Flexural Properties of Phenolic Resin Reinforced with Glass Powder: Preliminary Results

H. Ku, M. Trade, R. Nixon, P. Wong

Faculty of Engineering and Surveying, Centre of Excellence in Engineered Fibre Composites, University of Southern Queensland, Australia

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ABSTRACT: Phenol formaldehyde was filled with glass powder to increase its flexural strength for civil and structural applications by a research centre on composites, University of Southern Queensland (USQ). To reduce costs, the centre wishes to fill in as much glass powder as possible subject to maintaining sufficient strength of the composites in structural applications. This project varies the percentage by weight of the glass powder in the composites from 5 to 30%. The specimens were then subjected to flexural tests. The results show

that composite with 17.5% by weight of the glass hollow spheres produces the highest flexural strength and flexural modulus combined with a very good fluidity for casting. The maximum flexural strain was achieved with pure resin. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 116: 347–354, 2010

Key words: flexural strength; flexural strain; flexural modulus; phenol formaldehyde; phenolic resin; glass hollow spheres and glass powder

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.

In the resole process, the condensation polymerization is performed in an alkali solution with excess formaldehyde and is carefully controlled so that a linear, noncrosslinked polymer liquid, resole, is produced. The resole can then be molded. When molding, the crosslinking is achieved by heating the viscous liquid. Since a crosslinked part can be obtained by simply heating the resoles, which are called onestage resins.

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 mole to 0.85 mole of formaldehyde. This is the first stage of the reac-

tion 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 crosslinking 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

This research project is to investigate the flexural strength, flexural strain, and flexural modulus of phenol formaldehyde composites reinforced with varying percentage by weights of glass powder (GP) with a view to finding out the optimum percentage by weight of the filler used in the composites and at the same time minimize the cost.

PHENOL FORMALDEHYDE

The commercial resole resin used in this study is J-2027L produced by Hexion Speciality Chemicals Pty Ltd. Its official name is Hexion Cellobond J2027L.² The catalyst used to crosslink the resin is

Correspondence to: H. Ku (ku@usq.edu.au).

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phenolic resin hardener catalyst produced by the same company. The official name of the catalyst is Hexion Phencat 15.³ The ratio by weight of the resin to hardener is 30 : 1 for percentage by weight of glass powder from 5 to 15 %; the ratio became 20 : 1 for percentage by weight of glass powder of 20%; it became 12 : 1 for all other percentages by weight of glass powder. More catalyst had to be added to the resin as the percentage by weight of glass powder was increased because the filler hindered the hardening process of the composites.

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.¹

THE GLASS POWDER

The glass powder used is SPHERICEL[®] 60P18 (spherical) hollow glass spheres. They are used to enhance performance and reduce viscosity in paints and coatings and as lightweight additives in plastic

TABLE I Typical Properties of Hollow Glass spheres

Shape	Spherical			
Color	White			
Composition	Proprietary Glass			
Density	1.1 g/cc and 0.6 g/cc			
Particle Size	Mean Diameter 11 and 18 microns			
Hardness	6 (Moh's Scale)			
Chemical Resistance	Low alkali leach/insoluble in water			
Crush Strength	>10,000 psi			

parts. They are chemically inert, nonporous, and have very low oil absorption. Typical properties of the spheres are shown in Table I.⁴ SPHERICEL® 60P18 hollow spheres products offer formulators flexibility in polymer composites. The addition of hollow spheres to fiberglass reinforced plastics (FRP), epoxy, compounds, and urethane castings can provide weight reduction cost savings and improved impact resistance. Insulating features of hollow spheres also work to the chemists' advantage in thermal shock and heat transfer areas. Two densities available are 0.6 g/cc to 1.1 g/cc; it provides choices to best fit mixing and target weight requirements.⁵ The density of the hollow glass powder used in this research is 0.6 g/cc because the other filler, ceramic hollow spheres or SLG (fly ash) used in similar study is 0.7 g/cc; this will give a better basis for comparison of results obtained in the future. When used in polymer concrete, hollow spheres provide a cost-effective alternative without degrading physical properties. The material safety data sheet of SPHERICEL[®] 60P18 hollow spheres was also carefully studied to avoid unnecessary accidents.6

The particle size of the white glass powder ranges from 6 to 32 μ m with an average size of 20 μ m. They are therefore micron fillers. These fused inorganic oxides are spherical and nonporous.

THREE POINT BENDING TEST

The three point bending flexural test provides values for the modulus of elasticity in bending E_B , flexural stress σ_f , flexural strain ε_f 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. However, this method has also some disadvantages: the results of the testing method are sensitive to specimen and loading geometry and strain rate.

The standard used is ISO 14,125:1998(E) because the results can then be compared with the work of others.⁷ The centre uses a universal machine MTS Alliance RT/10 at 10 kN couple with the software TESTWORK 4. The dimensions of the specimens of

Weight of Materials required to Make 1000 g of PF/GP (30%)								
Parameters	Materials							
	Resin (R)	Catalyst (C)	R + C	Glass powder	Composite			
Percentage by weight	12	1	_	_	_			
Percentage by weight	-	-	70	30	-			
Weight of materials in 1000 g of PF/GP (30%)	646 (g)	54 (g)	700 (g)	300 (g)	1000 (g)			

 TABLE II

 Weight of Materials required to Make 1000 g of PF/GP (30%)

resins were 250 mm \times 10 mm \times 4 mm and tested at a crosshead speed of 1 mm/min.

The equation used for calculating the flexural stress,

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

The equation used for calculating the flexural strain,

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

The equation used for calculating the Young's modulus,

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

where $\sigma_{j:}$ stress in outer fibre at midpoint, MPa; $\epsilon_{j:}$ 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 delection 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 COMPOSITE SAMPLES

The reinforcer was glass powder, and they were made 5 to 30% by weight in step of 5% in the



Figure 1 Flexural strength of varying percentage by weight of glass powder reinforced phenolic resin. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

cured phenol formaldehyde composite, PF/GP (X %), where x is the percentage by weight of the filler; with 35% by weight of glass powder, the mixture became too viscous to be cast into moulds of specimens. As the raw materials of the composites are liquid and glass beads, the flexural test specimens were cast to shape. The resin is a dark brownish liquid and is first mixed with the dark brownish catalyst. After that the glass beads are added to the mixture and they are then mixed to give the uncured composite. Table II shows the mass in grams of resin, catalyst and glass powder required respectively to make 1000 g of uncured composite of 30% by weight of glass powder. The uncured composite was then cast into the moulds curing in ambient conditions. The number of samples used for each percentage by weight of glass powder will be six.

After initial 24-h curing, the test pieces were removed from the mould, they were postcured. This was achieved by baking the pieces in an oven. Oven temperatures and times were:

• 4 h at 50°C

• 2 h at 100°C

During the initial baking process of 4 h at 50° C, it was observed that a number of test pieces were developing a bow in middle. This bowing was between 1 and 4 mm in the middle of the piece and



Figure 2 Flexural strength of varying percentage by volume of lapinus reinforced cashew modified phenolic resin (adapted from Dong and Blum). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

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TABLE III Flexural Strength and its Standard Deviation of Varying Percentage by Weight of Glass Powder Reinforced Phenolic Formaldehyde Matrix Composite

		•	-				
Percentage by weight of glass powder	0	5	10	15	20	25	30
Flexural strength, MPa	24.26 (2.44) ^a	20.23 (2.20)	23.89 (3.75)	30.67 (3.94)	18.19 (2.37)	19.14 (5.44)	16.21 (3.60)

^a Standard deviation.

seemed to be exacerbated by the higher temperature baking processes. To counteract this, after they were removed from each baking 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 ~16 h as they cured overnight. The test pieces were then tested in the same manner as for hollow ceramics filled phenolic resin.⁸

RESULTS AND DISCUSSION

Figure 1 illustrates the flexural strength of varying percentage by weight of glass powder reinforced phenolic resin. The flexural strength of the neat resin was 24.26 MPa. At 15% by weight of the glass powder, the flexural strength is highest at 30. 67 MPa; at all other percentage by weight of glass powder, the flexural strengths were lower than that of neat resin. The bell shape of the curve suggests that the flexural strength increases steadily when the percentage by weight of glass powder ranges from 5 to 15% and then drops to a new low at 20% loading. The initial increase in the flexural strength with glass powder loading of 5 to 15% may be due to the better fusion of the glass powder with the resin matrix; with too much glass powder loading, i.e., over 15%, the better filler/resin fusion was disarrayed and the flexural strength dropped. It can be found that except at 10 to 15% by weight of glass powder, the higher the percentage by weight of the filler, the lower the flexural strength was. By having 15% of glass powder in the composite, the flexural strength was increased by 26%. Wang et al. found that the flexural strength of the neat resin was 71.3 MPa with a standard deviation of 13.5 MPa.⁹ It can be argued that the value obtained was not too reliable because of its high standard deviation. In this study, the flexural strength for the neat resin was 24.26 MPa and its standard deviation was 2.44 MPa.

Dong and Blum reinforced cashew modified phenolic resin by lapinus fibre and measured the flexural strength and modulus of the composites formed.¹⁰ The flexural strengths of the composites were illustrated in Figure 2. The flexural modulus of the neat resin was 90 MPa and it dropped to 76.8 MPa at 2% by volume of lapinus before rose up sharply to 101.6 MPa at 8% by volume of reinforcer. It then rose slowly to 102. 6 MPa at 13% by volume of filler. The absolute value of the flexural strength of composites studied by Dong and Blum were different from those of this study because they used cashew modified phenolic resin, (NC-126) with 5% (by weight) of hexamethylenetetramine (HMTA), USA, and lapinus mineral fibre RF 5164, a volcanic rock fibre of 125 µm in length.¹⁰ However, the tendency of the curve is similar to that of the composite in this study as depicted in Figure 1. In both cases, the flexural strengths were at their maximum when the percentage of the fillers were 15% by weight or 13% by volume respectively as illustrated in Figures 1 and 2. Dong and Blum did not measure the flexural strength beyond 13% by volume of lapinus but it can be argued that it will follow the trend of the curve of Figure 1.¹⁰

Wang et al. found that the flexural strength of the 20% glass powder filled resin was 90.5 MPa with a standard deviation of 10.8 MPa.9 This is better than the value obtained for the neat resin because of lower standard deviation. On the other hand, the flexural strength of the 20% glass powder filled resin in this project was 18.19 MPa with a standard deviation of 2.37 MPa. It was difficult to conclude who was correct because Wang et al. used ICI Fiberite resol-type CMXR-6055 phenolic formaldehyde resin; this research used Chemwatch Hexion Cellobond J2027L phenolic formaldehyde resin.9 On top of it, Wang et al. did not mention the way they cured the resin and its filler.⁹ The difference in flexural strength of the 20% glass powder reinforced phenolic formaldehyde composites for both studies is



Figure 3 Flexural strain of varying percentage by weight of glass powder reinforced phenolic resin. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Powder Reinforced Phenolic Formaldehyde Matrix Composite									
Percentage by weight of glass powder	0	5	10	15	20	25	30		
Flexural strain, mm/mm	0.025 (0.001) ^a	0.0096 (0.0023)	0.0084 (0.0017)	0.0074 (0.0012)	0.0102 (0.0022)	0.0126 (0.0018)	0.0074 (0.0014)		

TABLE IV Flexural Strain and its Standard Deviation of Varying Percentage by Weight of Glass Powder Reinforced Phenolic Formaldehyde Matrix Composite

^a Standard deviation.

400%, which is a significant difference. Wang et al. did not mention any information about the glass powder used.⁹ In this study, the diameters of the glass particles were from 6 to 32 μ m with an average size of 20 μ m. It can be argued that Wang et al. had used nano size glass powder.^{9,11,12} Redjel claimed that the flexural strength of neat phenolic resin was 68 MPa. The material he used was a pure phenolic resin 84,055 catalyzed by 3% of C 1650 and cured at 80°C for 8 days. It was produced and prepared by CDF-Chimie, France.¹³ The curing time was excessively long and would not be industrially viable and the energy consumption was enormous.

Table III shows the values of flexural strength mentioned above with their standard deviation. It can be found that the maximum flexural strength, 30.67 MPa, was obtained when the percentage by weight of filler is 15%. As the standard deviations flexural strengths obtained in this study were low, it can be argued that the values were valid for the resin used and the postcuring process used.

Figure 3 shows the flexural strain of varying percentage by weight of glass powder reinforced phenol formaldehyde matrix composite. The value (0.025 mm/mm) is highest with neat resin. At other percentages by weight of glass powder, the flexural strains varied from 0.007 mm/mm to 0.012 mm/mm; the change was not much. From Figure 3, it can be found that the flexural strain dropped dramatically from 0.0250 mm/mm for neat resin to 0.0096 mm/mm at 5 percent by weight of glass powder; it then dropped further to a low of 0.007 mm/mm at 15% by weight of filler; however, it then increased slightly again with higher percentage of reinforcer. The values found seem to be reasonable when they are compared with those of aluminum oxide (0.007 mm/mm) and glass (0.009 mm/mm). Redjel found the flexural strain of pure phenolic resin to be 0.0143 mm/mm.¹³ In this study, it was found that the flexural strain of neat phenolic resin was 0.0250 was much larger than that found by Redjel¹³; the value at 25% by weight of glass powder for this study was 0.0126, which was only 5% lower than that obtained by Redjel for neat resin.¹³ The reinforcement seemed to have no impact to the flexural strain of the composite.

Table IV illustrates the values of flexural strain mentioned earlier with their standard deviation. It

appears that except for the peak value, the flexural strain from 5 to 30% is around 0.010 mm/mm. It can be observed that at the maximum flexural strength of 30.67 MPa, i.e., 15% by weight of glass powder, the flexural strain was at the minimum. However, it can be argued by the same reasons for the flexural strength, their values were valid.

Figure 4 shows the flexural modulus of varying percentages by weight of glass powder reinforced phenol formaldehyde matrix composite. The flexural modulus increased from 1982 MPa (neat resin) to 3583 MPa (at 15% by weight of filler) and then dropped back to 1532 MPa (at 25% by weight of filler) and then increased again to 2215 MPa (at 30% by weight of filler). The curve of flexural modulus has the same bell shape of that of flexural strength. It can be argued that the increase in the flexural modulus with glass powder loading of 5% to 15% has the same reasoning as for the flexural strength.

With too much glass powder loading, i.e., over 15%, the better filler/resin fusion was disarrayed and the flexural modulus dropped. This phenomenon is uncommon because usually filled materials display a decrease of flexural strength and an increase of the flexural modulus when increasing the filler loading. The values found seemed to be a little bit low when they were compared with those of phenolic resins (2760 – 4830 MPa). Wang et al. found that the flexural modulus of neat resin was 2900 MPa and its standard deviation was 480 MPa⁹; they



Figure 4 Flexural modulus of varying percentage by weight of glass powder reinforced phenolic resin. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

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TABLE V Flexural Modulus and its Standard Deviation of Varying Percentage by Weight of Glass Powder Reinforced Phenolic Formaldehyde Matrix Composite

Percentage by weight of glass pourder	0	5	10	15	20	25	30
recentage by weight of glass powder	0	5	10	15	20	23	50
Flexural modulus, MPa	1982 (126) ^a	2167 (353)	2883 (534)	3583 (836)	1843 (427)	1532 (426)	2215 (348)

^a Standard deviation.

also found that the flexural modulus with 20% by weight of glass powder was 4,300 MPa and its standard deviation was 620 MPa. The flexural modulus of neat resin found by Redjel was 4401 MPa.¹³

Table V illustrates the values of flexural modulus mentioned above with their standard deviation. It can be found that the maximum flexural modulus, 3583 MPa, was obtained when the percentage by weight of glass powder was 15%. The maximum flexural strength of 30.67 MPa occurred at 15% by weight of filler. In addition the trend of the curves of Figures 1 and 3 was roughly the same.

One way to improve the flexural properties of particulate reinforced phenolic resin is to coupling agents as done by St John and Brown.¹⁴ On the other hand, using modified phenolic resin will also get the same result as carried out by Dong and Blum.¹⁰ By inspecting Figures 1, 3, and 4 carefully, one can obtain a set of compromised value for flexural strength (24.92 MPa), flexural strain (0.0087 mm/ mm), and flexural modulus (3135 MPa) at 17.5% by weight of glass powder. As the flexural strength of the composite with 17.5% by weight of filler was only 3% higher than that of neat resin, the 58% increase in flexural modulus was at the expense of the 65% decrease in flexural strain.

Figure 5 shows the bottom side of fractured flexural test specimen of 15% by weight of weight of glass powder reinforced phenolic composite, postcured conventionally, 400×. Hollow glass spheres of varying sizes of 5 to 30 µm could be viewed clearly. The sizes of the porosity were very small, ranging from 5 to 10 µm. Figure 6 illustrates the fractured flexural test specimen of neat phenolic resin postcured in an oven, $400\times$. The sizes of the porosity were bigger than those of the composite shown in Figure 5; their size varies from 20 to 30 μ m. It can be argued that the presence of hollow glass powder spheres had reduced the size of the porosity.¹⁵ Figure 7 shows the bottom side of fractured flexural test specimen of 15% by weight of glass powder reinforced phenolic composite, postcured conventionally, 1500×. Even at this magnification, the sizes of the porosities were still smaller than those found in neat resin in Figure 6. Fractures hollow glass spheres were also depicted in Figure 7. Figure 8 illustrates the bottom side of fractured flexural test specimen of 15% by weight of glass powder reinforced phenolic composite, post-cured conventionally, 6000×. It



Figure 5 Bottom side of fractured flexural test specimen of 15% by weight of glass powder reinforced phenolic composite, post-cured conventionally, \times 400. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



Figure 6 Fractured flexural test specimen of neat phenolic resin post-cured by microwave irradiation, \times 400. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



Figure 7 Bottom side of fractured flexural test specimen of 15% by weight of glass powder reinforced phenolic composite, post-cured conventionally, \times 1500. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

can be observed that the sizes of the porosities were much smaller than the larger hollow glass sphere and this was due to the presence of the glass powder.

Figure 9 shows the top side of fractured flexural test specimen of 15% by weight of glass powder reinforced phenolic composite, postcured convention-



Figure 9 Top side of fractured flexural test specimen of 15% by weight of glass powder reinforced phenolic composite, post-cured conventionally, $\times 400$.

ally, $400\times$; it can be found that the sizes of the porosities and glass hollow spheres were almost the same to those at the bottom side of the fractured sample (Fig. 5). Figure 10 depicts the top side of fractured flexural test specimen of 15% by weight of glass powder reinforced phenolic composite, post-cured conventionally, $1500\times$; holes due to air bubbles could be clearly viewed; partially fractured hollow glass sphere could also be found.



Figure 8 Bottom side of fractured flexural test specimen of 15% by weight of glass powder reinforced phenolic composite, post-cured conventionally, $\times 6000$. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]



Figure 10 Top side of fractured flexural test specimen of 15% by weight of glass powder reinforced phenolic composite, post-cured conventionally, \times 1500. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

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CONCLUSIONS

This study has evaluated the flexural strength, flexural strain, and flexural modulus of varying percentage by weight of glass powder reinforced phenolic resin; in all cases, the fluidity of the slurry composite was good and could be cast easily into moulds. The optimum percentage by weight of glass powder was 17.5% for compromised flexural properties of the composite. The value with no filler had also been compared with those found by other studies but they did not agree with each other. However, it is difficult to argue that which is better than the other because much experimental information employed by other researchers were not available. The values of this study were generally lower but they were reliable because their standard deviations of the properties obtained were low. It can be argued that when the fusion between phenolic resin (matrix) and glass (reinforcer) is improved by adding some other fillers and resins to the composite, its flexural properties will be improved.

The images produced by scanning electron microscopy (SEM) showed that the porosities of glass powder reinforced phenolic composites were much smaller than those found in the SLG reinforced phenolic composites.⁸ It can therefore be argued that glass powder spheres were better fillers for phenolic resins.¹⁶

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