

**Relationship between different Mechanical Properties of
Phenol Formaldehyde SLG Reinforced Composites**

H S Ku

*⁺Faculty of Engineering and Surveying, Centre for excellence in Engineered Fibre
Composites, University of Southern Queensland (USQ), Australia.*

Ku, H, Relationship between different Mechanical Properties of Phenol
Formaldehyde SLG Reinforced Composites, JRPC, 2011 Vol. 30, No. 15, pp. 1281-
1292.

Corresponding Author:

Title : Dr.
Name : Harry Siu-lung Ku (Corresponding author)
Affiliation : Faculty of Engineering and Surveying,
University of Southern Queensland.
Tel. No. : (07) 46 31-2919
Fax. No. : (07) 4631-2526
E-mail : ku@usq.edu.au
Address : Faculty of Engineering and Surveying,
University of Southern Queensland,
West Street, Toowoomba, 4350,
Australia.

Abstract: Phenol formaldehyde was filled with Envirospheres, ceramic hollow spheres (SLG) to increase the tensile strength, fracture toughness and flexural strength. This paper varies the percentages by weight of the SLG in the composites which are then subjected to the relevant mechanical tests. The results show that composite with 7.5 % by weight of the SLG produces the optimum combinations of tensile properties, flexural properties, fracture toughness and cost with a reasonable fluidity for casting. 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, tensile properties and flexural properties in other composites.

Keywords: Tensile strength, Young's modulus, fracture toughness, flexural strength, maximum flexural strain, phenol formaldehyde, phenolic resin, envirospheres and SLG.

1. Introduction

Phenolic thermosetting materials were the first major plastic material used by industry. They are still among the most widely used thermosets because they are some of the lowest-cost engineering material on a cost-per-volume basis. Phenolics are formed from the condensation of polymerization reaction between phenol and formaldehyde. The condensation reaction for phenolics can be carried out under two different conditions, resulting in two different intermediate materials. One of the intermediates is called resoles and the other novolacs [1, 2].

The novolacs are formed by reacting phenol and formaldehyde in an acid solution but with insufficient formaldehyde to complete the reaction at 100 °C (the opposite of forming resoles). About one mole of phenol is reacted with 0.7 to 0.85 mole of formaldehyde. This is the first stage of the reaction and a brittle thermoplastic resin is produced which can be melted but cannot crosslink to form a solid network. The addition of hexamethylenetetramine (hexa), a basic catalyst, to the first stage phenolic resin makes it possible to create methylene crosses linkages to form a thermosetting material. When heat and pressure are applied to the hexa-containing novolac resin, the hexa decomposes, producing ammonia which provides methylene cross linkages to form a network structure. On account that hexa, a second material, must be added to novolacs, they are called two-stage resins. The temperature required for the cross-linking of the novolac resin ranges from 120 to 177 °C. The various fillers used can vary from 50 to 80 % by weight. The fillers reduce shrinkage during molding, lower cost and improve strength. They are also used to improve electrical and thermal insulating properties and chemical resistance [1-4].

This research project is to find out the relationship between the tensile properties, flexural properties and fracture toughness of phenol formaldehyde composites reinforced with varying percentages by weight of Enviro spheres, the filler, with a view to finding out the optimum percentage by weight of the Enviro spheres used in the composites for different mechanical properties.

2. Phenol formaldehyde

The commercial resole resin used in this study was J2027 and manufactured by Borden Chemical Pty. Its official name is now Hexion Cellobond J2027L because the company had been taken by Hexion [5]. The catalyst used to crosslink the resin is phenolic resin hardener catalyst produced by the same company. The official name of the catalyst is Hexion Phencat 15 [6]. The ratio by weight of the resin to hardener is 50: 1, which may be changed when the resin is supplied by other manufacturer.

Most molded phenolic parts are made from novolacs. Without fillers or reinforcements, the parts are brittle and have high shrinkage in the mold because of the crosslinked nature of the cured resin. The most common filler is wood flour. Other common fillers and reinforcements are cotton fibres, fiberglass, chopped thermoplastic fibres, e.g. nylon.

The high number of OH groups in the resin gives it excellent adhesive qualities. However, this adhesive nature of phenolics causes molding problems. They tend to stick to the molds. Release agents have to be sprayed into the mold surface to solve this problem. The nonflammability of the resin leads to its wide applications. When phenol formaldehyde resin is subjected to a flame, they char rather than melt or burn. They are therefore widely applied in situations where avoiding flammability and smoke is vital. Furthermore, the char has a very low thermal conductivity so that surrounding materials are protected by the decomposed phenolic. Low thermal conductivity of the resin promote its used as bases for toasters and knobs for appliances. Most phenol formaldehyde parts are dark because the dark color is inherent to it and this also limits its use in some applications. A dark pigment is usually added to the resin to standardize the color and to decrease its sensitivity to UV light. Its high electrical resistance wins its applications in electrical switches and circuit breakers. The abrasive nature of the phenolic formaldehyde makes its machining difficult; they are therefore molded to near net shape. The resin is cured by condensation polymerization which results in the evolution of water as a by-product of the curing process and extensive microvoiding within the matrix. The microvoids have little effect on the composite properties except that significantly higher water absorption is observed. High water content can cause structures to delaminate when exposed to heat [3-4].

When it comes to performance under severe conditions phenolic resins are indeed irreplaceable in many modern and high technology applications. This includes their use for rubber reinforcement, especially in the production of modern radial tires. Depending on the pH value and the phenol/aldehyde ratio applied during the reaction either resol type resins or novolaks are formed. For resol resin, there should be excess of aldehyde and $\text{pH} > 7$; for novolaks resin, there should be excess of phenol and $\text{pH} < 7$ [7]

3. The Envirospheres

The Envirospheres (E-spheres) SLG, is a mineral additive that can improve product by reducing product's weight, improving its performance and lowering its cost. E-spheres are white microscopic hollow ceramic spheres that are ideal for a wide range of uses. The particle size of this general purpose E-spheres ranges from 20 – 300 μm with approximate mean of 130 μm . The relative density of E-spheres is 0.7. E-spheres are a combination of Silica, SiO_2 (55-60%), Alumina, Al_2O_3 (36-44%), Iron Oxide, Fe_2O_3 (0.4-0.5%) and Titanium Dioxide, TiO_2 (1.4-1.6%). E-sphere is an inert material similar to talc, etc (E-spheres, undated). The material may be prone to dusting in use. Grinding, milling or otherwise generating dust may create a respiratory hazard. In high dust areas the use of goggles and a National Institute of Occupational Health and Safety (NIOSH) approved dust respirator is recommended.

They are used in a variety of manufacturing applications because of their unique properties and they are [9]:

- extreme heat resistance;
- high compressive strength;
- pure, clean and white.

In addition to these unique features, E-spheres provide all the benefits you would expect from a microsphere. The typical applications in composites include casting, spray-up, hand lay-up, cold/hot press molding, resin transfer molding and syntactic foam.

4. Mechanical Properties

The mechanical properties discussed in this paper include yield strength, tensile strength, Young' modulus, fracture toughness, flexural strength, flexural strain and flexural modulus. Some of the characteristics of the above properties will be discussed in some detail.

4.1 Yield Strength

It is the strength at which a definite amount of plastic strain has occurred. For brittle material, proof load will substitute yield strength because no yield point can be identified in the stress vs. strain curve as depicted in Figure 1, which shows that 0.2 % proof load could not be determined because line passing the 0.2 % elongation and parallel to the most approximated linear portion of the curve will never cut the curve. 0.1 % proof load was therefore used to evaluate 0.1 % proof stress. Figure 1 also illustrates how the 0.1 % proof load was determined. The gauge length used should be the separation of the grips and 105 mm because the elongation of non-ductile material, e.g. this phenolic composite, when subjected to tensile force will spread along the sample evenly and not restricted to the conventional gauge length of 50 mm [10, 11].

When the intersection of the line passing the 0.1 % elongation and parallel to the most approximated linear portion of the curve was projected to the y-axis, the load found was 355 N which is the 0.1 % offset yield load. 0.1 % proof stress (yield strength) is calculated using the relationship below [9]:

The 0.1 % proof strength can be calculated as:

$$\sigma_y = \frac{\text{Yield load}}{\text{Original cross-sectional area}} \quad (1)$$

The 0.1 % proof strength of a sample illustrated in Figure 1

$$= \frac{0.1\% \text{ offset load}}{\text{Original cross-sectional area}} = \frac{355}{14.8 \times 3.8} = 6.31 \text{ (MPa)}$$

4.2 Tensile strength

The tensile strength is most sought after result of a tensile test. It is easy to determine and has become a familiar property and is useful for the purposes of specifications and quality control of a product.

4.3 Young's modulus

The Young's modulus (E) or modulus of elasticity is to measure the stiffness of the material. The Young's modulus can be calculated by calculating the slope of the initial linear portion of the stress-strain curve. As the force-extension curve of the material does not possess a perfectly linear portion (Figure 1), the Young's modulus quoted is the secant modulus at a strain of 0.1 percent [12, 13]. The Young's modulus [9]:

For example, the Young's modulus of a sample illustrated in Figure 1 was calculated using the data provided from Figure 2, in which a portion of the most linear part of the curve was selected; after projecting the top point of the selected linear portion into the

x- and y- axis respectively, the force (= 250 N) and the extension (= 0.186 mm) were obtained and used in the calculation.

4.4 Fracture toughness

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 3 shows a diagram of an edge crack with $Y \approx 1.1$ 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 $\text{MPa}\sqrt{m}$ [14].

The fracture toughness of composite material is extremely important in the design consideration in many engineering applications [15, 16]. 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 [1]. 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.

Measuring the fracture toughness of materials with high toughness, low yield strength and brittleness using ASTM (American Society for Testing and Materials) standards [16] may not be effective as the method is relatively expensive and the procedure is quite involved [15]. To overcome this problem, Baker 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 [17, 18]. 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 [18]. 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 3. The stress applied to the material is intensified at the flaw [19]. 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 [15].

Figure 4 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 5.

4.5 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) [20]. 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.

5. The Composite Samples

The reinforcer was E-sphere, SLG (ceramic hollow sphere) particulates and they were made 0 % to 35% by weight in the cured phenol formaldehyde composite PF/E-SPHERES (X %), where X is the percentage by weight of the filler. As the raw materials of the composites are liquid and ceramic hollow spheres, the three types of specimens were cast to shape. The resin is a dark brownish liquid and is first mixed with the dark brownish catalyst. After that the E-sphere SLG is added to the mixture and they are then 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 mixture of SLG, resin and accelerator was blended with mechanical blender to ensure a more homogenous mixture. The uncured composite was then cast into the moulds of different specimens and then cured in ambient conditions. After initial 24-hour curing when the test pieces were removed from the mould, they were post-cured. This was achieved by baking the pieces in an oven. Oven temperatures and times were:

- 4 hours at 50°C

- 4 hours at 80°C
- 2 hours at 100°C

The different test pieces were then tested accordingly.

6. Viscosity Measurements

Viscosity was measured using the Brookfield RDVD-II+ viscosity testing machine. Throughout the tests the viscosity was recorded at a constant temperature of 26 °C. The temperature of the composite (in liquid form) would rise as time went on due to the curing. Theoretically, one should allow the temperature to rise and viscosity to reduce (Figure 6) and then pour the liquid form composite into the moulds but this may not be achievable because the composite would have cured before one can properly cast the composite into the moulds. Viscosity has therefore to be measured as soon as the measuring probe was dipped into the composite.

7. Results and Discussion

Figure 7 illustrates the yield strengths, tensile strength, Young's modulus and fracture toughness of varying percentage by weight of E-sphere (SLG) reinforced phenol formaldehyde matrix composites. The yield strength of the neat resin was 13.00 MPa. The value remained stable from 0 to 15 % by weight of SLG; after that it dropped dramatically. From 20% to 35 % by weight of SLG, the yield strength values did not vary much but were relatively low and the composite will not be suitable for most applications.

The tensile strength of the neat resin was 15.00 MPa as depicted in Figure 7. The values of the tensile strength of the composites were steady from 0 to 15 % by weight of SLG. It dropped dramatically at 20 % by weight of SLG. After this the values remained steady again. The variation of tensile strength with respect to percentage by weight of SLG is the same as that of yield strength.

Figure 7 also illustrates the Young's moduli of varying by weight of E-sphere reinforced phenol formaldehyde matrix composite. The Young's modulus of the neat resin was 2.51 GPa and it decreased slowly as the percentage by weight of SLG increases (up to 15 %) and dropped more to 1.39 GPa when the percentage by weight of SLG was 20 %; after that the values did not change much. The values found seem to be reasonable when they are compared with those of phenolic formaldehyde (2.76 – 4.83 GPa) [20]. However, the Young's modulus of pure phenolic resin in this study is 2.51 GPa, which is 13.5 % lower than that found by a group of researchers for pure phenolic resin (2.9 GPa). The same team used ICI Fiberite resol-type CMXR-6055 phenolic formaldehyde resin; this research used Chemwatch Borden (Hexion)

Cellobond J2027L phenolic formaldehyde resin. On top of it, they did not mention the temperatures and duration of soak when they cured the resin and its filler [21]. They 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 [21]. In this study, the diameters of the ceramic particles were between 20 -300 μm . In general, the smaller diameters of the ceramic particles (SLG) can be wetted by and mixed with the resin better and this may result in higher Young's modulus value but this is not the case. More study has to be carried out to remedy this. In another study, it was found that the Young's modulus of the neat resin was 5.16 GPa. The material used was a pure phenolic resin 84055 catalyzed by 3 percent of C 1650 and cured at 80 °C for 8 days. The resin was produced and prepared by CDF-Chimie (France) [22]. It is not surprising that different types of phenolic resins with different curing conditions will give different values of Young's modulus.

Figure 7 also shows the of fracture toughness J2027 (Brendon Chemical) specimens filled with varying weight percentages of E-spheres SLG. It was found that the fracture toughness is highest with the neat resin and was 14.74 $\text{MPa}\sqrt{m}$. The value dropped to a low of 7.37 $\text{MPa}\sqrt{m}$ when the SLG by weight is 10%; after this the values varied from 8.08 to 8.81 $\text{MPa}\sqrt{m}$ as the percentage by weight of SLG increases from 15 to 25%. It then re-bounced back to 11.88 MPa at 35% particulate loading. Redjel found that the fracture toughness of pure phenolic resin was 1.51 $\text{MPa}\sqrt{m}$ [22]; the fracture toughness of neat resin by weight of SLG reinforced phenolic resin, PF/E-SHPERES (0%) in this study was 8.72 $\text{MPa}\sqrt{m}$, 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) and better post-curing method of the composite as compared to that of Redjel. By and large, it can be concluded that as far as tensile properties and fracture toughness as well as cost were considered, the best percentage by weight of SLG in phenolic resin should be 7.5 % as shown in Figure 7. At this particulate loading, the yield strength, tensile strength, Young's modulus and fracture toughness are 123 %, 100 %, 94 % and 71 % of the neat resin respectively; the cost would be reduced by 7%. The main drawback is the fracture toughness but for some applications like applications in electrical equipment, this will not matter much.

Figure 8 shows the flexural strength, flexural modulus, maximum flexural strain and fracture toughness of varying percentage by weight of E-sphere (SLG) reinforced phenol formaldehyde matrix composites. Figure 9 is the enlargement of Figure 8 to illustrate the flexural modulus and maximum flexural strain better. The flexural strength of the 5% SLG reinforced phenolic resin was 42.39 MPa. The value dropped significantly at 10 % by weight of SLG and remained low until there was a rebound at 25% by weight of SLG, where it was 26.48 MPa. It then dropped again.

The flexural modulus of the 5% SLG reinforced phenolic resin was 3.005 GPa as shown in Figure 9. The value dropped significantly at 10 % by weight of SLG and remained low until there was a rebound at 25% by weight of SLG, where it was 1.995 GPa. It then dropped again. Figure 9 also illustrates the fracture toughness of SLG reinforced phenolic composites with varying percentage by weight of SLG, which had been described earlier in Figure 7. In general, it can be concluded that as far as flexural properties and fracture toughness as well as cost were considered, the best percentage by weight of SLG in phenolic resin should be 5% as depicted in Figures 8 and 9. At this particulate loading, the flexural strength, flexural modulus, maximum flexural strain and fracture toughness are 29.1 MPa, 3.005 GPa, 0.014mm/mm and $13.8 \text{ MPa}\sqrt{m}$ respectively; the cost would be reduced by 5 %. The fracture toughness was greatly reduced after 5 % particulate loading and this contradicts to Waage et al. had found in their study, in which fillers were found to alter stress distribution in adhesive joints, thus improving fracture toughness [23]. Some extenders may be required to improve the fracture toughness of composites used in this study.

By viewing Figures 7 through 9, it can be argued that when tensile properties, flexural properties and fracture toughness as well as cost were considered, the best percentage by weight of SLG in phenolic resin should be 7.5 %. At this percentage by weight of SLG, the flexural strength, flexural modulus, maximum flexural strain and fracture toughness are 69%, 54%, 113% and 76 % of the 5% by weight of SLG composite respectively; the cost would be reduced by 7%. It can be argued that when all these properties and cost are considered the addition of SLG to phenolic resin is not viable as the reduction in cost cannot be offset by the reduction in properties. However, if flexural properties are not so significant, composite of phenolic resin with 7.5 % by weight can still be a viable option as it has cut cost by 7%.

Figure 10 shows the viscosity of 2027/E-sphere formulations as function of filler weight. This data shows that the viscosity increases with increasing percentage by weight of SLG. It has been found by experiment, and been confirmed by other researchers, that there is a viscosity limit of resin/SLG mixtures above which casting is not possible [24]. This is around 38-42% by weight and corresponds to viscosity in the range of 16,000 – 20,000 cps. This filler content is much higher than that previously determined to be best in terms of fracture toughness, 20% w/t. At 20% w/t, the viscosity of the resin/SLG mixture is around 4,550 cps [25]. At this viscosity, workability of the filled resin would be good. The viscosity of neat resin used by Waage et al. was 2,650 cps and it was found that if the initial viscosity of filler-resin mixture is greater than 6000 cps, difficulties will be encountered in achieving adequate adhesive distribution when spraying flakes [23]. The viscosity of neat resin used in this study was 3,240 cps therefore for the same particle loading, the initial viscosity of the filler-resin mixture of this resin is likely to be higher than its counterpart and the maximum percentage by weight of filler that could be added to this resin for

ease of casting would be less. The maximum particle (pecan shell flour) loading used by Waage et al. was 40% and the viscosity of the mixture was 15,400 cps, while that (SLG) in this study was 35 % and the viscosity of the mixture was 13,360 cps [23, 25]. If Figure 10 were extrapolated, it can be found that the viscosity of SLG filled phenolic resin would be over 20,000 cps and would not be suitable for casting [24, 25]. Therefore, the types of filler play an important part in the viscosity of the phenolic resin composite mixture and this is shown clearly in Table 2, which also illustrates that irrespective of the types of fillers, the higher the particulate loading, the higher the viscosity will be. However, for the 'best' composite of SLG (7.5 %) and phenolic resin, the viscosity is below 4,000 cps and there is no fluidity problem for casting the composites into moulds.

Figure 11 shows the scanning electron microscopy image of phenolic resin post-cured for 4 hours at 80 °C at a magnification of 3,500 times. Voids are found left by the evaporation of water formed during condensation polymerization of phenol formaldehyde. Despite the voids, its tensile strength was high because the structure is better than that shown in Figure 12, which illustrates the scanning electron microscopy image of phenolic resin reinforced by 20% by weight of SLG and post-cured for 4 hours at 80 °C at a magnification of 25,000 times. It can be found that the voids were partially filled by the SLG but the reinforcer did not fuse with the matrix and a gap was found between them. The lack of fusion brought about failure of the composite by cutting through the weak SLG when tensile load was applied. To improve the fusion between the reinforcer and the matrix, other fillers or resin will have to be added and this will also be research focus of the Centre of Excellence for Engineered Fibre Composites in the near future.

Figure 13 illustrates the scanning electron microscopy image of phenolic resin reinforced by 20% by weight of SLG and post-cured for 4 hours at 80 °C at a magnification of 15,000 X. It can be found that the voids were partially filled by the SLG but the reinforcer did not fuse with the matrix and gap was found between them. Its fracture toughness is $12.50 \text{ MPa}\sqrt{m}$ and it can be argued that if the reinforcer and the matrix mix homogeneously, the fracture toughness will even be higher. Figure 14 enables one to view the more serious gap of the composite (15%) clearer as the magnification of the image was increased to 25,000 X. With this magnification, it is clear that there is no fusion between the reinforcer and the matrix. To improve the fusion between the reinforcer and the matrix, other fillers or resin will have to be added and this will also be research focus for us in the near future.

The ceramic filler-reinforced phenolic resin friction materials increased slightly with increasing temperature up to 300 °C. However, the maximum temperature of 350 °C dramatically increased the wear rate of the material. This behaviour was attributed to the increased thermal decomposition of the phenol materials which further lowered

the bond strength of the fibre-matrix interface. As well as the shear strength of the phenolic resin matrix [26].

So and Rudin discovered that the final degree of cure of woodflour reinforced phenolic resin (resole) composites depended heavily on the curing conditions of resin pH, time and temperature with pH as the most important variable. By using acetone extraction technique, at a fixed curing temperature of 160 °C, condensation reaction time of 2 hours, Ba(OH)₂ catalyzed resole, F/P mole ratio of 1.37 and curing pH neutral, it was discovered that the longer the curing time, the higher the degree of cure. By keeping all material and curing conditions unchanged except pH value to 8.3, the curing time was nearly halved for the same degree of cure. Resins cured at neutral pH value were linked by ether linkages rather than the more stable methylene bridges. It was also discovered that the degree of cure increases with curing pH values. The same result was also shown by the CH₂/Ar₁ ratios (mole ratios of methylene bridges to phenolic residues). It was also discovered that curing phenolic resins should preferably be at temperatures below 180 °C to minimize degradation of the polymer [27]. The curing temperatures for composites used in this study were at or below 100 °C to ensure degradation will not happen but the curing time of 10 hours were too long and higher temperature and shorter curing time should be attempted in the future; the degree of cure as indicated by dynamic mechanical thermal analysis (DMTA) was 90% [28]. However, it was found that the curing process was unaffected by any additives. The differential scanning calorimetry (DSC) showed a single exothermic peak that was not influenced to a great extent by the fillers or extenders [23].

8. Conclusions

This study has evaluated the yield strength, tensile strength and Young's modulus of varying percentage by weight of SLG reinforced phenolic resin; in all cases, the fluidity of the slurry composite was high and could be cast easily into moulds. The values with no filler had also been compared with those found by other studies and some agreed with each other very well. The addition of SLG and perhaps other fillers to phenolic resins will not guarantee an optimum combination of all properties and cost. It can be argued that when the fusion between phenolic resin (matrix) and SLG (reinforcer) is improved by adding some other filler and resins to the composite, its tensile properties, flexural properties and fracture toughness will be improved.

References

1. Shackelford, J F, Introduction to Materials Science for Engineers, 3rd edition, Macmillan, 1992, pp.435-437, 459.
2. Smith, W F and Hashemir, J, Foundations of Material Science and Engineering, 4th edition, McGraw-Hill, 2006, pp. 523-525.
3. Strong, A B, Plastics: materials and processing, 3rd edition, Pearson/Prentice-Hall, 2006, pp. 182-183, 304-309, 323-333, 620-621.
4. Clarke, J L (Editor), *Structural design of polymer composites*, E & FN Spon, U.K., 1996, pp.59-62, 343-5, 357.
5. Chemwatch, Material Safety Data Sheet for Hexion Cellobond J2027L, 2005a, pp. 1-14.
6. Chemwatch, Material Safety Data Sheet for Hexion Phencat 15, 2005b, pp. 1-14.
7. Leicht, E and Sattelmeyer, R, Phenolic resins for rubber reinforcement, *Kautschuk und Gummi Kunststoffe*, Vol. 40, No. 2, 1986, pp. 126-129.
8. E-spheres, www.envirospheres.com.au, Envirospheres Pty Ltd., P O Box 497, NSW 2070, Australia, undated.
9. Callister, W D, Materials Science and Engineering, An Introduction, 6th edition, John Wiley, 2003 , pp. 126-7.
10. Ku, H, Trada, M and Puttgunta, V C, Mechanical Properties of Vinyl Ester Composites Cured by Microwave Irradiation: Pilot Study, *Key Engineering Materials*, 2006, Vol. 334-335, pp.537-540.
11. Ku, H, Puttgunta, V C and Trada, M, Young's Modulus of Vinyl Ester Composites Cured by Microwave Irradiation: Preliminary Results, *Journal of Electromagnetic Waves and Applications*, 2007, Vol. 21. No. 4, pp. 517 - 526.
12. Osswald, T A and Menges, G, Materials Science of Polymers for Engineers, Hanser publishers, New York, pp. 103-5, 229- 31, 1995.
13. John, V B, Engineering Materials, Macmillan, 1990, p. 209.
14. Callister, W D, Materials Science and Engineering: An Introduction, 7th edition, Wiley & Sons, Inc, 2007, pp. 217-218, 447-448.

15. Baker, L M, A simplified method for measuring plane strain fracture toughness, *Engineering fracture mechanics*, Pergamon press, United Kingdom, 1997, pp. 361-369.
16. ASTM: E399-78, Standard method of test for plane-strain fracture toughness of metallic materials, 1972.
17. Baker, L M, Development of the Short Road Method of Fracture Toughness Measurement, *Proceedings, Conference on Wear and Fracture Prevention*, 1980, 21-22 May, ASM, Metals Park, Ohio, pp. 163-180.
18. Baker, L M, Short Rod and Short Bar Fracture Toughness Specimen Geometries and Test Methods for Metallic Materials, *Proceedings, Fracture Mechanics: Thirteenth Conference*, ASMT STP 743, 1981, pp. 456-475.
19. Askeland, D R, *The Science and Engineering of Materials*, Third Edition, Stanley Thornes, New York, 1998, pp.163-164.
20. Callister, W D, *Fundamental of Materials Science and Engineering: An Integrated Approach*, 2nd edition, Wiley International, 2005, pp.184.
21. Wang, S, Adanur, S and Jang B Z, Mechanical and thermo-mechanical failure mechanism analysis of fibre/filler reinforced phenolic matrix composite, *Composites Part B*, 1997, 28B, pp. 215-231.
22. Redjel, B, *Mechanical Properties and Fracture Toughness of Phenolic Resin*, *Plastics, Rubber and Composites Processing and Applications*, 1995, Vol. 24, pp. 221-228.
23. Waage, S K, Gardnesr, D J and Elder, T J, Effects of fillers and extenders on the cure properties of phenol-formaldehyde resin as determined by the application of thermal techniques, *Journal of Applied Polymer Science*, 1991, Vol. 42, pp. 273-278.
24. Davey, S W, Heldt, T, Van Erp, G and Ayers, Stephen, *Vinyl Ester/Cenosphere Composite Materials for Civil and structural Engineering*, *FRP International*, Vol. 2, No. 3, 2005, pp. 2-5.
25. Ku, H, Rogers, D, Davey, R, Cardona, F and Trada, M, Fracture Toughness of Phenol Formaldehyde Composites: Pilot Study, *Journal of Materials Engineering and Performance*, 2008, Vol. 17, No. 1, pp.85-90.

26. Kim, H S and Huh, J S, Role of ceramic fillers in the friction and wear behaviour of phenolic resin composites, *Journal of Materials Science Letters*, Vol. 17, No. 6, 1998, pp. 501-502.
27. So, S and Rudin, A, Effects of resin and curing parameters on the degree of cure of resole phenolic resins and woodflour composites, *Journal of Applied Polymer Science*, 1990, Vol. 40, pp. 2135-2149.
28. Cardona, F, Rogers, D, Gurney, R, Trada, M and Ku, H, Flexural Tests of Phenol Formaldehyde and SLG Composites: Preliminary Results, *Journal of Reinforced Plastic and Composites*, 2009, Vol. 28, No. 22, pp. 2761-2769.

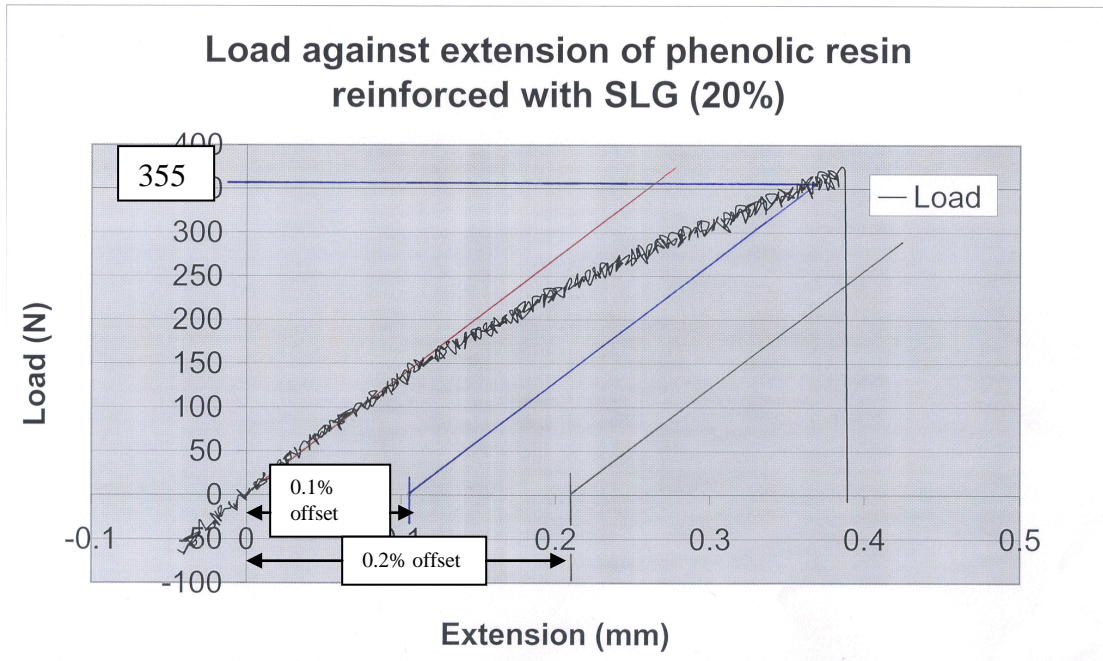


Figure 1: Load against extension of a sample showing the 0.1% proof load

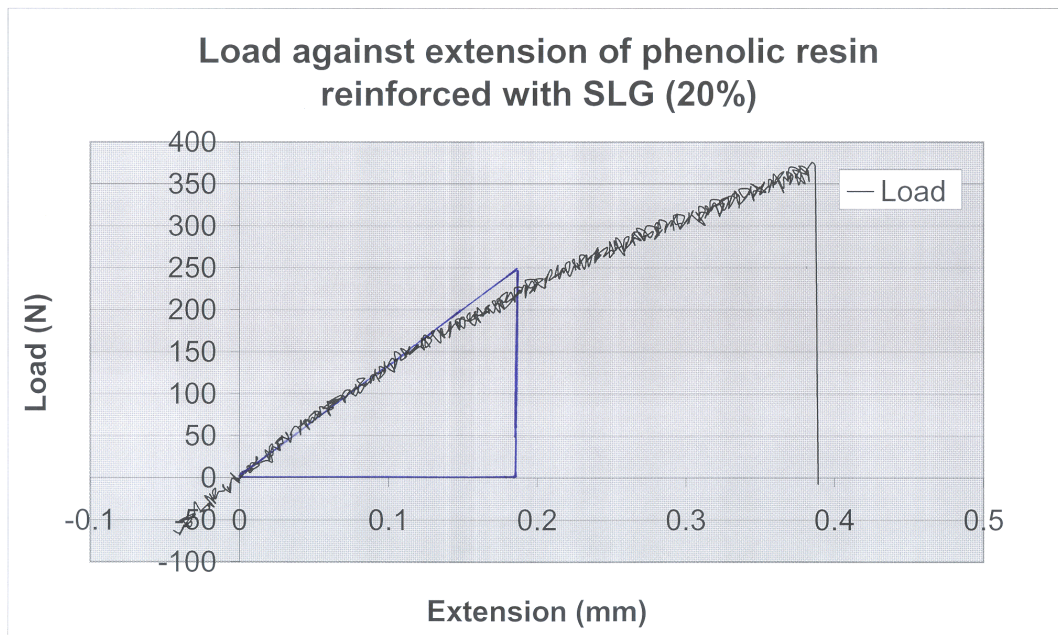


Figure 2: Graph showing how to get data for calculating Young's modulus in phenolic composite

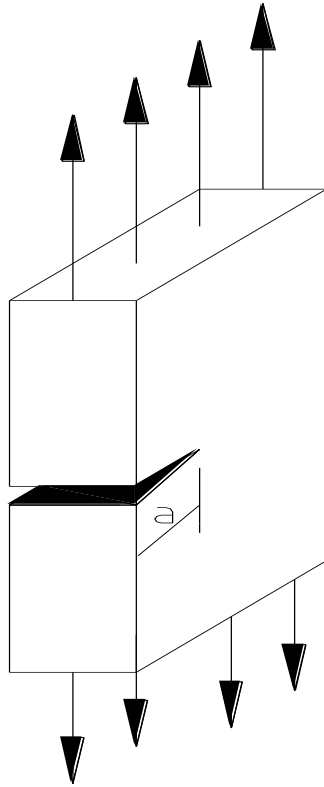
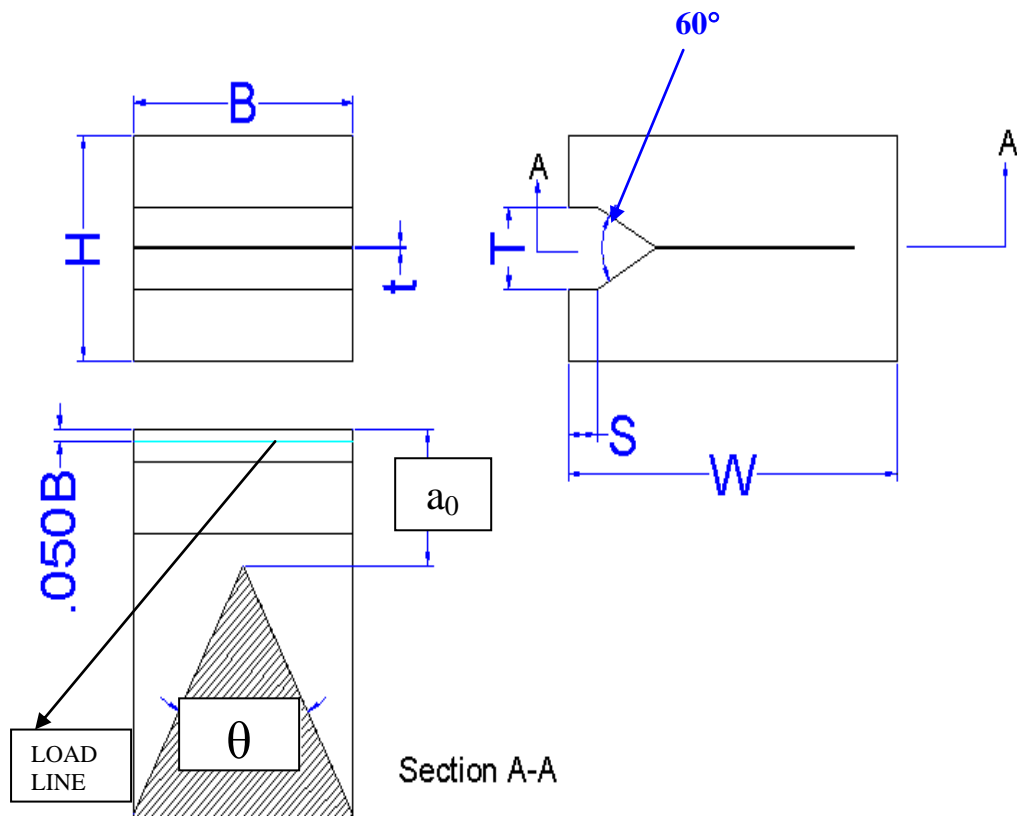


Figure 3: Drawing of fracture toughness specimen with edge flaw



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

Figure 4: Short Bar Specimen with Straight Chevron Slots.

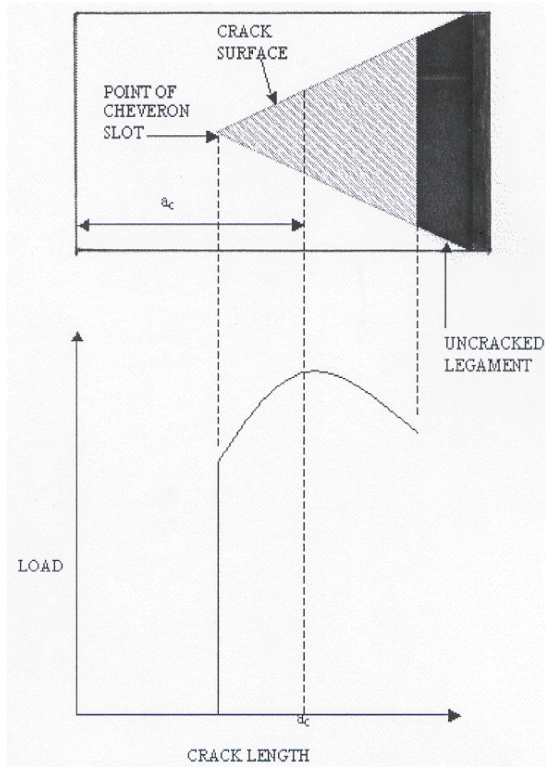


Figure 5: Variation of load versus crack length

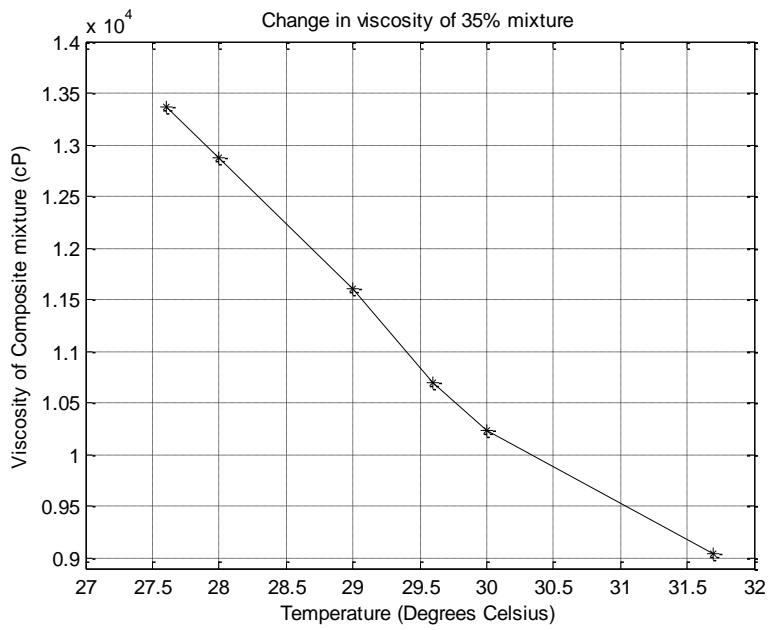


Figure 6: Viscosity change with temperature of 35% by weight of SLG composite

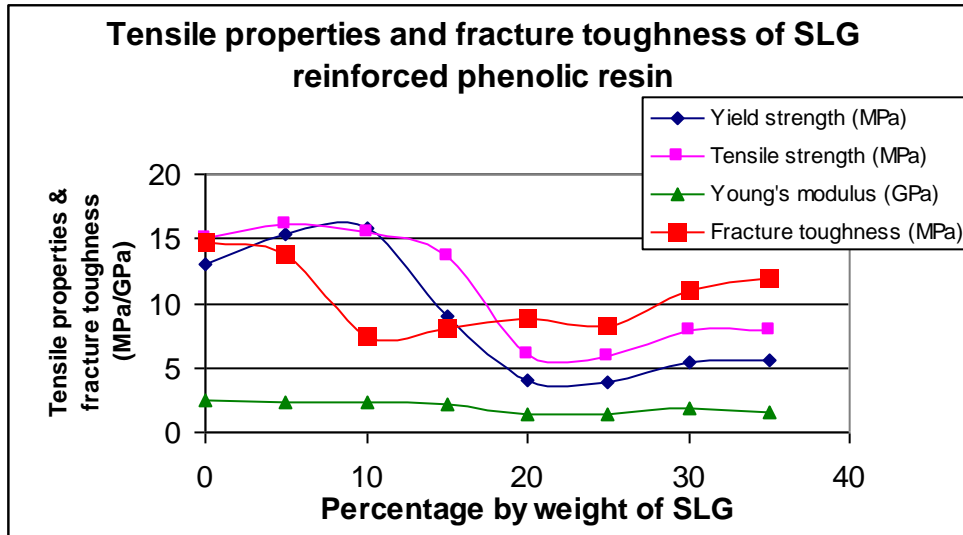


Figure 7: Tensile properties and fracture toughness of phenolic composite reinforced with varying SLG by weight

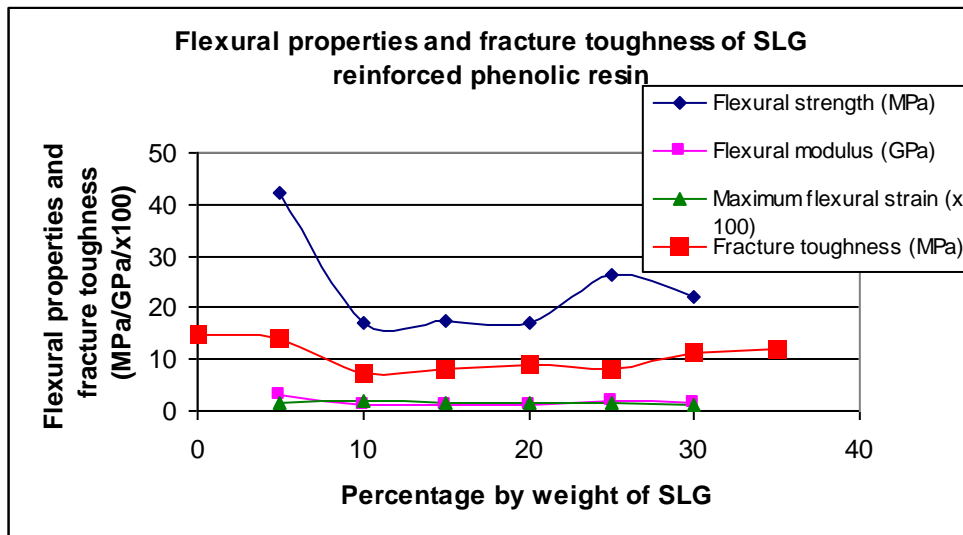


Figure 8: Tensile properties and fracture toughness of phenolic composite reinforced with varying SLG by weight

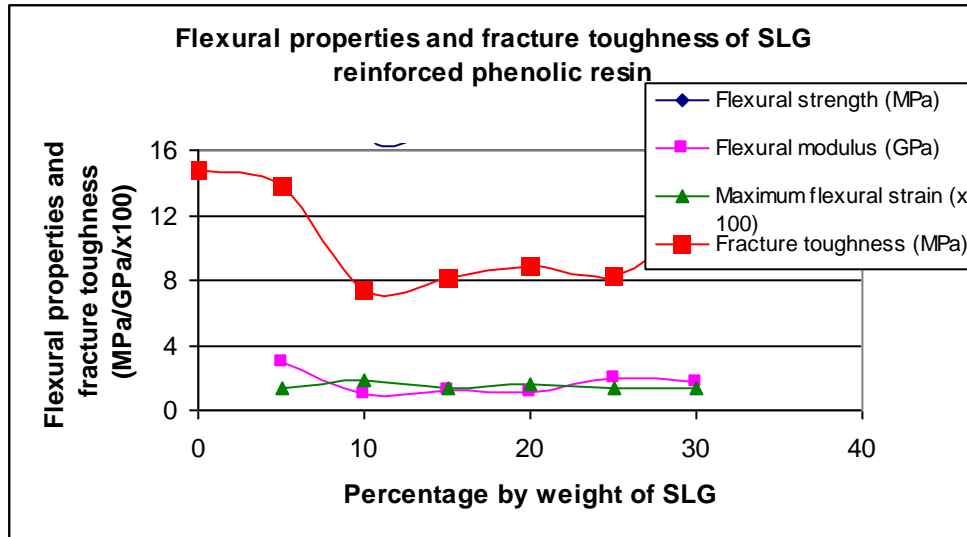


Figure 9: Truncated Figure 10 for flexural modulus and maximum flexural strain

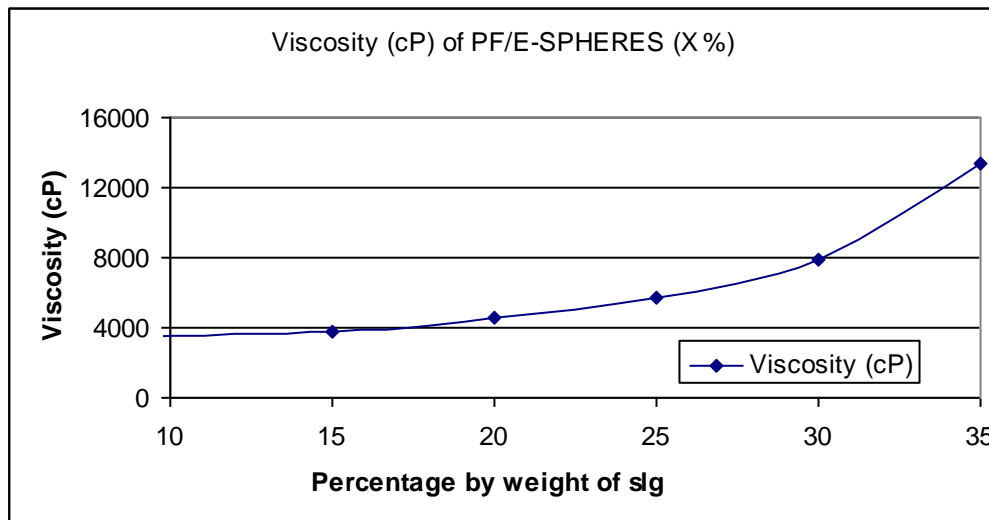


Figure 10: Viscosity of various composite mixtures at approximately 26°C

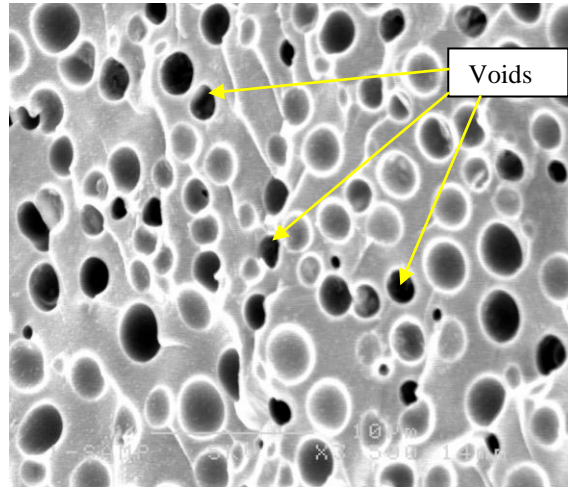


Figure 11: Phenolic resin post-cured for 4 hours at 80 °C at a magnification of 3,500 times

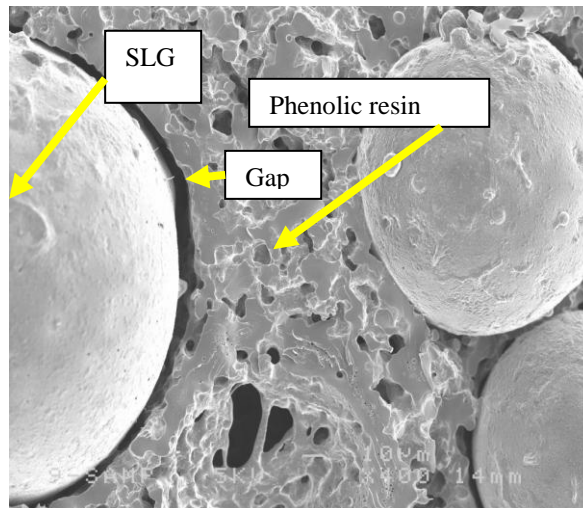


Figure 12: PF/E-SPHERE (20%) post-cured for 4 hours at 80 °C at a magnification of 25,000 times

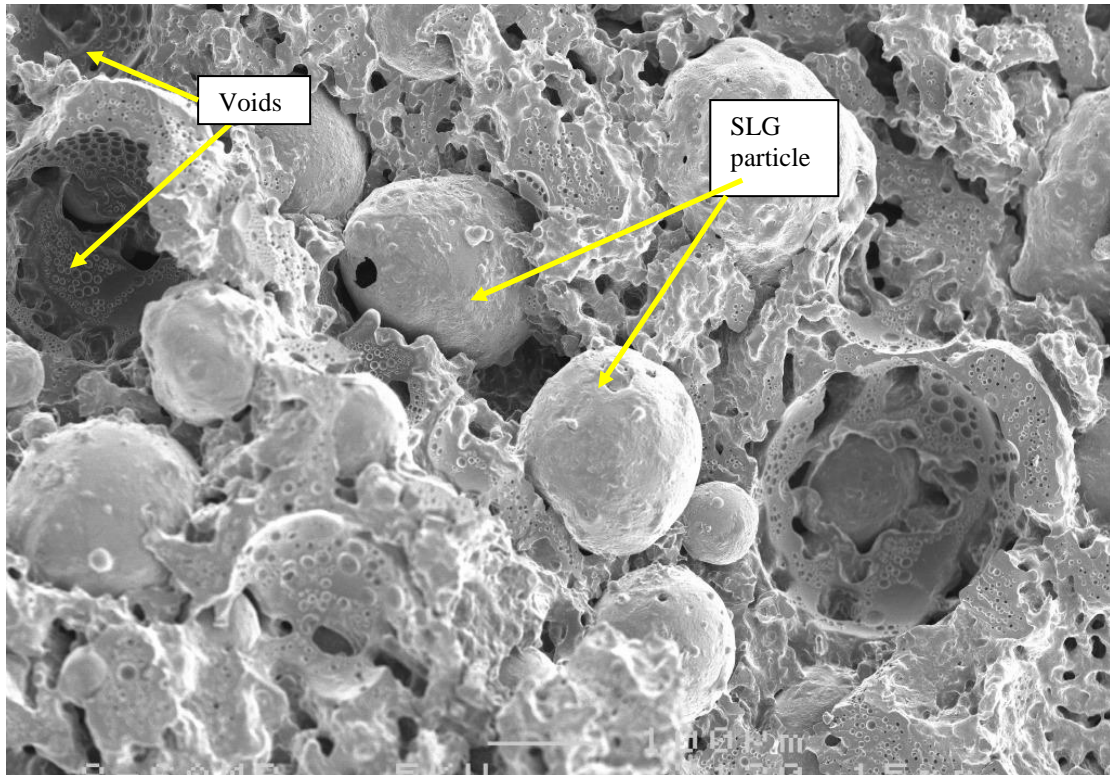


Figure 13: SEM image of phenolic resin reinforced by 20% by weight of slg and post-cured for 4 hours at 80 °C at a magnification of 15,000 X.

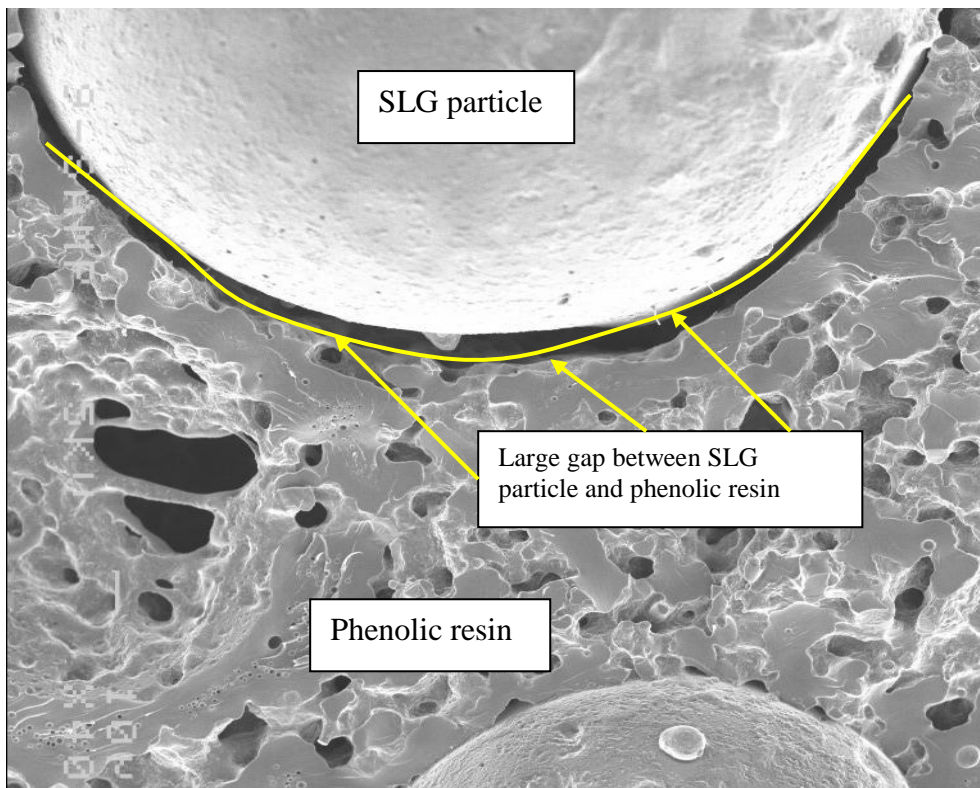


Figure 14: Closer look on more serious gap

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

	Materials	Resin (R)	Catalyst (C)	R + C	Slg	Composite
Parameters						
Percentage by weight		20	1	---	---	---
Percentage by weight		---	---	7	3	---
Weight of materials in 300 g of PF/SLG (10%)		686(g)	14 (g)	700 (g)	300 (g)	1000 (g)

Table 2: Viscosity and gel time measurements of phenol formaldehyde adhesives {adapted from [25]}.

Composites	Viscosity (cps) at 25 °C	Gel time (min) at 100 °C
PF resin (Waage et al)	2,650	23.1
25 % clay	7,000	27.6
25 % wheat flour	5,140	16.2
25 % pecan shell flour	3,700	24.2
33 % clay	12,000	29.5
33% wheat flour	6,600	2.0
40 % pecan shell flour	15,400	25.6
PF resin (this study)	3,240	-
25 % SLG	5,680	-
35 % SLG	13,360	-