An Investigation of Tensile and Thermal Properties of Epoxy Polymer Modified by Activated Carbon Particle

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Abstract. In this work, the effects of adding activated carbon (AC) powder with epoxy resin were investigated experimentally. The particulate epoxy composites are manufactured in vacuum technique with different weight fraction ratios of AC (0, 5, 10, 15, 20, 25, 30, 35 and 40) \(^\%\) wt. The particle size was measured during this work by laser particle size analyzer with an average size of about (14.74µm). The interaction between epoxy material and AC powder was examined by using Fourier Transform Infrared (FTIR) spectroscopy analysis. Moreover, the glass transition temperature (Tg) of the pure epoxy and composite material were measured by Differential Scanning Calorimeter (DSC). The tensile strength behavior and interaction strength between the matrix material and powder were investigated by conducting tensile test and SEM analysis. The results of FTIR test reveal that there is no a new peak after reinforcing epoxy with AC powder, which proves there is a strong interaction between epoxy resin and AC powder. The DSC results show that the increases by adding AC to epoxy will increase Tg temperature. The findings of FTIR analysis were supported by SEM analysis, which shows a good interaction and strong interfacial between matrix and particles. The tensile strength values increased with increasing AC content up to 15 \(^\%\) wt. with a max value of 26.34 MPa (19.16\%), then it decreased to 18.15 MPa at 40 \(^\%\) wt.

Keywords. Activated carbon, Epoxy, Tensile strength, DSC, FTIR, SEM.

1. Introduction
Classic materials like metals and metal alloy are not sufficient in modern industry requirements. For this reason, it is necessary to create material with specific characteristics. Composite material provides these characteristics like low-density, high fatigue, strength, toughness, high resistance to friction and wear, good mechanical and thermal properties. is a material consisting of two or more types of materials with different specification, physically or chemically. The resulting material will be of different specifications from the original materials [1]. The disadvantage of composite material is the high cost of matrix materials and reinforcement materials, difficult and high cost of fabrication [2]. The polymer matrix provides easy fabrication and low cost compared to metal and ceramic. Also, the particulate reinforcement provide these specifications compared with other reinforcement (fiber and laminate) [3]. Activated carbon powder is carbon rich material from 87\% to 97\% with high surface area larger than 1000 square meter over gram due to its porous structure pore diameter of less than 0.6 cc, this makes it excellent absorbent [4, 5]. The AC particles develop the mechanical properties of the
polymers due to their high porosity, which enables the matrix material to penetrate inside and thus form high bonding. A polymer composite reinforced with activated carbon AC particles is used as structure materials, marine application, fishnets, furniture and another household applications [6]. Few studies examined the effect of activated carbon particles on composite properties. Salleh et al. [7] studied the tensile effect on composite material containing activated carbon particles with weight fraction (2%, 4%, 6%) reinforced with polypropylene. The composite were then encapsulated with epoxy resin. The results revealed that tensile stress developed when activated carbon increased but the impact strength increased when polypropylene increased. Shakuntala et al. [8] studied the effect of 5, 10, 15, and 20 % wt. of wood apple shell particles on mechanical and erosive wear properties of epoxy composite. The results demonstrated that tensile, flexural, and erosive wear resistance strength increased with the increasing the particles. The loading of 15% was the best. Abass et al. [9] investigated the effect of orange peels particles on mechanical properties (Hardness, Impact, compression and tensile tests) of polyester composites with weight fraction (2, 4, 6, 8 and 10) %. The results indicated that the mechanical properties increased with the increasing of particles weight fraction. Khalil et al. [10] analyzed the effect of carbon black and activated carbon produced from bamboo reinforced polyester composite on tensile and flexural properties with (10, 20, 30, and 40) % weight fraction and particle size (100 mm). The results showed that tensile and flexural properties decrease after 10%. Salleh et al. [11] examined the effect of activated carbon powder in epoxy matrix on tensile properties. The activated carbon was prepared from three types of coconut shell carbon Komeng coconut shell (CKCS), carbon young coconut shell (CYCS), and carbon ripe coconut shell (CRCS) with weight fractions of (0, 5, 10, and 15) %. The results highlighted that the composite containing CKCS showed better tensile properties than other, and the tensile stress increased with the increase of activated carbon powder. The effect of carbon black particles with size of (14 µm), and weight fraction of (5, 7.5, and 10) % imbedded in polyester matrix on mechanical properties (tensile strength, hardness, impact strength, flexural strength, and density) was studied by Salman et al. [12]. The results pointed out that the mechanical properties developed when carbon black increased. Da costa et al. [13] explored the effect of carbon powder wastes embedded in epoxy matrix in different mass fractions of (0, 2.5, 5, 7.5, and 10) % on mechanical properties, which are elastic modulus and strength under tensile, compressive, flexural and impact loadings. The results disclosed that the addition of carbon powder develops tensile strength, compressive strength, and flexural strength.

Khalil et al. [14] considered the flexural properties of epoxy composite reinforced with weight ratio of 5% of carbon black prepared from three materials (oil palm fiber bench, coconut shells, and bamboo stem). The results designated that all types used had flexural properties better than epoxy resin alone, and coconut shell was better than the others. Mostafa et al. [15] examined the effect of activated carbon on tensile, flexural, impact strength, and strain-to-failure behaviour of polyester composite reinforced with jute fiber with weigh fraction (1, 3, 5 and 10) % of AC. The results established that the mechanical properties increased with AC fraction increasing.

In this study, activated carbon particles with a size of about (14.74μm), measured by laser particle size analyzer, to reinforce epoxy resin with different weight fraction ratios (i.e. 0, 5, 10, 15, 20, 25, 30, 35, and 40) % has been investigated. The main aim of this investigation is to study the Fourier-transform infrared spectroscopy (FTIR), and differential scanning calorimetry (DSC) test to find glass transition temperatures (Tg), as well as scanning electron microscope (SEM) of such composite. In addition, tensile test has been conducted to find the elastic modulus, ultimate tensile strength, and maximum elongation.

2. Materials
The epoxy resin was used as matrix material provided from Sikadur 52 Company. It was mixed with the hardener at a ratio of (2:1) via weight. The standard epoxy resin properties are shown in table 1, [16]. Micro activated carbon powder [17], with average size of particles about 14.74 µm was used as a reinforcement material within the epoxy matrix with various weight ratios.
### Table 1. Standard properties of epoxy [18].

| Property                  | Value  |
|---------------------------|--------|
| Tensile strength (MPa)    | ≤ 25   |
| Flexural strength (MPa)   | 53     |
| Modulus of elasticity (GPa) | 1.060  |
| Viscosity at 20 °C (mPa.s)| 500    |
| Density (kg/m³)           | 1100   |

### 3. Composite fabrication

Nine types of composites were fabricated during this work. The activated carbon particles reinforced the epoxy with different weight fraction ratios (0, 5, 10, 15, 20, 25, 30, 35, and 40) %. The purchased activated particles were ground and sieved by standard sieves with size between 8-38 μm. The sieve was placed in a shaker then the particles were analyzed by size analyzer device to obtain the average size of particles. The activated carbon particles were mixed with the epoxy resin and mechanically moved by using a laboratory shear mixture at 1000 rpm for mixing duration of 1 hour. The activated carbon particles dispersed in epoxy resin. For 30 min, the mixture was left to release trapped air bubbles. Good-dispersed activated carbon-epoxy mixture with stable suspension of the activated carbon particles within the epoxy matrix was obtained. To obtain the required composites specimens, a vacuum bag was used as shown in figure 1. The mold of tensile specimen was fabricated from PVC by CNC method. Then, the mixture of activated carbon with epoxy was poured into a closed mold and then the mold was placed inside a vacuum device for 24 hours to prevent air bubbles from the composite, where the pressure was reduced to lower than -30 psi. The composite specimens were left for 48 hr, to dry at room temperature and then the product specimens were left for 3 hr in an oven at 70°C for sufficient curing.

![Figure 1. Vacuum bag technology.](image)

### 4. Experimental setup

#### 4.1. Particle size analyzer

The Laser diffraction particle size analyzers device (Better size 2000, china) was used to calculate particle size from the angle of light scattered by a stream of particles passing through a laser beam.

#### 4.2. Fourier-transform infrared spectroscopy (FTIR)

A technique was used to analyze and identify materials (solid, liquid, and, gas). FTIR analyzing infrared light was used to scan material specimens to obtain the chemical properties and physical bonding of composite materials. The resulting signal at the detector was presented as spectrum from 4000 to 400 (1/cm) representing a molecular finger print of the specimen. The specimen mix with potassium bromide (KBr) was used as carrier for the specimen because it does not show any absorption and its transition is 100% between 4000 and 400 (1/cm). FTIR was used to determine
whether the chemical structure remains the same between epoxy composites and neat epoxy (physical interaction). The specimens were scanned by IRAffinity-1 Fourier-transform infrared spectroscopy (FTIR), Shimadzu.

4.3. Differential scanning calorimetry (DSC)
The glass transition temperatures (Tg) of neat epoxy and epoxy/activated carbon composites were determined by differential scanning calorimeter (DSC). To obtain glass transition temperatures Tg, Differential scanning calorimetry (DSC-60 SHIMADZU) were used. It is a thermo analysis method, which measures the change for heat needed to increase the temperature of a specimen and reference.

4.4. Tensile behavior test
The tensile specimens were manufactured according to the specification of ASTM D638 [18], and performed using a universal testing machine of type H50KT-0404, Tinius Olsen, UK, with a cross-head speed of 2 mm/min. For more accuracy, three specimens for each composite have been tested and the average value of each weight fraction was taken.

4.5. Scanning electron microscope (SEM)
Microstructures of the composites were assessed with scanning electron microscopy. FEI Quanta 450 scanning electron microscope (USA), was used to examine the prepared composites specimens to show the nature of fracture.

5. Results and Discussions

5.1. Laser particle size analyze results
Particle size distribution of analyzing a specimen with different size ranges and giving the number of particles of several sizes is preset in the specimen, as shown in figure 2. Distribution of particle size D50 is defined as the average value of the distribution of particle size, which is particles size at 50% in cumulative distribution. The particle diameter of the AC powder was measured during this work. It was found that D10 =2.945 µm is a cumulative 10% point of size, D50 =14.74 µm is also defined as the average particle diameter, and D90 =28.7 µm.

![Figure 2. Size distribution of activated carbon particles.](image)

5.2. Fourier-transform infrared spectroscopy (FTIR) results
Figure 3 shows the results of FTIR test, between 4000 (1/cm) & 400 (1/cm) wave length. The FTIR spectra of pure epoxy where the functional group appeared was: OH appeared at wave number 3433.29 (1/cm) [19] [20], the band at 2925.58 (1/cm) is characterizing to CH2[20], the band at 2870.08 (1/cm) gave CH3 [17], at 1612.49(1/cm) assigned C=O, at 1512.19 (1/cm) and 825.53(1/cm) assigned aromatic bonds [19][20], at 1242.16 (1/cm) and 1033.85 (1/cm) indicate C-O-O ether bonds [20]. The FTIR results of the specimens, pure epoxy and epoxy composites reinforced with various weight fraction ratios of activated carbon powder of (5, 10, 15, 20, 25, 30, 35 and 40) %, are clarified.
in figure 3. It can be noticed that there are no new peaks after reinforcing by the AC powder. Also, there was no shifting in any of these peaks, and the intensity of all characteristics composite peaks increased with the increasing in concentration of AC particles. It reaches the maximum value at (40 % wt.). This behavior refers to physical bonds in composite constituents. This is a good indication of improvement of the miscibility state between composite constituents, and absence of any residual monomer. This finding is in agreement with [21].

Figure 3. FTIR spectra of all specimens, where x-axis represent wave length in (1/cm) and the y-axis represent the absorbance (Abs).

5.3. Results of differential scanning calorimetry (DSC)

The glass transition temperatures (Tg) of neat epoxy and epoxy/activated carbon composites are calculated by differential scanning calorimeter (DSC). Tg is an important factor in composites and epoxy resins which is define the temperature of applications. It is normally below Tg. It was determined for each specimen from the midpoint of the corresponding glass transition regions [22]. Table 2 shows glass transition temperature for neat epoxy and epoxy reinforced with (5, 10, 15, 20, 25, 30, 35, 40) % wt. activated carbon. From this analysis, it can be indicated that Tg of the composite specimens increased continuously with increasing the weight fraction ratios of activated carbon. It is observed from this table that the neat epoxy has Tg of 147.76 °C, the same type of trend is obtained by [23]. The increase in the Tg with AC addition may be attributed to high cross link strong interfacial between the particles and epoxy, which reduces the mobility of the chains around the high surface area microporous particles. Finally, the increase in the crosslink density may result in higher Tg.

Table 2. Glass transition temperature for neat epoxy and epoxy composite.

| AC % wt. | Tg (°C) |
|----------|---------|
| 0%       | 147.76  |
| 5%       | 151.39  |
| 10%      | 162.13  |
| 15%      | 177.48  |
| 20%      | 177.50  |
| 25%      | 179     |
| 30%      | 186.8   |
| 35%      | 190.11  |
| 40%      | 192.83  |
5.4. Tensile test result
Figures 4 & 5 and Table 3 show the results of tensile properties of neat epoxy and epoxy reinforced by activated carbon powder specimens with (5, 10, 15, 20, 25, 30, 35, 40) % wt. It is clear that there is nonlinear behavior of all specimens. The ultimate tensile stress increases with increasing weight fraction ratios of activated carbon particles until reaching the maximum value at 15% (which was 26.34 MPa). This is because of the high connection between epoxy and activated carbon particles due to the high surface area of this particle with the addition of the excellent adhesion of epoxy. This finding is consistent with . After that, the ultimate tensile stress begins to decrease to reach the lowest value at 40%, which was 18.15 MPa. This is attributed to the de-bonding between AC Particles and epoxy matrix and the number of agglomerations increasing and becoming more present as a result of increasing AC content where it acts as a stress concentration which contribute to decreasing strength. The increase loading of AC to 15 % wt. led to increase in the tensile modulus of the composite to the max value of 2.902 MPa due to the addition of AC particles which impart stiffness. After 15 % wt. of activated carbon loading, deterioration occurred in the tensile modulus of the material. This is due to the increasing of brittleness of composite as a result of the AC particle brittleness nature.

![Figure 4. Stress-strain diagram.](image)

(a) The effect of activated carbon % wt. on ultimate tensile strength.  (b) The effect of activated carbon % wt. on modulus of elasticity.

![Figure 5. The effect of activated carbon % wt. on tensile tests.](image)
Table 3. Tensile properties for all specimens.

| Activated carbon %, wt. | Ultimate tensile strength (MPa) | Yield stress (MPa) | Modulus of elasticity (GPa) | Strain at break point % | Poison ratio |
|--------------------------|---------------------------------|-------------------|---------------------------|------------------------|-------------|
| 0%                       | 21.34                           | 15                | 1.01                      | 9.77                   | 0.34        |
| 5%                       | 25.3                            | 18                | 1.945                     | 9.5                    | 0.3         |
| 10%                      | 23.304                          | 17                | 1.539                     | 9                      | 0.3         |
| 15%                      | 26.34                           | 19                | 2.902                     | 8.97                   | 0.32        |
| 20%                      | 24.6                            | 15                | 2.42                      | 7.3                    | 0.31        |
| 25%                      | 23.004                          | 14                | 2.254                     | 7.24                   | 0.3         |
| 30%                      | 20.16                           | 13.41             | 2.030                     | 7.03                   | 0.3         |
| 35%                      | 19                              | 13                | 1.919                     | 6.99                   | 0.32        |
| 40%                      | 18.15                           | 12                | 1.818                     | 6.82                   | 0.3         |

5.5. Results of scanning electron microscope (SEM)

Scanning electron microscope (SEM) micrograph shows the fracture surface and a morphological inspection to investigate the microstructure of tensile specimens of activated carbon filled epoxy composites with 10, 15 and 20 % wt. as can be seen in figure 6 (a, b, and c respectively). The SEM test was conducted to examine the effect of activated carbon powder loading and the adhesion of particles–matrix interphase of the composites and the distribution of activated carbon particles in matrix. The composite failed by brittle way as shown in Figure 6. The micrograph of 10 % wt. specimen shows agglomeration and inhomogeneous distribution of activated carbon powder which appears as elliptic white colour due to the bad mixing as can be seen in figure 6 (a). Meanwhile, the mixing seems to have better distribution in 15 % wt. as shown in figure 6 (b). The micrograph of 20 % wt., seen in figure 6 (c) shows there is a de-bonding or detached phenomenon between the activated carbon powder and epoxy matrix. Moreover, the numbers of carbon clusters increased due to the increasing in carbon powder content which acts as stress concentration regions leading to decrease in the strength of the composites.

Figure 6. Micrographs by Scanning electron microscope (SEM) images of fracture surfaces for (a) 10 % wt. of activated carbon (b) 15 % wt. of activated carbon (c) 20 % wt. of activated carbon.

6. Conclusion

In this work, the effects of adding activated carbon (AC) powder with 5, 10, 15, 20, 25, 30, 35, 40 % weight fractions on epoxy resin were investigated experimentally. According to the results obtained from laser particle size analyzer, FTIR, DSC, tensile, and SEM tests, it can be concluded that:
1. There is no new peak or peak shifting after reinforcing the epoxy with activated carbon powder and the bonding type between the matrix and the reinforcement material was a physical bonding, which indicates that there is no new material formed.

2. The glass transition temperature increased with increasing the activated carbon content. The maximum value of $T_g$ is $192.83^\circ C$ which is obtained for composite specimen reinforced with 40% of activated carbon powder.

3. The ultimate tensile strength and elastic modulus of pure epoxy improved with the addition of activated carbon powder until reaching the maximum value at 15% wt. with a percentage increase of 16.6% and 65.19% for ultimate tensile strength and elastic modulus, respectively, as compared with pure epoxy.

4. The strain decreases with the increasing of activated carbon powder because of the brittleness nature of activated carbon.

5. The composites failed in the brittle manner, and 15% wt. of activated carbon powder had the best structure.

| Symbols definitions |
|---------------------|
| **Symbol** | **Definition** |
| AC | Activated carbon. |
| FTIR | The Fourier transform infrared |
| DSC | Differential Scanning Calorimeter |
| SEM | Scanning electron microscopy |
| $T_g$ | The glass transition temperature |
| Cumu. | Cumulation |
| Diffra. | Diffraction. |
| E | Modulus of elasticity |
| OH | Hydroxide. |
| CH$_2$ | Methylene. |
| CH$_3$ | Methyl group |

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