Effect of Three Different Mineral Components on the Properties of Wood Flour/High-density Polyethylene Composites: A Comparison

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To investigate the properties of wood flour/high-density polyethylene composites (WF/HDPE) filled with different kinds of mineral components ($CaCO_3$, talc powder, and wollastonite), WF/HDPE was prepared *via* a molding method. The thermal properties of WF/HDPE were analyzed with a comprehensive thermal analyzer, and the mechanical properties were measured. The moisture resistance was indicated by 24 h water absorption. The results indicated that both the mechanical and physical properties of WF/HDPE samples were improved remarkably by adding $CaCO_3$, talc powder, or wollastonite. The tensile strength with talc powder was 83% higher than the composites without the additive. The maximum residual weight of the composites with added talc powder was 21.8% at 600 °C, which was 7.47% higher than that of the composites without additive. The composites with added talc powder exhibited better water resistance. Additionally, scanning electron microscopy (SEM) showed that the addition of talc powder enhanced the interaction between the components.

Keywords: WF/HDPE; Mineral components; Thermal properties; Water resistance

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INTRODUCTION

Wood-plastic composite material is a new environmentally friendly material that is based on wood fiber (*e.g.*, wood flour, bamboo flour, and rice husk powder, *etc.*) and plastic, and prepared by molding and extrusion. It is a composite material made from thermoplastic polymer plastic as the matrix and filled with wood fiber particles (Borah and Kim 2016). The wood powder and thermoplastics can be discarded by the forestry branch, old wood, sawdust, and plastic garbage recovery. Therefore, it is of great significance to reduce the environmental pollution and the utilization of resources (Kordkheili *et al.* 2013). A wood-plastic composite has the advantages of low density, low cost, high stiffness, and availability of renewable natural resources (Kaseem *et al.* 2015). It is widely used in outdoor flooring, garden building, furniture, automobile interior, and other fields. Wood flours also have some disadvantages, such as moisture absorption, quality variations, low thermal stability, and poor compatibility with the hydrophobic polymer matrix (Farsheh *et al*. 2011). Therefore, wood modification is needed to improve the weakness of wood and strengthen the properties of wood for attaining proper interfacial interaction between wood flour particles and the polymer matrices (Rahman *et al.* 2017).

Mineral particles can improve dimensional stability, electromagnetic and thermal stability, impact strength and cold resistance, modulus of elasticity, and tensile properties of composites (Fabiyi *et al*. 2009; Ghasemi and Farsi 2010). Composites with minerals as reinforcing phase have been the subject matter of many discussions in recent years. Due to the high improvement of the interfacial adhesion between the wood fibers and the plastic, it is ideal for reinforcement and enhancing the properties. Deka *et al.* (2014) examined the properties of the WF/HDPE with addition of nanoclay, SiO_2, and ZnO. SiO_2 and ZnO showed an improvement in mechanical properties, thermal stability, and a decrease in water uptake capacity. Tabar *et al.* (2015) using silicon dioxide (SiO_2) as reinforcement for walnut shell flour/HDPE composite materials, found that an increasing nano-SiO_2 loading (4 wt%) led to agglomeration and inefficient transfer of stress. Dey and Tripathi (2010) found that the thermal expansion coefficient of high-density polyethylene (HDPE) composites with silica powder (volume fraction of approximately 3% to 20%) increased linearly with the increase of temperature, and that a higher silica content resulted in a smaller thermal expansion coefficient of the composites. The Pan *et al.* (2014) study found that the addition of nanosilica can improve the thermal stability of a wood powder/HDPE wood-plastic composite. Devi and Maji (2013) studied the effect of nanometer titanium dioxide and silicon dioxide nanoclay on composite material physical properties of styrene/acrylonitrile wood; they found that two kinds of nanoparticles can improve the thermal stability of the composites, as well as the flexural and tensile properties of composite materials. Deka and Maji (2010) found that nanoclay can enhance the heat resistance of HDPE, low-density polyethylene (LDPE), polypropylene (PP), and polyvinyl chloride (PVC) hybrid wood-plastic composites. The reason is that silicate in clay plays a role in inhibiting the thermal decomposition of wood-plastic composites.

The present study aimed to better understand the effect of minerals on the thermal and mechanical properties of high-density polyethylene (HDPE) and wood flour (WF). The aim was also to study the effect of these particles on other properties such as water uptake capacity of the composites. In this paper, wood powder is used as filling material, HDPE is recycled as the matrix, and three kinds of minerals ($CaCO_3$, talc powder, and wollastonite) were added. The wood-plastic composite was prepared by a molding method. The effects of different minerals on the thermal and mechanical properties of WF/HDPE are considered, providing a theoretical basis for developing a high-performance composite material.

EXPERIMENTAL

Materials

Recycled HDPE was purchased from Nanjing Dongnan Tongye Co., Ltd. (Jiangsu, China). The poplar powder recovery was from Nanjing Yongxin Agricultural Waste Utilization Co., Ltd. (Jiangsu, China). Wood flour (WF) was chilled and ground in a hammer mill to pass through sieves measuring 100-mesh. The $CaCO_3$ powder (<800-mesh; Shanghai Huijinya Materials Co., Ltd., Shanghai, China), the talc powder (<800-mesh; Nanjing Chemical Material Co., Ltd., Nanjing, China), and the wollastonite (<800-mesh; Zhejiang Fenghong New Material Co., Ltd., Zhejiang, China) were used as received. Zinc stearate (industrial grade; PT. Nubika Jaya Co., Shanghai, China) was used

as a mixing lubricant. The maleic anhydrite (MA) was supplied by Nantong Rizhisheng Co., Ltd. (Jiangsu, China) and was industrial grade.

Composites Preparation

Before processing, all the materials were naturally dried, crushed over 100-mesh screens. Then the powder was dried with electric heat and constant temperature dryness in a box for 4 h at 110 °C to achieve a moisture content of less than 3%. To provide the same matrix for reinforcement of mineral components ($CaCO_3$, talc powder, and wollastonite), the amount of HDPE, MA, and lubricant, was held constant at 35 wt%, 3 wt%, and 2 wt%, respectively. Mineral components were used as reinforcing filler with loading levels at 6 wt% (Table 1). First, HDPE and MA were mixed in the mixer (X(S)K-160; Jiangdu Tianyuan Testing Machinery Co., Ltd., Jiangdu, China) at approximately 210 °C for 10 min. After that, MA grafted HDPE, WF, and mineral were added and mixed less than 5 min to prevent carbonization of the wood fiber. The compounded materials were then molded using a plate vulcanizing machine (QLB-20 D/Q; Jiangdu Tianyuan Testing Machinery Co., Ltd., Jiangdu, China) then placed into a mold at approximately 190 °C, and pressed under approximately 9 MPa for 8 min. The specimens were machined to the dimensions required for testing.

Table 1. Formulations of the Wood Plastic Composites Filled with Various Contents of Mineral (wt%)

WF	HDPE	MA	Lubricant	$CaCO_3$	Talc powder	Wollastonite
60	35	3	2	-	-	-
54	35	3	2	6	-	-
54	35	3	2	-	6	-
54	35	3	2	-	-	6

Methods

Mechanical property testing

All of the tests were performed with a universal testing machine (SANS CMT6104; Meitesi Industry System Co., Ltd., Shanghai, China). Tensile strength tests were carried out in terms of GB/T 1040.4 (2006) with a speed of 2 mm/min. Bending strength were tested according to the GB/T 9341 (2008) standard with the loading rate 2 mm/min. Impact strength tests were performed following the GB/T 1043.1 (2008) standard. All of the tests were conducted at room temperature, and the results were averaged 5 times.

Thermogravimetric (TG) analysis

A thermal analyzer (Nezsch STA 449 F3, Bavaria, Germany) was used for the TG and differential scanning calorimetric (DSC) measurements testing. The 8 mg specimens were sieved to obtain particles with a mean size of 2 mm under Ar protection. The heating rate was 10 °C/min from 30 °C to 600 °C.

Water absorption test

Specimens were conducted according to the GB/T 17657 (2013) standard. The specimens were entirely immersed in distilled water at 25 °C for 24 h. When the mass was almost unchanged, it could be regarded as a moisture balance. The weights of the

specimens before and after soaking were recorded. The water absorption (Q; %) was calculated using Eq. 1,

$$Q\ (\%) = [(M_2 - M_1) / M_1] * 100 \quad (1)$$

where M_1 and M_2 denote the oven-dried weight (g) and weight (g) at a given immersion time (t), respectively.

Scanning electron microscopy

The interfacial morphology of WF/HDPE was analyzed with a SEM (S-4800, Hitachi, Tokyo, Japan). The interfacial morphology of wood powder/HDPE wood composite materials with the addition of inorganic filler was observed and the surface of the composite material was sprayed with gold (Hitachi, Tokyo, Japan) at a voltage of 17 kV.

RESULTS AND DISCUSSION

Mechanical Properties of WF/HDPE

The mechanical properties of WF/HDPE were improved to different extents after the addition of mineral components.

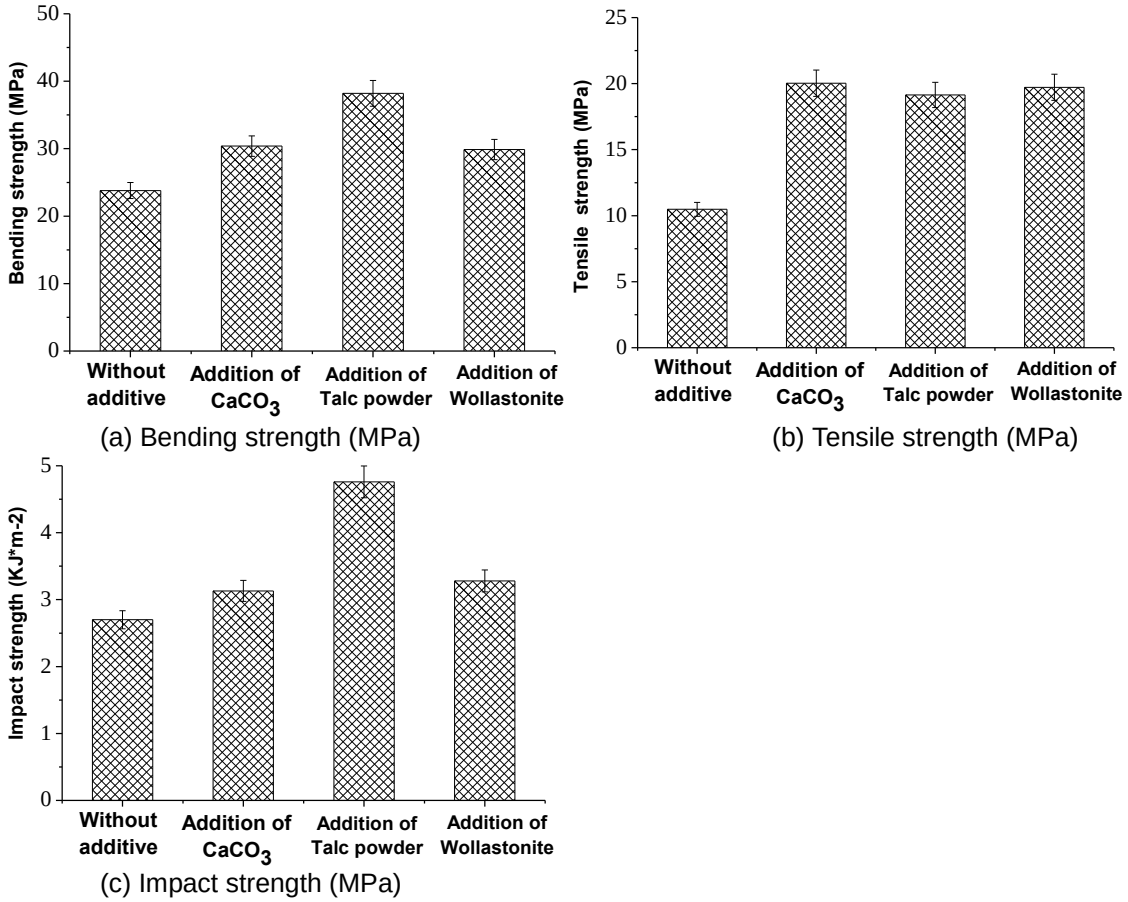


Fig. 1. Mechanical properties of WF/HDPE filled with different mineral

According to the results of the mechanical tests in Fig 1(a), the bending strength of WF/HDPE with talc powder led to the largest increase compared to others, the composites adding $CaCO_3$ showed the second largest increase, and the composites adding wollastonite showed the lowest increase. Specifically, the tensile strength, bending strength, and impact strength of WF/HDPE with talc powder was 83%, 61%, and 76%, respectively, higher than the composites without additive. Talc particles can be closely combined with the physical and chemical actions of HDPE macromolecular chains (Piekarska *et al.* 2016). The physical entanglement with macromolecules can inhibit the sliding of molecular chains when composite materials are subjected to external forces (Asgari *et al.* 2017). It can produce crazing when subjected to impact, and consume part of the impact energy to prevent crazing from developing into cracks, resulting in material failure; thereby such mechanisms can increase the impact toughness of composite materials. The mechanical properties of WF/HDPE with wollastonite increased the least compared to the others, which may be due to the extrusion pressure of wollastonite in the mixing process (Jia *et al.* 2009). As a result, the number of HDPE molecular chains and fiber molecular chains that intercalated wollastonite was reduced, and it was easier to break under the action of external force (Zolfaghari *et al.* 2013).

Thermal Stability of WF/HDPE

The TG and DSC thermograms for WF/HDPE are shown in Fig. 2. The weight loss of the four kinds of WF/HDPE were divided into three stages. In the first stage, the temperature was less than 260 °C, and the weight loss of the wood-plastic composite was smaller, which is mainly caused by the release of water and small molecular compounds (Fei *et al.* 2016). In the second stage (260 °C to 360 °C), the weight loss of WF/HDPE was more noticeable, and this was attributed to the thermal decomposition of the three major components of fiber (cellulose, hemicellulose, and lignin).

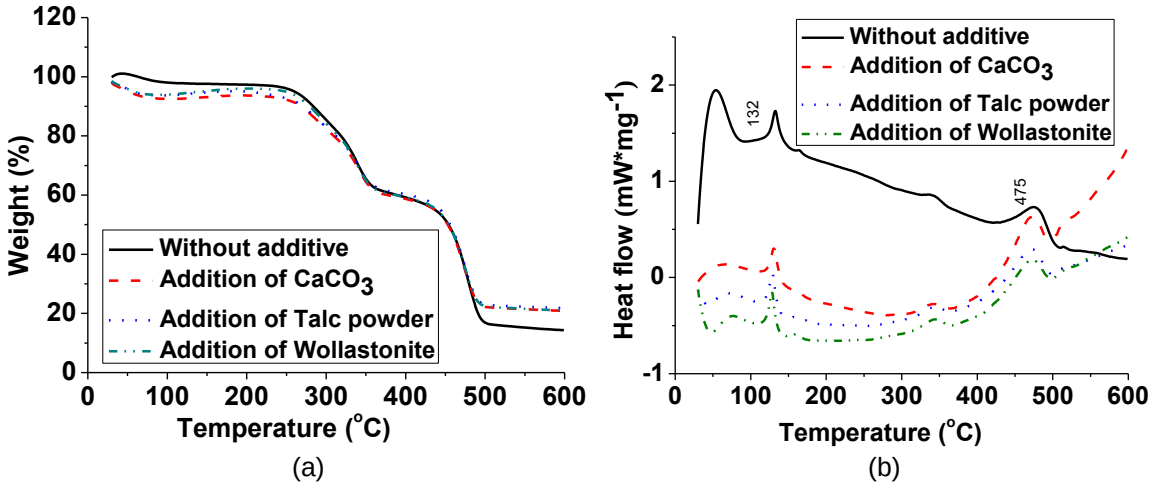


Fig. 2. (a) TG and (b) DSC curves of WF/HDPE filled with different minerals

The decomposition of the material at 380 °C to 450 °C was relatively slow, which indicated further thermal decomposition of lignin (Tufan *et al.* 2016). In the third stage (450 °C to 500 °C), the thermal decomposition of HDPE was produced and the fracture of carbon chain skeleton occurred in the HDPE molecular chain at 416 °C to 477 °C. The decomposition of the composite was basically completed above 500 °C (Zhou *et al.* 2018). The maximum residual weight of the composites that contained talc powder was

21.78% at 600 °C, which was 7.47% higher than that of the composites without additive. The residual mass was higher than that of the composite without additive, because the three kinds of mineral did not produce volatile products above 600 °C (Li *et al.* 2007).

There were first noticeable absorption peaks from 129 °C to 132 °C, and there was no obvious weightlessness in the TG curve, which indicated that the HDPE melted at this temperature range. A small absorption peak appeared at approximately 350 °C in the wood-plastic composite, and the corresponding TG curve was weightless, which may be due to the degradation of the hemicellulose and cellulose in the fiber content of the composites (Tufan *et al.* 2016). The third absorption peak appeared at 475 °C, and the corresponding TG curves entered the third weightlessness stage at the temperature range of 450 °C to 500 °C. The WF/HDPE with different minerals in the third stages of T_i (initial pyrolysis temperature), T_p (decomposition peak temperature), and T_f (end temperature of weightlessness) were increased (Table 2), which showed the higher enthalpy to dissociate their crystallinity when compared to the WF/HDPE without additive because some of the amorphous region of composites was converted to crystalline region. The WF/HDPE specimens with talc were the highest, which was attributed to the strong interaction between talc and the matrix molecules that reduced the mobility of HDPE chains and made the thermal decomposition of wood-plastic composites difficult (Lei *et al.* 2007).

Table 2. Thermogravimetric data of WF/HDPE Filled with Different Minerals

Type of Filler	Wood Fiber Decomposition			HDPE Decomposition			Residual Mass (%)
	T_i (°C)	T_p (°C)	T_f (°C)	T_i (°C)	T_p (°C)	T_f (°C)	
-	291.1	339.4	355.2	455.0	478.4	493.0	14.31
$CaCO_3$	295.6	339.4	355.5	451.4	473.9	488.4	20.82
Talc powder	289.1	340.1	356.4	450.5	474.3	488.8	21.78
Wollastonite	288.5	338.7	355.7	451.3	474.5	489.0	20.99

Effect of Different Mineral Components on the Moisture Absorption of WF/HDPE

Results for water absorption at different time intervals showed that water absorption values of WF/HDPE filled with different minerals were greatly reduced compared to the samples without additive (Fig. 3). This indicates that the incorporation of fillers resulted in a decreased wettability of the composites due to the decreasing amount of hygroscopic WF (Turku and Karki. 2014). The water absorption rate of $CaCO_3$- and wollastonite-filled composites was almost equal, and the water absorption rate of composites was approximately 47.9% lower than that without the additive. The particles were uniformly distributed in the composites, which had certain protective effects on the hydrophilic hydroxyl groups in the fibers, and the water could not penetrate into the composite material through the voids (Qin *et al.* 2004). The water absorption rate of WF/HDPE with talc powder was obviously smaller than that of the wood-plastic composite without additive, and the water absorption rate of composites was approximately 54.9% lower than without additive. This indicates that the lamellar talc played a certain role in covering the wood flour, reducing the contact area between wood powder and water, and thus reduced the water absorption of composites (Nourbakhsh *et al.* 2010).

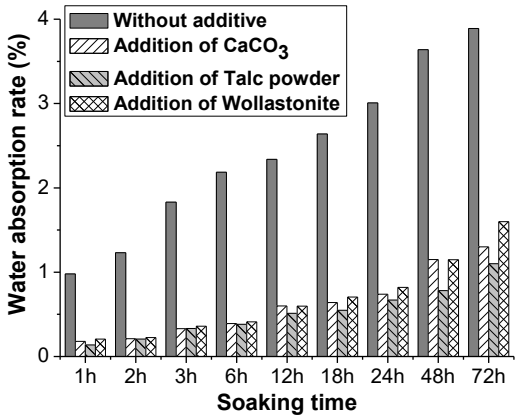
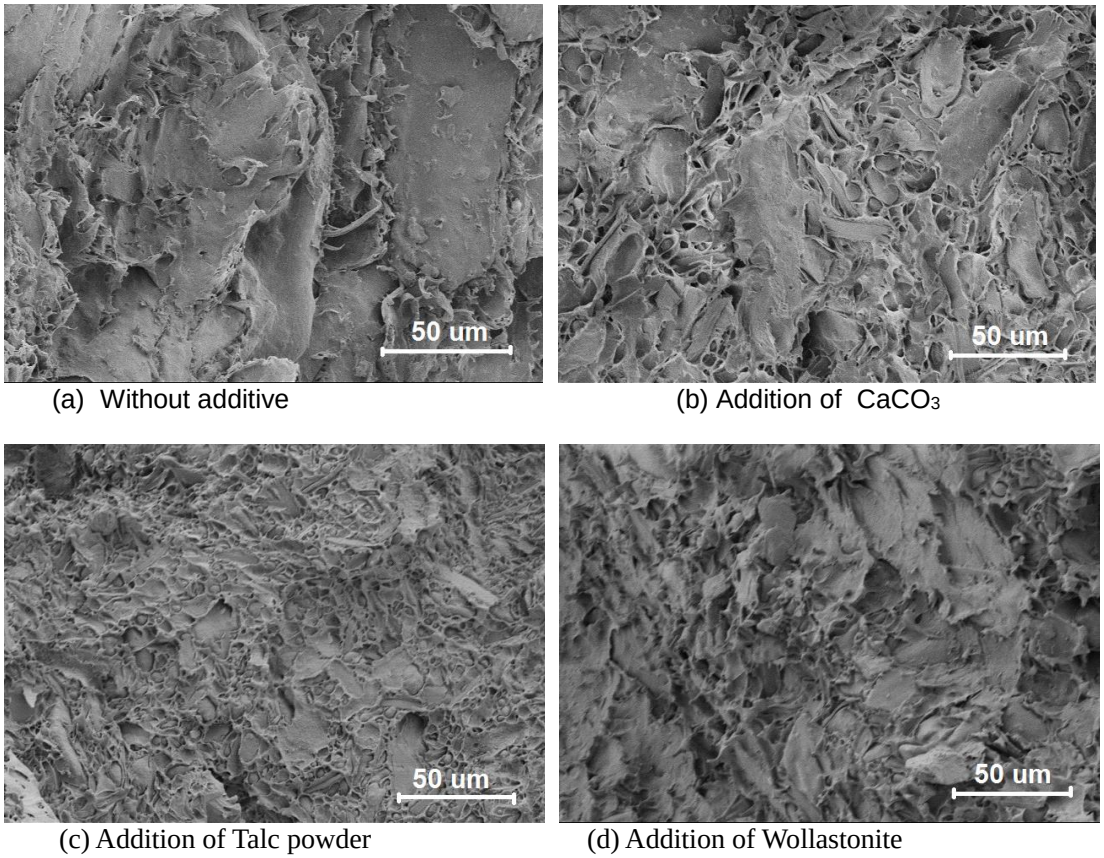


Fig. 3. Water absorption rate of WF/HDPE filled with different mineral components

Microstructure of WF/HDPE Filled with Different Mineral Components Content of Minerals



(a) Without additive (b) Addition of $CaCO_3$

(c) Addition of Talc powder (d) Addition of Wollastonite

Fig. 4. Microstructure of WF/HDPE filled with (a) without additive, (b) $CaCO_3$, (c) talc powder, and (d) wollastonite

Figure 4 shows the SEM micrographs of the fracture surface for WF/HDPE filled with different mineral components after mechanical testing. Figure 4(a) shows that the impact section of WF/HDPE without additive appeared hollower, which suggests that some cavities in the surface can absorb water or reduce mechanical properties. This

indicates that the level of interfacial bonding between the WF and HDPE in the composites without additive is weak (Tabar *et al*. 2015). Figure 4(b) illustrates that the impact cross-section of WF/HDPE with $CaCO_3$ had good smoothness, and there was a small amount of $CaCO_3$ granule agglomeration, so that calcium carbonate particles were evenly dispersed in the composite material and compatibility became better, which resulted in improved mechanical properties. Figure 4(c) shows that no fiber was pulled out, which indicated a stronger adhesion between the talc particles and the HDPE matrix, which is consistent with the measured higher tensile and flexural strength for the composites containing 6% talc particles (Hamed Mosavian *et al.* 2012). Figure 4(d) illustrates that the impact section of the composite material was rough, and that many interpenetrating fibers formed on the section of the system. It is difficult for the wollastonite particle to be fully compatible with the WF/HDPE matrix, so the improvement of mechanical properties is not obvious (Ou *et al.* 2014). Thus, it can be seen that the WF/HDPE with talc powder had optimal impact properties. The voids formed in the impact section of the wood-plastic composite with talc powder were less, which may have been due to talc powder having a layered structure and electric charge; it also has a lubrication effect on the matrix (Li *et al.* 2018). Therefore, the interaction between the minerals and polymer chains forming a physical cross-linking point to improve the toughness of WF/HDPE (Tabari *et al*. 2011).

CONCLUSIONS

1. Three kinds of mineral components ($CaCO_3$, talc powder, and wollastonite) improved the mechanical properties of WF/HDPE. Among them, the optimal mechanical properties of composites were those with the addition of talc powder. The tensile strength, bending strength, and impact strength of WF/HDPE with talc powder were 83%, 61%, and 76%, respectively, higher than the composites without additive.
2. The three kinds of mineral components increase the crystallinity of WF/HDPE. Among them, the effect of adding talc powder was the most noticeable, the maximum residual weight of the composites was 21.8% at 600 °C, which was 7.47% higher than that without additive.
3. The three kinds of mineral components reduced the moisture absorption. The water absorption rate of $CaCO_3$ and wollastonite filled composites was almost equal, and the water absorption rate of composites was approximately 47.9% lower than that without additive. The water absorption rate of WF/HDPE with talc powder was obviously smaller than that of the composites without additive, and the water absorption rate of composites was approximately 54.9% lower than without additive.

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