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Modification of Epoxy/Recovered Carbon Black (rcB): The Effect of Defoamer and Dispersing Agent

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ABSTRACT

This study focused on recovered carbon black (rCB), obtained from the pyrolysis of end-of-life (EOL) rubber tyres. The rCB was mixed with epoxy resin and diethyl toluene diamine (DETDA) as a hardener to produce epoxy/rCB composites intended for electrostatic discharge (ESD) applications. A defoamer was added to the pre-mixed solution to restrict air bubble formation produced from mixing, while a dispersing agent was used to ensure more uniform dispersion of rCB in the epoxy matrix. This work studied the mechanical, morphological, and electrical properties of untreated epoxy/rCB systems and treated systems with defoamer and dispersing agent. Outcomes showed that the addition of defoamer reduced bubble formation and improved the composite properties. However, adding a dispersing agent led to the formation of more bubbles, which deteriorated the properties of the composite system.

Keywords: *Epoxy, recovered carbon black, defoaming agent, dispersing agent*

1. INTRODUCTION

Electrostatic discharge (ESD) is an event where electric charges are transferred between two bodies with different potential. EDS is a type of failure caused by electric overstress (EOS) resulting from charge imbalance when two objects come in contact and are then separated [1]. This event is commonly seen in semiconductor devices, where charge imbalance produced by integrated circuits (ICs) with other objects, especially during transportation, can lead to damage of semiconductor devices. To minimize charges, voltages, and electric fields, electrostatic discharge (ESD) trays are used in the packaging, shipping, and storing of electronic parts and components. ESD trays help to dissipate the accumulated static electricity in the components and are usually made of polymeric materials due to their robustness, lightweight, processability, and low-cost properties. These polymers are usually filled with carbon-based conductive fillers to improve mechanical and thermal properties as well as impart electrical conductivity to the polymer [2].

The most commonly used polymer is epoxy, which reacts with curing agents to form a highly crosslinked network exhibiting outstanding resistance towards moisture and solvents, excellent adhesion to different substrates, and relatively low curing temperatures [3]. Recently, epoxy has gained popularity in electronic applications due to these exceptional properties. However, resulting from its insulative nature, conductive fillers are added to impart electrical conductivity to the epoxy resins. These fillers also reinforce the matrix, enhancing mechanical properties [4]. Conductive fillers provide electrical conductivity to the insulative polymer matrix by forming segregated conductive networks known as percolated pathways [5]. These pathways enable electron tunneling along the filler-polymer interface, where delocalized electrons can move freely. In this study, recycled carbon black (rCB) was used as an alternative for commercial carbon black fillers. These rCBs are obtained from pyrolysis of end-of-life tires, typically containing 80-90% of recovered carbon black and 10-20% foreign substances used in rubber compounding. However, the presence of impurities in rCB lowered the properties of the filler as compared to the commercial carbon black, restricting its use to applications not requiring high strength [6].

The mixing process prior to incorporating fillers into the resin often produces air bubbles. In addition, incorporation of fillers increases the viscosity of the mixture, making the air bubble harder to remove. Besides, rCB has a high tendency to aggregate rather than forming intpolymer forces with the polymer chains, due to its small size resulting in a high surface area in contact [6]. These factors can negatively affect composites by forming stress concentration regions. To overcome these issues, additives such as defoamers and dispersing agents can control bubble formation and improve filler dispersion [7].

In this study, a silicone-based defoaming agent and an acrylic-based dispersing agent were used to suppress bubbles and achieve more uniform dispersion of rCB. The silicone-based defoamer acts as a surfactant to reduce the surface tension and destabilizing bubbles, facilitating their removal [8]. The dispersing agent was used to give better alignment of rCB particles. The effects of these additives on mechanical and electrical properties were evaluated, and fracture surface morphologies were examined via scanning electron microscopy (SEM).

2. EXPERIMENTAL APPROACH

2.1 Materials

Epoxy DGEBA grade 331 DER was supplied by Euro Chemo-Pharma Sdn Bhd. Recovered carbon black (rCB) was provided by Eco Power Synergy Sdn Bhd (from pyrolysis of old tires). Diethyl toluene diamine (DETDA) was obtained from Shandong Aonuo New Material Company Ltd., and the silicone-based defoamer and acrylic-based dispersant were supplied by AFCONA Chemicals Sdn Bhd.

2.2 Compounding of Epoxy/rCB

Different rCB loadings (0, 5, 10, 15, and 20 vol.%) were used. Mixing was carried out with a mechanical stirrer at 300 rpm. rCB was added gradually into the epoxy resin, followed by the addition of DETDA (24 phr), and stirring continued for 2 minutes. The mixture was cast into a mold for degassing and then cured overnight at 100°C. Final curing was conducted at 120°C for 4 hours. For samples with defoamer, it was added after all other components. For samples with dispersant, it was mixed into the epoxy resin before adding rCB.

3. TESTING AND CHARACTERIZATION

Mechanical tests included a flexural properties (three-point) test and a fracture toughness test. The electrical properties were identified via impedance test using an LCR meter. The morphologies of fractured surfaces were examined through scanning electron microscopy (SEM).

The dimensions of rectangular specimens used for flexural and fracture toughness tests were 60 mm x 12.7 mm x 3 mm (length x width x thickness). Flexural tests were performed using Instron Universal Testing Machine 5569 following ASTM D790, with a crosshead speed of 2.38 mm/min and a support span fixed at 50 mm. The fracture toughness test was done according to ASTM D638, with a pre-crack of 4 mm and a loading speed of 1 mm/min. Fracture toughness (K_{IC}) was calculated according to the formula given in Equation (1) and (2)

$$Fracture\ toughness, Kic = \frac{F_{max}\sqrt{\pi a}}{BW}\ f(\frac{a}{w}) \quad (1)$$

$$f\left(\frac{a}{w}\right) = 1.99 - 0.41\frac{a}{w} + 18.7\left(\frac{a}{w}\right)^2 - 38.48\left(\frac{a}{w}\right)^3 + 53.85\left(\frac{a}{w}\right)^4 \quad (2)$$

where,
Kc: Fracture toughness, $MPa * m^{1/2}$
Fmax: Maximum force in the force deflection trace, N
a: Total notch length, m
B: Thickness of specimen, m
W: Width of specimen, m

$f\left(\frac{a}{w}\right)$: Geometric factor near unities that depend on the details of sample geometry

The microscopy analysis was performed using JEOL JSM-6460 LA at 10 kV. The fracture surface of the specimens was coated in a thin layer of palladium to prevent accumulation of electrostatic charges at the surface of the specimens. Electrical properties were identified by the LCR meter HIOKI-IM 3536 with frequency ranging from 10 Hz to 1 MHz at ambient temperature. The impedance (Ω) and conductivity (S) were measured.

4. RESULTS AND DISCUSSION

4.1 Effect on Flexural Properties

Flexural properties identify the combined properties of both tension and compression stress on the surfaces of the specimen. Flexural properties reflect resistance to bending. As reported [9], increased filler content typically increases flexural properties due to the reinforcement effect from filler particles, resulting from interfacial bonding between the polymer matrix and the filler that allows the composite to withstand higher strength before breaking [10].

However, as observed in Figure 1, pure epoxy gave the highest flexural strength values, which were 130.70 MPa and 162.50 MPa, respectively. The addition of rCB reduced strength, making composites more prone to fracture. For untreated composites, the flexural strength reduced by 72.05%; the system with defoaming agent by 69.85%; and the system with dispersing by 59.52% when rCB loadings increased from 0 to 20 vol.%. Impurities deposited on the surface of pyrolytic CB reduced the effective surface area, resulting in weak polymer-filler interaction [6]. Besides, the small particle size of rCB promotes aggregation, creating stress concentration area that reduced the effectiveness of load transfer and thus weakening the material.

Presence of voids created stress concentration region and thus lower bending force needed to break the specimen [11]. This was proved when defoamer was introduced into the composite system, showed slightly improvement in flexural strength especially at high rCB loadings (e.g., 15.01% and 25.45% increases at 15 and 20 vol.%, respectively) as shown in Figure 3. Choi et. al. [7] and Li et. Al. [12] reported that use of defoaming agent suppressed the formation of pores.

Dispersing agent promoted more homogenous dispersion of rCB due to its hydrophilicity, helping to reduce aggregation [7]. However, it led to increased void formation, reducing flexural strength at lower rCB loadings (17.65% and 21.56% reductions at 5 and 10 vol.%) as shown in Figure 1 [12]. Presence of air bubbles dominated the flexural properties as there were less rCB for the dispersing agent to react with. At higher loadings, improved dispersion partially offset strength losses.

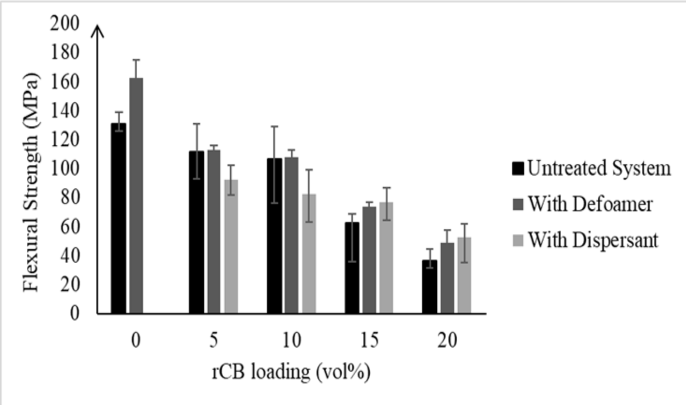

Figure 1. Effect of rCB loading on flexural strength of epoxy/rCB composites.

The flexural modulus was illustrated in Figure 2. It can be observed that the flexural modulus increased with rCB loading up to 15 vol.% for untreated and defoamer systems due to enhanced stiffness from polymer-filler interactions. However, as the rCB loading increased to 20 vol.%, a slight drop in modulus of the composite systems were observed due to agglomeration of rCB [6]. For dispersant system, modulus increased smoothly, 2.35% at 5 vol.% and 5.65% at 10 vol.%, resulted from more uniform dispersions of rCB particles that gave better polymer-filler interaction. However, flexural modulus thereafter as concentration of fillers exceeded the amount required for optimal dispersion [7].

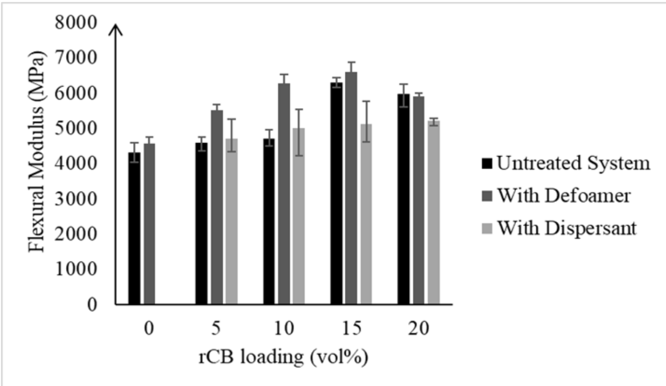

Figure 2. Effect of rCB loading on flexural modulus of epoxy/rCB composites.

4.2 Effect on Fracture Toughness

Fracture toughness defines the ability of material to resist fracture through presence of notch as stress concentration region [13]. As shown in Figure 3, fracture toughness of all systems decreased with increasing rCB. Presence of impurities and high aggregation tendency of rCB decreased the load transfer efficiency [6]. Despite the deterioration, defoamer system showed highest fracture toughness. Air bubble removal eliminated stress concentration zone, enhanced the fracture toughness of polymer composites. Dispersant system showed lowest fracture toughness. The hydrophilic nature of dispersant triggered formation of more void, weakening the properties of the composite systems despite improving filler dispersion [7].

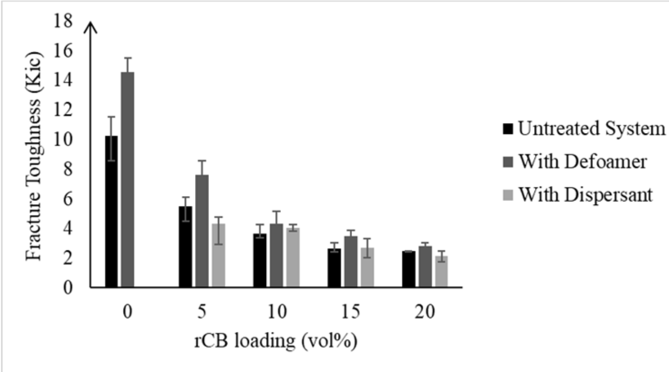

Figure 3. Effect of rCB loading on fracture toughness of epoxy/rCB composites.

4.3 Scanning Electron Microscopy

Figure 4 shows morphologies of fractured surfaces of pure epoxy and different systems of epoxy/rCB at 20 vol.%. (a) illustrated fractured surface of pure epoxy. The smooth surface indicated no sign of plastic deformation during rupture. (b), (c) and (e) illustrated the SEM images of untreated, defoamer and dispersant systems respectively. (d) showed magnified images of agglomerates whereas (f) showed close-up of void in dispersant system. Lumps of were observed as signs of agglomeration (b) and (c). Voids were observed in (b) without addition of defoamer. However, (e) displayed smooth surface despite observation of voids, showing that dispersant enhanced the overall dispersion of rCB fillers.

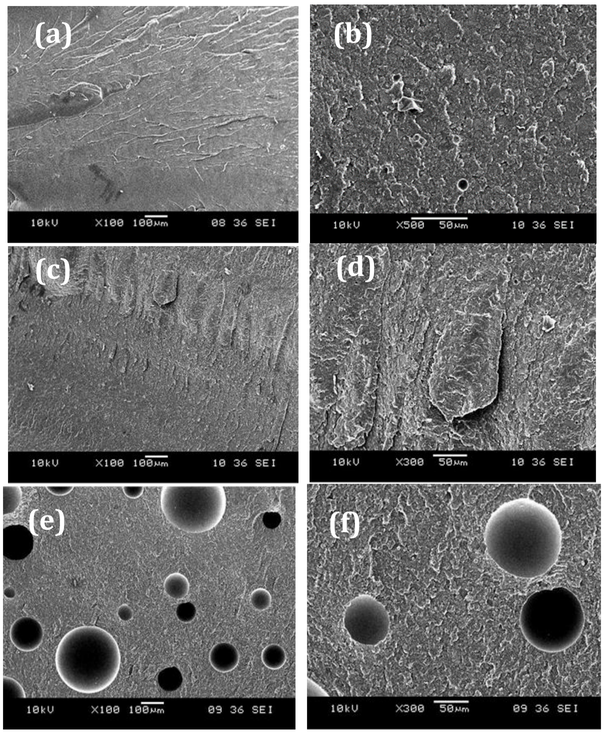

Figure 4. SEM micrographs of **(a)** Pure epoxy, **(b)** untreated epoxy/rCB, **(c)** epoxy/rCB with defoamer, **(d)** close up image of agglomerates from system with defoamer, **(e)** epoxy/rCB with dispersing agent and **(f)** close up image of voids from system with dispersing agent.

4.4 Effect on Electrical Conductivity Properties

The effect of rCB loading on the impedance for all epoxy systems were investigated with frequency fixed at 2371.4 Hz. Figure 5 shows reduction of impedance from 5.98×10^6 Ω to 3.42×10^6 Ω for untreated system; 3.55×10^6 Ω to 2.31×10^6 Ω for defoamer system and 4.16×10^6 Ω to 2.41×10^6 Ω for dispersant system. Increasing rCB loading formed conductive pathway, led to increase in number of continuous networks, allowing electrons to move freely throughout the epoxy matrix [5].

Defoamer system showed lowest impedance, followed by dispersant and untreated systems. This showed that reduction of voids gave more effective electron transportation. Although dispersant was proved to aid in formation of more segregated network, its anionic surfactant nature led to void-induced pathway isolation [12].

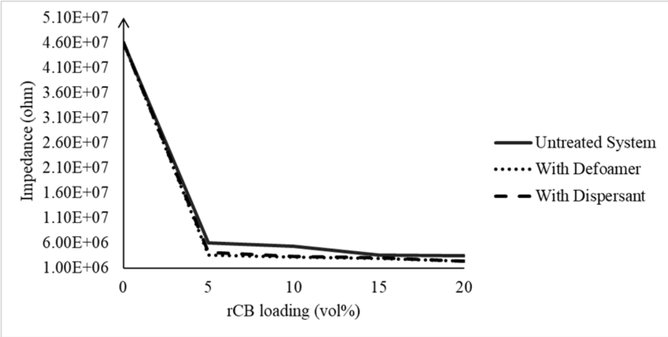

Figure 5. Effect of rCB loading on impedance of epoxy/rCB composites.

Electrical conductivity of a conductive polymer composite is highly associated to the theory of percolation, where the transition from insulative to conductive polymer composite was identified [14]. Figure 6 shows that electrical conductivity of the epoxy systems increased with increasing rCB. Defoamer. (4.70×10^{-4} S/cm to 7.22×10^{-4} S/cm) and dispersant systems (4.0×10^{-4} S/cm to 6.92×10^{-4} S/cm) displayed higher electrical conductivity as compared to the untreated one (2.79×10^{-4} S/cm to 4.87×10^{-4} S/cm). Defoamer suppressed voids formation that reduced electric field strength of the systems [15], whereas dispersant gave more homogeneous dispersion of rCB. Both factors contributed to higher electrical conductivity as compared to the untreated system.

A sudden increment from 10 vol.% to 15 vol.% of rCB marked the percolation of composite systems. Both defoamer and dispersant systems showed noticeable increase in conductivity at 20 vol.%. This might cause by the elimination of air bubbles and more uniform dispersion of rCB that further increased the effectiveness of electrical conductivity by providing void-free continuous pathways as compared to the untreated system [6, 7].

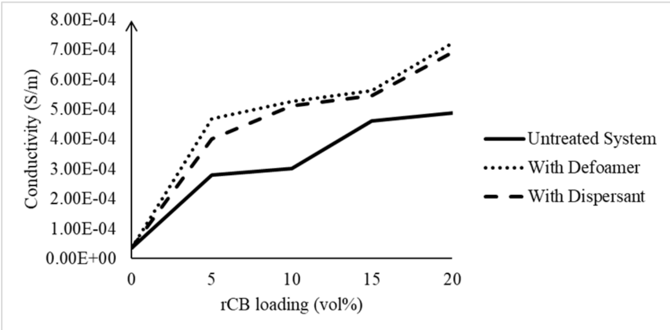

Figure 6. Effect of rCB loading on electrical conductivity of epoxy/rCB composites.

5. CONCLUSION

Recovered carbon black imparted electrical conductivity to epoxy systems but reduced mechanical properties due to impurities and aggregation. Adding defoamer improved mechanical performance and maximized conductivity by minimizing voids. Dispersing agents improved filler distribution but introduced bubbles that weakened mechanical properties. Further purification, such as modification using strong acids and higher loadings, is recommended to optimize percolation thresholds and improve overall properties.

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