The Effect of Graphite on the Water Uptake, Mechanical Properties, Morphology, and EMI Shielding Effectiveness of HDPE/Bamboo flour composites

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This goal of this work was to study the effect of graphite on the water uptake, mechanical properties, morphology, and electromagnetic interference shielding effectiveness (EMI SE) of HDPE/Bamboo flour composites using the material mechanical testing machine, scanning electron microscopy (SEM), and EMI shielding apparatus. The water uptake of the composites was improved by graphite. Compared with the neat HDPE/bamboo composites, the flexural strength of the graphite-filled composites showed a small decrease, but the flexural modulus was enhanced greatly, indicating that graphite could effectively elevate the stiffness of the composites. An obvious result was that the toughness of composites was improved considerably by the graphite. The notched impact strength of the composites was increased from 5.18 to 9.0 KJ/m^2 as graphite content was increased from 0 to 40 %. A large amount of graphite would increase the conductivity property and the EMI SE. The HDPE/bamboo composite with 40% of graphite exhibited electrical resistivity of 31.2 Ω.cm and the EMI SE of 20 dB in the frequency range of 30 - 3000 MHz.

Keywords: HDPE; Bamboo; Graphite; Water uptake; EMI SE

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INTRODUCTION

Wood-polymer composites (WPC), based on wood, bamboo flour, and other plant fillers and polymer, came into being in the 1960s (Chavooshi and Madhoushi 2013). Due to the low cost, easy processing and environment friendly character, WPC had obtained attention all over the world, and these merits have led to wide application of WPC in outdoor and indoor construction, as well as in the automobile industry (Segerholm *et al.* 2012; Fang *et al.* 2013; Binhussain and El-Tonsy 2013; El Mansouri *et al.* 2012; Wu *et al.* 2013; Yu *et al.* 2014).

In order to further improve the WPC product’s additive value, WPC materials should be endowed with special functions. Conductive WPC materials are regarded as a promising development direction in the future. If conductive, WPC materials could be used in antistatic materials and electromagnetic shielding materials. Unfortunately, unmodified WPC tends to be a good insulator. This means that some conductive filler, such as carbon black, graphite, carbon nanotubes, or metal powder, should be incorporated into WPC to elevate the conductivity.

Along with the electrical and electronic equipment entering into people's lives, electromagnetic pollution came into being (Sapurina *et al.* 2005; Liang *et al.* 2013).

Research has confirmed that electromagnetic pollution could leak out information, interrupt other apparatus operation, and damage people's health. The solution to this problem is to develop electromagnetic shielding materials. It was reported that materials with high conductivity usually exhibit excellent electromagnetic shielding effectiveness; therefore, all kinds of conductive fillers have been incorporated into polymer, such as graphite, carbon black, carbon nanotubes, carbon fiber, and semiconductors. Among these conductive fillers, graphite was deemed as having better commercial prospects due to its very low cost and high conductivity. Research indicated that PE and PP composites with 15% of graphite exhibited the optimal EMI SE of 16 dB and 11 dB in the range 20 to 40 GHz (Volkov *et al.* 2010). But Panwar *et al.* (2010) fabricated the PP/graphite composites using a melt mixing method, and the maximum value of EMI SE was measured to be as high as 44.12 dB at 2.76 GHz for the composites with 25% of graphite. Higher EMI SE was found for ABS/composites with 15% of graphite (fabricated using a long mixing time and the pressure of 75 MPa) where the EMI SE was measured to 60 dB in the range 8 to 12 GHz (Sachdev *et al.* 2011). Other research involving polyaniline/graphite composites with 90% graphite showed EMI SE values of only 28 dB or so (Saini *et al.* 2009). To the best of our knowledge, no article with respect to graphite-based WPC in the field of electromagnetic shielding has been published, especially in the low frequency range (< 3 GHz).

Consequently, in light of the developing trend of WPC and the requirement of electromagnetic shielding materials, in this paper, HDPE/bamboo/graphite composites were produced using a melt-blending method, and the effect of graphite on EMI SE of the composites was studied. Also, the water uptake, mechanical properties, and morphology of HDPE/bamboo/graphite composites were investigated in detail.

EXPERIMENTAL

Materials and Preparation

High-density polyethylene (HDPE) (DMDA-8008) was bought from the Dushanzi Company of Petro China Co. Ltd. Ethylene acrylic acid copolymer (EAA) (9005), containing 6.5 wt% of acrylic acid content, is produced by the Mitsui Corp, Japan. Bamboo flour, 60 mesh, was bought from the Ling'an Mingzhu Wood Flour Co. Ltd., China. Natural graphite, with size of 2.8 to 40 μm and the specific surface area of 9609.5 cm^2/cm^3, was produced by the Qingdao Golden Day Graphite Co. Ltd. China. The morphology of graphite is shown in Fig. 1. It is clear that the graphite was composed of many graphite layers, as indicated by the white arrows. Before use, all the components were dried at 70 ^oC in a vacuum oven for 12 h.

The melting blending method described in the literature (Dányádi *et al.* 2007; Wang *et al.* 2011; Bledzki *et al.* 2012; Kazemi-Najafi and Englund 2013; Kordkheili *et al.* 2013) was adopted to fabricate homogeneous HDPE/bamboo/graphite composites using an internal mixer (the Changzhou Suyan Technology Co., Ltd, China)

Firstly, HDPE, graphite, EAA, and bamboo powder were added into the internal mixer and were compounded at 175 ^oC for 10 min with the speed of 40 rpm, and then the mixtures were taken out and smashed using a plastic mill (produced by the Tianjin City Test Instrument Co., Ltd. China). Finally, the HDPE/graphite/bamboo/EAA composites were injected to the specimens (for flexural strength and impact strength measurement) by using an injection molding machine (Ningbo Haiying Plastics Machinery

Manufacturing Co., Ltd. China) according to the ISO standards 178:2001 and 180:2000. The injection temperature and pressure were 175 °C and 90 bars, respectively.

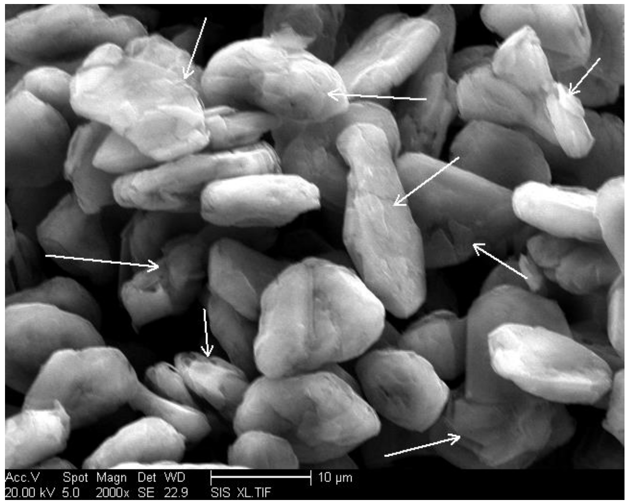

Fig. 1. SEM image of natural graphite

In order to study the effect of graphite content on the electrical resistivity and EMI SE of the composites, in the present experiment, the bamboo content and EAA content were controlled to 30% and 5%, respectively. When the graphite content in the composites increased from 0 to 40%, the HDPE content changed from 65% to 25%. All component contents are listed in Table 1.

Table 1. Components and Electrical Resistivity of Composites

	HDPE %	Bamboo %	Graphite %	EAA %	ρ (Ω.cm)
1	65	30	0	5	＞40000
2	55	30	10	5	＞40000
3	45	30	20	5	＞40000
4	35	30	30	5	626.4
5	25	30	40	5	31.2

Characterization

Viscosity of composites

The viscosity of the HDPE/bamboo/graphite composites was measured using a Rotary Rheometer MARS III, produced by the Thermo Fisher Scientific Corporation, America. The data was collected from 1 Hz to 100 Hz, and the temperature and shear stress were fixed to be 175 °C and 300 Pa, respectively.

Water uptake

The water uptake experiments were performed on the HDPE/bamboo/graphite composites. Before measurement, the weight of each specimen was measured. Specimens were soaked in distilled water at room temperature for 96 h. Then the specimens were taken out and water was removed from the specimen surface using a dry cloth. Finally, the weights of specimens were measured again. Each value obtained represented the average of 5 specimens. The water uptake was calculated by the following equation,

$$W = [(W_2 - W_1)/W_1] \times 100\% \quad (1)$$

where W was the water uptake, W_1 was the weight of the specimen before water treatment, and W_2 was the weight of the specimen before water treatment.

Mechanical properties

The flexural properties of the composites were measured using a Material Mechanical Testing Machine (the Shenzhen SANS Company of China). The specimens were tested at a crosshead speed of 10 mm/min at 20 °C. The notched impact strength of composites was evaluated by means of an Impact Testing Machine ZBC-25B (the Shenzhen SANS Company of China). All the reported values for the tests were the average values of 5 specimens for each group.

SEM observation

The morphology of fracture surface of the composites were carried out using a Scanning Electron Microscope (SEM, QUANTA 200, FEI corporation) with an accelerating voltage of 20 kV. Firstly, the composites were dipped into liquid nitrogen and fractured, and then the fracture surface was coated with gold particles.

Electrical resistivity

The electrical resistance R (Ω) of the HDPE composites was determined using a Milliohmmeter VC480C+, produced by the Shenzhen City Vichy Technology Co. Ltd. China. The electrical resistivity, ρ (Ω.cm), was calculated using the following relation:

$$\rho = R \times S/L \quad (2)$$

where L is the sample thickness (L= 0.2 cm) and S (cm^2) represents the surface area of the electrodes (S=5 cm^2).

EMI properties

EMI shielding measurements were conducted at room temperature over a frequency range of 30 MHz to 3000 MHz using an Electromagnetic Shielding Measuring Instrument (DR-S02). The sample was pressed into a disc with 12 cm of outer diameter and 1 mm of thickness using a molding machine with 170 °C and 10 MPa for 10 min.

RESULTS AND DISCUSSION

Viscosity

Figure 2 shows the variation of viscosity of the HDPE/bamboo composites as a function of graphite content. It was clear that the HDPE/bamboo composite presented the highest viscosity in all composites at a given frequency. Importantly, the viscosity of composites at first quickly started to decrease when 10% of graphite was added. This effect was attributed to the small amount of graphite in matrix, which weakened the intermolecular forces among the components. However, when graphite content was increased to 20%, the viscosity of the composites increased and reached the maximum value (except for neat HDPE/bamboo composite), indicating that the intermolecular force was enhanced and that the movement of polymer chains became more difficult than before. This was ascribed to the formation of conductive graphite network and the dispersion of graphite (Al-Hartomy *et al.* 2011). After that, the viscosity of the composites linearly decreased as graphite content increased further, and the minimum viscosity of the composite was observed for the composite with 40% of graphite. Such a case could be related to the layer structure of graphite, and the fact that adjacent graphite layers were easy to slide under the influence of an external force.

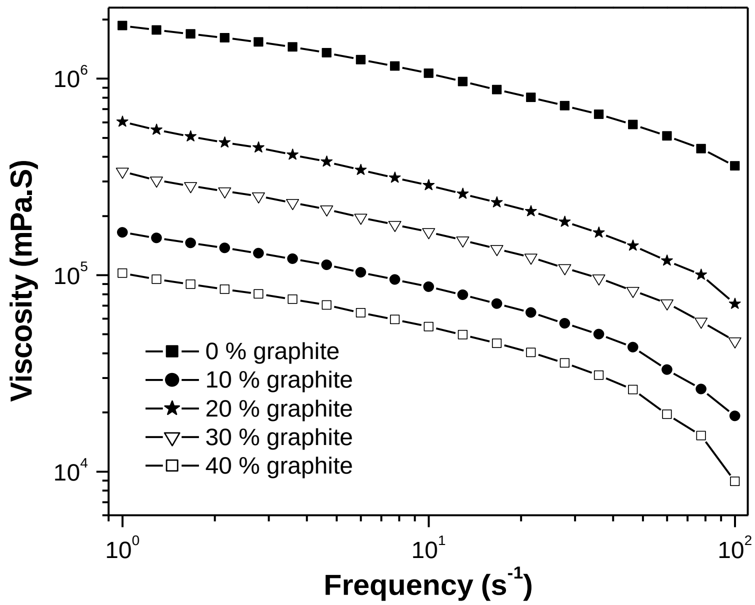

Fig. 2. Viscosity of the HDPE/bamboo/graphite composites with different graphite contents

Water Uptake

Figure 3 shows changes of the water uptake of the composites as a function of graphite content. By nature, the HDPE is hydrophobic and exhibits very low water uptake, but when bamboo flour was added into the polymer matrix, the water uptake of neat HDPE/bamboo composite was observed to increase to 0.95% due to many polar groups in the structure, such as –OH and –C=O groups. Once graphite appeared in the matrix, the water uptake of the composite was observed to drop to 0.59% for the composite with 10% of graphite. As graphite content was increased to 20%, the water uptake of the composite reached a maximum value of 1.1%; this was attributed to the increase in void quantity due to the poor interface between the HDPE and graphite (as shown in SEM section). After that, the water uptake of the composites dropped as graphite content was further increased. The minimum value of the water uptake of the composite was found to be 0.40 % at 40 % graphite; this was attributed to the hydrophobic nature of graphite (Du *et al.* 2008) and the reduction in the number of voids. The very low viscosity of the composites allowed the HDPE to fill voids more easily. The reduction in voids was also confirmed by the decrease in electrical resistivity with increasing graphite content (in the following Electrical Resistivity section). Once the number of voids began to increase, the electrical resistivity of composites should increase (Thongruang *et al.* 2002). Such a result suggested that graphite could effectively improve the water uptake property of HDPE/bamboo composites.

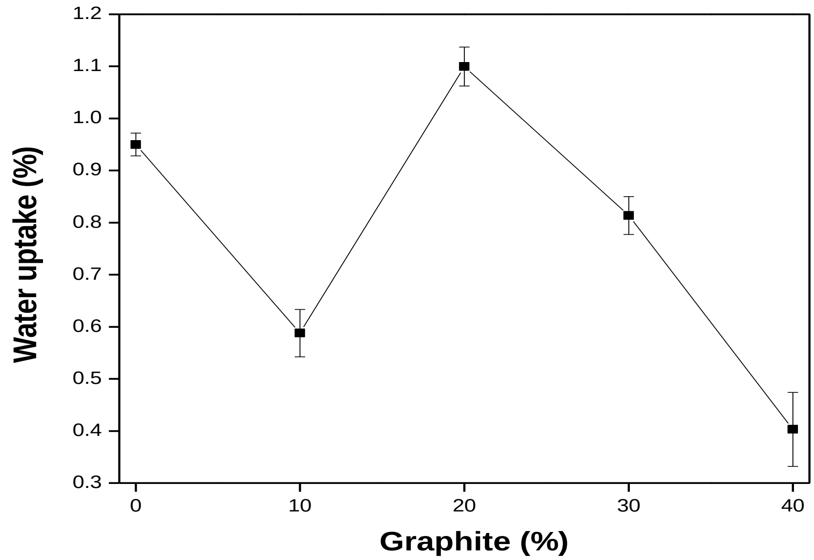

Fig. 3. Variation of the water uptake of the HDPE/bamboo/graphite composites as a function of graphite content

Mechanical Properties

In order to evaluate the effect of graphite on the mechanical properties of the composites, the flexural strength, flexural modulus, and notched impact strength of the composites were carried out, and the results were shown in Figs. 4 and 5. Figure 4a exhibits the changes of the flexural strength and flexural modulus of the composites as a function of graphite content. The neat HDPE/bamboo composites presented the flexural strength of 38.8 MPa, but when graphite was added, the flexural strength of the composite was observed to drop to 35.5 MPa at 10% of graphite. Such a phenomenon was also observed in the HDPE/expanded graphite composite and was ascribed to the poor interface and the aggregation of graphite (Yasmin *et al.* 2006; Király and Ronkay 2013). However, as graphite content increased to 20%, the flexural strength of the HDPE/bamboo/graphite composites also reached the maximum value of 37.8 MPa. Such an increase has been ascribed to the formation of graphite network and the strong interface adhesion (Al-Hartomy *et al.* 2011).

As mentioned above, we attributed the high water uptake of the composite with 20% of graphite to the presence of voids, and it was understood that the voids would in turn lead to the decrease in flexural strength of the composites. However, the flexural strength of composite with 20% of graphite was the highest; the reason for this was due to the reinforcing effect of graphite network, which overcame the weakening effect of voids. At the same time, strong intermolecular forces also enhanced the composites. When graphite content was increased further, the flexural strength of the composites exhibited a large decrease, which was ascribed to the sliding of graphite layers.

The flexural modulus of the composite with graphite content is shown in Fig. 4b. Different from the flexural strength, the flexural modulus of the composites increased from 2.2 GPa to 4.3 GPa when graphite content was increased from 0 to 20 %. Compared with the neat HDPE/bamboo composite, the flexural modulus of the composites was elevated almost two times. As graphite content exceeded 20%, the flexural modulus of the composites began to show a small decrease, but it was still far higher than that of neat

HDPE/bamboo composite. Such a result indicated that the stiffness of HDPE/bamboo composite was improved greatly by graphite.

Figure 5 displays the notched impact strength of composites with different graphite content. The impact strength of HDPE/bamboo composite was determined to be 5.18 kJ/m^2, which was far lower than that of neat HDPE. The addition of 10% graphite did not improve the toughness of composites; in contrast, it caused impact strength to decrease to 4.8 kJ/m^2. When the graphite content continued to increase, the impact strength of the composites was observed to increase as well, and it reached a maximum value of 9.0 kJ/m^2 (composite with 40% of graphite), increasing 73% above the HDPE/bamboo composite. The reason for this was due to the characteristics of graphite. Graphite is a kind of multilayer structure material, and there is only Van der Waals' force among layers maintaining such multilayer structure. These traits allow graphite layers to move easily and absorb energy under the external force. As a result, the impact strength of the composites was found to increase as graphite content increased. Another phenomenon should be noted that within 20% graphite, the improvement in impact strength was not obvious, but once graphite content exceeded 20%, the impact strength quickly increased and exceeded that of the neat HDPE/bamboo composite, and this is possibly related to the formation of graphite networks. When a small amount of graphite was added into HDPE/bamboo matrix, graphite particles were situated far from each other and did not form effective network; at the same time, the rigid bamboo flour surrounding graphite particles also inhibited graphite from deforming under external force, so the impact strength of the composite did not show any obvious increase. But when graphite content exceeded 20%, a contiguous graphite network started to form, determining the physical properties of the composites, and thus the impact strength was observed to increase substantially. Additionally, the flexural modulus of the composites started to decrease above 20% graphite, which is due to graphite's characteristic of easy sliding.

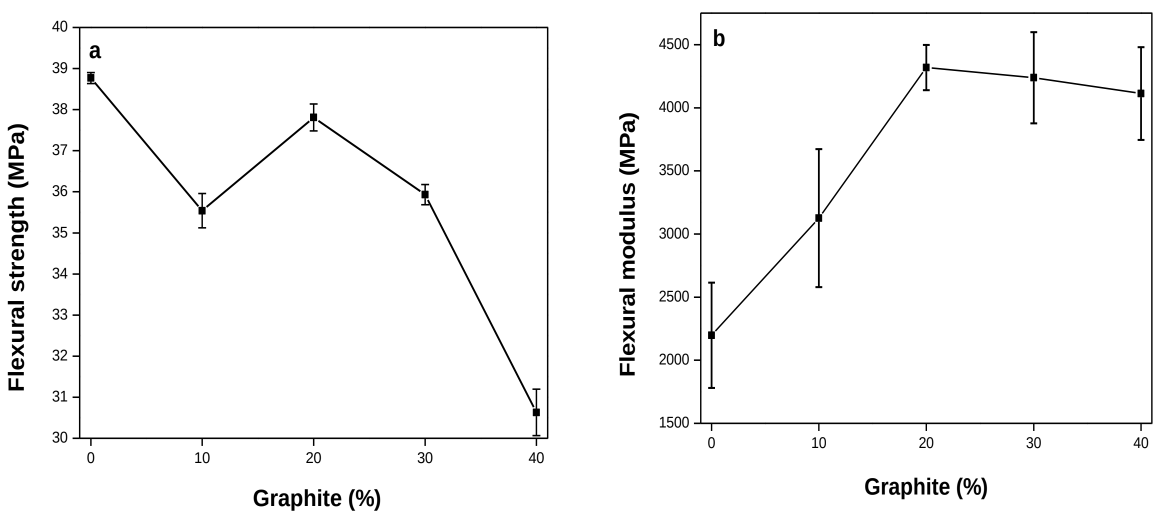

Fig. 4. Changes of the flexural strength (a) and flexural modulus (b) of the HDPE/bamboo/graphite composites as a function of graphite content

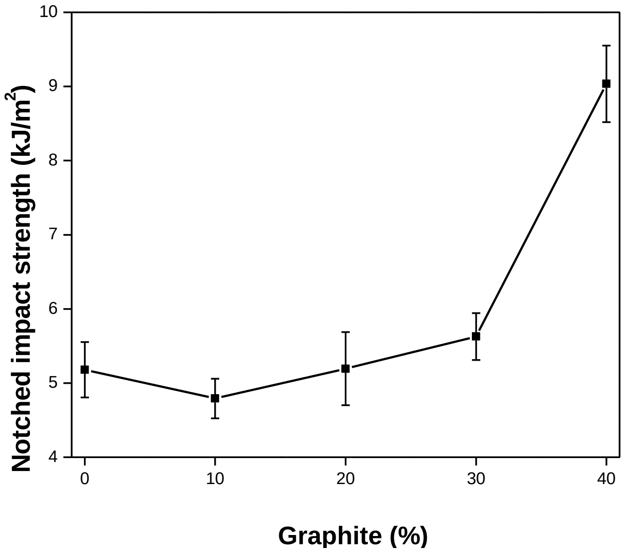

Fig. 5. Changes of notched impact strength of the HDPE/bamboo/graphite composites as a function of graphite content

Morphology

Through the SEM study, the distribution of graphite and compatibility between the graphite and HDPE matrix could be observed (Fig. 6).

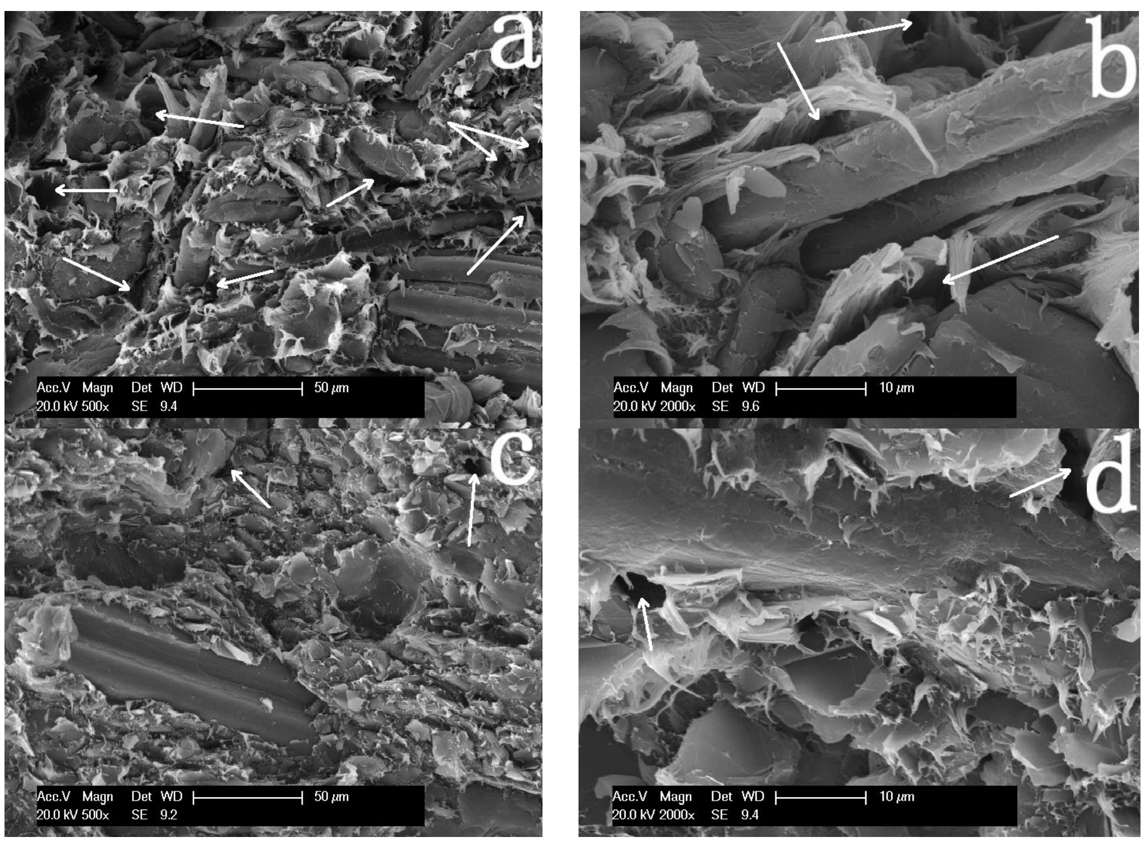

Fig. 6. SEM images of the HDPE/bamboo/graphite composite: (a) 20 % of graphite 500×; (b) 20 % of graphite 2000×; (c) 40 % of graphite 500×; (d) 40 % of graphite 2000×.

As can be seen from Fig. 6, there were considerable differences between the composites with 20% and 40% of graphite. It was found that the interface between the graphite and HDPE in Fig. 6a was clearer than that in Fig. 6c, indicating that the interface in 20% of graphite composite was poorer, and in such case led to the formation of void, as marked by the white arrow. Without doubt, these voids would cause water uptake to increase, and this was in agreement with the above results from the water uptake. In contrast, in Fig. 6c, the graphite could disperse uniformly in the HDPE phase and were almost wrapped well by the polymer, and the interface between the graphite and HDPE was very obscure. Also, the number of voids was obviously reduced, which meant that the composite with 40% of graphite exhibited lower water uptake. Concurrently, the massive graphite in the matrix began to form a dense graphite network, and it was expected that the electrical resistivity and mechanical properties of the composites should be improved (Al-Hartomy *et al.* 2011). In Fig. 6 b and d, the surface of bamboo flour was coated with a layer of resin; this indicated that the coupling agent of EAA could effectively improve the compatibility between the bamboo flour and HDPE. A related study has been reported in literature in which the graphite particles were found to contact with each other and to form conductive networks in the HDPE/graphite composites (Thongruang *et al.* 2002).

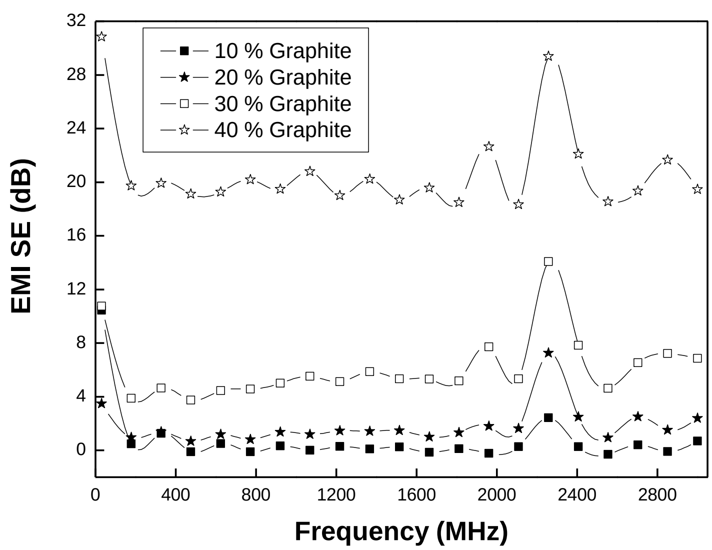

Fig. 7. The EMI SE of HDPE/bamboo/graphite composites as a function of graphite content

Electrical resistivity and EMI SE

It is known that the WPC is a good insulator by nature, so conductive fillers should be added to improve the conductivity. In our experiments, graphite was selected as a conductive filler since it is cheap and exhibits good conductivity. Table 1 shows the electrical resistivity of the HDPE/bamboo/graphite composites. Because the resistance values of the composites were still high and out of range of the apparatus, the electrical resistivity of the composites having less than 20% graphite could not be obtained. As graphite content increased to a key weight fraction (20%), the electrical resistivity dropped abruptly due to the formation of a conductive graphite network (conductive path). The composite became electrically conductive. This key weight fraction was defined as the electrical percolation threshold. It was determined to be 626.4 Ω.cm for 30% of graphite composite, which was lower by at least 64 times than that of the composites with

20% and 10% of graphite. When the graphite content reached as high as 40%, the electrical resistivity continued to decrease and reached a minimum value of 31.2 Ω.cm. This meant that more and more conductive paths were produced by graphite. Similar results were also found in the LDPE/graphite composites and the HDPE/graphite composites, where the electrical resistivity values were achieved as 3.8×10^{-3} Ω.m and 1.2 $\times10^{-3}$ Ω.m at 40 vol% of graphite or so, respectively (Krupa *et al.* 2004). Also, it was reported the graphite/poly(urethane acrylate) composites, fabricated by *in-situ* polymerization, presented the electrical resistivity of 20 Ω.cm when graphite content was 40 wt% (Otieno and Kim 2008).

The EMI SE is used to evaluate the ability to reflect and absorb electromagnetic wave, and it was found that the EMI SE was dependent on electrical resistivity. Usually, a specimen with high electrical resistivity will exhibit excellent EMI SE. The EMI SE of the specimens was calculated according to the following equation,

$$SE = 10\log\frac{P_0}{P_t} \tag{3}$$

where P_0 is the incident electromagnetic power and P_t is the transmitted electromagnetic power. In general, the EMI SE is expressed in decibel (dB) units. For example, if the EMI SE is equal to 20 dB, it means that 99% electromagnetic wave power was shielded.

Figure 7 presents the EMI SE of the composites with different graphite contents in the frequency range from 30 MHz to 3000 MHz. It was observed that the EMI SE of the composites increased as graphite content increased at a constant frequency range. The neat HDPE/bamboo composites did not present the EMI function (not shown in Fig. 6). Owing to the high electrical resistivity, composite with 10% and 20% graphite exhibited poor EMI SE (not more than 4 dB). Similar to the electrical resistivity, the EMI SE was found to increase when the graphite content exceeded 20%. The highest EMI SE appeared at the 40% of graphite composite and was determined to be 20 dB or so over the frequency range of 30 to 3000 MHz, especially at about 2250 MHz, the EMI SE for the 40% of graphite composite was up to 29 dB, and this result was higher than that of PP/CNT composites (Kim *et al.* 2013). Importantly, the EMI SE of composites did not present linear changes with graphite content. When graphite content exceeded 20%, the EMI SE of composites increased greatly, which was indicative of the EMI SE percolation threshold. The reason for this is because the graphite started to contact each other and form the graphite network. Such a conclusion was in agreement with results of electrical resistivity and mechanical properties. These results indicated that the 40% of graphite composites had a good market prospect in the electromagnetic shielding field.

CONCLUSIONS

The HDPE/bamboo/graphite composites were prepared by a melt-blending method, and the influence of graphite on the water uptake, mechanical properties, and viscosity of the resulting composites were investigated. Some meaningful results were obtained, as follows:

1) The water uptake of the HDPE/bamboo/graphite composites at first decreased with increasing graphite content and reached a maximum value when the graphite

content got to 20%, and then it in turn decreased as graphite content increased from 20% to 40%.

2) Like water uptake, the composites exhibited the highest flexural strength for composites with 20% of graphite, and then it decreased as graphite content increased. The flexural modulus of composites was found to increase as graphite content at below 20% graphite, after that it show a little decrease. The toughness of composite was improved largely by graphite. It was observed that the notched impact strength of the composites reached a maximum value of 9.0 kJ/m^2 at 40% of graphite, which was far higher than that of the composite without graphite (5.18 kJ/m^2)

3) The electrical resistivity of the composites decreased as graphite content increased, which caused the EMI SE of the composites to increase with graphite content. The 40% of graphite composite exhibited electrical resistivity of 31.2 Ω.cm and the EMI SE of 20 dB in the frequency range of 30 to 3000 MHz.

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