

# Rheological properties, morphology, mechanical properties, electrical resistivity and EMI SE of cyclic butylene terephthalate/graphite/carbon black composites

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**Abstract** According to traits of cyclic butylene terephthalate (CBT) oligomer polymerizing into poly(butylene terephthalate) (PBT), the composites with high electromagnetic interference shielding effectiveness (EMI SE) were obtained based on CBT oligomer, graphite (GR) and carbon black (CB). The effects of GR and CB on rheological properties, morphology, mechanical properties, and electrical resistivity and EMI SE of PBT composites have been investigated using rheological rheometer, scanning electron microscope (SEM), material mechanical testing machine and electromagnetic shielding measuring instrument, respectively. The synergistic action of GR and CB was observed, which made PBT/GR/CB composites exhibit good mechanical properties, low electrical resistivity, and high vicat softening temperature (VST). Also, the incorporation of CB into PBT/GR composites improved largely EMI SE of composites, and the optimal EMI SE was determined to be ~60 dB over the frequency range of 30–3,000 MHz.

**Keywords** CBT · Graphite · Carbon black · EMI SE

## Introduction

With more and more electrical and electronic equipments entering human life, the electromagnetic pollution, following water, air and noise pollutions, starts to influence the people's health as well as interrupt the equipment and leak out

important information. As a consequence, considerable investigation had centered on the development of electromagnetic shielding materials [1–4]. Among EMI materials, metal-based materials are a good candidate due to high conductivity and strong ability to reflect the electromagnetic wave, but the traits of high cost and easy corrosion limit its application in EMI fields. The polymer composite is deemed to be promising EMI materials due to its low cost, resistance to corrosion and easy processing; however, unfortunately, the insulating trait of polymer makes polymer incorporated a large amount of conductive fillers so as to improve conductivity of materials [5, 6]. King et al. showed that 15 wt% of exfoliated graphite nanoplatelets caused the electrical resistivity of polycarbonate (PC) to decrease to be  $2.79 \times 10^4 \Omega \cdot \text{cm}$  [7]. Generally speaking, the materials with low electrical resistivity and good magnetic properties usually exhibited excellent EMI SE [8]. Zhang et al. [9] presented that the carbon fiber reinforced composites exhibited EMI SE of 25 dB in the frequency range from 30 to 1,200 MHz. Ameli et al. [10] presented that foamed polypropylene/carbon fiber composites with 10 vol% of carbon fiber exhibited EMI SE of 25 dB in X band. But from a commercial point of view, the addition of carbon nanotubes, carbon fiber or graphene will undoubtedly greatly increase the EMI production cost, and thus the low cost and high EMI SE of materials are vital to application of EMI materials.

Recently, cyclic butylene terephthalate (CBT), oligomer of PBT, is a new type of processing agent and has been applied widely in polymer fields [11–14]. The most noticeable trait is the very low viscosity, which makes CBT exhibit strong wetting ability for inorganic or organic fillers. Also, CBT will polymerize into linear polybutylene terephthalate (PBT) within 3~5 min at above 190 °C by ring-opening polymerization [15]. The high temperature can lead to the reduction of polymerization time obviously. Due to the reaction process is athermal and entropically driven, there are no hot spots in the mold that result in thermal degradation. Concurrently, the

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byproducts are not produced during polymerization process [15].

In this paper, carbon-based conductive polymer composites with high EMI SE are prepared using melt blending method. Here, PBT polymerized from cyclic butylene terephthalate oligomer is selected as a polymer matrix, and graphite (GR) and carbon black (CB) are chosen as conductive fillers. The electrical resistivity, rheological properties, mechanical properties and morphology of PBT composites are also studied in detail.

## Experimental

### Materials and preparation

Cyclic butylene terephthalate (CBT 160 resin) was bought from Cyclic Corporation (USA), and it can polymerize into PBT with  $M_w=22,000$ , measured using gel permeation chromatography (GPC) method [16]. A mixture of chloroform and hexafluoro-2-propanol was used to dissolve PBT, and in order to calibrate the molecular weight, the standard polystyrene was adopted.

Nature graphite (1,500 meshes) was produced by Qingdao Golden Day Graphite Co. Ltd. (China); Carbon black (Printex XE-2) was purchased from Degussa-Hüls Company of Germany. Before experiments, all the materials were dried at 70 °C in vacuum oven for 12 h.

First, CBT were melted at 250 °C for 5 min using an internal mixer (Changzhou Suyan Technology Co., Ltd, China) with a mixing speed of 40 rpm, and then the graphite and carbon black with a certain mass ratio were added into an internal mixer and another 10 min was needed to mix all the ingredients fully, subsequently, the mixtures were smashed using a plastic mill (produced by Tianjin City Test Instrument Co., Ltd. China). Finally, PBT/GR/CB composites were injected into the specimen (for flexural strength measurement) in accordance to the standards of ISO 178:2001 using an injection molding machine. The injection temperature and pressure were 250 °C and 10 MPa, respectively. In order to achieve low electrical resistivity, the above 50 % of conductive fillers were required to be incorporated into the PBT. The addition of carbon black is to improve the conductivity, whereas the aim of the addition of graphite is to decrease the viscosity of composites and electrical resistivity [17]. All component contents are listed in Table 1.

### Characterization

#### *Rheological properties*

The rheological properties of PBT/GR/CB composites were measured using a rotary rheometer MARS III, produced by

Thermo, Germany. The storage modulus, loss modulus, and complex viscosity of composites were collected in the frequency range from 1 to 100 Hz, and the temperature and shear stress were fixed to be 250 °C and 400 Pa, respectively.

#### *SEM observation*

The morphology observation of fracture surface of PBT composites were carried out using a scanning electron microscope (SEM, Quanta 200, FEI Corporation) with an accelerating voltage of 20 kV. Firstly, composites were dipped into liquid nitrogen and fractured, and then the fracture surface was coated with gold particles.

#### *Electrical resistivity*

The electrical resistance  $R$  ( $\Omega$ ) of PBT composites was measured using a milliohm meter VC480C+, produced by Shenzhen City Vichy Technology Co. Ltd. China. The electrical resistivity,  $\rho$  ( $\Omega\cdot\text{cm}$ ), was calculated using the following relation:

$$\rho = R \times S/L \quad (1)$$

where  $L$  is the sample thickness ( $L=0.2$  cm) and  $S$  ( $\text{cm}^2$ ) represents the surface area of the electrodes ( $S=5$   $\text{cm}^2$  in our experiments).

#### *EMI properties*

EMI shielding measurements were conducted at room temperature over a frequency range of 30 to 3,000 MHz using electromagnetic shielding measuring instrument (DR-S02), produced by EMC Technology Company, China. The PBT samples were pressed into disc with a 12 cm of outer diameter and 1 mm of thickness using a molding machine at 250 °C and 10 MPa for 10 min.

#### *Mechanical properties*

In the light of standard of ISO 178:2001, the flexural properties of PBT composites were evaluated with a material mechanical testing machine (Shenzhen SANS Company of China). The specimens were tested at a cross-head speed of 10  $\text{mm min}^{-1}$  at room temperature.

#### *Heat resistance*

The heat resistance of PBT/GR/CB composites was assessed by the Vicat soften temperature (VST), which was carried out in Vicat Softening Point Meter of Shenzhen SANS Corporation (China), and the heating rate was 120 °C  $\text{h}^{-1}$ .

**Table 1** The component contents and the data of PBT/CB/GR composites

Number	PBT wt%	GR wt%	CB wt%	Flexural strength (MPa)	Flexural strength (MPa)	VST (°C)
CBT1	50	50	0	30.08	3,907.1	117.1
CBT2	50	45	5	35.29	3,994.2	124.9
CBT3	50	40	10	43.82	3,151.82	137.5
CBT4	50	35	15	36.74	2,698.19	147.6
CBT5	40	60	0	33.93	2,958.85	121.4
CBT6	40	55	5	34.68	3,828.99	130.6
CBT7	40	60	10	26.91	2,861.21	143.2

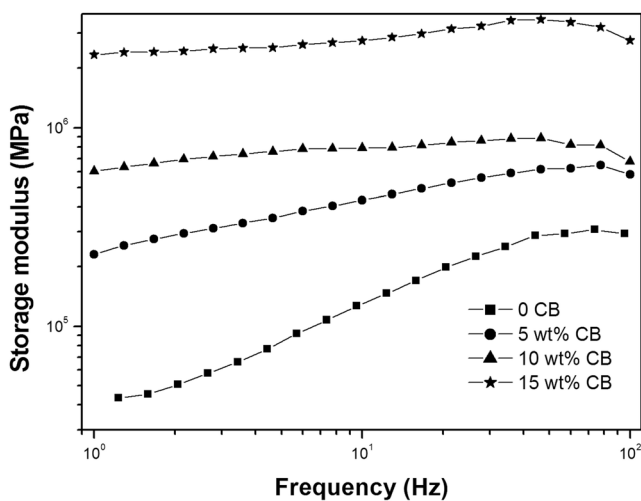
**Results and discussion**

**Rheological properties**

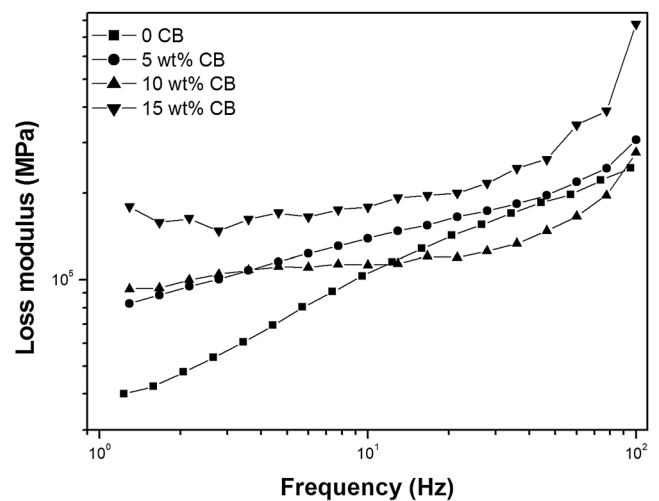
Polymer melt rheology property is very important for fundamental and technology investigation since it can make us understand well microstructure of composite and dispersion of fillers in matrix. In fact, many factors can influence the rheological properties of composites [18], for example, the content, orientation, shape, dispersion of filler in matrix and the interfacial interaction between filler and the polymer. Figures 1, 2, 3, 4, 5 and 6 show the storage modulus, loss modulus and viscosity of PBT composites with different contents of graphite and carbon black. In this study, as expected, the rheological properties of PBT composites changed significantly due to the addition of graphite and carbon black.

Figures 1, 2 and 3 are for the PBT composites with 50 % of filler. The storage modulus of PBT composites were found to increase as graphite was replaced by carbon black gradually till 10 % of CB, and the loss modulus of composites had the same tendency with storage modulus. Obviously, the storage modulus of composite was far higher than loss modulus over the whole frequency range. Also, the increased magnitude of loss

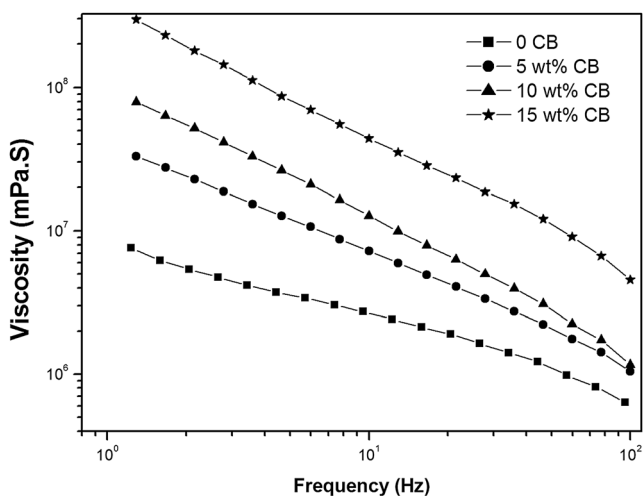
modulus of PBT composites with CB content was far lower than that of storage modulus, indicative of solid-like behaviors. The increased storage modulus was thought to result from better dispersion of carbon black in the matrix, providing interaction between filler and polymer [19, 20]. In addition, The storage modulus of PBT composites increased as frequency increased at below 10 % of CB, however, when CB content got to above 10 %, the storage modulus of PBT composites was independent of frequency, and a platform appeared in the low frequency, as shown in Fig. 1, such platform grew taller with further increasing CB contents. Importantly, the storage modulus of composite with 10 and 15 % of CB were observed to be independent on applied frequency, which suggested that the polymer molecular chains relaxation was inhibited [20]. For the plateau in low frequency, researchers [7] believed it was due to filler network in matrix, which could prevent molecular chains from relaxing. Also, other researchers [21] deemed such plateau was a useful tool to evaluate the filler dispersion in matrix; the appearance of plateau was indicative of good dispersion of filler in composites. In the light of such conclusion, there were reasons to believe the graphite and carbon black could disperse in PBT matrix very well and formed conductive network at above 10 % of CB.



**Fig. 1** The variation of storage modulus of PBT composites with 50 % of filler as a function of CB content



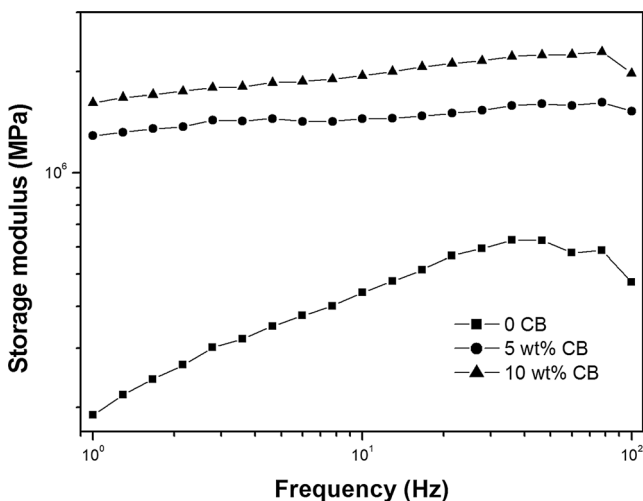
**Fig. 2** The variation of loss modulus of PBT composites with 50 % of filler as a function of CB content



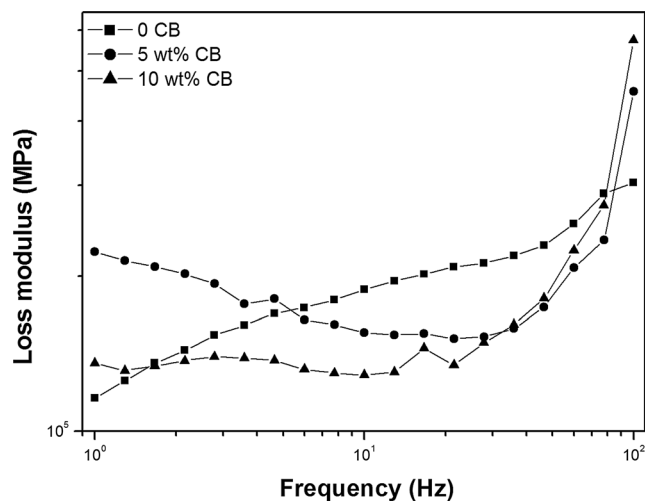
**Fig. 3** The variation of viscosity of PBT composites with 50 % of filler as a function of CB content

Figure 3 is the variation of complex viscosity of PBT composites as a function of CB contents. The viscosity of PBT composites declined as applied frequency increased, indicative of shear-thinning traits. Also, as expected, the viscosity of composites increased as graphite was replaced by carbon black gradually, which is due to the enhancement in intermolecular interaction between the filler and polymer. Certainly, the increase in viscosity meant that the processing property of PBT/GR/CB composites began to become poor.

For PBT composites with 60 % of filler, the storage modulus, loss modulus and viscosity of composites with CB content were shown in Figs. 4, 5 and 6. Like composites with 50 % of fillers, the storage modulus and loss modulus of composites with 60 % of fillers also showed the plateau at 5 % of CB, indicative of good dispersion and formation of conductive network. Additionally, at the same frequency and CB loadings, the composites with 60 % of filler exhibited



**Fig. 4** The variation of storage modulus of PBT composites with 60 % of filler as a function of CB content

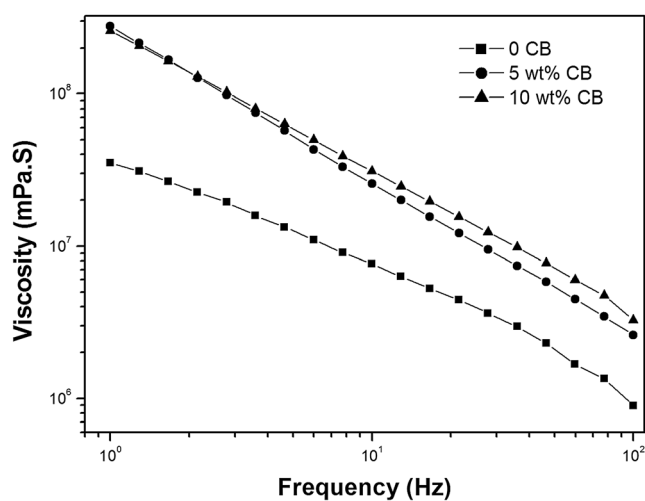


**Fig. 5** The variation of loss modulus of PBT composites with 60 % of filler as a function of CB content

higher storage modulus and viscosity than composites with 50 % of filler, which means that the movement ability of molecular chains was further weakened. i. e. the composites became more rigid.

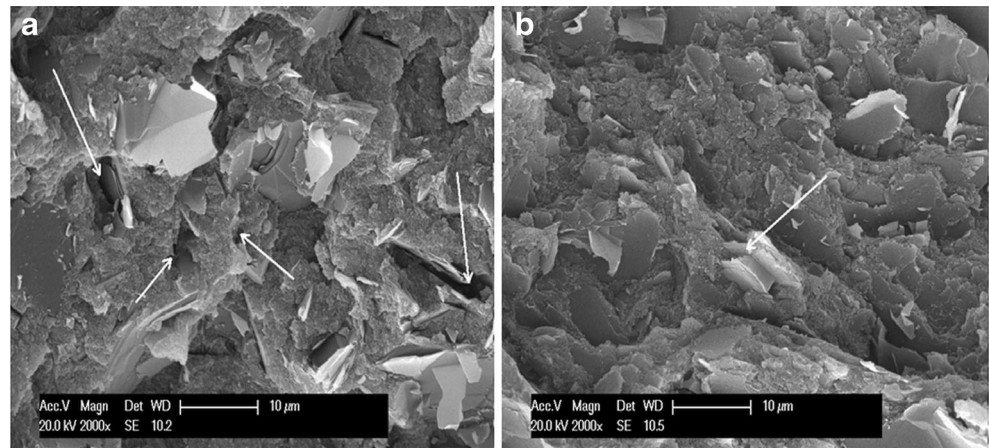
#### SEM analysis

Figures 7a and b show the fracture surface of CBT composite with 50 % graphite and CBT composite with 35 % of graphite and 15 % of carbon black, respectively. The addition of a large amount of graphite and carbon black made the fracture surface of composites become coarser, and no plastic deformation was found, which indicated that PBT/GR and PBT/GR/CB composites were brittle. Especially, it was observed in Fig. 7a that the interface between graphite and PBT is so poor that many voids are produced, as indicated by a white arrow. Concurrently, graphite layers were clearly observed. When 15 % of



**Fig. 6** The variation of viscosity of PBT composites with 60 % of filler as a function of CB content

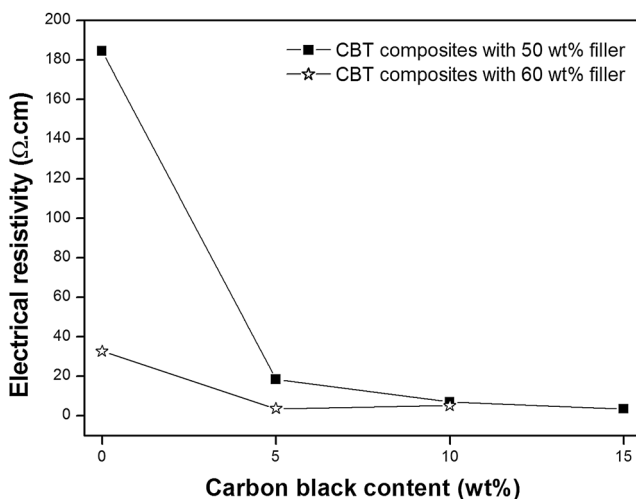
**Fig. 7** The SEM images of PBT composites: a. with 50 % of GR; b. with 35 % of GR and 15 % of CB



graphite is replaced by 15 % of carbon black, the microstructure of composites changed significantly (shown in Fig. 7b), some voids, as well as gaps of graphite layers in matrix, were filled with PBT phase containing carbon black (as shown by a white arrow in Fig. 7b), and those changes in morphology would bring positive impact on conductivity and mechanical properties of the composite.

#### Electrical resistivity

In order to endow polymer with high conductivity, all kinds of conductive fillers were needed to be added into the polymer matrix. Figure 8 shows the variation of electrical resistivity of PBT composites as a function of graphite and carbon black contents. It was expected that the conductivity of PBT composites was improved largely due to the addition of graphite and carbon black. The literature reported that the electrical resistivity of PBT was about  $10^{18} \Omega \cdot \text{cm}$  [22], which meant that neat PBT was a typical insulating material. After the addition of 50 % of graphite, the electrical resistivity of PBT/graphite



**Fig. 8** The variation of electrical resistivity of PBT composites as a function of CB content

composite was found to be reduced to  $184.5 \Omega \cdot \text{cm}$ , and as graphite content increased to 60 %, the electrical resistivity of PBT/graphite composite was further dropped to  $32.8 \Omega \cdot \text{cm}$ , and this was related to the formation of more conductive paths in PBT composites. A large decrease in electrical resistivity appeared when some graphite was replaced by carbon black. For 50 % of filler composites, the electrical resistivity of PBT composites decreased from  $184.5 \Omega \cdot \text{cm}$  to  $18.5 \Omega \cdot \text{cm}$ , decreasing approximately 10-fold, when 5 % of graphite was replaced by the 5 % of carbon black, which indicated that the carbon black has major effect on the conductivity properties of the PBT composite. The minimum electrical resistivity appeared at the composite with 35 % of graphite and 15 % of carbon black with  $3.5 \Omega \cdot \text{cm}$ . The improvement in electrical resistivity was believed to be due to carbon black filling voids or pores in matrix and connecting with different graphite layers, and those results demonstrate that graphite and carbon black present a good synergistic effect. But for 60 % of filler composites, the lowest electrical resistivity ( $3.7 \Omega \cdot \text{cm}$ ) of composites appeared at the composite with 5 % of CB, not the composite with 10 % of CB. The reason for this is due to excessive fillers were not wetted by polymer completely and voids or pores formed in matrix, leading to the increasing electrical resistivity. Similar results had been reported by Madani et al. where the electrical resistivity of natural rubber/butyle rubber/carbon black composites was achieved to be the minimum value of  $10^{-2} \Omega \cdot \text{m}$  at 50 % of fillers [2]. Gupta et al. [4] also found that the electrical resistivity of polyurethane/CNTs composites was measured to be  $7.9 \Omega \cdot \text{cm}$  at 22 % of CNTs. Pang et al. also observed that the electrical resistivity of functionalized graphene oxide/carbon nanotube/polyethylene composite exhibited the good value of  $4.8 \times 10^{-3} \text{ S/cm}$  with only 1.6 % of CNTs [23].

#### EMI SE

The EMI SE is used to evaluate the ability to reflect and absorb electromagnetic wave, and it was reported that the

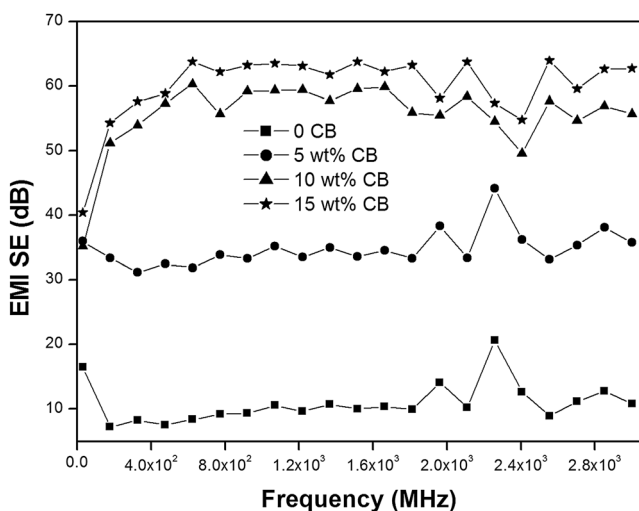


EMI SE was dependent on electrical resistivity. Usually, a specimen with high electrical resistivity exhibited excellent EMI SE. The EMI SE of the specimens was calculated according to the following equation:

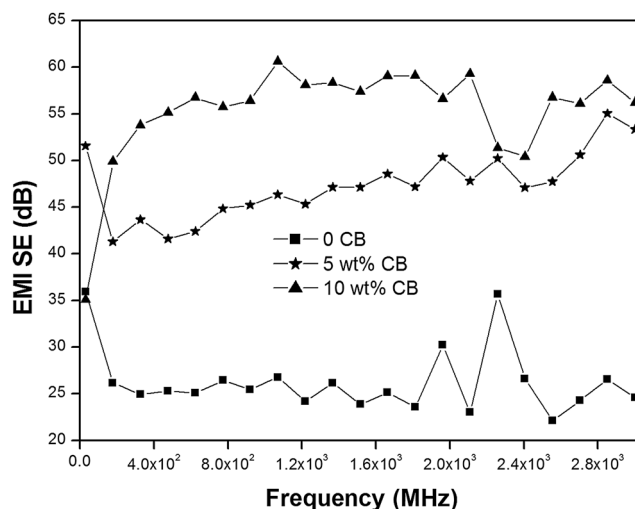
$$SE = 10 \log \frac{P_0}{P_t} \quad (2)$$

Where  $P_0$  is the incident electromagnetic power and  $P_t$  is the transmitted electromagnetic power. In general, EMI SE is expressed in decibel (dB) units. For example, if SE is equal to 20 dB, it means that 99 % of electromagnetic wave power was shielded, and a SE of 60 dB indicates that 99.9999 % of electromagnetic wave power was dissipated. In practice, the materials with at least 30 dB meet commercial requirement.

Figures 9 and 10 present the EMI SE of composites with different graphite and carbon black content in the frequency range from 30 to 3,000 MHz. It was observed that EMI SE of CBT composites increased with carbon black content increased at a given frequency. Owing to the high electrical resistivity, composite with 50 % of graphite exhibited low EMI SE of 10 dB (shown in Fig. 9). When graphite was replaced by carbon black gradually, the EMI SE was observed to increase to 40–64 dB for composite with 35 % of graphite and 15 % of carbon black, which indicated 99.9999 % of electromagnetic energy was dissipated. For composite with 60 % of graphite, the EMI SE was determined to be ~25 dB for composite with 60 % of graphite over the frequency range of 30–3,000 MHz. The highest EMI SE appeared at composite with 50 % of graphite and 10 % of carbon black, and the EMI SE was measured to be 35–60 dB over the frequency range of 30–3,000 MHz, and those results were higher than that of PE/carbon nanofibers composites and PS/CNT composites [1, 24]



**Fig. 9** The EMI SE of PBT composites with 50 % of fillers with variation of CB content



**Fig. 10** The EMI SE of PBT composites with 60 % of fillers with variation of CB content

where the EMI SE were only 14 and 20 dB, respectively. These results indicated that the method of preparing the EMI materials based on graphite and carbon black not only obtained materials with high EMI SE, but also reduced the largely production cost significantly, presenting a good market prospect in the electromagnetic shielding field.

#### Flexural properties

If EMI materials were used, the good mechanical properties were also needed except for high EMI SE. Here, the flexural strength and flexural modulus of PBT composites were tested. Table 1 shows the variation of flexural strength and modulus of PBT composites as a function of CB content. For composite with 50 % of filler, the flexural strength of PBT composites increased with CB content and achieved the maximum value of 43.8 MPa at 10 % of CB, and this was ascribed to the enhanced molecular interactions due to high specific surface area of CB, and then the flexural strength of composite reduced to 36.7 MPa when CB content got to 15 %. The reduction in flexural strength was believed to be due to poor interface properties between the filler and polymer. For composites with 60 % of filler, the flexural strength of composites exhibited a little increase as CB content increased and reached the maximum value of 34.7 MPa at 5 % CB, and then it decreased with further increasing CB content. The flexural modulus of composites with 50 and 60 % of filler firstly increased with CB content, indicative of enhancement in stiffness, and then decreased largely when CB content exceeded 5 %, which suggested the rigidness of composites began to decline. The maximum flexural modulus appeared at composite with 45 % of graphite and 5 % of carbon black and was determined to be 4.0 GPa. These results were better than that of polyamide 6/graphite composites where the maximum flexural strength was only 4.6 MPa [25].

## Heat resistance

In order to evaluate the thermal stability of composite, the vicat softening temperature (VST) was used, and such parameter is defined as the temperature of sample pressed into 1 mm depth by using 1 mm<sup>2</sup> needles at a certain load and a certain temperature. The high vicat softening temperature means the materials possessed excellent heat resistance property. Table 1 shows the changes of VST of PBT composites with different contents of CB. The PBT composites with 50 and 60 % of graphite exhibited the VST at 117.1 °C and 121.4 °C, respectively. As CB content increased, the VST of PBT composites were observed to increase to maximum values of 147.6 °C for composite with 35 % of GR and 15 % of CB, to be 143.2 °C for composite with 50 % of GR and 10 % of CB, respectively, which indicated that the addition of CB effectively improved the heat resistance of composites. The reason could be attributed to the fact that the graphite and carbon black could form more networks in matrix, which effectively inhibit the movement of polymer chains.

## Conclusion

In this study, the conductive PBT/GR/CB composites were prepared using melt blending method. The effect of GR and CB on rheological properties, morphology, mechanical properties, electrical resistivity and EMI SE of composites were studied.

The storage modulus, loss modulus and viscosity of composites increased with carbon black content, and the appearance of a plateau in low frequency range indicates that good dispersion of conductive fillers in matrix and conductive network are formed. However, the addition of CB also caused the viscosity to increase, which is not helpful to the processing properties. SEM confirmed that the addition of carbon black could fill the voids of graphite and formed a compact conductive network. The electrical resistivity of composites declined as CB loadings increased, and the minimum electrical resistivity was observed to be 3.5 Ω.cm for composite with 35 % of graphite and 15 % of carbon black. Like electrical resistivity, the EMI SE of composites increased with carbon black content, and the composite with 35 % of graphite and 15 % of carbon black presented the highest EMI SE of 40~64 dB over the frequency range of 30–3,000 MHz. The flexural strength of composites firstly increased with CB content till to 10 % of CB, and then decreased with further increasing CB content. The flexural modulus of composites with 50 and 60 % of filler firstly increased with CB content, and then decreased largely when CB content exceeded 5 %. The heat resistance of PBT composite was improved due to the graphite being replaced by carbon black, and the VST was elevated from 117.6 to

147.6 °C (composites with 50 % of filler) and from 121.4 to 143.2 °C (composites with 60 % of filler), respectively.

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