Contrasts on Fracture Toughness and Flexural Strength of Varying Percentages of SLG reinforced Phenolic Composites

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Abstract: *Many previous studies had reported the improvement in the mechanical properties of vinyl ester resin reinforced with SLG*. Among these material properties, fracture toughness and flexural properties are important material characteristics. This paper investigates the relationship between these two set of material properties in *enviroshperes* (SLG) reinforced phenolic composites. The material properties of the phenolic resin composites containing different percentage by weight of SLG are experimentally measured using the short bar method and the tree-point test. The findings indicated that the PF/E-SHERES (30%) constitute the best compromise with respect to cost, fracture toughness and flexural strength. *It is hoped that the discussion and results in this work would not only contribute towards the development of SLG reinforced phenolic composites with better material properties, but also useful for the investigations of fracture toughness and flexural strength in other composites.*

Keywords: Phenolic resin, SLG, fracture toughness, flexural strength and Young's modulus.

Introduction

Fracture toughness, K_{IC} is a property that measures the material's resistance to brittle fracture when a crack is present. From principles of fracture mechanics, the critical stress for crack propagation (σ_c) is related to the crack length (a) by $K_{IC} = Y\sigma_c\sqrt{\pi a}$. For thin specimens, the value of K_C will depend on the thickness of the material. K_{IC} becomes independent of the thickness of the material when the specimen thickness is much larger than the crack. Figure 1 shows a diagram of an edge crack with Y≈1.1 when the crack is much smaller than the semi-infinite width of the plate. The value of

Y will approach 1.0 for a plate of infinite width having a through-thickness crack. Plane strain condition then exists and the K_{IC} value is known as the plain strain fracture toughness, $K_{IC} = Y\sigma_c\sqrt{\pi a}$ and its unit is MPa $\sqrt{m}$ (Callister, 2007).

The fracture toughness of composite material is extremely important in the design consideration in many engineering applications (Baker, 1977; Callister, 2007). Other critical factors include the material's strength and modulus values. For reinforced polymeric resins, these properties can be affected by the resin, catalyst, filler and constituents. There are many means to determine the fracture toughness and strength of composite materials. For example the three- or four- point bending tests are normally used to determine the stress-strain behaviours of particulate reinforced resins. These traverse bending tests are flexural investigations in which rod specimens having either circular or rectangular cross sections are bent until fracture using three- or four- point loading techniques. The stress at fracture using this test is known as flexural strength, which is frequently quoted together with the flexural modulus (Shackelford, 1992). The flexural strength will depend on the specimen size. By increasing the specimen volume under tensile stress, there is greater probability of having a crack-producing flaw and consequently, a decrease in flexural strength. Under these circumstances the magnitudes of flexural strengths for composites are likely to be greater than measurements obtained from tensile tests.

This paper investigates the fracture toughness and the flexural strength of SLG particulate reinforced phenolic resin composite containing different percentage by weight of SLG. *Phenolic resins present excellent dimensional stability, thermal stability, chemical resistance, and load-bearing capability at elevated temperatures. It*

is fire resistant and has many applications especially in electrical devices. This commonly used resin is used in this study. SLG is cheap and commonly used in a variety of manufacturing applications because of their unique properties, which include extreme heat resistance and high compressive strength. Many previous studies had reported the improvement in the mechanical properties of vinyl ester resin reinforced with SLG (Cheng et al., 2004; Ku et al., 2005a; 2005b; 2006a; 2006b; 2007a; 2007b).

The objective of this paper is to determine the effects of different percentage by weight of SLG on the mechanical properties of SLG reinforced phenolic resin. The material properties are experimentally measured using the short rod and short bar method and the three point bending test. Based on the results of these tests, the paper proposes approaches to improve the composite material properties by adjusting the percentage by weight of SLG. The discussions in this paper will be useful for producing SLG reinforced phenolic composites with good fracture toughness and flexural strength. The approach may also be applicable to other composite materials.

Measurement of Fracture Toughness

Measuring the fracture toughness of materials with high toughness, low yield strength and brittleness using ASTM (American Society for Testing and Materials) standards (ASTM, 1978) may not be effective as *the method is relatively expensive and the procedure is quite involved (Barker, 1977).* To overcome this problem, Baker (1981) designed the short rod and short bar method. This cost effective approach eliminates the residual stress effects as a source of error in the fracture toughness measurement

(Barker, 1980). It uses a real crack and reduces the size of the specimen. It does not require fatigue precracking. The method is also applicable to a wide range of materials, including metals, ceramics, polymers and rocks. This method was also found to be suitable for the particulate reinforced phenolic resins (Barker, 1981). A typical fracture toughness test may be performed by applying a tensile stress to a specimen prepared with a flaw of known geometry and size as shown in Figure 1. The stress applied to the material is intensified at the flaw (Askeland, 1998). By performing a test on a specimen with a known flaw size, the value of K_{IC} that causes the flaw to grow and the peak force to cause failure can be determined without using the load versus deflection plot (Baker, 1977).

Figure 2 shows a sample short bar specimen with straight chevron slot. The specimen breath is indicated by parameter B. The short bar test uses an opening load applied near the mouth of the specimen, causing a crack to initiate at the point of the chevron slot. The load line is the line along which the opening load is applied in the mouth of the specimen. Ideally, the opening load should be less than the load that will be required to further advance the crack. A continually increasing load must be supplied until the crack length reaches the critical crack length, a_c. Beyond a_c, the load should decrease, as shown in Figure 3.

Measurement of Flexural strength

The three point bending flexural test provides values for the flexural stress σ_f, flexural strain ε_f, modulus of elasticity in bending E_B and the flexural stress-strain response of the material. The main advantage of a three point flexural test is the ease of the

specimen preparation and testing. The standard used is ISO 14125:1998(E) (ISO, 1998). A Material Testing Systems (MTS) 810 was used for the tests. The dimensions of the specimens of resins were 100mm x 10mm x 4mm and tested at a crosshead speed of 1 mm/min.

The flexural stress can be calculated as:

$$\sigma_f = \frac{3PL}{2bh^2} \tag{1}$$

Flexural strain can be calculated as:

$$\varepsilon_f = \frac{6Dh}{L^2} \tag{2}$$

and the modulus of elasticity can be calculated as:

$$E_B = \frac{L^3 m}{4bh^3} = \frac{Flexural\ \ strength}{Flexural\ \ strain} \tag{3}$$

where: σ_f : stress in outer fibre at midpoint, MPa;

ε_f : strain in the outer surface, %;

E_B: modulus of elasticity in bending, MPa;

P: load at a given point on the load deflection curve, N;

L: support span, mm;

b: width of test beam, mm;

h: depth of test beam, mm;

D: maximum deflection of the centre of the beam, mm;

m: slope of the tangent to the initial straight line portion of the load deflection curve, N/mm.

The samples

The reinforcers used were E-sphere (SLG, ceramic hollow sphere) particulates. They were made 5 % to 35 % by weight in step of 5 % in the cured phenol formaldehyde composite PF/E-SPHERES (X %), where X is the percentage by weight of the filler. The short bar and flexural test specimens were cast from the raw materials. The resin was first mixed with the catalyst. After that the E-sphere SLG was added and mixed to give the uncured composite. Table 1 shows the mass in grams of resin, catalyst and *SLG* required respectively to make 1000 grams of uncured composite of 30 % by weight of SLG. The moulds were made from PVC (poly vinyl chloride) sheets with 6 pieces of short bar, and of bending test specimens each. These are depicted in Figures 4 and 5 respectively. The slots in short bar specimen were made by inserting plastic sheets of suitable thickness. Figure 6 shows some of the PF/E-SPHERES (X %) short bar specimens. After preliminary curing, the samples were taken out of the moulds and post-cured in an oven at 50 ^{o}C for 2 hours followed by 80 ^{o}C for 4 hours and finally by 100 ^{o}C for 4 hours. The specimens were then subjected to short bar and flexural tests. Six samples were tested for each percentage by weight of E-spheres. An MTS 810 Material Testing Systems was used for the tests. The rate of extension was made at 1 mm per minute. The specimens for flexural tests were post-cured in the same way as those for short bar tests. The dimensions of the flexural test specimens were 250mm x 10mm x 4mm and tested at a crosshead speed of 1 mm/min.

Results and discussions

Figure 7 shows the of fracture toughness of the PF-E-SHERES with varying percentage by weight percentages of SLG. It was found that the fracture toughness is highest when the percentage by weight of the filler is zero. The peak fracture toughness value is 14.74 MPa$\sqrt{m}$. The value dropped slightly to 13.80 at 5% by weight of SLG. It then dropped sharply to 7.37 MPa at 10% by weight of SLG. The value remained steady at around 8.1 to 8.8 MPa at percentages by weight of SLG from 15 to 25%. It rose back to 11.88 MPa at 35% by weight of SLG. The standard deviation of the sample is small indicating that the values of fracture toughness obtained are reliable (Ku et al., 2008a). By adding 35% by weight of SLG particulates to the phenolic resin, the price of the PF/E-SPHERES (35%) composite would be lowered. *The cost of the phenolic resin used in this research is Australian dollar $7.00 per kg and that of SLG is $0.30. For 1 kg of the composite with 35% by weight of SLG, the cost for the resin is $4.55 and that of the SLG is $0.10. The total cost is $4.65 as compared to $7.00 for unfilled resin. This is a reduction in cost of 34%. However, the fracture toughness was only reduced by 19%. This would give a lower cost composite with medium fracture toughness.* To further determine if the PF/E-SPHERES (35%) composite is really advantageous, it is necessary to investigate other mechanical properties like flexural strength.

Redjel (1995) found that the fracture toughness of pure phenolic resin was 1.51 MPa$\sqrt{m}$*; the fracture toughness of neat resin by weight of SLG reinforced phenolic resin, PF/E-SHPERES (0%) in this study was 8.72 MPa*$\sqrt{m}$*, which is 5.78 times the fracture toughness of pure phenolic resin, an increase of 478%. This may be due to*

the improved resin used (the work was carried out eleven years later) with better post-curing facilities.

Figure 8 illustrates the flexural strength of varying the percentage by weight of E-SPHERE (SLG) reinforced phenol formaldehyde matrix composite. At 5 percent by weight of the SLG, the flexural strength is highest at 42.39 MPa. At 10 – 20 percent by weight of SLG reinforcement, the values of flexural strength drop and vary from 17.22 to 17.18 MPa. At 25 % by weight of SLG the flexural strength increases again to 26.48 MPa. It drops back to 22.07 MPa at a weight reinforcement of 30 percent. The trend of the flexural strength curve in Figure 8 does not completely correlates to the fracture toughness curve in Figure 7. The flexural strength of the composite at 5% by weight of SLG is too high when compared with its fracture toughness at the same percentage by weight of filler. This phenomenon was also reported in another study (Ku et al., 2008b).

The flexural strength determined in this study is highest at 5% by weight of SLG reinforcement. It seemed that at this low percentage of reinforcement, the SLG does not have significant impact on the flexural strength of the composite. The finding that the unfilled phenolic formaldehyde has a higher flexural strength than its filled counterpart had been examined by Wang et al (1997). In their study, Wang et al (1997) found that the flexural strength of unfilled phenolic formaldehyde was 71.3 MPa, and that of 20% by weight of ceramic powder reinforced phenolic formaldehyde matrix composite was 10.5 MPa. In this research the corresponding flexural strength by weight of SLG reinforced phenolic formaldehyde matrix composite was 17.18 MPa. The difference in flexural strength of the unfilled phenolic formaldehyde for

both studies is about 2.6. The difference in magnitudes in the two separate studies could be attributed to the different phenolic resin used in the investigations. Wang et al (1997) used ICI Fiberite resol-type CMXR-6055 phenolic formaldehyde resin, while this work used Chemwatch Hexion Cellobond J2027L phenolic formaldehyde resin. The curing of the resin and filler could also contribute to the difference in results.

The difference in flexural strength of the reinforced phenolic formaldehyde composites for both studies is 64%, which is a significant difference. Wang et al (1997) used ceramic particles of diameters between 300 – 600 µm with a specific gravity of 1.05 g/cm^3; no other details of the filler were mentioned. In this study, the diameters of the ceramic particles were between 20 -300 µm; it can be argued that the smaller diameters of the ceramic particles (SLG) can be better wetted and mixed with the resin and this could lead to higher flexural strength. Moreover, the post-curing processes could also contribute significantly to the flexural strength.

Considering both fracture toughness and flexural strength, the PF/E-SPHERES (35%) composite has a fracture toughness of 11.88 MPa$\sqrt{m}$ but its flexural strength was a low 17.18 MPa (extrapolated). Decreasing the percentage by weight of SLG to 30% (i.e. PF/E-SPHERES (30%)) increases its flexural strength to 22.50 MPa but reduces its fracture toughness to 11.06 MPa$\sqrt{m}$, which is 7 % below that of PF/E-SPHERES (35%). A compromise has to be made and PF/E-SPHERES (30%) composite should be regarded as the best combination of cost, fracture toughness and flexural strength.

Figure 9 shows the Young's modulus of the SLG reinforced phenolic composites. The graph follows the same pattern of the flexural strength and indicates that the PF/E-SPHERES (30 %) is the best compromise without adding additives or other resins.

At 5% by weight of SLG, the Young's modulus is highest at 3,005 MPa; at 10 – 20% by weight of SLG reinforcement, the values of flexural strength drop and vary from 979 to 1,114 MPa. At 25% by weight of SLG the Young's modulus increased again to 1,995 MPa; it dropped back to 1,707 MPa at a weight reinforcement of 30% SLG. The values found seem to be reasonable when compared with the results from Callister (2005). The extrapolated value of Young's modulus of pure phenolic resin in this study is 5,730 MPa, which is quite close to that found by Redjel (1995) for pure phenolic resin (4,401 MPa).

The structure of the composites could also be modified by other means other than changing the percentage by weight of SLG. For example, adding other epoxy resins to PF/E-SPHERES (30%) could be investigated. The epoxy resins to be selected should be adhesive to the other substances and are supposed to cure and fill in the gaps between the phenolic resin and the SLG particulates as well as the voids in the phenolic resin (Cardona et al., 2008; Ku et al., 2008a; Smith and Hashemi, 2006). The proportion of phenolic and epoxy resins by weight could be varied until optimum mechanical properties are attained. Other works such as Hepworth et al. (2000) had shown that mixing the resins with pre-treated SLG in 50% PVA solution could improve the stiffness and strength of the composites. *These pre-treatments not only enable more SLG particulates to be added to the composites, but also facilitate the*

fusing of the SLG with the matrix (phenolic resin). This could further reduce cost, while at the same time, maintains or even improves the mechanical properties.

Cost is always an important factor in industrial applications. *In the manufacturing industries, generally more than one materials or composites can be found to be appropriate for the manufacture of the goods. Under such conditions, cost is the deciding factor to be considered.* Cost reduction could be achieved up to a certain limit by adding more SLG. The high viscosity of the composite at around 40% by weight of SLG could be the limiting factor. With suitable epoxy resins, it may be possible to maintain the fracture toughness and the flexural strength at higher percentage by weight of SLG.

Conclusion

This study has made a compromise between the fracture toughness and the flexural strength of SLG particulate reinforced phenolic resin composite by appropriately adjusting the percentage by weight of SLG. The optimum percentage by weight of SLG is found to be 30%. The result from this work paves the way for further research to improve the properties of PF/E-SPHERES (30 %) with additives or other resins. Future work will investigate the pre-treatment of SLG with 50% PVA solution for better adherence to the resins.

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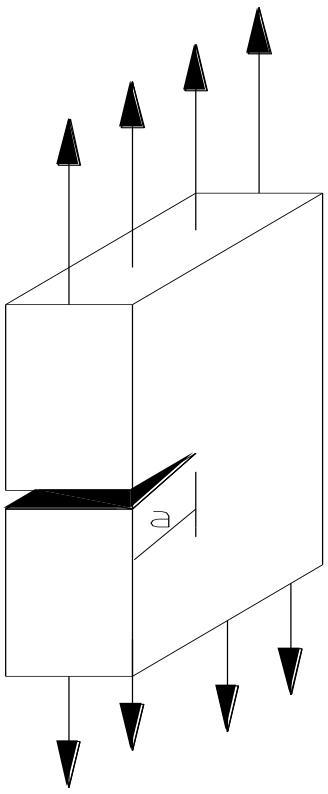
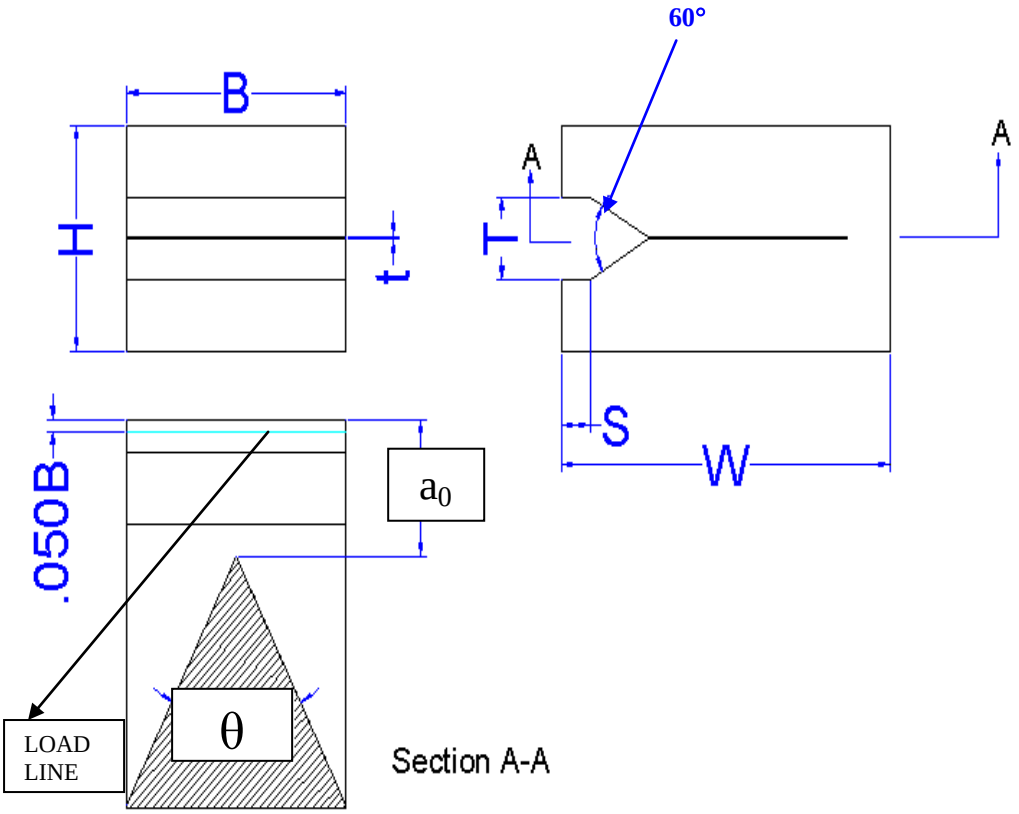


Figure 1: Drawing of fracture toughness specimen with edge flaw



SYMBOL	DEFINITION	VALUE	TOLERANCE
B	BREADTH	B	
W	LENGTH	1.5B	± .010B
H	HEIGHT	.870B	± .005B
a_0	INITIAL CRACK LENGTH	.513B	± .005B
θ	SLOT ANGLE	55.2°	± 1/2°
T	SLOT THICKNESS	SEE TABLE III (of Barker, 1981)	
S	GRIP GROOVE DEPTH	.130B	± .010B
T	GRIP GROOVE WIDTH	.313B	± .005B
R	RADIUS OF SLOT CUT	SEE FIG 4 (of Barker, 1981)	±2.5B

Figure 2: Short Bar Specimen with Straight Chevron Slots.

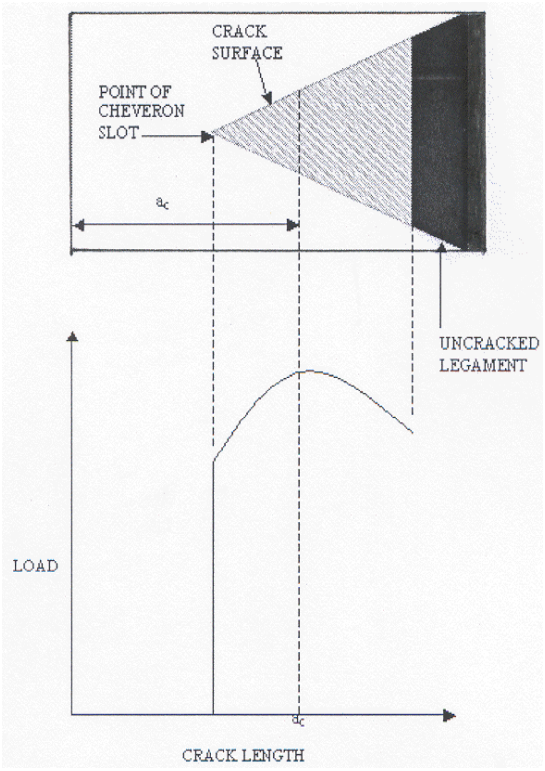


Figure 3: Variation of load versus crack length

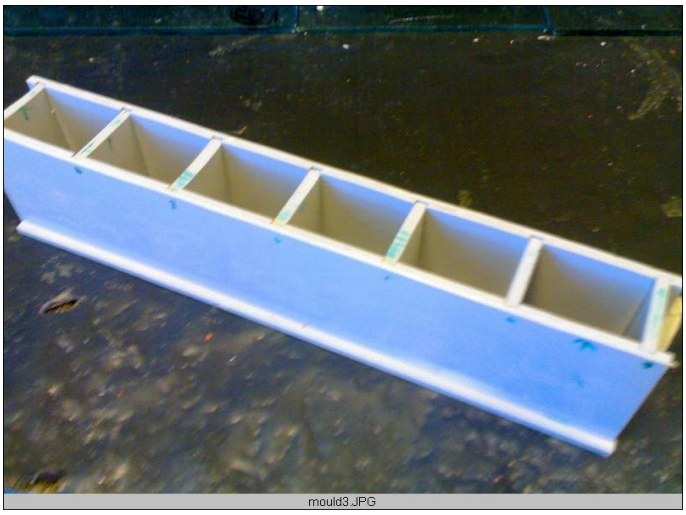


Figure 4: The mould for short bar specimens

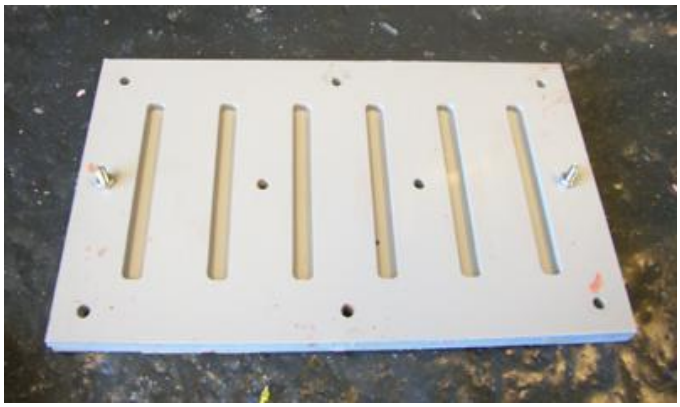


Figure 5: The mould for bending test

Figure 7: The short bar specimens

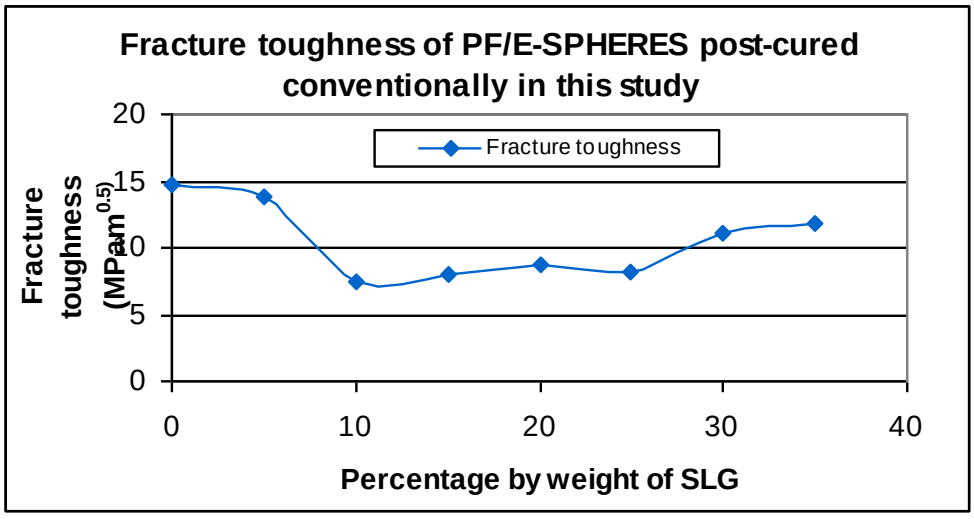


Figure 7: Fracture toughness of PF-E-SPHERES with varying percentage by weight of SLG

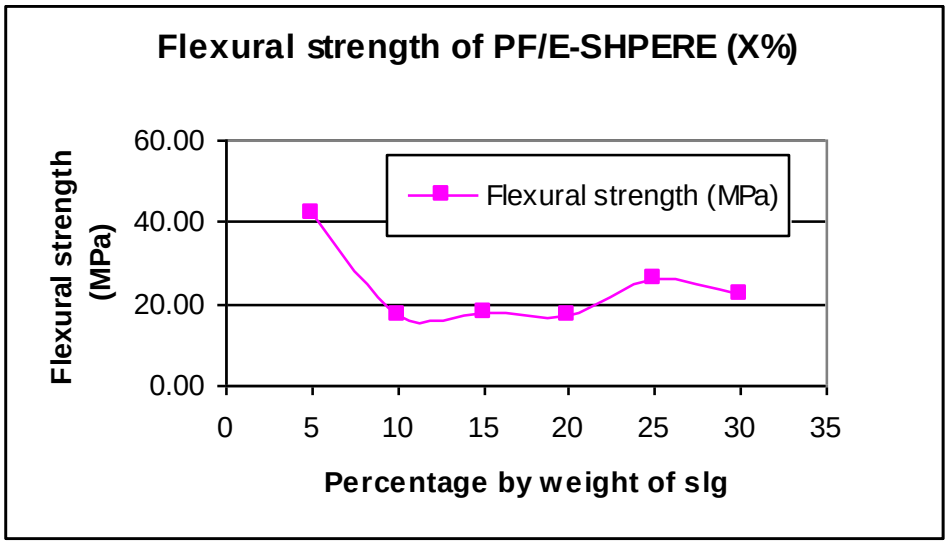


Figure 8: Flexural strength of varying percentage by weight of E-sphere reinforced phenolic formaldehyde matrix composite

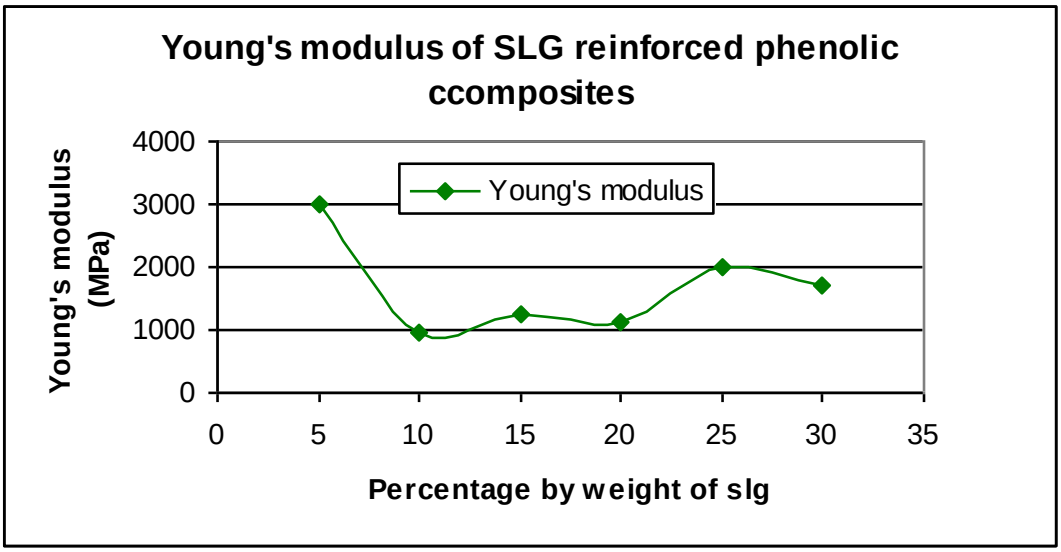


Figure 9: Modulus elasticity of varying percentage by weight of SLG reinforced phenolic formaldehyde matrix composite

Table 1: Weight of materials required to make 1000 g of PF/SLG (30%)

Parameters	Resin (R)	Catalyst (C)	R + C	SLG	Composite
Percentage by weight	20	1	---	---	---
Percentage by weight	---	---	70	30	---
Weight of materials in 1000 g of PF/SLG (30%)	667 (g)	33 (g)	700 (g)	300 (g)	1000 (g)