

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