

EFFECTS OF EXPANDABLE GRAPHITE ON FLAMMABILITY, THERMAL AND MECHANICAL PERFORMANCE OF PALM EMPTY FRUIT BUNCH FIBRE REINFORCED COMPOSITE

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Abstract

Natural fibre reinforced composites (NFRC) possess the tendency of being highly flammable in nature. The current paper investigates the effectiveness of expandable graphite (EG) on the enhancement of the flame retardancy of palm empty fruit bunch (EFB) fibre filled diglycidyl ether of bisphenol A (DGEBA) thermoset epoxy composites. The EFB was treated with 5 wt% sodium hydroxide (NaOH) to improve fibre surface adhesion with the epoxy matrix. In this research, two approaches were employed to introduce EG with concentration ranging from 1 to 5 wt% into the epoxy composite filled with 20 wtn% of EFB. The first approach involved infusion of a pre-mixed EG filled epoxy resin into EFB mat during composite fabrication while the second approach considered was the application of EG filled epoxy resin as a surface coating onto post cured EFB epoxy composite. The flammability, thermal and mechanical properties were studied through a series of experiments, such as the Bunsen burner test, dynamic mechanical analysis (DMA), tensile, flexural and impact tests. The results showed that increasing EG concentration capable of enhancing the flame retardancy and mechanical behaviour in both approaches, with 5 wt% EG demonstrated superior performance in fire resistant and mechanical properties amongst other formulations.

Keywords: Empty fruit bunch, Epoxy, Expandable graphite, Flame retardant, Palm fibre.

1. Introduction

The demand to introduce lightweight, eco-friendly and sustainable structural materials has gained significant momentum in recent years, particularly in transportation and building industries such as the automotive, aerospace and construction. This is backed by the growing awareness of environmentalists' campaign and various governmental incentives in offering tax reduction. For those products traditionally made from fibre-reinforced composites, numerous manufacturers and material scientists have initiated intense research collaboration to incorporate natural fibres as reinforcement owing to their green credential and other advantages in material properties as compared to fibres of synthetic origin. These include the lower cost, lightweight, biodegradable, less abrasive to processing equipment and availability as sustainable renewable resources [1-3].

Malaysia is the world second largest in palm oil plantation after Indonesia. Approximately 3.1 million hectares of land were used for oil palm cultivation and EFB waste generated from the palm oil industry was about 8 million tons annually [4]. It has been reported that these EFB fibres are suitable to be incorporated into a composite for their high cellulose content and high toughness [5]. In order to minimise the residual EFB biomass that could end up as industrial waste or landfill, palm EFB was chosen as the natural reinforcement for the NFRC. Care must be taken in scientific research utilising natural sources such as the EFB, the quality of the fibre could be greatly influenced by plant species and environmental factors, such as climate conditions, soil characteristics, and seasonal change [6]. In addition, natural fibre has the tendency to absorb moisture, which affects the properties of the composite. The hydrophilic nature of EFB fibre that arises from the presence of hydroxyl group could also lead to poor interfacial adhesion with hydrophobic polymer matrices [5]. Therefore, natural fibres are often subjected to surface pre-treatment with a chemical such as alkali, which removes fibre constituent such as dirt and oil residue, leaving a higher concentration of cellulosic compounds to enhance fibre matrix bonding and improve the overall mechanical properties [3, 7].

Thermosetting polymers are commonly used in fibre reinforced composite as the resin is readily in liquid or viscous form at room temperature, which facilitate a facile route to fully wet out the fibre mat through impregnation. Among the vast variety of thermosetting resins, epoxy is one of the most popular thermosets used to produce fibre-reinforced polymer composite. Epoxy itself has wide applications as coatings, adhesives and its composite has shown great potential in the aerospace industry [8].

Nevertheless, both palm EFB fibre and epoxy are highly combustible and there is an absolute need to reduce the flammability characteristic of the composite. In the current research, flame retardant was incorporated into the material formulation and study the resulting flammability characteristic of the composite. Flame-retardants work by disrupting the combustion cycle of the composite [9].

Expanded graphite (EG) is often used as a flame retardant additive or in the form of coating [10]. It is a kind of intumescent flame retardant, which is capable to experience significant expansion and form char layer on the surface of the composite when exposed to heat. The reason for this is the existence of an expansion agent, which is usually acid. For example, sulfuric acid (H_2SO_4) are intercalated between the carbon layers of EG. This causes EG to be expanded to a worm-like shape due to the reaction at high temperature. The char formed by

expanded graphite act as an insulation layer. It extinguishes the flame by protecting the composite inside from radiant heat and flame in the condensed phase [11-13].

This research studied the effect of EG on palm EFB fibre reinforced epoxy composite with respect to the flammability, thermal and mechanical properties. Two approaches were employed. The first approach was based on composite filled with EG, and the second approach consists of composite covered with EG and epoxy as a surface coating. It is worth noting that this research was not emphasized on the mechanical properties of the coating composite because the coating had no EFB fibre as reinforcement and it had an insignificant effect of the coating of the composite.

2. Experimental

2.1. Materials

The palm EFB fibre was pulverised and supplied by Polycomposite Sdn. Bhd. The short strand fibre had an average length of approximately 3 mm. NaOH in pellet form was purchased from R&M Chemicals. The thermoset epoxy resin and hardener were of the trade name Ultimeg 2020 purchased from AEV Ltd. The ratio of the mixture is 1 part hardener to 5 part the resin in weight. The specific grade of the epoxy resin had a high viscosity at room temperature and high bonding strength after curing. The EG was supplied by Qingdao Kropfmuehl Graphite Co. Ltd. The grade name of the EG is "ES 350 FS". Based on the analysis data obtained from the department of quality control of the supplier, the particle size of EG was a mixture of 80% above 300 μm and 1.2% below 150 μm , while the remaining 18.8% fell in between the two sizes. The expansion rate of EG was 510 ml/g, with the activation temperature ranging from 200 to 230°C.

2.2. Fibre treatment

The palm EFB fibre was treated with NaOH before the composite fabrication to reduce surface contaminants and oil residue without damaging the fibre properties. First, a solution containing 5 wt% of NaOH and 95 v% of distilled water was prepared. The palm EFB fibre was soaked in the solution for 24 hours. After that, the solution was disposed and the palm EFB fibre was rinsed with distilled water until the pH value dropped to approximately 7. Finally, the wet palm EFB fibre was dried in an oven at 70°C for another 24 hours.

2.3. Fabrication of EG filled palm EFB fibre reinforced composite

The method selected for fabrication of the composite was based on a technique similar to solution casting. Table 1 summarises the formulation of the composite with varying concentration of EG (in term of weight, wt%). Prior to the fabrication, EG, palm EFB fibre and beakers were dried in an oven maintained at 70°C for 24 hours to remove the moisture. This was to prevent the formation of voids in the composite as the exothermic curing process may cause the water molecules to turn into gaseous form [14].

The first step was mixing the materials. In order to achieve homogeneous distribution, a predetermined amount of EG and epoxy resin were initially mixed in a beaker with a spatula. The mixture was placed under ultrasonication with Hielscher Ultrasonic Processor UP400S at amplitude 50% and frequency of 24 kHz for 15 minutes. The palm EFB fibre was added into the mixture and placed under

Fisher Scientific mechanical mixer operated at 250 rpm for 10 minutes. For the control sample (0% EG), the ultrasonication was not included in the process.

Table 1. Formulation for EG filled composite.

EG	EFB	Epoxy & hardener	Amount of EG	Amount of EFB	Amount of epoxy & hardener
(wt%)	(wt%)	(wt%)	(g)	(g)	(g)
0	20	80	0	18	72
1	20	79	0.9	18	71.1
3	20	77	2.7	18	69.3
5	20	75	4.5	18	67.5

The second step was conducted to remove the air induced during material mixing. The mixture was degassed for 10 minutes inside a catch pot using a vacuum pump. The hardener was subsequently added into the mixture and gently mixed for 5 minutes. The final mixture was degassed for another 5 minutes. The reason of multiple degassing stages was to remove the air in the mixture as much as possible after mixing.

The third step was setting up the mould for casting. The mould was made of four rectangular aluminium and two acrylic plates, where the aluminium plates were used to form the boundary frame on an acrylic plate, forming a cavity of 200 mm × 100 mm × 2 mm. The frame held in position with the use of masking tape. A release agent was applied on the frame and acrylic plates for the ease of demoulding the composite.

The fourth step was casting the final mixture into the mould. The process of transferring the mixture had to be gentle to prevent air from being induced into the composite. The mixture was manually spread across the mould and left for 30 minutes to achieve a partially cured state. Subsequently, a second acrylic plate was placed on top to encompass the partially cured composite with the cavity of a closed mould. Finally, 70 kg of weight was applied and the composite was allowed to cure for 24 hours.

2.4. Fabrication of palm EFB fibre reinforced composite with EG coating

The base composite was the control sample, 0 wt% EG, which was fabricated with the same method as mentioned in section 2.3. Table 2 summarises the formulation of EG coating. The coating was fabricated by simple mixing, e.g. EG and epoxy resins were mixed in a beaker and ultrasonicated at amplitude 50% and frequency 24 kHz for 15 minutes. The hardener was subsequently added into the mixture, then stirred and degassed for 5 minutes.

Table 2. Formulation for EG coating.

EG	Epoxy & hardener	Amount of EG	Amount of epoxy & hardener
(wt%)	(wt%)	(g)	(g)
1	99	0.4	40
3	97	1.2	40
5	95	2.0	40

The coating was applied on the surface of the fully cured composite by brushing and allowed to cure for 24 hours. With reference to the method by Wang et al. (2005),

after the first layer of coating was cured, the composite was coated second time to cover the surface for better finishing [15]. The combined average thickness of the coating on any one surface was kept at 0.5 mm, which was measured by digital Vernier calliper.

2.5. Scanning electron microscopy (SEM)

SEM was conducted to study the fracture surface of the composite. It was also used to observe the dispersion of EFB fibre and EG in the composite. The model of SEM was FEI Quanta 400F FESEM, which featured low vacuum and high vacuum mode. Low vacuum mode allowed imaging of non-conductive materials without the needs of conductive coating, while high vacuum mode allowed imaging of various materials and conditions. In this study, the high vacuum mode was used. This model of SEM had EDX built in which capable of identifying the elemental composition of the samples.

2.6. Flammability test

The flammability characteristic of the composite was determined by Bunsen burner test. The test was conducted based on Federal Aviation Regulations, FAR 25.855 vertical Bunsen burner test for cabin and cargo compartment materials. The specimen was subjected to flame made of 99% methane fuel for 12 seconds and subsequently removed. The flame time, flame drip time and burn length of the composite were recorded for each test.

2.7. Dynamic mechanical analysis (DMA)

The viscoelasticity properties of the composite at different temperature were determined by DMA 8000 dynamic mechanical analyser from PerkinElmer. DMA applied an oscillatory force at a set frequency and temperature to the sample. It showed the changes in stiffness in terms of storage modulus and loss modulus as well as the damping in term of the tan delta. The setting for the DMA was single cantilever mode, heating rate 3°C/min with temperature range from 30 to 100°C at 1 Hz frequency.

2.8. Mechanical test

The tensile and flexural tests were conducted with the use of Lylod Instruments LR50K Testing Machine. The tensile test was carried out in accordance to ASTM D3039. The crosshead speed was set to 2 mm/min and proceed with 5 kN load cell. The flexural test was conducted according to ASTM D790. The crosshead speed was set to 5 mm/min and proceed with 5 kN load cell. Impact test was conducted with the use of MH-358 Impact Tester. The test was in accordance to ASTM 6110.

3. Results and Discussion

3.1. Dispersion and morphology

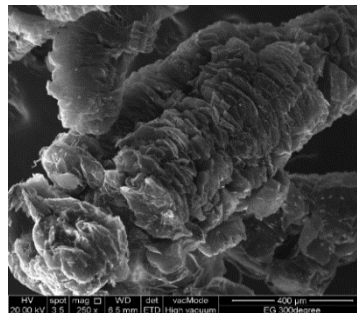
The morphology of the composite is depicted by the SEM images of fractured samples in Fig. 1. The palm EFB fibre and EG appear to be randomly distributed without any preferential orientation. The control sample in Fig. 1(a) shows only a low degree of microcracks and no noticeable gap can be observed between the bonding surface of the palm EFB fibre and epoxy matrix. This implied the presence of strong bonding due to the alkali treatment, which was similar to the results reported in the work of Azwa and Yousif [1] and Pickering et al. [3].

Figure 1(b) shows the EG after heating up to 300°C. EG slowly expanded and increased its thickness when the temperature was raised gradually from 200°C to 300°C. The thickness continued to grow proportional to the graphite layer and formed a worm-like shape when it was fully expanded [16].

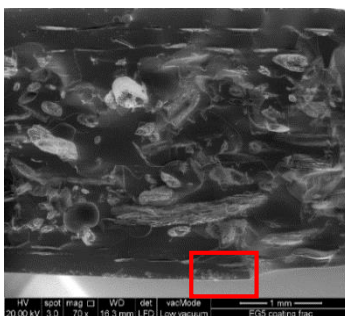
The layer of EG 5 wt% coating can be observed on the top and bottom surface of the composite in Fig. 1(c). The EG was difficult to identify, but a small amount of EG can be observed close to the surface of the coating layer, which was concentrated in the highlighted region. On the other hand, the dispersion of the constituents in the composites are shown in Fig. 1(d) -1(f). It was observed that the EG and palm EFB fibre were homogeneously dispersed without aggregation in the epoxy matrix. It can also be observed that the palm EFB fibre and EG became more compact as EG content in the composite was increased.



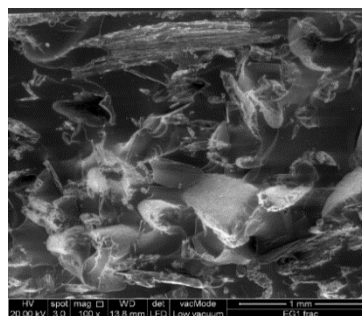
(a) Control sample EFB and epoxy composite.



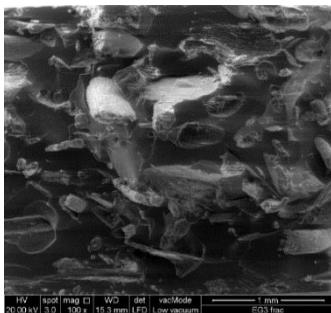
(b) Expanded EG at 300 °C.



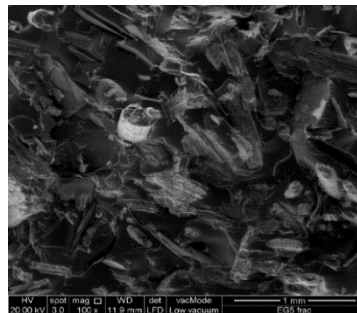
(c) EG 5wt% coating.



(d) EG 1 wt% composite.



(e) EG 3 wt% composite.



(f) EG 5 wt% composite.

Fig. 1. SEM images of samples.

3.2. Flammability

The result of the Bunsen burner test is summarised in Table 3. The flame time indicated the time of the sample remains on flame after 12s. On the other hand, drip flame time denoted the burning time of the composite that was dripping down during the combustion. Three out of seven samples were burned completely, which included the control sample, EG 1 wt% composite and EG 1 wt% coating sample.

Table 3. Bunsen burner test result.

Sample	Flame time (s)	Drip flame time (s)	Burn length (mm)	Thickness (mm)
EFB 20% composite	174	106	the entire length	2
EG 1% composite	195	92	the entire length	2
EG 3% composite	16	0	1.7	2
EG 5% composite	0	0	1	2
EG 1% coating composite	310	76	the entire length	3
EG 3% coating composite	33	0	1.8	3
EG 5% coating composite	0	0	1	3

It can be observed that EG 1 wt% coating had the longest flame time and flame drip time. This result can be explained by its burning behaviour and char formation. During combustion, the coating layer was first in contact with flame and EG was expanded in the process. Due to an insufficient amount of EG, the char formed was not able to protect the inner composite. Meng et al. (2009) pointed out that char yield and fire resistance had a strong correlation, where the char inhibits the propagation of flame by acting as a thermal barrier [17, 18]. EG 3 wt% and 5 wt% for both composite and coating samples showed good result in flame resistance. Flame on both EG 5 wt% samples distinguished immediately as soon as the EG expanded. In addition, a composite containing 5 wt% EG showed shorter burn length than those with 3 wt% EG, which indicated less flame spread.

Bhagat (2011) emphasized that the EG selected as flame retardant needed to have low expansion temperature [19]. This allowed the EG to expand at the critical temperature of the composite matrix, where decomposition, exothermal reaction and ignition occur [19]. Epoxy DGEBA has a critical temperature range from 150 °C to 200 °C.

3.3. Thermal properties

The storage modulus and tan delta of all samples at the variation of temperature are illustrated in Figs. 2 and 3, respectively. It can be observed that the storage modulus decrease with the increment of temperature, resulting from degradation of dynamic mechanical properties.

With the introduction of 1 wt% EG into the formulation, the storage modulus was observed to be lower compared to that of the control sample. However, when the concentration of EG content was increased to 3 wt% and 5 wt%, the storage modulus was greater than the control sample, with EG 5 wt% being slightly higher than EG 3 wt%. The initial decrease in storage modulus when EG was introduced into the composite can be attributed to the EG being intercalated between palm EFB fibres

and epoxy matrix, leading to increasing amount of pores fraction in the composite and hence the reduction of bonding surface between the fibre and matrix. [20, 21].

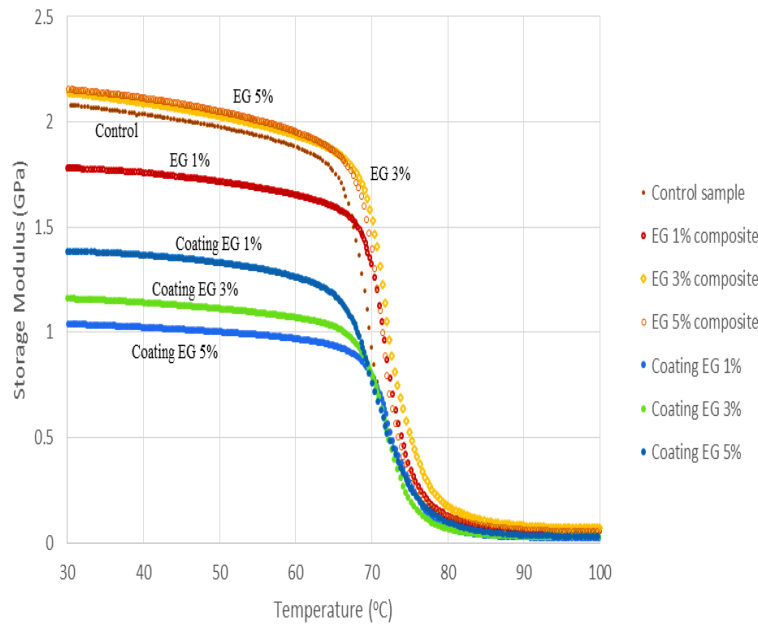


Fig. 2. DMA result: Storage modulus of samples at 30 °C to 100 °C.

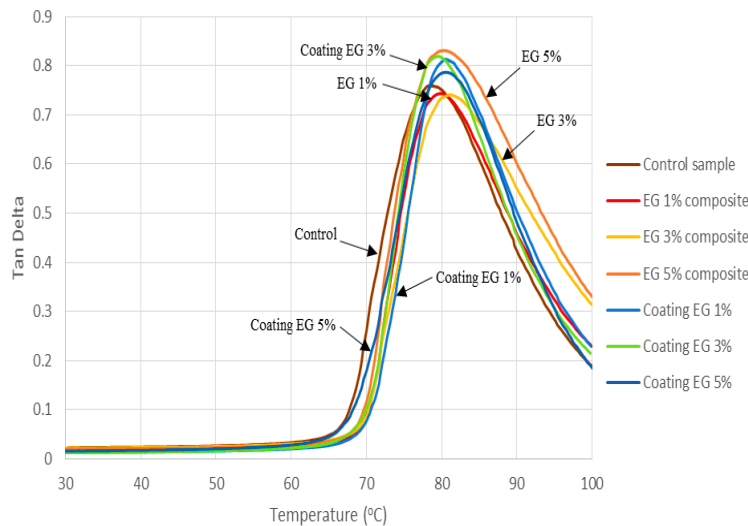


Fig. 3. DMA result: Tan delta of samples at 30 °C to 100 °C.

The measured behaviour as seen in EG 1 wt% composite showed coherence with the research works of other researchers. However, the trend of increasing storage modulus strength with a progressively higher concentration of EG, as seen in the 3 and 5 wt% EG composites was in contradiction to the other research works. For instances, Meng et al. (2009) and Saba et al. (2015) reported that storage

modulus decreased at high filler loading [17, 22]. A possible reason for this could be attributed to the homogenous distribution of EG being achieved in the composite, which in turn offer better cohesiveness and intercalation of the EG, palm EFB fibre and matrix. On the other hand, the storage modulus of coating samples was supposedly close to the control sample, but all three coating samples resulted in lower storage modulus. It is believed that the measured storage modulus was largely reflecting those of the EG epoxy resin mixture on the surface coating, where the thickness ratio of the coating to the base composite was 1 to 2.

The sudden drop of storage modulus in the vicinity of 80°C was due to glass transition temperature of the composite, which was determined by the middle point of storage modulus curve and the peak of the tan delta. It can be observed that the glass transition of the composite was ranging from 78 to 80°C. The peak of tan delta decreased as EG filler increased, which can be attributed to the increasing viscous domination at elevated temperature. Tan delta relates the defects in the composite, which included dislocations, grain boundaries, and bonding interfaces [22, 23].

3.4. Mechanical properties

The results of the tensile test are shown in Fig. 4. It can be observed that increasing amount of EG tends to improve the overall performance in elastic modulus, tensile strength and elongation at break.

The homogenous distribution of EG disrupted the bonding area of palm EFB fibre and epoxy matrix. This provided a strengthening effect on the composite. This matched the findings reported in the research by Jana and Zhong (2009) and Shalwan and Yousif (2014) [23, 24]. The chart in Fig. 4(c) shows the almost linear relationship of elongation at break and EG content, representing improving toughness at higher EG concentration.

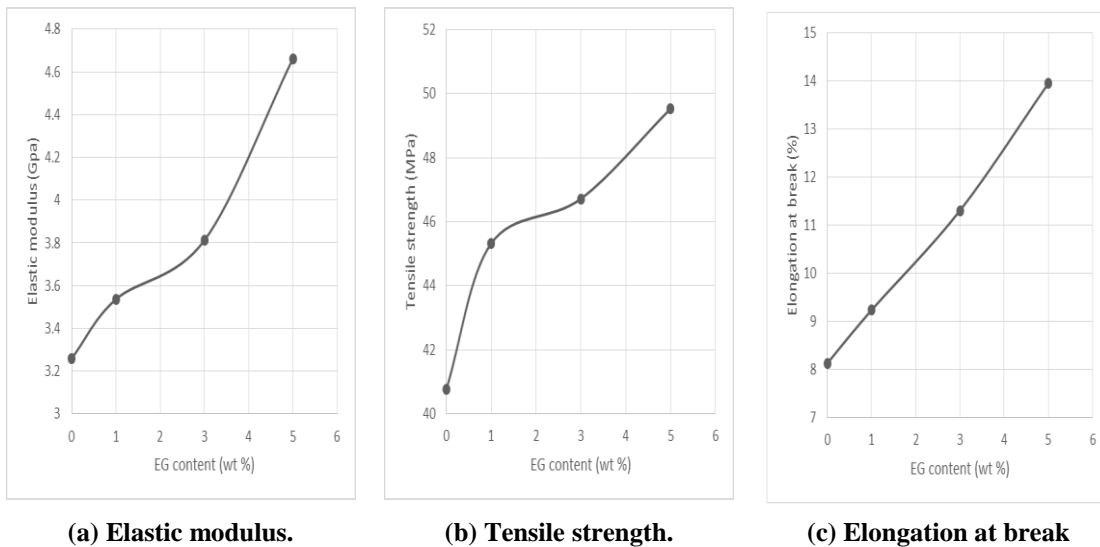


Fig. 4. Tensile test.

The result of the flexural test is presented in Fig. 5. The flexural strength showed improvement as the EG content increased, which was represented by the bending stress on the composite. The inclusion of EG seems to offer a reinforcing effect to the composite in flexural behaviour, which was in agreement with the research works from Jana and Zhong (2009) and Shalwan and Yousif (2014) [23, 24]. The measured flexural strength and bending strain were observed to approach an asymptote when the EG content was increased from 3 wt% to 5 wt%.

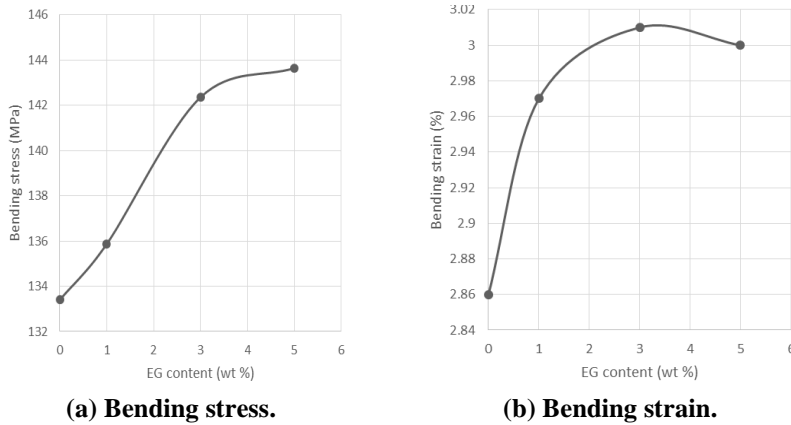


Fig. 5. Flexural test.

The result of the impact test is depicted in Fig. 6. The impact strength improved with the increasing EG from 1 to 3 wt%. These results were coherence with the findings from other researchers. Similar to the observed behaviour as in flexural tests, the impact strength seems to reach an asymptote when the EG concentration was increased from 3 to 5 wt%. The impact strength or fracture toughness is dependent on the interfacial strength of EG and palm EFB fibre-matrix bonding interface. For EG 5 wt% sample, slippage among EG was possible to cause stress concentration at the crack tip [2]. This could be the reason for the leveraging in impact strength at higher concentration of EG.

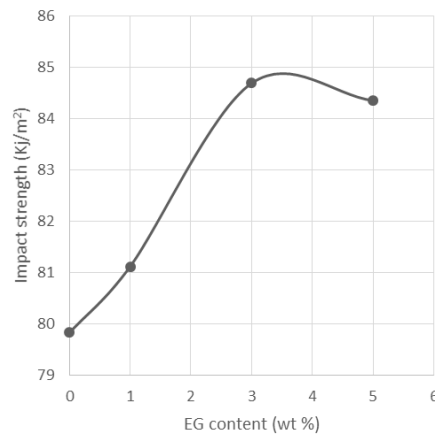


Fig. 6. Impact test: Impact strength.

4. Conclusions

The current research set to examine the effect of EG on the flammability, thermal and mechanical properties of EFB fibre. The addition of EG as a flame retardant in EFB composite had shown improvement in overall performance. Some concluding observations from the research are given below.

- For flammability, EG 1 wt% was proven to be insufficient to offer an acceptable level of flame retardancy. On the other hand, EG 5 wt% showed the optimum flame retardant effects for both filler and coating. This indicates the concentration of EG play a crucial role in determining the flame retardancy amongst other such as dispersion and expansion temperature.
- For thermal properties, the inclusion of EG at a concentration of 1 to 5 wt% does not cause many variations to the glass transition temperature of the EFB epoxy composite.
- For mechanical properties, the tensile strength, flexural strength and impact strength were improved with increasing EG content. At 5 wt% of EG, both the flexural and impact properties of the composites seems to reach an asymptote due to possible slippage among EG at a higher concentration in the test sample.

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Abbreviations

DGEBA	Diglycidyl Ether of Bisphenol A
DMA	Dynamic Mechanical Analysis
EFB	Empty Fruit Bunch
EG	Expanded Graphite
NFRC	Natural Fibre Reinforced Composites

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