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# Comparison study between recovered carbon black and commercial carbon black filled epoxy conductive materials

Huai M. Ooi[1], Pei L. Teh*[1,2], Cheow K. Yeoh[1,2], Wee C. Wong[3], Chong H. Yew[3], Xue Y. Lim[1], Kai K. Yeoh[1], Nor A. Abdul Rahim[1] and Chun H. Voon[4]

[1]*Faculty of Chemical Engineering & Technology, Kompleks Pusat Pengajian Jejawi 2, Universiti Malaysia Perlis, Taman Muhibbah, 02600 Arau, Perlis, Malaysia*
[2]*Frontier Materials Research, Centre of Excellence (FrontMate), Universiti Malaysia Perlis (UniMAP), Perlis, Malaysia*
[3]*Ecopower Synergy Sdn. Bhd., 1A, Jalan Kenari 9, Bandar Puchong Jaya, 47100 Puchong, Selangor*
[4]*Institute of Nano Electronic Engineering, Universiti Malaysia Perlis, Lot 106, 108 & 110, Blok A, Taman Pertiwi Indah, Jalan Kangar-Alor Setar, Seriab, 01000 Kangar, Perlis, Malaysia*

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**Abstract.** Waste tire management and recycling have grown to be significant issues because they bring up a global environmental concern. Thus, turning recycled waste tires into useful products may help tackle the environmental issue. This research aims to study and compare the effect of recycled carbon black (rCB) and commercial carbon black (CB) at certain 15 vol. % of filler loading on the mechanical, thermal, morphology and electrical properties of epoxy/CB composites. For this project, epoxy resin, diethyltoluenediamine (DETDA), recovered carbon black (rCB) and commercial carbon black (CB) graded N330, N550, N660 and N774 were mixed and compared accordingly to the formulation determined. The CB content was dispersed in the epoxy matrix using the mechanical mixing technique. The distribution and dispersion of CB in the epoxy matrix affect the characteristics of the conductive composites. rCB content at 15 vol% was selected at fixed content for comparison purposes due to the optimum value in electrical conductivity results. The flexural strength results followed the sequence of rCB>N774>N660>N550>N330. As for electrical conductivity results, epoxy/N330 exhibited the highest conductivity value, while the others achieved a magnitude of $X10^{-3}$ due to the highest external surface area of N330. In terms of thermal stability, epoxy/N330 and epoxy/N774 were slightly more stable than epoxy/rCB.

**Keywords:** conductive materials; epoxy; recovered carbon black

## 1. Introduction

Nowadays, soaring automotive industries result in increasing driving vehicle demand for tire production. To solve the problem of waste tire management, the pyrolysis process is introduced to transform the waste tires into commercial-grades recovered carbon black. Pyrolysis is thought to be an effective thermochemical procedure to deal with waste tires among other resource-saving techniques. In the review by Xu *et al.* (2020), the pyrolysis of carbon black has received special focus due to its widespread applicability and anticipated monetary quality in the future. There are

*Corresponding author, Associate Professor, E-mail: plteh@unimap.edu.my


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also various grades of commercial carbon black that differ in structure, namely, N110, N220, N330, N440, N550, N660 and N770 exist in the current market. Referring to the ASTM D1765 standard, the alphabet 'N' represents the normal rate of curing. The first number immediately after 'N' denotes the particle size range, whereas the two remaining numbers are randomly chosen. Most of the special-graded carbon black is created to satisfy the precise product requirements for introducing qualities like dispersion, coloring, and electrical conductivity that are necessary for certain end products (Wang *et al.* 2003). Additionally, current developments in electron microscopy that examine the composition of CB and its electrical and physiochemical properties in nanocomposites have rekindled the researchers' interest in future technological breakthroughs.

On the other hand, thermosetting polymers started to draw manufacturers' attention as an alternative to metal-made products. Thermosets are widely applied in filled or reinforced structures in order to save costs, adjust physical properties, serve as particles or fibers binder, minimize shrinkage during the curing process, and improve fire resistance. Thermosets often have good electrical insulation performance, chemical resistance, dimensional and thermal stability (Gotro and Prime 2017, Alameri and Oltulu 2023). Currently, the rising semiconductor industry is seeing an increased demand for epoxy conductive composites that are both electrically and thermally conductive. This is due to the composite's superior thermal conductivity which introduces effective heat dissipation needed in the electronic system (Mousavi *et al.* 2022). However, the epoxy itself without the addition of fillers provides poor electrical and thermal conductivities. Hence, a second conductive phase such as carbon fillers, needs to be added to the epoxy to manufacture a composite equipped with conductivity (Kam *et al.* 2019).

This research aims to produce a composite with good electrostatic discharge (ESD) and mechanical properties for ESD tray applications. The electrostatic discharge may degrade or completely destroy a semiconductor by altering its electrical properties. Contaminants can be drawn to be held by the charged surfaces, thereby making it difficult to remove the material (Phua *et al.* 2016). In this research, the characterization of epoxy with recovered carbon black between different commercial grades of virgin carbon black on mechanical, thermal, morphology, and electrical conductivity is studied. The performance of rCB is compared to that of different grades of commercial CB to determine how wide the gap between rCB and virgin CB is. So far, there has been no research done on the comparison study of different grades of virgin CB with the rCB in the epoxy system. The cured epoxy containing rCB and different grades of CB fillers is prepared. The epoxy/rCB and epoxy/CB composites are prepared to investigate the effect of rCB or CB on the epoxy resin and the characterization is focused on the electrical conductivity, physical and mechanical properties.

## 2. Methodology

### *2.1 Material preparation*

The compounding of composites in this section includes epoxy, hardener, and different rCB (1500 mesh) loadings at 0, 5, 10, 15, and 20 vol%. 1500 mesh of rCB is selected in this study because it is the commercial market grade from the company. The recovered carbon black filler used in this research is supplied by Eco Synergy Sdn. Bhd. The rCB powder was ground into a 1500 mesh size via pyrolysis from recycled tyres. First, the epoxy resin is mixed with different vol % of rCB loading, respectively. The epoxy/rCB mixture is stirred for 15 minutes using the mechanical stirrer. Then,

the epoxy/rCB mixture is treated by the ultrasonic method at room temperature for around 15 minutes. The ultrasonic process is intended to provide better dispersion of rCB in epoxy. Next, the DETDA is added to the epoxy mixture and stirred for 10 minutes. The compound then underwent the degassing process for 10 to 15 minutes approximately. The degassed compound is poured gently into the mould with dimensions of 100mm×100mm×4mm and goes through the curing process. The compound is then placed in the oven for 5 hours pre-cure at 100°C and 24 hours post-cure at 100°C to obtain the solid specimens with different vol% rCB. The compounding of composites in this section includes epoxy, hardener, and different grades of CB N330, N550, N660 and N774 with a fixed loading of 15 vol%. The mixing procedure used for this section is the same as the previous one. After being cured, the samples are cut into different dimensions for different testing and to compare to the rCB grade.

### *2.2 Electrical conductivity test*

The purpose of the electrical bulk resistance measurement is to quantitatively measure the electrical current that can go across the material. 5V of direct voltage supply at room temperature with 50% relative humidity is applied to measure the electrical conductivity. This test proceeds with the aid of the ASTM D-257 procedure on circular specimens using the digital multimeter. The dimensions of the specimen are fixed at a diameter of 20 mm and a thickness of 1mm. The specimens were painted with a conductive silver coating to lower the contact resistance. Eq. (1) shows the calculation of bulk conductivity.

$$\text{Bulk conductivity, } \sigma = \frac{1}{R} \tag{1}$$

Where, $R$=bulk resistivity

### *2.3 Flexural test*

Flexural testing determines a material's stiffness or resistance to bending by measuring the amount of force needed to bend a plastic beam (Shrivastava 2018). In a 3-point flexural test, the sheet or plate's convex side is held in tension, putting the outer fibres under the most stress and strain. Flexural testing in this section is performed by the Instron Universal Testing Machine 5569 and is specified by the ASTM D790 standard. The crosshead speed is 2.38 mm/min and the support span is about 50mm. The specimen dimension is fixed to be 60 mm×12.7 mm×3 mm (L×W×T). Eq. (2) was used to calculate the flexural strength.

$$\text{Flexural strength, } \sigma_f = \frac{3PL}{2bd^2} \tag{2}$$

Where, $P$=load at a specified point on the curve of load-deflection
$L$=support span length
$b$=sample width
$d$=sample thickness

### *2.4 Scanning electron microscopy (SEM)*

A concentrated stream of high-energy electrons is utilized by the scanning electron microscope (SEM) to produce a range of signals at the solid surface of the specimens. The signals resulting from

electron-sample interactions also provide information including the chemical composition, morphology, crystalline structure and orientation of the constituent materials of the sample (Swapp 2017). A thin layer of palladium will be coated on the specimen's fracture surface by a sputter coater machine before it is inserted onto the aluminum stab. SEM JEOL JSM-6460 LA is used to carry out the analysis of microscopy at 10 kV and 50 kV of activation voltage.

### *2.5 Fracture toughness test*

The ability of brittle materials to resist the spread of flaws in the presence of applied stress is known as "fracture toughness." The critical stress intensity factor Kc will be used to express the fracture toughness, with the former being more widely applied. Briefly described, a material's fracture resistance increases with higher Kc, which may be interpreted as a better damage tolerance (Mouritz 2012). Instron Universal Testing Machine 5569 and ASTM D638 procedures are used to conduct the test. 4mm of initial crack is cut on the specimens with dimensions of 60 mm×12.7 mm×3 mm. The loading speed is set at 1 mm/min in tensile opening mode. Eq. (3) shows the calculation of fracture toughness.

$$\text{Fracture toughness, } Kc = \alpha\sigma_{app}\sqrt{\pi a_c} \tag{3}$$

Where, $\alpha$=parameter depends on specimen and crack theory
$\sigma_{app}$=stress applied
$a_c$=critical crack length

### *2.6 Thermogravimetric analysis (TGA)*

Thermogravimetric analysis is introduced to analyse the weight change that takes place as a sample is heated at a constant pace to assess the thermal stability and the percentage of volatile components in a material (Rajisha *et al.* 2011). In this section, the ASTM 1131 procedure is used to carry out the testing using Q500 TA instruments. A 5 mg specimen is prepared in a platinum pan and placed in the heating chamber with temperatures ranging from 50 ℃ to 500 ℃ at a 10 ℃/min heating rate. Nitrogen purging is applied to the specimen at 50 ml/min.

## 3. Results and discussion

### *3.1 Electrical conductivity test*

The electrical conductivity of epoxy/rCB composites filled with 0 to 20 vol % of rCB content is shown in Fig. 1. The electrical conductivity depicts an increasing trend for the epoxy composites with increasing rCB content. Based on literature studies, the electrical bulk conductivity starts to increase when 5 vol % of rCB is incorporated and will further increase when the rCB content rises. From the demonstrated data, the percolation threshold of epoxy/rCB is determined at 15 vol % rCB loading. Therefore, rCB content at 15 vol % was used for comparison between different grades of rCB later. The increasing trend of electrical conductivity is caused by the increasing physical contact between the conductive rCB particles and the non-conductive epoxy matrix with increasing rCB loading (Kam *et al.* 2019).

The bulk electrical conductivity of epoxy/rCB composites incorporated with different grades of

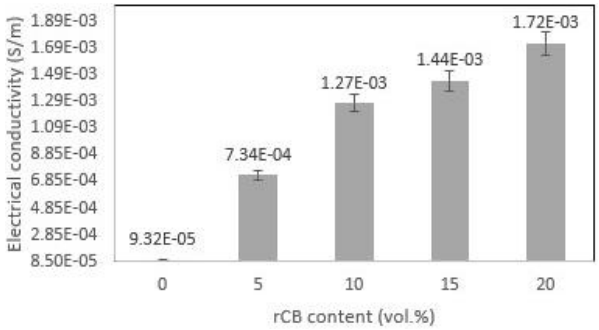



Fig. 1 Electrical conductivity of different rCB loading incorporated epoxy composites.

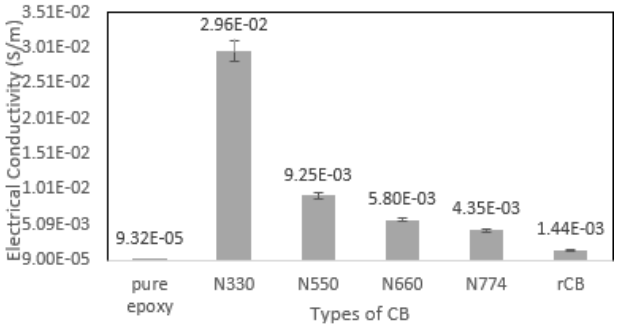



Fig. 2 Electrical conductivity of epoxy/CB composites filled with different grades of CB at fixed 15 vol% of CB loading.

Table 1 Comparison of STSA and OAN values for different grades of CB (Norris *et al.* 2023)

| CB grades | STSA [$m^2/g$] | OAN [ml/100g] |
|---|---|---|
| Vulcan®3 (N330) | 76.0 | 122.0 |
| Sterling®SO (N550) | 38.9 | 120.5 |
| Sterling®V (N660) | 37.7 | 90.1 |
| Sterling®NS(N774) | 30.0 | 72.0 |

CB fixed at 15 vol% is illustrated in Fig. 2. The electrical conductivity for different grades of CB is predicted using the value of the external surface area (STSA) and oil absorption number (OAN) (Table 1). Referring to the literature studied (Choi *et al.* 2019), the increasing STSA value suggests higher electrical conductivity. Thus, the bulk electrical conductivity of CB-graded N330 and N550 are higher than that of CB N660 and N774 as they have a higher STSA value (smaller particle size). However, the oil absorption number is also another factor that affects the electrical conductivity of different grades of CB. In addition, the electrical conductivity is also correlated to the OAN value, and a rising OAN causes the percolation threshold to decrease (Spahr *et al.* 2016). Dropping the percolation threshold may lead to an increase in the electrical conductivity value for the epoxy/CB composites. Hence, the trend of electrical conductivity of the epoxy/CB composites is portrayed by combining both OAN and STSA values. Overall, the epoxy/N330 composite obtained the highest electrical conductivity, while the epoxy/rCB recorded the lowest electrical conductivity among the others at 15 vol% CB content.

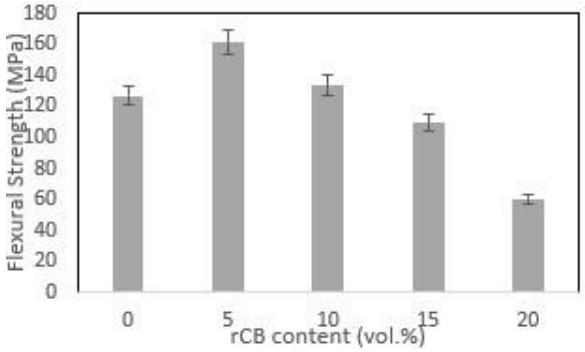



Fig. 3 Flexural strength of epoxy/rCB (recovered carbon black) composites at different rCB content

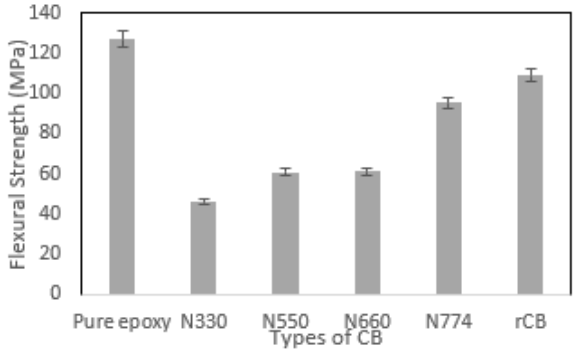


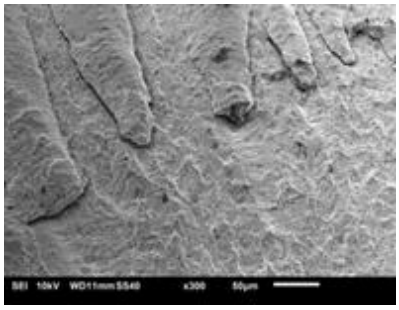

Fig. 4 Flexural strength of epoxy/CB (carbon black) with different grades of CB at 15 vol % loading

### *3.2 Flexural test*

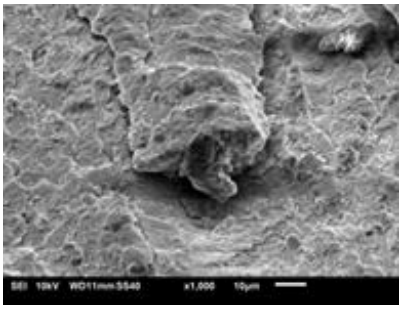
Fig. 3 shows the flexural strength of epoxy/rCB composites varied from 0 to 20 vol % rCB content. The flexural strength of epoxy/rCB composites achieved an optimum value at 5 vol % of rCB content but showed a decreasing trend with further incorporation of rCB. This happens due to the low agglomeration and better dispersion of rCB particles in the epoxy matrix. At low filler loading, rCB promotes good dispersion of filler which helps to distribute stress from matrix to filler between the epoxy matrix and rCB particles (Phua *et al.* 2016). Hence, the composites may possess higher flexural strength. In short, further increasing rCB filler loading causes an increment in the agglomeration of particles, which results in poor dispersion and weak interface interaction, thus lowering the epoxy/rCB composites flexural strength.

Fig. 4 depicts the flexural strength of epoxy/CB composites fixed at 15 vol% CB loading of different grades of CB. The flexural strength recorded an increasing trend from N330 to N774, and also rCB, respectively. This condition happens due to different grades of CB having different particle sizes. From CB grade N330 to N774, the particle size is generally increasing which leads to a decreasing external surface area which reduces the agglomeration of CB particles as referred to Table 1. This indicates that low agglomeration of CB particles has good dispersion which improves the flexural strength of epoxy/CB composites (Bera *et al.* 2018).

### *3.3 Scanning electron microscopy (SEM)*

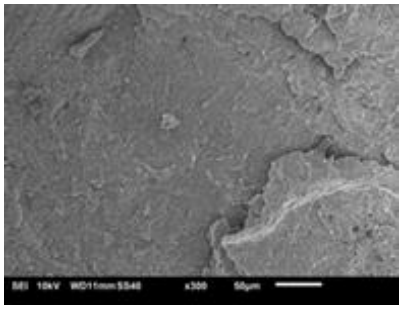


(a) At ×300 magnification

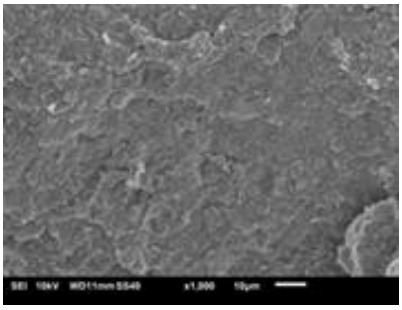


(b) At ×1000 magnification

Fig. 5 Fracture surface of 5vol% recovered carbon black filled epoxy composites

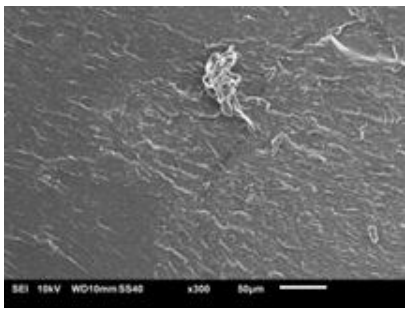


(a) At ×300 magnification

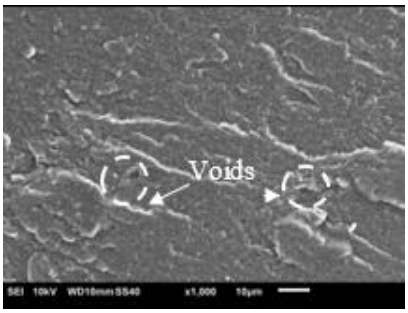


(b) At ×1000 magnification

Fig. 6 Fracture surface of 15vol% recovered carbon black filled epoxy composites.



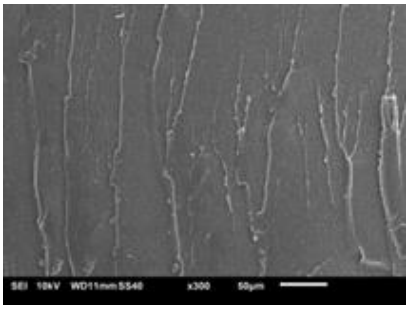
(a) At ×300 magnification



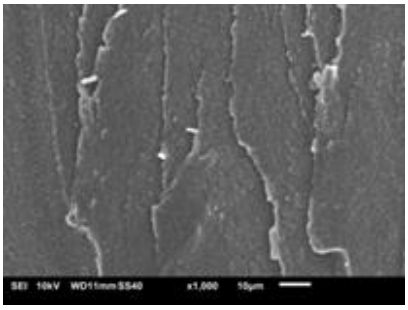

(b) At ×1000 magnification

Fig. 7 Fracture surface of epoxy/N330 composite

Figs. 5(a) and (b) depict the fracture surface of a 5 vol% rCB-filled epoxy composite at varied magnification. It is obvious that the rCB particles are evenly dispersed across the matrix of epoxy resin. With the low filler loading added, the dispersion appears quite thorough and spaced out, as seen in the photograph. As a result, there is no agglomeration of the carbon black fillers in this situation because the adhesion forces at the matrix-filler interface are greater than the cohesion forces of the fillers (Verma *et al.* 2020). Next, Figs. 6(a) and (b) indicate the fracture surface for a 15 vol% rCB-filled epoxy composite at different magnifications. The figures show how the rCB particles dominate the epoxy matrix, resulting in a drop in the composite's flexural strength. Within



(a) At ×300 magnification



(b) At ×1000 magnification

Fig. 8 Fracture surface of epoxy/N774 composite

the layers of epoxy, the rCB particles clump together and form clusters. This agglomeration causes a stress concentration point to occur, which reduces the flexural strength of the composite. Fig. 6(b) shows there are a few tiny voids that occurred on the fracture surface which suggested higher interaction behaviour. Overall, the main reason for the epoxy/rCB composite with 15 vol% loadings having weak strength was said to be the existence of multiple clusters at various locations within the matrix.

The SEM micrographs of Figs. 7(a) and (b) depict the surface morphology of EP/N330 at 15 vol% loading with different magnifications. Fig. 7(a) demonstrates that the matrices and CB were mixed homogeneously but were present with bubble voids. Fig. 7(b) shows the agglomeration of particles for EP/N330 morphology and the occurrence of some voids. The clustered structure has imparted the composite with high electrical conductivity as the agglomeration of particles has formed a conductive pathway. On the other hand, the agglomeration of CB particles and the bubble voids present have introduced poor particle distribution in matrices and poor flexural strength to the EP/N330 composite. The non-uniform distribution of CB particles in the epoxy matrix fails to provide uniform strength to withstand the load applied. The agglomerated particles have proved that the CB N330 has a high external surface area which increases the intermolecular interactions between the CB particles and allows them to stick together. This is the reason epoxy/N330 recorded the highest electrical conductivity among all samples.

SEM images Figs. 8(a) and (b) show the surface morphology of EP/N774 at 15 vol% CB content with different magnifications. Fig. 8(a) demonstrated good distribution and dispersion for the morphology and no bubble voids were observed. This depicted the high interaction behaviour between the CB particles and the epoxy matrix (Verma *et al.* 2020). There are fewer conductive pathways shown in Fig. 8(b) as it pictures less agglomeration of CB particles. Hence, the electrical conductivity imparted in EP/N774 is lower than EP/N330. Less CB particle agglomeration has confirmed that the CB N774 has a lower external surface area with lower intermolecular interaction which reduces the chance for the CB particles to stick together. Thus, epoxy/N774 has lower electrical conductivity compared to epoxy/N330. Since the EP/N774 composites have good morphological distribution and dispersion, the composite has better flexural strength than the EP/N330.

### *3.4 Fracture toughness test*

The fracture toughness of epoxy/rCB composites at filler contents of 0, 5, 10, 15 and 20 vol % is

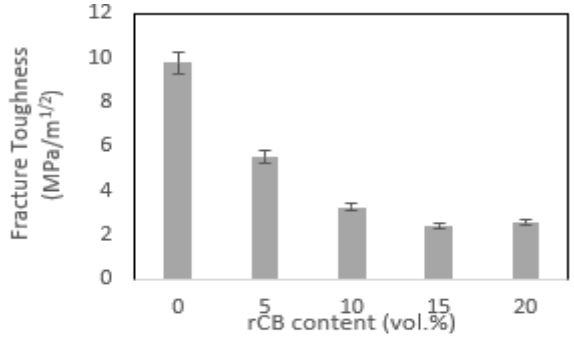



Fig. 9 Fracture toughness of epoxy composites added with different loading of recovered carbon black (rCB)

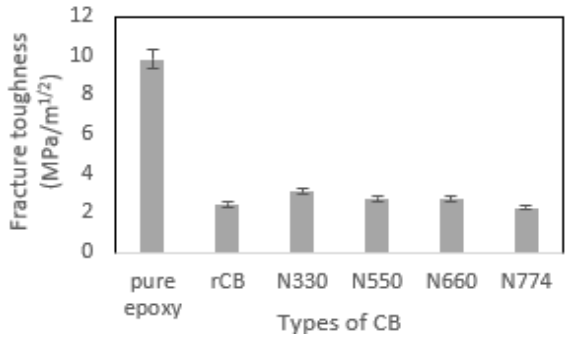



Fig. 10 Fracture toughness of epoxy composites added with different grades of carbon black (CB) at 15 vol %

shown in Fig. 9. In several studies, the fracture toughness shows a decrease with increasing loading of rCB in the epoxy matrix. This situation is caused by the uneven dispersion and distribution of rCB aggregates into the epoxy matrix. The decrease in fracture toughness is caused by the primary aggregates developing a loose secondary structure of agglomeration by weak Van der Waals forces as the rCB particles accumulate in grape-like form (Phua *et al.* 2016).

Fig. 10 demonstrates the fracture toughness at 15 vol % of CB loaded with different grades of CB-filled epoxy composites. The fracture toughness shows a decreasing trend from CB N330 to N774, however, the epoxy/rCB exhibited slightly higher fracture toughness than N774. Based on the literature, the fracture toughness of epoxy composites may be affected by the particle size of CB. The particle size of CB N330 to N774 increases accordingly causing the crack resistance to decrease as well as the fracture toughness (Lauke 2008).

### *3.5 Thermogravimetric analysis (TGA)*

Fig. 11 indicates the thermogram of a pure epoxy and 15 vol% rCB-filled epoxy composite. Both pure epoxy and epoxy/rCB show thermal stability until approximately 200°C. Thermogravimetry analysis (TGA) of the samples is recorded in Table 2. The initial temperature for decomposition of pure epoxy is slightly higher than the epoxy/rCB, this shows that the addition of rCB has deteriorated the decomposition temperature of the epoxy. Pure epoxy experienced drastic weight loss at 372.21 ℃ while epoxy/rCB was at 362.05 ℃. The reduction in thermal stability after adding the rCB

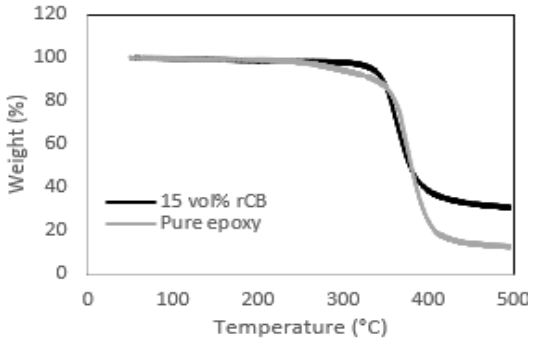



Fig. 11 Comparison of weight loss percentage between pure epoxy and 15 vol% rCB filled epoxy composites

Table 2 TGA of pure epoxy, epoxy/CB and epoxy/rCB fixed at 15 vol% filler content

| Samples | $T_{on\ set}$ [°C] | $T_{max}$ [°C] | $T_{end\ set}$ [°C] | Weight loss [%] |
|---|---|---|---|---|
| Pure epoxy | 354.23 | 372.21 | 398.04 | 86.23 |
| Epoxy/rCB | 343.80 | 362.05 | 386.26 | 67.51 |
| Epoxy/N330 | 356.93 | 372.13 | 397.63 | 68.39 |
| Epoxy/N774 | 361.99 | 372.39 | 395.24 | 67.37 |

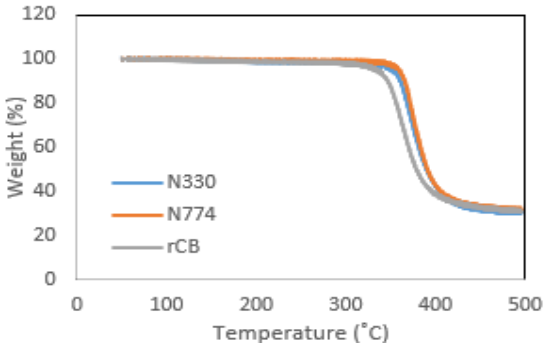



Fig. 12 Comparison of weight loss percentage between different grades of CB and rCB filled epoxy at 15 vol% loadings

suggested that the impurities in the rCB have affected the thermal performance of the samples as the rCB used is recovered from waste tires. Next, the $T_{max}$ of epoxy/rCB is lower than the pure epoxy. Since it is highly correlated to the mechanical properties of the composites, the TGA result has confirmed the flexural properties of pure epoxy are better than epoxy/rCB at 15 vol% loadings. Overall, the residue left after the thermal decomposition of epoxy/rCB is higher than the pure epoxy.

Fig. 12 indicates the thermogram of different grades of CB and rCB-filled epoxy composites at 15 vol% loadings. The composites show thermal stability until approximately 300 °C. Thermogravimetric analysis of the samples is recorded in Table 2. The initial temperature for the decomposition of the composites is arranged following the sequence: N774>N330>rCB. This shows that the epoxy filled with N774 has the highest thermal stability compared to the other samples. Epoxy/N774 recorded radical weight loss at 372.39 °C while epoxy/N330 is at 372.13 °C. The reduction in thermal stability indicates similar thermal behaviour for N774 and N330-filled epoxy

composites and they have better thermal stability compared to the epoxy/rCB. Overall, the residue left after thermal decomposition for all the composites is almost the same.

## 4. Conclusions

This paper studied the effect of rCB content and compared the mechanical, thermal, morphology and electrical conductivity properties of epoxy/rCB conductive materials between rCB and different grades of commercial CB. As conclusions:

- The percolation threshold is determined at 15 vol% CB loading, N330 has recorded the highest bulk electrical conductivity which is confirmed by the SEM image which has demonstrated the agglomeration of CB particles, distribution and intermolecular interactions.
- 5 vol% rCB-filled epoxy indicates better flexural properties by comparing different loadings of rCB. Also, the flexural properties of epoxy/rCB are better than the epoxy filled with different CB contents at 15 vol%.
- Furthermore, the epoxy/N330 recorded the highest fracture toughness as it has a smaller particle size which enhances the crack resistance. Last but not least, epoxy/N774 has the best thermal stability and the highest rate of weight loss among all the samples.

## Acknowledgments

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