Carbon Black Filled Shell of High Density Wood-Polyethylene Composites to Balance the Antistatic Properties and Mechanical Properties

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Carbon black (CB) was used to fill the shell of wood high-density polyethylene (HDPE) composite to improve its antistatic properties and simultaneously maintain its mechanical properties. The conductivity analysis revealed that the percolation threshold of the CB in coextruded WPC was between 15% and 20%, and the surface resistivity of the sample containing 20% CB (CW-20) achieved the floor antistatic requirements of an electronic information system room and its support area according to GB/T 50174 (2017). The addition of CB into the shell of the coextruded WPC inhibited its crystallization. The mechanical properties of coextruded WPC were increased when the CB content was increased. The flexural strength, flexural modulus, and impact strength of the CW-20 sample were increased by 21.6%, 10.8%, and 26.1%, respectively. In addition, the dosage of CB in regular WPCs was 6.71 times as much as that of coextruded WPCs, which achieved similar antistatic and mechanical properties of the two structural WPCs. These results demonstrated that the low CB filled shell of HDPE composite exhibited a large reduction in its surface resistivity and a significant enhancement in its mechanical properties, which expands the application of coextruded WPCs in flammable and explosive uses while also improving the antistatic and mechanical properties.

Keywords: Wood-flour composites; Coextrusion; Carbon black; Mechanical properties; Antistatic properties

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INTRODUCTION

Wood plastic composites (WPCs), which have emerged as constructive and decorative materials, are rarely used in flammable and explosive applications due to their electrostatic discharge. Electrostatic charge is easily produced on the surface of WPCs by rubbing, as thermoplastics and lignocellulose fibers show excellent electrical insulation properties (Yossef *et al.* 2001; Gao *et al.* 2010). WPCs prepared by thermoplastics and wood flour are poor conductors of electricity, and they easily generate electrostatic accumulation to cause dust, electric shock (discharge), burning, and even explosions. It is necessary to conduct an antistatic treatment for WPCs before their use in certain applications, such as program-controlled equipment rooms, operating rooms, or electronic components production workshops (Luo *et al.* 2000). In addition, the antistatic properties of WPCs are crucial, especially for indoor environments such as conference rooms or costly computer rooms, because excessive accumulation of electrostatic charge is detrimental to human health and electronic devices (Yu *et al.* 2017). There is a large amount of research on antistatic polymers. In contrast, the antistatic properties of WPCs are rarely reported,

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and their products have not been widely used in the market (Pinto *et al.* 2004; Zheng *et al.* 2004; Zhang *et al.* 2010; Cao *et al.* 2013). To improve the antistatic properties of polymers, various conductive fillers have been added, including metal powder or fiber (Malliaris and Turner 1971; Aharoni 1972), carbon black (CB) (Abdel-Bary *et al.* 1979; Miyasaka *et al.* 1982; Narkis *et al.* 2010), and carbon fiber (Wang and Peng 1997; Wu *et al.* 1999).

CB is one of the most widely used conductive fillers because of its light weight, low cost, and permanent conductivity (Song and Zheng 2005; Chen et al. 2006; Liao and Wang 2014). CB has a strong electrical conductivity and can be dispersed in the composites in a relatively uniform fashion due to its small particle size, which can form a continuous conductive network in the matrix, thereby improving the conductivity of the composites (Liu et al. 2010). The addition of CB is effective for decreasing the volume resistivity of polypropylene (PP), epoxy, and glass fiber composites (Tchoudakov et al. 1996). The electrical conductivity of polymer/CB composites increases sharply at a critical filler concentration, called the percolation threshold, due to the formation of a three-dimensional conductive network throughout the polymer matrix (Yu et al. 2017). Previous research has shown the percolation values for the critical transition in filler-based composites are 6.2 wt% for PP/CB (Zois et al. 1999) and 9 wt% for nylon 6/CB (Pinto et al. 2004). However, high loadings of CB (15 wt% to 20 wt%) in the polymer matrix was required to achieve the conductive percolation threshold, leading to the high cost, poor mechanical properties, and complex processing of the composites (Narkis et al. 1999; Rosner 2001; Cheah et al. 2015). The CB distribution was affected by the surface tension (Sumita et al. 1991; Tchoudakov et al. 1996) and interfacial free energy (Asai et al. 1992). It also depends on the polymer crystallinity (Gubbels et al. 1994), viscosity (Feng and Chan 2000; Mironi-Harpaz and Narkis 2001a; Mironi-Harpaz and Narkis 2001b), and processing (Gubbels et al. 1994; Cheah et al. 2015). When the CB concentration reaches the percolation level in the continuous CB-rich phase, double percolation is achieved, and the percolation threshold is lowered (Tchoudakov et al. 1997; Breuer et al. 2015; Foulger 2015). For instance, Gubbels et al. reported that the electrical percolation threshold can be lowered to 0.2 to 0.3 v/v% when the CB particles were selectively localized at the interface of a cocontinuous polyethylene/polystyrene (PE/PS) blend (Jamshid 1998).

However, introducing conductive fillers into an immiscible polymer blend, due to its weak interfacial interactions, has caused a decline in mechanical properties, especially in tensile properties (Gao et al. 2012; Cheah et al. 2015). Therefore, it is essential to find an optimal balance between the antistatic and the mechanical properties for the design of the polymer composites. Coextrusion technology, a method to make a protective surface, can produce a multilayer board with different properties in the outer and inner layers. Since electrostatic charge primarily occurs at the surface of a material (Yossef et al. 2001; Gao et al. 2010), pursuing a cost-effective means to cope with electrostatic charge would not favor adding conductive fillers into the bulk of a material instead of the surface. The coextruded WPCs with a high-density polyethylene (HDPE) cap layer significantly optimized the water resistance and flexural strength in comparison to the regular WPCs (Jiang et al. 2007; Rauwendaal 2010; Liu et al. 2018). Adding 3% CB into the shell layer of PP based WPC improved the tensile strength and hardness by 45% and 25%, respectively (Turku and Kärki 2014). The addition of CB has been shown to be effective in enhancing the antistatic properties of virgin plastic (Mamunya et al. 1996; Ishigure et al. 1999; Dudler et al. 2000; Pinto et al. 2004), however, the addition of CB to the HDPE shell of coextruded WPC has not yet been demonstrated to simultaneously improve its antistatic and mechanical properties.

The objective of this study was to reduce the composite cost and balance the antistatic and the mechanical properties of the composites through adding CB into the shell of coextruded WPCs. To achieve this, the effects of CB (0%, 5%, 10%, 15%, and 20%) on the antistatic, thermal, and mechanical properties of the coextruded and regular WPCs were studied using an ultrahigh resistance meter, universal testing machine, scanning electron microscopy (SEM), and differential scanning calorimetry (DSC). Comparative analysis of regular and coextruded WPCs was done to confirm the advantages of cost and properties in coextruded WPCs. The obtained results gave a comprehensive understanding of how to seek a balance between the antistatic properties and the mechanical properties, and to apply coextruded WPCs in flammable and explosive applications.

EXPERIMENTAL

Materials

The wood flour (WF) passing through a sieve of 80-mesh, but retained on the 100mesh sieve, was prepared by Pucheng Wood Industry Co. Ltd. (Xuzhou, China). The HDPE (density: 0.951 g/cm^3 , melt flow index: 0.095 g/min, grade: 5000 s) and the coupling agent (MAH-g-PE) were supplied by Yangzi Petrochemical Co. Ltd. (Nanjing, China). The CB (Type F900B) with a specific surface area of 0.75 to $1.1 \text{ m}^2/\text{g}$ and a particle size of 9 to 17 nm, as a conductive filler, was obtained from Yiborui Chemical Co. Ltd. (Tianjin, China). The anhydrous ethanol, with a density of 0.79 g/mL, was obtained from Nanjing Chemical Reagent Co. Ltd. (Nanjing, China). Talcum with a particle size of 500 mesh was supplied by Jinqiang Limestone Co. Ltd. (Nanjing, China) and its main components were SiO₂ and MgO. The polyethylene wax was obtained from Hongsheng Chemical Co. Ltd. (Dongguan, China).

Preparation of Coextruded HDPE / Wood-Plastic Composites

The composition and the labeling of the studied formulations are shown in Table 1. CW denotes coextruded WPC, and RW denotes regular WPC. The modified CB was prepared by suction filtration after stirring 100 g CB, 3 mL KH-570, and 200 mL anhydrous ethanol in a 500 mL three-necked bottle for 1 h at 80 °C (Bai and Li 2014; Guo et al. 2015). WPC with a core-shell structure was prepared using coextrusion technology. The HDPE and the CB were dried in an oven at 103 ± 2 °C for 24 h, and then they were placed in an open mill (Zhenggong Electromechanical Equipment Technology Co. Ltd., Type ZG-160, the speed of the main engine at 8 rpm, Dongguan, China) to mix at 150 °C for 10 min. The mixed materials were disposed in a claw crusher (Nagin Machinery Co. Ltd., Type NPCY-50J, Dongguan, China) to break down the particles of the shell layer composites. The WF was dried in an oven at 103 ± 2 °C for 8 h. The formulation of the core layer composites contained 51.3% wood powder, 34.2% HDPE, 4.3% MAH-g-PE, 8.5% talcum, and 1.7% polyethylene wax, based on the total weight of the composites. The blended core layer composites were placed into the high-speed mixer (Huaming Machinery Co., Type SHR-10A, with the speed of the main engine at 1500 rpm, Zhangjiagang, China) for 10 min to disperse the particles uniformly, and then were put into a conical twin screw extruder (Jinwei Machinery Manufacturing Co., Type SJZ45/100, Shenzhen, China) for granulation. Then, the core layer composites particles were fed through the core layer extruder (Type SJZ45/100, the speed of the main engine at 5 rpm, the range of the temperature from 150 to 185 °C), and the particles of the shell layer composites were placed in the shell extruder

(Type JWS35/25, the speed of the main engine at 8 rpm and the range of the temperature from 100 to 210 °C). The preparation of regular WPC was similar with the preparation of the core layer composites in coextruded WPC. The only difference was that different proportions of CB (0%, 5%, 10%, 15% and 20%) were added to the regular WPC, based on the total weight of the wood poplar and the HDPE. The cross-sectional dimension was 100 x 8 mm (length x width) and the shell thickness was 0.5 mm, as shown in Fig. 1.

Labels	WF (wt%)	HDPE (wt %)	CB (wt %)	MAH-g-PE (wt %)	Talc (wt %)	Polyethylene wax (wt %)
CW-0		98.5	0			1.5
CW-5		93.5	5			1.5
CW-10	-	88.5	10	-	-	1.5
CW-15		83.5	15			1.5
CW-20		78.5	20			1.5
RW- 0/5/10/15/20	WF : HD)PE=6 : 4	0/5/10/15/20	5	10	2

Table 1. Compositions and Labeling of the Studied Formulations



Fig. 1. Schematics of the cross section of regular WPC and coextruded WPC

Characterization and Testing

According to GB/T 1410 (2006), the surface resistivity of the specimen was tested with a ultrahigh resistance meter (Guance Testing Instrument Co. Ltd., Type EST121, Beijing, China). The size of the specimen was $100 \times 100 \times 8$ mm (length x width x height) and the results from three specimens were averaged. The formula for calculating the surface resistivity is shown in Eq. 1,

$$\rho_s = R_x \left(P/g \right) \tag{1}$$

where $\rho_s(\Omega)$ is the surface resistivity, $R_x(\Omega)$ is the measured surface resistivity, P (cm) is the effective perimeter of a protected electrode in a particular electrode device, and g (cm) is the distance between the two electrodes

Tensile testing was conducted according to the GB/T 29418-2012 standard using an electronic universal testing machine (Sans Material Co. Ltd., Type CMT4204, Shenzhen, China). The specimen size was $210 \times 20 \times 8$ mm (length x width x height), the two fulcrum span was 200 mm, and the loading speed of the machine was 2 mm/min. According to the GB/T 1043-2008 standard, the specimens in the impact strength test were cut using a die from a compression mold with dimensions of $100 \times 10 \times 8$ mm (length x width x height) by a pendulum impact testing machine (Sans Material Co. Ltd., Type ZBC1251-1, Shenzhen, China). The experiment results were taken as the average of the six measurements.

The section morphology of the sample after impact testing was observed using a

scanning electron microscope (SEM, FEI Co., Type Quanta 200, Hillsboro, OR, USA). The section of the sample was sprayed with gold before SEM observation at an acceleration voltage of 15 kV.

The crystallization and thermodynamic properties were measured with a differential scanning calorimeter (DSC, NETZSCH Co., Type DSC 200F3, Bavaria, Germany) in a N₂ atmosphere (20 mL/min). The heating rate was 10 °C/min and temperature range from room temperature to 200 °C. In addition, the degree of crystallinity was determined from the total specific enthalpy of melting or crystallization, according to Eq. 2 (Gaur and Wunderlich 1982),

$$X_c = \Delta H_{\rm m/} \Delta H_{\rm m}^{+} \tag{2}$$

where $\Delta H_{\rm m}$ (J/g) is the specific enthalpy of melting and $\Delta H_{\rm m}^+$ is the specific enthalpy of melting for 100% crystalline polyethylene. The $\Delta H_{\rm m}^+$ value is 293 J·g⁻¹ in the equation.

RESULTS AND DISCUSSION

Antistatic Properties

Conductivity analysis

Figure 2 shows that the surface resistivities of coextruded and regular WPCs varied with the CB content. The surface resistivities of all samples were decreased while the CB content increased, indicating that the addition of CB reduced the surface resistivity of the composites and thus resulted in the disappearance of the surface electrostatic discharge. The surface resistivity of the CW-0 sample was $10^{14} \Omega$, which was higher than that of the RW-0 sample by two orders of magnitude. This result was due to the hydrophobicity of the HDPE shell in coextruded WPC and the hydrophilicity of the wood fiber in regular WPC (Klason and Kubát 1984). In addition, the critical value of the conductive filler to form continuous conductive networks, causing a dramatic resistivity decrease is known as the percolation threshold (Li et al. 2005). According to the standard GB 50174-2008, the surface resistivity of the antistatic floor in the computer room and its support area was 2.5 x 10⁴ to 1.0 x 10⁹ Ω (*i.e.* below the dotted line in Fig. 2). The surface resistivity of the RW-15 sample was $10^8 \Omega$, which could achieve the antistatic requirements of the floor in the electronic information system room and its support area. However, the surface resistivity of the RW-20 sample was $10^4 \Omega$, showing a dramatic decrease in comparison with that of the previous sample. These results indicated that the percolation threshold of the CB in regular WPC was between 15% and 20%, which could form continuous conductive networks to rapidly dissipate the charge, thereby decreasing its surface resistivity. Similar results were also observed in coextruded WPCs with added CB. Moreover, although the surface resistivity of the RW-20 sample was almost equal to that of the CW-20 sample, the dosages of CB added into the two structural WPCs were different. It can be seen from Fig. 1 that the thickness ratio of the shell to core layer in coextruded WPCs was 1:14. The dosages of CB in regular WPCs were much higher than those in coextruded WPCs under the premise of the same surface resistivity in the two structural WPCs. For instance, since the densities of the two structural WPCs were approximately equal, 71.1 g/m³ CB was required for coextruded WPCs while 476.8 g/m^3 CB was required for regular WPCs. Therefore, to achieve the similar antistatic properties of the two structural WPCs, the dosage of CB in regular WPCs was 6.71 times as much as that of the coextruded WPCs.



Fig. 2. Effects of CB contents on the surface resistivity of the two structural WPCs

Thermal properties

Figure 3 shows the endothermic heat flow peak for the two structural WPCs containing different CB contents. The eigenvalues obtained from the DSC curve are summarized in Table 2. The determination of the melting point (T_m) showed that the CB particles slightly influenced this parameter, which is caused by the change in lamellar thickness of the crystallites (Chodák 1998). However, this influence was not monotonic for coextruded WPCs. To a certain extent, CB was added to the matrix as a nucleating agent. The addition of CB narrowed the grain size of the crystal, and its crystals were more regular than before (Sever et al. 2013). Therefore, the crystals in coextruded WPCs with the addition of CB completed the melting process within a short range of temperatures, showing the $T_{\rm m}$, and the full width at the high maximum (FWHM, *i.e.*, $\Delta T_{\rm m}$) of the crystalline peak decreasing with the increase in the CB content. The onset melting temperature (T_{m-o}) , T_m , and $\Delta T_{\rm m}$ of the RW-0 sample were obviously decreased by 5.20 °C, 4.30 °C, and 2.45 °C in comparison with those of the CW-0 sample, respectively. The obtained results indicated that the presence of WF in the RW-0 sample led to the melting of the HDPE matrix in the smaller temperature range than the CW-0 samples (Yang et al. 2015). A decrease in the degree of crystallinity was observed with an increase in CB content in the case of the coextruded WPCs. The crystallinity of the CW-20 sample was decreased by 12.5% in comparison with that of the CW-0 sample, demonstrating that the addition of CB into the shell inhabited the crystallization of the HDPE matrix. This result also indicated that the CB particles represent defect centers, which impeded the folding of macromolecular chains, similar to crosslinking in polymers (Lazár et al. 1990). Simultaneously, the crystallinity of the HDPE matrix decreased greatly with the addition of wood flour, showing that the wood flour hindered crystallization. Also, the crystallinity of the RW-20 sample was the smallest value in all samples, showing that the combination of CB and wood flour hindered the crystallization of the HDPE matrix.



Fig. 3. DSC curves of coextruded WPCs with different CB contents (0%, 5%, 10%, 15% and 20%) and the two structural WPCs (coextruded WPCs and regular WPCs) with the same CB contents (0% and 20%)

Samples	T _{m-o} /°C	<i>T</i> _m /°C	∆7 _m /°C	$\Delta H_{\rm m}/({\rm J/g})$	Xc/%
RW-0	117.5	130.0	7.50	33.98	11.60
CW-0	122.7	134.3	9.95	105.40	35.97
CW-5	120.8	131.4	8.05	76.12	25.98
CW-10	120.7	131.3	7.70	72.55	24.76
CW-15	119.8	129.9	7.05	69.71	23.79
CW-20	119.6	130.5	6.90	68.66	23.43
RW-20	122.8	130.8	4.60	32.11	10.96

Table 2. DSC Parameters for the Two Different Structural WPCs

Section morphology

Figure 4 shows the section morphology of the RW-20 sample and the shell of the coextruded WPC when 0%, 10%, and 20% of CB was added. Its magnification is 1200 times. For coextruded WPCs, the shell section morphology of the CW-0 sample demonstrated the appearance of the filament (i.e., the arrow in Fig. 4). With the addition of CB, the shell section morphology of the CW-10 sample changed significantly, showing a certain interval of the lamellar structure. Also, it can be seen from Fig. 4 that the small shape of the lamellar structure in the section morphology of the CW-10 sample disappeared when the CB content increased to 20%. The lamellar structure in the section morphology of the CW-20 sample became larger, which can effectively transfer the static electricity generated by the HDPE shell through the free moving electrons and reduce the electrostatic effect on the surface of the composites. The CB can conduct electricity since the carbon atoms in the crystal structure of CB formed 3 covalent bonds (*i.e.*, a π bond), and each carbon was left with a free electron, which can transport electric charges (Yang et al. 2015). The addition of CB into the composites can form a conductive path within the composites to transfer the redundant charges, thereby achieving the purpose of being antistatic. This result was consistent with the surface resistivity of the samples decreasing with the increase

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in the CB content in the conductivity analysis. The section morphology of the CW-20 sample had a large lamellar structure and its surface was rough. The fractured burr of the shell HDPE was small, as indicated by the white arrow in Fig. 4. The section morphology of the RW-20 sample had a more obvious lamellar structure and its fractured burr was larger than those of the CW-20 sample. These results indicated that CB was mainly combined with the HDPE matrix in the three-phase system of CB/HDPE/wood flour, which formed a lamellar structure at the section and a conductive network, thereby dissipating the redundant charge (Breuer *et al.* 2015). In addition, it is only needed to build the conductive network in the shell HDPE matrix by adding CB to the coextruded WPCs; however, the addition of CB was much more in regular WPCs and thus met similar antistatic requirements.



Fig. 4. The section morphology of the coextruded WPCs added 0%, 10%, and 20% CB (CW-0, CW-10, CW-20), and the regular WPC added with 20% CB (RW-20)

Mechanical Properties

Figure 5 shows the mechanical properties (flexural strength, flexural modulus, and impact strength) of the two structural WPCs added with different CB contents. The flexural strength and flexural modulus of the CW-0 sample were higher than these of the RW-0 sample by 1.9% and 5.9%, respectively. The impact strength of the CW-0 sample was also increased by 18.8% in comparison with that of the RW-0 sample. These results were due to the HDPE shell withstanding part of the external stress to effectively hinder the diffusion of the stress-induced cracks (Zhang *et al.* 2005). As shown in Fig. 5, the addition of CB into the shell of coextruded WPCs can improve the mechanical properties of the composites. The flexural strength, flexural modulus, and impact strength of the CW-0 sample were increased by 21.6%, 10.8%, and 26.1% in comparison with those of the CW-0 sample, respectively. This result is due to the HDPE through the coupling agent with the effective combination of CB (Chen *et al.* 2017), which was also consistent with the inhabitation in

the crystallization of the HDPE matrix by adding CB into the shell in DSC analysis. In addition, the flexural strength and flexural modulus of the RW-20 sample was increased by 28.0% and 1.3%, respectively, in comparison with those of the CW-20 sample. This result is due to the silane coupling agent on the CB surface combined with the wood fiber and HDPE in the process of blending and melting, which led to the tight integration of each component (Sever *et al.* 2013; Gong *et al.* 2016). However, the impact strength of the RW-20 sample decreased by 14.7% in comparison with that of the CW-20 sample. This result was because the HDPE shell can effectively delay the diffusion of the stress-induced cracks, and thus, the regular WPCs were more brittle than the coextruded WPCs.



Fig. 5. Mechanical properties of the two structural WPCs (coextruded WPCs and regular WPCs)

CONCLUSIONS

- 1. The percolation threshold of the carbon black (CB) in regular wood-polymer composites (WPC) or coextruded WPC was between 15% and 20%, at which point the conductive filler can form continuous conductive networks to rapidly dissipate the charge, thereby causing the surface resistivity to decrease. The CW-20 sample had the lowest value of surface resistivity in coextruded WPCs and the RW-20 sample had the same results in regular WPCs, which can achieve the floor antistatic requirements of the electronic information system room and its support area.
- 2. The differential scanning calorimetry (DSC) analysis revealed that the crystallinity of the CW-20 sample decreased by 12.5% in comparison with that of the CW-0 sample, demonstrating the inhibition on the crystallization of the high-density polyethylene (HDPE) matrix by adding CB to the shell. This result is consistent with the large lamellar structure in the section morphology of the CW-20 sample, which formed a conductive network to eliminate the static electricity due to the uniform distribution of CB in the HDPE matrix.
- 3. The flexural strength, flexural modulus, and impact strength of the CW-20 sample were

increased by 21.6%, 10.8%, and 26.1%, respectively, in comparison with those of the CW-0 sample. This is due to the HDPE through the coupling agent with the effective combination of CB, and thus improving the mechanical properties of the composite.

4. Adding CB into the shell of coextruded WPC can improve its antistatic properties and simultaneously maintain its mechanical properties. Moreover, under the premise of the similar antistatic and mechanical properties of the two structural WPCs, the dosage of CB in regular WPCs was 6.71 times as much as that in coextruded WPCs.

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