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Morphology and Conductive Properties of Carbon Black- and Graphite- Filled Conductive Epoxy Micro-porous

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Abstract. Conductive composite requires a well dispersion of conductive filler, which is this difficult to be achieved low content of filler for high density conductive filler. This work investigates the effect of carbon black and graphite on the morphology and conductive properties of conductive epoxy micro-porous. The conductive epoxy micro-porous was prepared by using single emulsion technique. It involved the drop of epoxy-hardener-blowing agent-conductive filler mixture into corn oil at 160 °C and followed by the leaching process to remove excess corn oil. Results show the addition of conductive filler in epoxy matrix lead to increases porous structure in epoxy micro-porous. Carbon black filled epoxy micro-porous possessed smaller particles compared to graphite filled epoxy micro-porous. For skeleton density and total pore, both of the value increase with filler loading due to packing sphere effect. However, the skeleton density for carbon black filled epoxy micro-porous is higher compared to graphite filled epoxy micro-porous. For electrical conductivity, graphite filled micro-porous was higher than that of carbon black filled epoxy micro-porous.

1 Introduction

Nowadays, conductive polymer composites have gained a lot of interests in many applications such as electromagnetic interference (EMI) shielding, conductor, anti-static materials and sensor [1-3]. The application of bipolar plates in polymer electrolyte membrane fuel cells (PEMFCs) requires compound with a high conductive filler loading to achieve high electrical conductivity [4]. However, high filler loading can reduce both mechanical strength and process-ability of conventional composite. In order to overcome this problem, metal coated cenospheres is considered as one of the most advanced

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conductive materials to reduce the conductive filler used in conductive composites [5]. Nevertheless, there are some limitations in applications of these conductive cenospheres as well as the difficulties encountered during the process. Metal coated cenospheres have two layers, namely cenosphere wall and metal coated layer. The adhesion of metal coated layer and cenospheres is expected to be strong enough to avoid the debonding mechanism of metal particles at the coated layer during mixing process and preparation of composites. Moreover, the use of metal coated cenospheres have to be cared on the wettability of metal surface with the matrix binder. In addition, the metal coating process onto the cenospheres such as electroless plating, magnetron sputtering deposition and fabrication of cenospheres (ceramic microballoons) are complex [6-8]. This research proposes one step process to produce conductive epoxy micro-porous through single emulsion technique. The process is simple and economic. The conductive epoxy micro-porous have one cell wall containing dispersed conductive filler, hence it could provide good wettability and well adhesion to epoxy matrix. Moreover, the effect of different type of conductive fillers used on morphology and conductive behavior is also investigated.

2 Experimental

2.1 Materials

Epoxy resin DER 331 and polyamide A062 from DOW chemical were used. Epoxy resin DER 331 has epoxide equivalent weight of 182-192, density and viscosity of 1.16 g/cm³ and 11-14 Pas at 25 °C. Polyamide A062 has equivalent weight per H active of 110, density and viscosity of 0.96 g/cm³ and 35–45 Pas at 25 °C, respectively. White crystalline powder of sodium bicarbonate from HmbG Chemicals was used as the blowing agent with density of 2.22 g/cm³. Corn oil was purchased from Yee Lee Corporation Sdn. Bhd. and contained free polyunsaturated fatty acids. Carbon black N300 (HAF) was purchased from Chemicals. Inc. Carbon black with particles size 100 µm has iodine absorption number of 82 ± 5 g/kg and CTAB surface area of 79~87 10³m²/kg. Graphite powder from Sigma Aldrich was purchased from AR Alatan Sdn Bhd. Graphite powder was a synthetic graphite with < 20 µm particles size. For conductive testing, epoxy resin DER 331 and polyamine were used as binder. Polyamine has equivalent weight per H active of 95, density and viscosity of 1.03 g/cm³ and 3–6 Pas at 25 °C, respectively.

2.2 Preparation of Conductive Epoxy Micro-porous (CEMP)

In this study, two different conductive filler content (10 phr and 20 phr) and two types of conductive filler (carbon black and graphite) were used. The formulation and nomination of conductive epoxy micro-porous are depicted in Table 1. The ratio of epoxy and polyamide was 1E:2P (as hundred part resin - phr). Epoxy was mixed with 4 phr of sodium bicarbonate by using over-head stirrer with the speed of 300 rpm for 3 minutes. Conductive filler was varied from 10 phr and 20 phr and added and stirred for 2 minutes at the same speed. Then, polyamide was added and mixed at the speed of 300 rpm for another 3 minutes. Epoxy mixture was dropped into corn oil (weight ratio of 1:9) at 160 °C and stirred at 1000 rpm for 1 hour. The mixture was then allowed to cool down to 80 °C. The micro-porous was filtered and washed five times with detergent solution at 60 °C. The ratio of detergent to water of 1:20 was used. The detergent solution was changed every 15 minutes. Drying and post-cure process were carried out at 80 °C for 4 hours in air-circulating oven.

Table 1. Nomination of conductive epoxy micro-porous.

Nomination	Control	CBEMP	CBEMP	GPEMP	GPEMP
		10	20	10	20
Epoxy: Polyamide	1 : 2	1 : 2	1 : 2	1 : 2	1 : 2
Sodium Bicarbonate (phr)	4	4	4	4	4
Carbon Black (phr)	-	10	20	-	-
Graphite (phr)	-	-	-	10	20

*CB= carbon black, GP= graphite, EMPn= epoxy micro-porous with n phr of conductive filler content, phr= part per hundred resins.

2.3 Preparation of Epoxy Composite Filled CEMP for Conductive Testing

The amount of CEMP in 20 phr was added into 100 phr of epoxy-polyamine binder (weight ratio of 10:5). The mixture was casted into PP mold with dimension of 24 mm x 20 mm x 4 mm and allowed to harden at room temperature for 24 hours.

2.4 Characterization and Testing

Particle density of CEMP was measured by using gas pycnometer density analyser, AccuPyc II 1340 V1.0.5. Testing was carried out at room temperature using the chamber of 10 cm³ and Helium gas.

The morphology analysis of CEMP was carried out using a scanning electron microscope, model JSM 6260 LE JOEL. The CEMP was coated with gold/platinum layer to avoid electrostatic charging during examination.

Electrical conductivity test of CEMP was obtained by using Keithley 4200 Semiconductor Characterization System. The samples were measured using two probes I-V measurement system. The distance between two probes was 5 mm. The voltage was varied from 0 to 10 V.

3 Results and Discussion

3.1 Morphology

Figure 1 shows the variation morphology of carbon black filled epoxy micro-porous (CBEMP) and graphite filled epoxy micro-porous (GPEMP) at different concentration. As seen in Figure 1(a), epoxy micro-porous without adding conductive filler possessed large micro-porous particle which adhered to one another [9]. In contrast, the addition of conductive filler led to the reduction of the particle size of CEMP as shown in Figure 1. Results also indicated that the particle size of CEMP was smaller than that of control sample. The addition of conductive filler increased the viscosity of epoxy mixture droplets and consequently the matrix less expanded. An increased viscosity of epoxy mixture droplets caused it difficult to expand when sodium bicarbonate decomposed [10]. However, as the content of conductive fillers increased from 10 phr to 20 phr, the particle size of the CEMP reduced. This was due to strong interaction between fillers which complicated the

separation of epoxy droplet [11]. Hence, CEMP with 20 phr conductive fillers has smaller particle size compared to CEMP with 10 phr conductive fillers. Furthermore, higher conductive filler loading increased the viscosity of the epoxy mixture. The increased viscosity of epoxy mixture in emulsion system will also affect the distribution of droplet size and increase the tendency of the droplet to aggregate [12]. On the other hand, GPEMP20 produced large particle compared to CBEMP20. It can be implied that the reduction of particle size of conductive filler will result in increased viscosity of epoxy mixture droplets and thus difficult to blow [13].

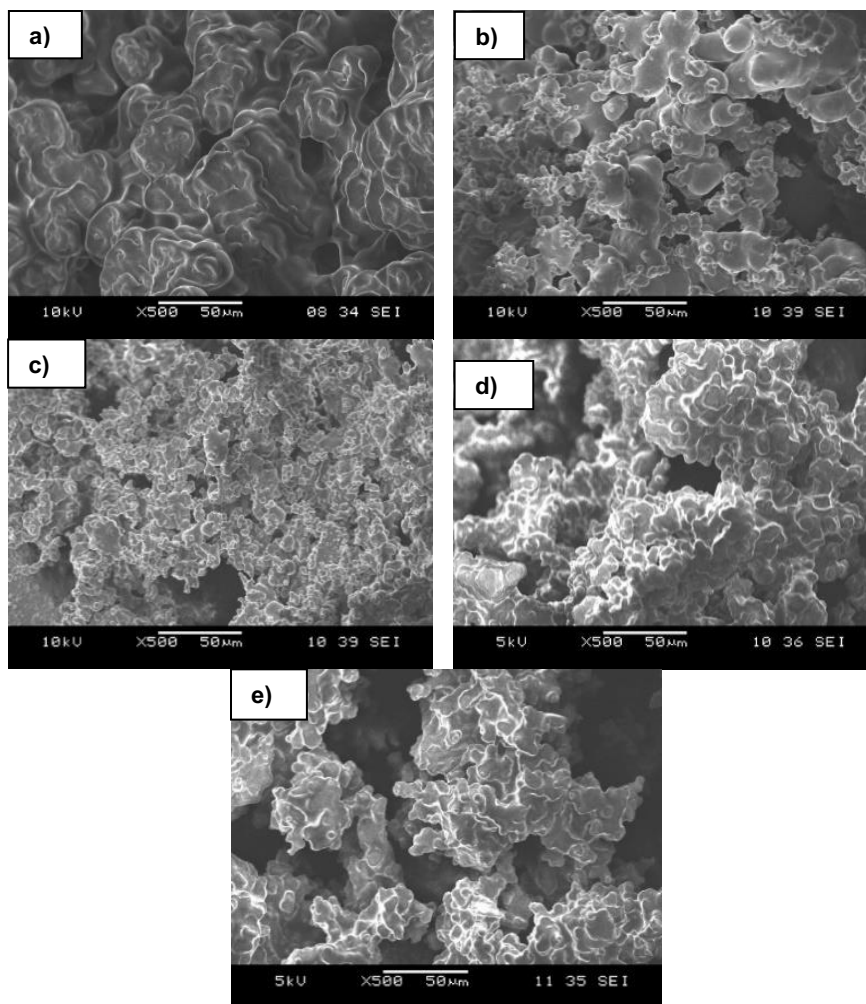


Fig. 1. SEM image for CEMP at magnification of 500x: (a) Control, (b) CBEMP10, (c) CBEMP20, (d) GPEMP10, (e) GPEMP20.

3.2 Density

Figure 2 shows the skeleton density and total pore volume of CEMP with variation of conductive filler and content. It showed that an increase of conductive fillers content from 10 phr to 20 phr increased the micro-porous skeleton density. The increased micro-porous density was due to high loading of conductive fillers. Besides, CEMP with carbon black of 10 phr showed the highest skeleton density than that of CEMP with graphite of 10 phr. This

result could be influenced by larger particle size of carbon black than the graphite. The total pore volume of CEMPs increased compared to that of control sample. In both types of conductive filler, an increase in conductive filler content increased total pore volume. This was greatly related to the packing sphere effect [14]. Furthermore, total pore volume GPEMP was slightly increased compared to CBEMP due to high density of graphite which led to increment of total pore volume compared to carbon black.

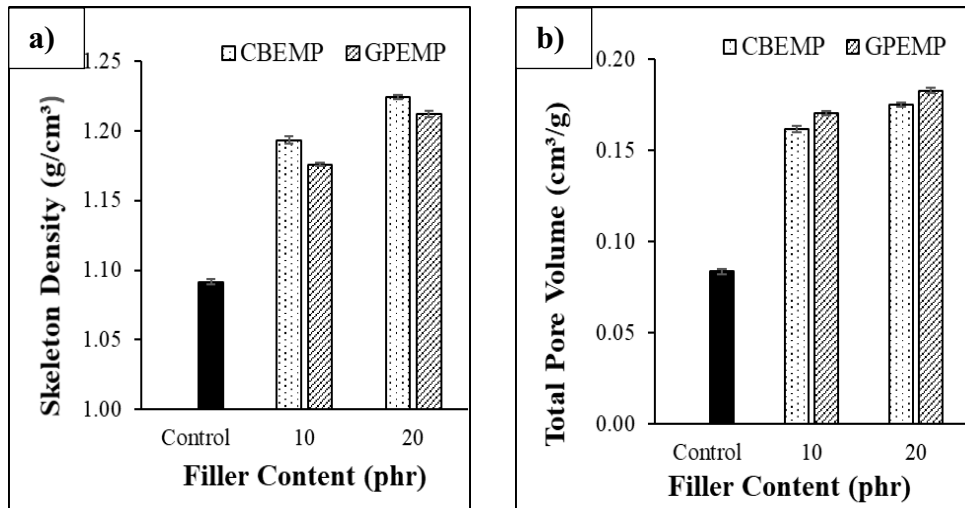


Fig. 2. The effect of different conductive fillers of CEMP on (a) skeleton density and (b) total pore volume.

3.3 Conductivity Behavior

The electrical conductivities of epoxy composite filled CEMP (epoxy/CEMP) are presented in Figure 3. The figure depicted that electrical conductivity increased with the increasing of conductive fillers content which reduced the particles size of CEMP as reported in Figure 1.

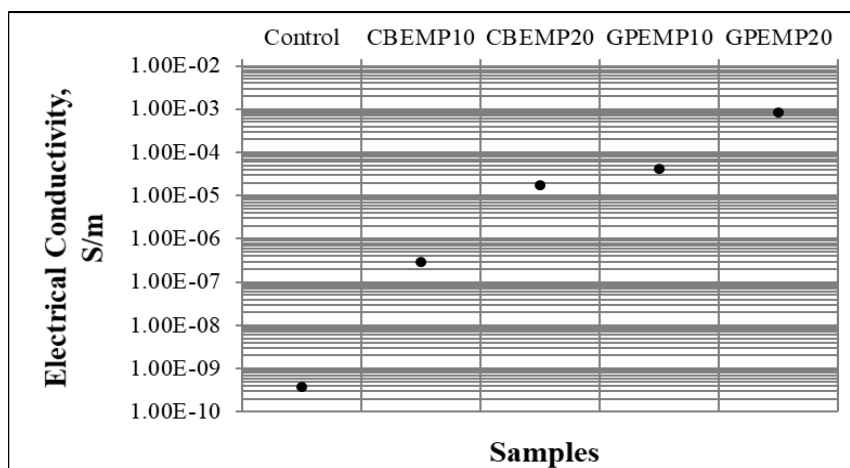


Fig.3 . Electrical conductivity of epoxy/CEMP composite.

The reduction of particles size of CEMP promotes higher surface area and better particles dispersing in polymer matrix [15]. Hence, more electrical pathway provides better conductivity behaviour. Moreover, the use of different types of conductive filler also affected the electrical conductivity properties. GPEMP20 showed higher electrical conductivity compared to CBEMP20 because of different chemical structure bonding arrangement between carbon black and graphite [16]. Graphite has each atom connected evenly to three carbons formed sp² hybridization. The sp² set forms the hexagonal lattice typical of a layer of graphite [17]. On the contrary, carbon black has disorder crystal structure [18]. Hence, the bonds between carbon in graphite were stronger compared to carbon black. Therefore, GPEMP20 has better electrical conductivity properties.

4 Conclusions

CEMPs were prepared through single emulsion technique. Higher conductive fillers loading increased the skeleton density, and contributed to high total pore volume and excellent electrical conductivity of CEMP. Besides, high density of graphite powder resulted in higher skeleton density and total pore volume of GPEMP compared to CBEMP. The dispersion of conductive fillers has been proven by electrical conduction of epoxy composite filled CEMP. It was found that GPEMP produced excellent electrical conductivity compared to CBEMP.

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