

# Tensile Tests of Phenol Formaldehyde Glass-Powder-Reinforced Composites: Pilot Study

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Received 11 January 2009; accepted 31 August 2009

DOI 10.1002/app.31424

Published online 13 November 2009 in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** Phenol formaldehyde was filled with glass powder (GP) to optimize the strength and impact toughness of the composite for structural applications by a research center at the University of Southern Queensland. To reduce costs, the center wished to fill as much of the glass microspheres as possible to maintain sufficient strength and impact toughness in the composites in structural applications. In this project, we varied the weight percentages of the GP in the composites, which were then subjected to tensile tests. The best weight percentage

of GP that could be added to the phenolic resin to give the optimum yield, tensile strengths, Young's modulus, and cost was found to be about 10%. The contribution of this study was the finding that if the tensile properties are the most important factors to be considered in the applications of the composites, GP is not a suitable filler. © 2009 Wiley Periodicals, Inc. *J Appl Polym Sci* 116: 10–17, 2010

**Key words:** composites; mechanical properties; resins

## INTRODUCTION

Organic–inorganic hybrid materials consisting of inorganic materials and organic polymers are a new class of materials that has received much attention in recent years. The composite materials exhibit the characteristics of both inorganic materials and organic polymers. It has been established in recent years that polymers reinforced with a small percentage of strong fillers can have significantly improved mechanical and thermal properties.<sup>1</sup> 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 materials on a cost-per-volume basis. Phenolics are formed from the condensation of a polymerization reaction between phenol and formaldehyde. The condensation reaction for phenolics can be carried out under two different conditions, which result in two different intermediate materials. One intermediate is called *resoles*, and the other is called *novolacs*.<sup>2,3</sup>

The novolacs are formed by the reaction of phenol and formaldehyde in an acid solution but with insufficient formaldehyde to complete the reaction at 100°C (the opposite of the formation of resoles). About 1 mol of phenol is reacted with 0.7–0.85 mol

of formaldehyde. This is the first stage of the reaction, and a brittle thermoplastic resin is produced; this 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 for one to create methylene crosslinkages to form a thermosetting material. When heat and pressure are applied to the hexa-containing novolac resin, the hexa decomposes and produces ammonia, which provides methylene crosslinkages to form a network structure. Because hexa, a second material, must be added to the novolacs, these are called *two-stage resins*. The temperature required for the crosslinking of the novolac resin ranges from 120 to 177°C. The various filler amounts used can vary from 50 to 80 wt %. The fillers reduce shrinkage during molding, lower cost, and improve strength. They are also used to improve the electrical and thermal insulating properties and chemical resistance.<sup>2–5</sup>

In this research project, we investigated the yield strength, tensile strength, and Young's modulus of phenol formaldehyde (PF) composites reinforced with various weight percentages of glass powder (GP), the filler, with a view to determining the optimum weight percentage of GP used in the composites.

Dekkers and Heikens<sup>6</sup> found that the tensile strengths of glass-bead reinforced composites decreased steadily with increasing weight of filler if the interfacial adhesion between the matrix and the filler was excellent. If the interfacial adhesion of the matrix and the reinforcer was poor, the tensile strengths of glass-bead reinforced composites

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decreased abruptly with particulate loading. The incorporation of a small amount of BaSO<sub>4</sub> particles in polypropylene did not significantly affect the yield strength of the composites, whereas a relatively high content of BaSO<sub>4</sub> particles (>16%) had a negative effect on the yield strength.<sup>7</sup> The tensile strength of polyimide/SiO<sub>2</sub> hybrid films increased first to a maximum and afterward decreased with increasing silica content.<sup>8</sup> This was in line with the results of a study by Ku et al.,<sup>9</sup> in which phenolic resin was reinforced with SLG (ceramic hollow spheres). Spanoudakis and Young<sup>10</sup> discovered that the Young's modulus of glass-bead-filled epoxy composites decreased with increasing particulate content. This was in line with the curve of this study. The Young's modulus of phenolic-resin-filled nylon 6-polyesteramide block copolymers increased with increasing weight percentage of phenolic resin.<sup>11</sup> This time, the curve was the opposite of that found in this study, but in this case, the phenolic resin was the reinforcer. Qi et al.<sup>1</sup> improved the mechanical performance, including the tensile strength and impact strength, of a composite of a SiC-powder-filled phenolic resin by glass fiber.<sup>1</sup> However, if GP were added instead of glass fiber, it can be argued that the fracture toughness and flexural properties would improve, but the tensile properties would deteriorate.

### PF

The commercial resole resin used in this study was J2027 and was manufactured by Borden Chemical Pty (Columbus, OH). Its official name is now Hexion Cellobond J2027L because the company was taken over by Hexion.<sup>12</sup> The catalyst used to crosslink the resin was Hexion Phencat 15 and was produced by the same company.<sup>13</sup> The weight ratio of the resin to hardener varied from 30 : 1 to 12 : 1 depending by the weight percentage of GP; the higher the weight percentage of the filler was, the more catalyst was required.

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 fibers, fiberglass, and chopped thermoplastic fibers (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 application. When PF resin is subjected to a flame, it chars rather than melts or burns. They are,

therefore, widely applied in situations where the prevention of flammability and smoke is vital. Furthermore, the char has a very low thermal conductivity so that the surrounding materials are protected by the decomposed phenolic. The low thermal conductivity of the resin promotes its use as bases for toasters and knobs for appliances. Most PF 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 gives it 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 byproduct 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. A high water content can cause the structures to delaminate when they are exposed to heat.<sup>4,5</sup>

### GP

The GP used was SPHERICEL 60P18 (spherical) hollow glass spheres. They are used to enhance the performance and reduce the viscosity in paints and coatings and as lightweight additives in plastic parts. They are chemically inert and nonporous and have very low oil absorption. Typical properties of the spheres are shown in Table I.<sup>14</sup> SPHERICEL 60P18 hollow-sphere products offer formulators flexibility in polymer composites. The addition of hollow spheres to fiberglass-reinforced plastics, epoxy compounds, and urethane castings can provide weight reduction, cost savings, and improved impact resistance. The insulating features of hollow spheres also work to chemists' advantage in thermal shock and heat-transfer areas. The two densities available are 0.6 and 1.1 g/cc; this provides choices to best fit mixing and target weight requirements.<sup>15</sup> The density of the hollow GP used in this research was 0.6 g/cc because the density of the other filler, ceramic hollow spheres, or SLG used in a similar study was 0.7 g/cc. This will give a better basis for comparison for results obtained in the future. When used in polymer concrete, hollow spheres provide a cost-effective alternative without degradation of the physical properties. The material safety data sheet of SPHERICEL 60P18 hollow spheres was also carefully studied to prevent unnecessary accidents.<sup>16</sup>

The particle size of the white GP ranged from 6 to 32 μm, with an average size of 20 μm. They were, therefore, micrometer fillers. These fused inorganic oxides were spherical and nonporous.

**TABLE I**  
**Weight of Materials Required to Make 1000 g of PF/GP (30%)**

Parameter	Material				
	R	C	R + C	GP	Composite
wt %	12	1	—	—	—
wt %	—	—	7	3	—
Weight of materials in 1000 g of PF/GP (g; 10%)	646	54	700	300	1000

R = resin; C = catalyst.

### STRESS–STRAIN CURVE

In the tensile test, the force and extension of the test pieces were recorded. Figure 1 shows a typical curve for the specimen undergoing testing. This graph gives information for the tensile force versus tensile elongation. A Material Testing Systems 810 was used for the tests. The capacity of the testing machine was 100 kN. The rate of extension, 1 mm/min, was in accordance with an Australian standard (Australian Standard 1145.2, 2001).<sup>17</sup>

#### Yield strength

The yield strength is the strength at which a definite amount of plastic strain occurs. The gauge length used should be the separation of the grips and 110 mm because the elongation of a nonductile material (e.g., this phenolic composite) when subjected to tensile force will spread along the sample evenly and will not be restricted to the conventional gauge length of 50 mm.<sup>18,19</sup> Figure 1 also illustrates how the 0.05% proof load was determined.

When the intersection was projected to the *y* axis, the load found was 881 N, which was the 0.05% offset yield load. The yield strength was calculated with the following relationship:<sup>20</sup>

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

For example, the yield strength of the sample illustrated in Figure 1 was calculated as follows:

$$\begin{aligned} \text{Yield strength} &= \frac{0.05\% \text{ offset load}}{\text{Original cross-sectional area}} \\ &= \frac{881}{14.67 \times 5.19} = 11.57 \text{ MPa} \end{aligned}$$

#### Tensile strength

We calculated the tensile strength by dividing the maximum load by the original cross-sectional area of the specimen as follows:<sup>20</sup>

$$\text{Tensile strength} = \frac{\text{Maximum load}}{\text{Original cross-sectional area}} \quad (2)$$

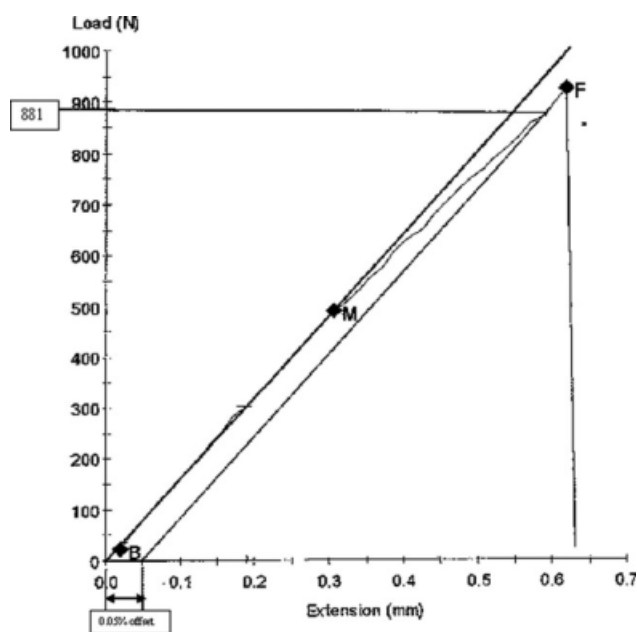
For example, the tensile strength of sample illustrated in Figure 1 was calculated as follows:

$$\text{Tensile strength} = \frac{924}{14.67 \times 5.19} = 12.14 \text{ MPa}$$

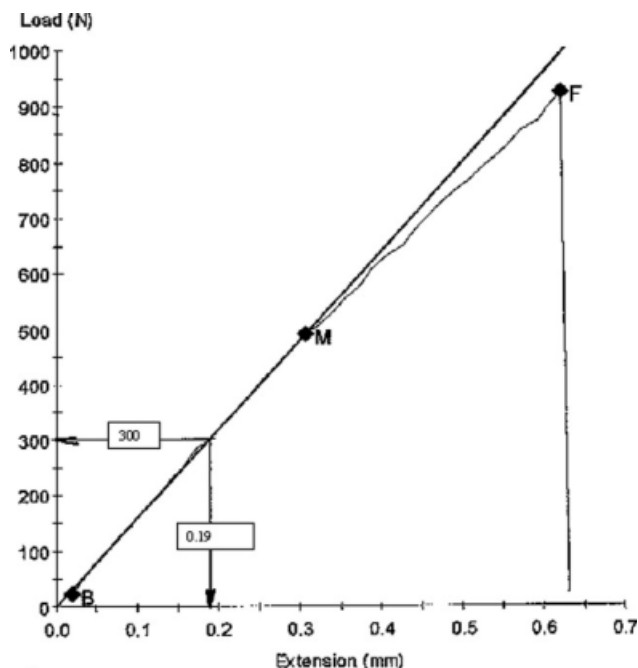
The tensile strength is the most sought after result of a tensile test. It is easy to determine, has become a familiar property, and is useful for the purposes of specifications and quality control of a product.

#### Young's modulus

The Young's modulus or modulus of elasticity is used to measure the stiffness of a material. The Young's modulus can be calculated by the calculation of the slope of the initial linear portion of the stress–strain curve. As the force–extension curve of the material did not possess a perfectly linear portion (Fig. 1), the Young's modulus quoted was the secant



**Figure 1** Load versus extension of a sample showing the 0.05% proof load. F, final; M, middle; B, beginning.



**Figure 2** Graph showing how to obtain data to calculate the Young's modulus in the phenolic composite.

modulus at a strain of 0.1%.<sup>21,22</sup> The Young's modulus ( $E$ ),<sup>20</sup> from eqs. (1) and (2) becomes

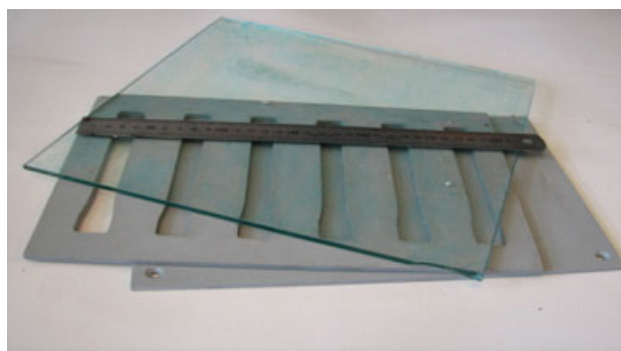
$$E = \frac{F}{\frac{\Delta L}{L_0}} \quad (3)$$

For example, the Young's modulus of the sample illustrated in Figure 1 was calculated with the data provided from Figure 2, in which a portion of the most linear part of the curve was selected; after the top point of the selected linear portion into the  $x$  and  $y$  axis was projected, the force (300 N) and the extension (0.19 mm) were obtained and used in the following calculation:

$$E = \frac{\frac{300 - 0}{14.67 \times 5.19}}{\frac{0.019 - 0}{110}} = 2285 \text{ MPa}$$

### COMPOSITE SAMPLES

The reinforcer was GP (glass hollow-sphere) particulates, and they were 0–30 wt % in the cured PF/GP composite ( $X$  %), where  $X$  is the weight percentage of the filler. Above 30 wt % filler, the slurry would be too sticky to be cast into molds. As the raw materials of the composites were liquid and glass hollow spheres, the tensile test specimens were cast to shape. The resin was a dark brownish liquid and was first mixed with the dark brownish catalyst. After that, the GP was added to the mixture, which was then mixed to give the uncured composite.

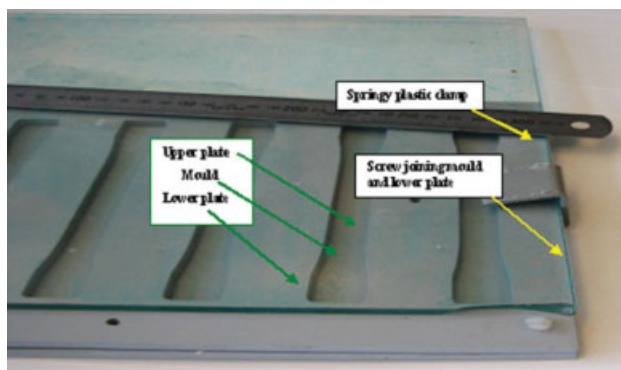


**Figure 3** Molds for the specimens. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

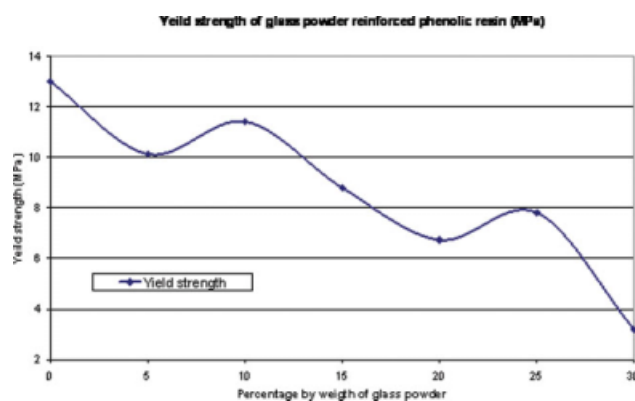
Table I shows the mass in grams of the resin, catalyst, and SLG required to make 1000 g of the uncured composite with 30 wt % GP.

The mixture of GP, resin, and accelerator was blended with a mechanical blender to ensure a more homogeneous mixture. The upper and lower plates and the mold are illustrated in Figure 3. They were clamped by nine screws and springy plastic clamps, as illustrated in Figure 4. This proved to be effective, and no seeping of the slurry took place when the samples were cured under ambient conditions. The screwed and tightened mold combination was slightly vibrated to facilitate the escape of the gases, and this certainly reduced the porosity of the specimens. Finally, before the uncured composite was poured into the mold, the upper surface of the lower plate, the cavities of the mold, the two faces of the mold, and the lower surface of the upper plate were sprayed with more releasing agent (wax) to enable easy release of the samples after curing. The uncured composite was then cast into the molds (Fig. 3) to cure under ambient conditions.

After the initial 72-h curing, when the test pieces were removed from the mold, they were postcured.



**Figure 4** Screwing and clamping of the mold, upper and lower plates. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



**Figure 5** Yield strength of the phenolic composite reinforced with various SLGs by weight. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

We achieved this by curing the pieces in an oven. The oven temperatures and times were

- 4 h at 50°C.
- 4 h at 80°C.
- 2 h at 100°C.

During the initial curing process of 4 h at 50°C, we observed that a number of test pieces developed a bow in middle. This bowing was between 1 and 4 mm in the middle of the piece and seemed to be exacerbated by the higher temperature baking processes. To counteract this, after they were removed from each curing session, all test pieces were subject to an approximate 2-kg load while between two pieces of toughened glass. The time for this weighting was approximately 16 h as they cured overnight. The test pieces were then tested.

## RESULTS AND DISCUSSION

Figure 5 illustrates the yield strengths of various weight percentage of PF matrix composites reinforced with glass hollow spheres. The yield strength of the neat resin was 13.00 MPa, which was higher than those of the composites with any weight percentage of GP. After dropping to 10 MPa at 5 wt % filler, it rebounded back to 11.43 MPa at 10 wt % GP. In general, the higher the weight percentage of

GP was, the lower the yield strength was. Table II shows the values of yield strength mentioned previously with their standard deviations in parentheses.

Figure 6 shows the tensile strengths of the phenolic composite with various GP contents; the tensile strength of the neat resin was 15.00 MPa, which was higher than those of the composites with any weight percentage of GP. At 5 wt % filler, the tensile strength dropped to 10.14 MPa; it then rebounded to the highest value, 12.6 MPa, at 12.5 wt % GP. After this SLG reinforcement dragged the values of the tensile strength down, it dropped dramatically from 12.08 to 5.38 MPa when the weight percentages of the filler were 10–30%, respectively. The variation of the tensile strength with respect to the weight percentage of GP was the same as that of the yield strength. If the cost and tensile strength were considered at the same time, the composite with 12.5 wt % filler was the best. Table II shows the values of the tensile strength mentioned previously with their standard deviations in parentheses.

Redjel<sup>23</sup> found that the tensile strength for a neat resin was 27 MPa, which was really very high compared to our results. The material he used was pure phenolic resin 84055 catalyzed by 3% C 1650 and cured at 80°C for 8 days. It was produced and prepared by CDF-Chimie, France. The curing time was excessively long and would not be industrially viable, and the energy consumption was enormous.

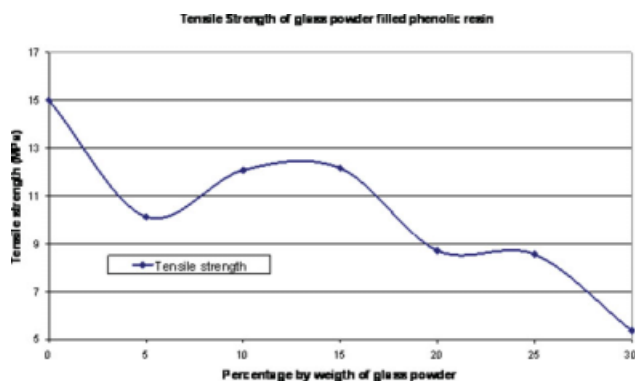
The trend of the tensile strengths of this research was in line with those of glass-bead-reinforced polystyrene; that is, the tensile strengths decreased with increasing particle loading.<sup>24</sup> However, the trend was just the opposite of that of glass-bead-filled epoxy resin.<sup>25</sup>

Figure 7 shows the Young's modulus of the glass-hollow-sphere-reinforced PF matrix composites varying by weight. The Young's modulus of the neat resin was 2.51 GPa, and it dropped to 2.23 GPa when the weight percentage of GP was 5%. It then bounced back to 2.38 GPa at 10 wt % filler; after that, the values did not change much but still dropped slightly until the filler weight percentage was 20%. The value then dropped significantly, and it was 1.31 GPa when the weight percentage of GP was 25%. It dropped further to 1.29 MPa when the weight percentage of the filler was 30%. The cost of

**TABLE II**  
Yield Strength, Tensile Strength, and Young's Modulus of Phenolic Composite Reinforced with GP

Mechanical property	SLG (wt %)						
	0	5	10	15	20	25	30
0.05% yield strength (MPa)	13.00	9.63 (1.37)	11.43 (1.20)	8.79 (1.37)	6.74 (2.15)	7.82 (1.20)	3.19 (0.80)
Tensile strength (MPa)	15.00	10.14 (1.45)	12.08 (1.11)	12.16 (2.85)	8.72 (2.01)	8.56 (0.71)	5.38 (0.46)
Young's modulus (MPa)	2510 (118) <sup>a</sup>	2230 (130)	2380 (126)	2189 (240)	2230 (1085)	1310 (277)	1290 (147)

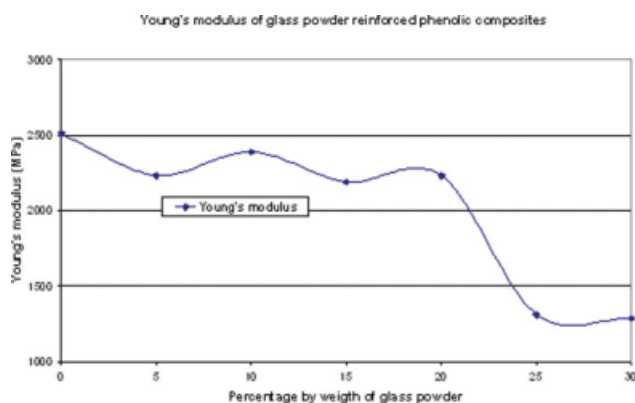
Standard deviations are in parentheses.



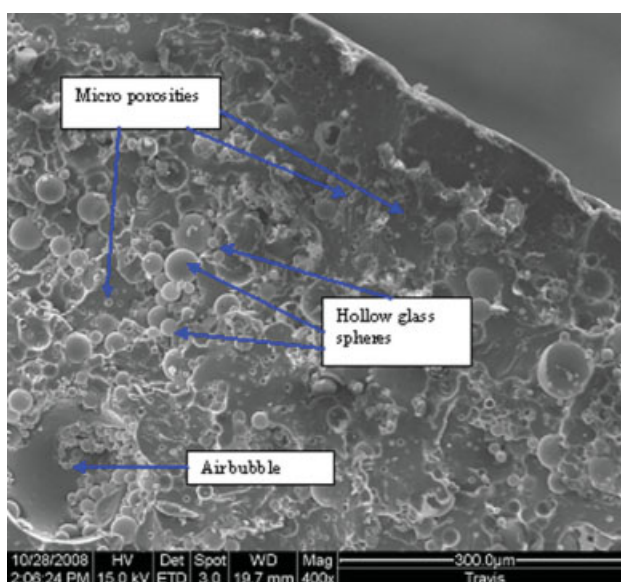
**Figure 6** Tensile strength of the phenolic composite reinforced with various SLGs by weight. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

the resin was \$7/kg, whereas that of GP was \$3/kg. For 10 wt % GP, the cost of 1 kg of the composite was equal to  $0.9 \times \$7 + 0.1 \times \$3 = \$6.60$ . The reduction in cost was equal to  $(\$7 - \$6.60)/\$7 = 5.7\%$ , whereas the reduction in the Young's modulus was equal to  $(2500 \text{ MPa} - 2386 \text{ MPa})/2500 \text{ MPa} = 4.8\%$ . For other weight percentages of filler, the gain from the reduction in cost was offset by a larger loss because of a reduction in Young's modulus; if cost and Young's modulus were considered at the same time, it can be argued that the composite with 10 wt % filler was the best.

The values found seemed to be reasonable when they were compared with those of phenolic formaldehyde (2.76–4.83 GPa).<sup>20</sup> However, the Young's modulus of the pure phenolic resin in this study was 2.51 GPa, which was 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; we used Chemwatch Borden (Hexion) Cellobond J2027L phenolic formaldehyde resin. On top of it,



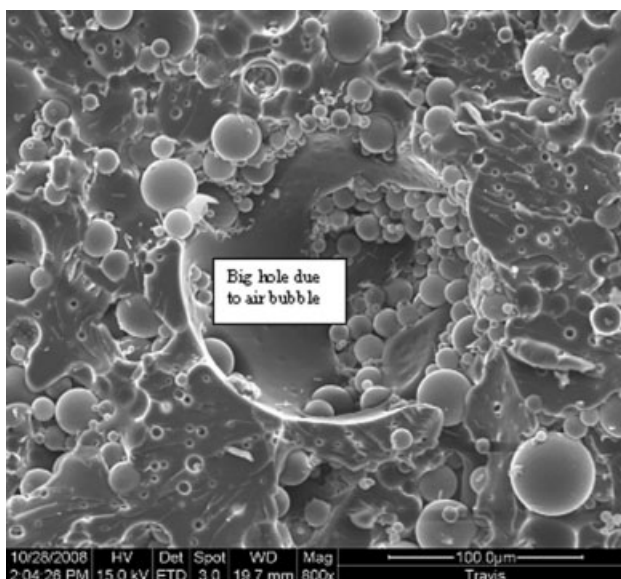
**Figure 7** Young's modulus of the phenolic composite reinforced with various SLGs by weight. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



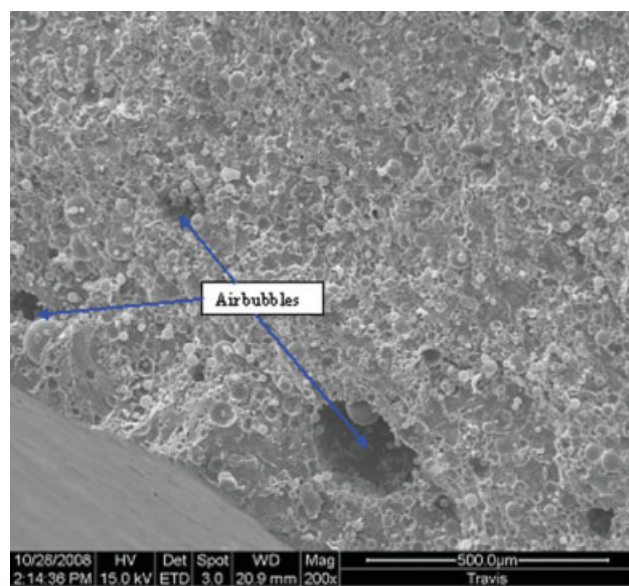
**Figure 8** Top surface of the fractured flexural sample of the phenolic resin reinforced by 10 wt % hollow glass spheres cured conventionally (400 $\times$ ). [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

they did not mention the temperatures and duration of soaking when they cured the resin and its filler.<sup>18</sup> Table II shows the values of Young's modulus mentioned previously with their standard deviations in parentheses. Redjel<sup>23</sup> found that the Young's modulus for the neat resin was 5.16 GPa, which was really very high compared to our results and those found in literature.<sup>21,23</sup>

Figure 8 shows the top surface of the fractured flexural sample of phenolic resin reinforced by 10 wt %

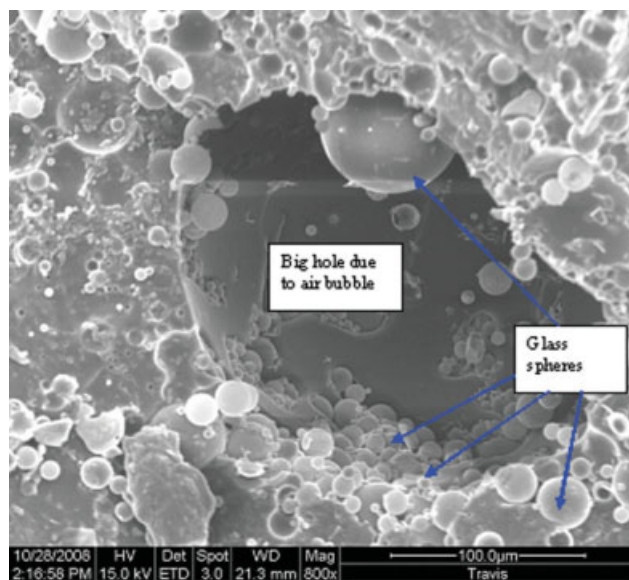


**Figure 9** Magnified image of the left-hand corner of Figure 8 (800 $\times$ ).

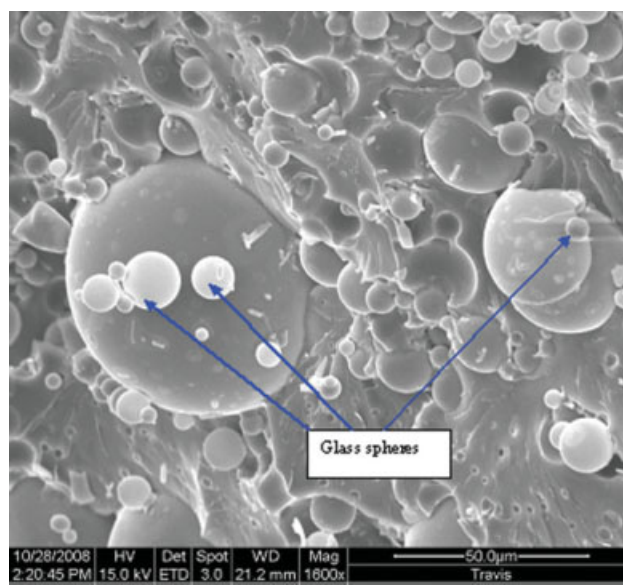


**Figure 10** Bottom surface of the fractured flexural sample of the phenolic resin reinforced by 10 wt % hollow glass spheres cured conventionally (200 $\times$ ). [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

hollow glass spheres cured conventionally at 400 $\times$ . A large hole due to air bubbles was found in the bottom left-hand corner of the figure. The sizes of the microporosities were generally very small, around 2–10  $\mu\text{m}$ . This could not be avoided because the molecular weight of the resin increased because of crosslinking, and the water already present in the resin and that produced during the reaction became incompatible with the curing resin and phase-separated to produce water domains, which were



**Figure 11** Magnified image of the hole of Figure 10 (800 $\times$ ) [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



**Figure 12** Higher magnification of the fractured surface (1600 $\times$ ). [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

observed as microvoids.<sup>26</sup> The sizes of the hollow glass spheres varied from 5 to 30  $\mu\text{m}$ . Because of the small size of the porosities, the quality of the composites produced was good, and accurate tensile properties could be obtained. Figure 9 illustrates the magnified image of the left-hand corner of Figure 8 at 800 $\times$ . Many smaller hollow glass spheres were found in the hole.

Figure 10 depicts the bottom surface of the fractured flexural sample of phenolic resin reinforced by 10 wt % hollow glass spheres cured conventionally at 200 $\times$ ; a big hole is shown in the middle of the lower part of the figure, which might have been due to a large air bubble. Figure 11 shows the magnified image of the hole of Figure 10 at 800 $\times$ ; the sizes of the hollow glass spheres varied significantly. Figure 12 illustrates the higher magnification of fractured surface at 1600 $\times$ ; many small glass spheres were on larger glass spheres or bubbles.

## CONCLUSIONS

In this study, we evaluated the yield strength, tensile strength, and Young's modulus of phenolic resins reinforced with various weight percentages of GP; in all cases, the fluidity of the slurry composite was high, and the composites could be cast easily into molds. The values with no filler were also compared with those found by another study, but they did not agree with each other very well. Because the sizes of the porosities of the composites found in this study were very small, the values of the tensile properties obtained were very good and reliable as their standard deviations were low. Some air bubbles were

found because of the manual manufacturing of the samples. The strengths would likely improve if semiautomatic or automatic manufacturing were used. It can also be argued that if the fusion between the phenolic resin (matrix) and GP (reinforcer) was improved by the addition of some other fillers and resins to the composite, the tensile properties would be improved.

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