University of Southern Queensland

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Best Percentage Weight of Microspheres as Fillers in Resin Using Three-Point Bending Test

A dissertation submitted by

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Abstract

This project sought to find the best percentage by weight of microspheres as fillers in phenolic resin using a three-point bending test to determine material properties.

With cost becoming a progressively important issue in the construction and engineering industry, finding and employing methods to reduce these costs are becoming increasingly more important. One cost reduction method that has been implemented is the introduction of microspheres as fillers when using phenolic resins for construction. These fillers can be used to reduce weight, while also reducing the cost of the final construction. The fillers are cheaper than the resin that they are 'replacing', and therefore cost reduction is seen as an immediate result.

The objectives of this research were as follows:

- 1. Research the use of fillers in plastics
- 2. Create moulds of test components and pour resin
- 3. Test components using 3-Point bending test and ascertain best percentage weight of filler
- 4. Ascertain relationships between strength and percentage weight of filler
- 5. Analyse the fracture surfaces of the test specimens (if time permits)

Versatile material properties provide thermosets with the ability to be used in many different applications. Phenolics can also be purchased in flake, powder and liquid form. According to CFAP (2003) the different forms that Phenolics may be purchased in facilitate a wide range of manufacturing scenarios, such as:

- Combining Phenolics with reinforcement as composite materials
- Electrical components and assemblies
- Heat shielding, such as exhaust shielding in cars
- Household applications such as power tool casings or hot water jugs.

The everyday use of Phenol formaldehyde resins is widespread and varied, meaning that any reduction in their costs will be very effective and felt in a wide number of industry areas. Testing was conducted using the three point bending test as specified by ISO14125: Fibre reinforced plastic composites – Determination of flexural properties.

It was found that the mixture of 5% slg-filler by weight in phenol formaldehyde resin was the strongest, though there was a rise in strength at the 25% slg-filler by weight. This percentage was suitable as a final result as it still provided reasonable flexural strength and modulus of elasticity while the amount of filler will still provide a decent reduction in cost.

This research provides the basis for future study into the strengths and effects of fillers on phenol formaldehyde resins.

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Date

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Abbreviations and Symbols

PF = Phenol formaldehyde

Phenolics = The broad term given to phenol formaldehyde resins

 σ_f = Flexural Stress (MPa)

 $\varepsilon_f =$ Strain in the outer surface

 $E_f =$ Modulus of Elasticity in bending (MPa)

P = Load at failure (Newtons)

L = Distance between supports (for this research = 64mm)

b = Width of test specimen

h = Depth of test specimen

D = maximum vertical deflection in the centre of the beam (mm)

slg = Micro-sphere filler used in this study

Filler = A material mixed into a resin to change material properties or lower cost

Chapter 1 – Introduction

1.1 Outline of study

This project seeks to find the best percentage by weight of microspheres as fillers in phenol formaldehyde resin using a three-point bending test to determine material properties.

1.2 Introduction

This chapter provides an outline of the reasoning behind this dissertation, the objectives involved in completing the research and the research conducted in determining the best percentage weight of slg microspheres as fillers in phenol formaldehyde (phenolic) composite resin.

1.3 The problem

The overall aim of this research project was to find the best percentage, by weight, of microspheres as fillers in resin. This was accomplished by using a three-point bending test to determine the material properties and therefore, physical effects.

With cost becoming an increasingly important issue in the construction and engineering industry, finding and employing methods to reduce these costs is becoming ever more important. One cost reduction method that has been implemented is the introduction of microspheres as fillers when using resin for construction. These fillers can be used to reduce weight, while also reducing the cost of the final construction. The fillers are cheaper than the resin that they are 'replacing', and therefore cost reduction is seen as an immediate result.

The introduction of these fillers also has an effect on the strength of the resin. Presently, information is not available regarding the effects that microspheres have on the resin properties. The scope of this project not only includes finding the best percentage by weight of microspheres as fillers in resin, but also to provide reliable and accurate information in terms of the effects these microspheres have on the strength of resin.

1.4 Research objectives

The following research objectives intend to fulfil the purpose of this research project, this being finding the best percentage, by weight, of microspheres as fillers in resin.

- 1. Gather background information for three point bending tests and standard testing procedures
- 2. Gather information on phenolic resins, their properties and uses
- 3. Ascertain final test specimen dimensions
- 4. Design mould for test specimens
- 5. Use the constructed mould to create samples and then test these samples using a three point bending test and record the data
- 6. Measure the viscosity of the phenol formaldehyde and slg filler mixture
- 7. Analyse collected data and calculate stress, strain and modulus of elasticity of the material. From these results the best percentage weight of the filler will be derived.

1.5 Conclusions – Chapter 1

This chapter has provided the background and research objectives involved in calculating the best percentage weight of slg microspheres as fillers in phenolic resins. The following chapter provides a literature review that addresses existing research and past studies into phenolic resin, its uses and the use of slg fillers in phenolic resins.

Chapter 2 – Literature Review

2.1 Introduction

The literature review has been divided into areas covering phenol formaldehyde resins, their material properties and applications, fillers and their uses, the three point bending test, beam loading equations safety and the consequences, and effects of this research.

2.2 Phenol Formaldehyde (Phenolic) resin

First synthesized by Leo Bakeland in 1907, phenol – formaldehyde resins (otherwise called Phenolic resins) were the first thermosetting plastics and are considered to be the first commercially available plastic resins. They are also the second most widely used thermosetting plastic on the market today (Goodman 1998).

Phenolic resins are made of a variety of elements. The three major components of phenolic resins are (1) Phenol (C_6H_5OH), which is created by the fractional distillation of tar, (2) Formaldehyde (CH_2O) and (3) Hexamethylene Tetramine ($[CH_2]_6N_4$) sometimes called hexa. Hexa is the curing agent required to harden the resin. The formation of resin is viewed as an extremely complex process.

Resin is formed by the chemical reaction between the phenols and the formaldehyde solution (Goodman 1998), which simultaneously forms cross-links between the molecules in the plastic and polymer linkages (Strong 2006). Other phenols, such as Cresols, Xylenols and Alkylated-phenols can be used as an alternative to the basic phenol. This method of substitution however is very limited due to the more widespread availability of the cheaper synthetic phenols as stated above (cost is also more prohibitive).

Depending upon the amounts of resin and filler used, this mixture gives it a paleamber to dark brown colour that appears as opaque.

2.3 Material properties of phenolics

Due to the cross-linking between molecules in Phenolic resin, they are inherently very stiff. As the amount of cross-linking increases, so does the stiffness of the resin. This stiffness leads to a very brittle material compared to thermoplastics. According to Strong (2006) this brittle nature has a number of advantages associated with it, such as very high creep resistance, high deformation resistance, and good dimensional accuracy.

In addition to these above mentioned direct material properties, thermosets are also associated with good heat resistance and can be easily formed into very complex and intricate shapes (CFAP 2003) These shapes then have little to no surface finishing required after extraction from the mould (CFAP 2003). Due to these material properties phenolic thermosets are one of the most common plastics used all over the world.

2.4 Use of phenolic thermosets

The previous mentioned material properties provide thermosets with the ability to be used in many different applications. Phenolics can also be purchased in flake, powder and liquid form (CFAP 2003). According to CFAP (2003), the different forms that Phenolics may be purchased in facilitate a wide range of manufacturing scenarios, such as:

- Combining phenolics with reinforcement as composite materials
- Electrical components and assemblies
- Heat shielding, such as exhaust shielding in cars
- Household applications, such as power tool casings or hot water jugs.

Although phenolic thermosets have many uses, as previously mentioned, as an engineering strength-based material they are inherently stiff and brittle, which limits their use in this regard. The brittleness and stiffness of phenolic thermosets can be greatly improved with the addition of reinforcement to the mixture, such as fibres or fillers (Goodman 1998).

2.5 Resin utilized in this research

The resin used in this research was phenol formaldehyde resin solution J-2027L, produced by Hexion Specialty Chemicals Pty Ltd. The name given to it by Hexion is Hexion Cellobond J2027L (Chemwatch 2005). The same company, Hexion Specialty Chemicals; produced the catalyst used in this research to cross-link the resin its official name being Hexion Phencat 15 (Chemwatch 2005).

Figure 1: First stage of formation of Phenol formaldehyde



Figure 2: Second stage of formation of phenol formaldehyde



Phenolformaldehyde

From Figure 1, there are five hydrogen atoms in the benzene ring however due to the limited space there are only three sites for reaction. In this case, the phenolic molecule

is said to have a functionality of three and this is shown in Figure 2. As the functionality of the phenolic molecules is more than two, the molecules can react to form a three-dimensional network polymer with the formaldehyde molecules (Sheckelford 1992).

2.6 Fillers

Fillers are used in composites to change material properties and add favourable characteristics to the material, such as a higher impact strength or lower cost. The type of filler used depends upon the application and what the material has to achieve (Strong 2006). More common fillers and their general material changes are outlined by Goodman (1998) and include:

- Wood flour: Improves tensile strength
- Silica: Abrasive qualities, improves insulation
- Mineral Fillers: Insulation & dimensional stability
- Kevlar & Nomex: Improved tensile strength and penetration avoidance
- Cellulose: Improved impact strength
- Glass & Graphite fibres
- Paper
- Fabrics.

The above mentioned fillers can be combined for multiple property enhancements. A key example of this would be combining carbon fibres and Kevlar to gain a very lightweight, strong, composite material with multiple uses.

2.7 Filler utilized in this research

This research project focused on the use of the filler Envirospheres (E-Spheres) slg and its subsequent impact on strength. E-Spheres are microscopic, hollow, ceramic particles that have a wide range of uses. Particle sizes range from 20-300µm with an approximate mean of 130µm for this general purpose E-Sphere while their relative density is approximately 0.7.

Envirospheres are made up of Alumina, Al_2O_3 (35%-45%), Silica, SiO₂ (50%-60%), Iron Oxide, Fe₂O₃ (0.4%-0.5%) and Titanium Dioxide, TiO2 (1.5%-1.6%). Key advantages of using E-Spheres include good compressive strength, heat resistance and a clean appearance (white in colour). These aspects made E-Spheres suitable for use as the filler for this study as they are widely used and have good material properties while also being inert.

Material properties and changes resulting from this filler have been ascertained using the three-point bending test conducted in accordance with International Standard ISO14125; Fibre-reinforced plastic composites – Determination of flexural properties. (See Section 3.10.2 for information on conducting the three point bending test)

2.8 Viscosity

According to Fox, McDonald & Pritchard (2003), when a solid is deformed, stresses arise due to the object being deformed or put under strain. Similarly in a liquid, shear stresses arise due to the solid being viscous (able to flow). Therefore, the viscosity of a fluid is a measure of the resistance of the fluid to deform when subject to a shear stress (Fox 2003).

As an example, water has a very low viscosity (and therefore low resistance to shear) and flows very easily. Some oils have a high viscosity and will therefore take more force to flow. The viscosity of a liquid, or pertaining to this research, a resin, can have an effect on moulding procedures. If a liquid has a low viscosity, then it will be able to be poured easily. On the other hand, if the viscosity is high then problems can arise with the fluid not permeating to the edges of the mould. In these cases other moulding methods may have to be utilized, such as the use of positive pressure.

2.9 Three-point bending test

The three-point (3-point) bending test is a test that is useful for finding the flexural properties of a material, such as un/reinforced plastic composites. The results of such tests are useful for quality control purposes and specification analysis. The test is carried out on a simple bar-shaped element, though sometimes the test piece can be notched (see Figure 3).

This research has been carried out as per International Standard ISO14125: Fibre reinforced plastic composites – Determination of flexural properties, details of which are set out by the International Organization for Standardization. (ISO14125 1998). Please note that this standard is an expansion on ISO178.

The test was conducted on a beam-type test specimen, supported at both ends (at specified points) and was deflected up to a pre-determined point, either complete fracture or some value of interest (see Figure 4). Deflection was carried out at a constant rate and was transferred to the beam midway along its length for the 3-point test. This test has been designed to determine flexural stress-strain information and properties of the test specimen material.

Figure 3: Dimensions of test piece



2.10 Test Machine

The requirements of the test machine were that it had to be able to maintain the speed of testing as the load increases (ISO14125 1998). Tolerances of $\pm 5\%$ for the loading rate were set out in ISO14125 as were the full dimensions for the support points and loading point for the beam. See Appendix C for dimensions of supports.



Figure 4: Three-Point bending test machine

2.11 Specimen/Beam loading calculations

During the test, the upper support contacted the top surface of the test specimen, applying a load, which forced the piece to bend. This force was increased until the test specimen failed, at which point the maximum load (Peak Load (N)) that the specimen supported was recorded and the deflection at the mid point was noted.

From this peak load, the flexural strength (or flexural stress) (see ISO14125) of the material can be found using the following equation (1) and was measured in mega Pascals (Mpa). From this, calculations of Flexural strain and then Young's modulus of Elasticity in Bending (E) were conducted. Please note it is possible to use other values of load at different points of deflection to find the stress at a given point, though this research only dealt with load at failure.

(1) Flexural Stress (MPa)

$$\sigma_f = (3FL) / (2bh^2)$$

(2) Flexural Strain

$$\epsilon_{f} = (6Dh) / (L^{2})$$

(3) Young's Modulus, E (Pa)

$$E = (L^3m) / (4bh^3) = \sigma_f / \varepsilon_f$$

From these values of peak load and flexural stress sustained, the best percentage weight of filler in the resin was ascertained.

2.11.1 Stress (σ)

Stress is a method of defining the load on a certain object and is expressed in Pascals (Pa) (Beer, Russell Johnston & DeWolf 2002). Simple compression is defined as being equal to the force or load, divided by the area on which that load is applied (Beer, Russell Johnston & DeWolf 2002). In SI units the force is expressed in Newtons (N) and the area is expressed in square metres (m²). This gives the following basic equation:

$$\sigma = F/A$$

2.11.2 Strain (ϵ)

Strain is defined as the deformation of the member per unit length (Beer, Russell Johnston & DeWolf 2002). It is also a dimensionless value due to being a percentage. In the case of a tensile load, and in light of the above statement, the equation becomes:

$$\epsilon = \delta / L$$

Where δ (delta) is the change in length and L is the original length (Beer, Russell Johnston & DeWolf 2002). The relationship that strain provides is a percentage change that can be compared with stress to give a value of **E**, or Young's modulus of elasticity.

2.11.3 Modulus of Elasticity (E)

$$E = \sigma / \epsilon$$

The above equation shows the relationship between stress and strain and how it relates to the modulus of elasticity (Beer, Russell Johnston & DeWolf 2002). As the stress is given in Pascals, the value of \mathbf{E} is also in Pascals.

The modulus of elasticity can be used to ascertain not only how strong a material is, but when compared to stress and strain can show what sort of behaviour the material will exhibit when a load is applied. Please note that for this research the specimens were loaded to failure and therefore the fracture stress of the resin was examined, though this method can be used in other cases not leading to failure.

2.12 Consequences and effects

2.12.1 Safety

Safety considerations when carrying out this research required considering the safe use of the resin and hardener mixture when pouring into moulds for the test specimens. Safety precautions such as gloves, air filtration mask and appropriate eye shielding were enforced whenever handling the resin mixture.

In the case of ingestion of the resin mixture in any way, first aid measures (as per Chemwatch information dealing with appropriate material) were to be immediately put into practice and medical assistance sought. Chemwatch information also contains notes to physicians.

Disposing of the resin mixture and test pieces was conducted at the University of Southern Queensland's appropriate facility.

In the case of a major spillage, guidelines set out by Chemwatch in the appropriate resin/hardener safety information were followed. These guidelines cover areas such as isolation distance, downwind protection distance and evacuation directions and were followed accurately. Minor spills were cleaned up immediately.

2.12.2 Environmental considerations

The effects of this research in the future involve phenolic thermosets being used more often as engineering load-bearing materials. Currently, thermoset plastics are the second most widely used plastic on the market so they are very widespread and in use all over the world. Therefore, the possibility that this use will increase dramatically is unlikely as there is very little room for expansion on the market.

If in the future an increase in their use does occur, it may be found that the increase in production levels of the plastic has an effect on the environment. Close monitoring will need to be undertaken upon the use of thermosets, though once again, the chances of this increase are quite slim due to their present widespread use.

2.13 Conclusions – Chapter 2

This chapter has provided a literary review of modern phenolics and their applications, the use of fillers in light of this research, the three point bending test and relevant equations, and the implications and consequences of this research. The following chapter will provide the methodology that this research has followed in order to obtain relevant results for further study.

Chapter 3 – Research design and methodology

3.1 Introduction

The methodology for this research has been divided into sections relating to the mould and test specimen, the three point bending test and the collection of data. Each section has been discussed in detail and covers mould design and its manufacture and relevant detail, the test specimen, curing and preparation for testing and the testing procedure used to obtain relevant, useful results.

3.2 Mould design

Two major options were available when deciding what type of mould system to use when casting the test pieces.

The first was a mould that could be disassembled fully to enable easy removing of the resin test piece. This consisted of multiple pieces of 6mm plastic sheeting bolted together in such a way as to leave a space in the correct shape of the test piece. After disassembling the mould, the test pieces would be left free with no manual removal required after this.

This option was not used in this research, due to the larger amount of components in the mould and the fact that the mould needed to be reconstructed every time that a new test piece was moulded. Having to rebuild the mould so many times may have lead to inaccuracies in dimensions and deterioration of the surface finish.

The second option consisted of three sheets of 6mm plastic sheet bolted together on top of each other with the middle sheet containing cut outs of the test pieces (see Figure 5). After the bolts were removed the mould could be split into the three parts, with the middle containing the cast resin pieces. These would then have to be removed manually from the sheet. This method yields a higher dimensional accuracy and surface finish while retaining very few pieces and ease of use. For dimensions of this mould please refer to Appendix A: Mould dimensions.

Figure 5: Mould



3.3 Mould Fastening

The mould was fastened together with $10 \times M5$ bolts with wing type nuts to allow quick fastening without the need for tools. These were spaced evenly around the surface of the plastic, with three bolts on the top, four in the middle and three along the bottom (See Figure 6). Refer to Figure 31 of Appendix A for detail of moulds.





3.4 Test pieces and porosity

Porosity is a phenomenon that occurs when pouring and drying resin pieces. As the hardener combines with the resin, the chemical reaction that takes place produces gas, which is usually forced to the surface of the piece and appears as bubbles and gas just under the surface and as indentations on the surface.

The amount of porosity generally depends upon the type of resin and the amount of hardener used, though it can also depend upon the temperature at drying.

The design of the mould was required to take the possible creation of porosity into account. Therefore the use of 6mm sheet plastic added 2mm to the top of the required 4mm test piece to allow for the removal of material containing porosity on the upper surface of the test piece.

3.5 Manufacture of mould

The mould was manufactured from three sheets of 6mm PVC plastic (see Figure 7 and Figure 8). The top and bottom sheets are identical, while the middle sheet had the cut outs of the components required. The University of Southern Queensland Engineering Workshops machined all parts of the mould.



Figure 7: Manufactured mould

Figure 8: Mould bolted together



3.6 Mould preparation

No cleaning or preparation was required to make the mould ready for use, as the finish on the mould was adequate after receipt from the USQ Engineering Workshop. The only change that was required was to the centreline of boltholes. On the top sheet, the outer two holes had to be increased in size due to permanent fastening of the bottom two sheets together using an M5 nut (see Figure 9). When these were bolted together prior to pouring, a larger hole had to be allowed so that the upper sheet mated to the middle without these nuts holding it clear of the surface.



Figure 9: Permanent fastening with M5 nut

After the mould was used, it was cleaned and prepared for the next batch of parts. This involved cleaning any excess resin off surfaces and wiping them with a thin layer of oil (cooking oil in a spray can was appropriate) to facilitate removal of the resin pieces.

3.7 Preparation of mould

Before the resin can be poured, the mould was cleaned and checked for traces of previous resin mixture or dirt. The presence of any surface contaminants or old resin disrupted the strength characteristics if dried into the test piece as this would introduce sections into the piece that had different flexural strengths, leading to either less space for the material to fail or areas of stronger or weaker material.

After being cleaned, the mould was sprayed with cooking oil (aerosol type spray can). After coating all surfaces of the mould, most excess oil was wiped away with a piece of absorbent paper towel to reduce the amount that could interfere or mix with the resin.

Oil was applied to reduce the surface friction when removing the test pieces from the mould. This made pieces easier to remove while avoiding breaking them in the process.

3.8 Manufacturing of test pieces

3.8.1 Mixing of resin

Cost plays a significant role in decision making today, as there are an increasing number of different research areas becoming available for institutions to support. The cheaper one area of research is the more funds can be placed in other areas of study. Therefore, as the resin Hexion Cellobond J2027L and catalyst Hexion Phencat 15 are the most expensive part of this research study, the need to reduce its cost is high and any gains would be desirable.

With this factor in mind, the amount of resin by weight that would be made was decided to be 250g. This allowed ample resin to fill each of the six gaps in the mould while not having much left over.

The resin Hexion Cellobond J2027L and the hardener/catalyst Hexion Phencat are to be mixed in a ratio of 20:1 by weight respectively. Therefore, using the mass of the constituent parts, the following table was obtained for values of 5, 10, 15, 20, 25 and 30 percent (see Table 1).

250g	MIXTURE			
%	Resin (g)	Hardener (mL)	Filler (g)	Weight (g)
5	226.19	10.71	12.5	250
10	214.29	10.15	25	250
15	202.38	9.58	37.5	250
20	190.48	9.02	50	250
25	178.57	8.46	62.5	250
30	166.67	7.89	75	250

Table 1: Resin mixture

This weight provided sufficient amounts of mixture for the 6 spaces in the mould while minimizing waste. The resin was first measured into a container, then the filler measured out and added to this. This was then mixed thoroughly making sure that all the filler was added. Next, the hardener was measured using a syringe and added to the resin/filler mixture. The hardener was stirred through quickly, to allow ample time for pouring without the mixture hardening/curing. See Figure 10 for an example of how the resin was mixed for this project. As the mixture mixed through, it was seen that it would turn to a pale cream colour.

Figure 10: Mixing of resin



3.8.2 Pouring

This mixture was poured into each space in the mould using a small plastic spoon. Slight excess was allowed in each space to minimize the formation of air bubbles while the resin cured (see Figure 11).



Figure 11: Pouring of resin

3.8.3 Tightening of fasteners

Due to the possibility of air becoming entrapped between the middle and top layer of the mould, as the mould was being sealed a sequence was developed to tighten the M5 bolts and wing nuts to force as much air as possible from between the layers of the mould as the bolts were being tightened.

Just after pouring the resin, the top layer of the mould was placed over the bottom two layers and was located in the correct position using the two pre-fastened bolts and M5 hex nuts. Next, the other bolts were put in place and the wing nuts were attached and tightened up until they were just above the surface of the mould, making sure that none were tight enough to apply pressure. The nuts were then tightened in the order shown in Figure 12.



Figure 12: Procedure for tightening bolts

The procedure depicted in Figure 12 was used to move as much air from underneath the top of the mould and reduce the amount of air trapped and then cast into the resin. The reasoning behind this was that tightening in this way would cause a "pressure wave" to move through corresponding to the tightened bolts.

This is shown in Figure 13 with air moving from the top right to the bottom left hand side as the bolts are tightened in the aforementioned way. Tightening in this way also

served to spread the excess resin smoothly over the surface of the test pieces, leading to a consistent finish.



Figure 13: Movement of air underneath mould

3.8.4 Removal of specimens from mould

Allowing a minimum time of 24 hours for preliminary curing immediately after pouring the resin, the six test specimens could be removed from the mould ready for curing in the oven. After this initial cure, the specimens were very tight and therefore had to be removed carefully to avoid breaking too many of them. A number of methods were tried before settling upon the final method of removal.

The removal method involved using the handle of a screwdriver lying across the face of the test specimen (to distribute the force uniformly over the length of the specimen) and while applying slight pressure, a short, sharp hit was applied to the handle which dislodged the specimen while stopping some of the specimen edges grabbing on the inner mould surface and cracking the resin. This allowed for the greatest number of test pieces available for testing, therefore increasing the overall accuracy and consistency of the results.

3.9 Test pieces

3.9.1 Size and Dimensions of test pieces

The International Standard ISO14125 gives the dimensions (80mm x 10mm x 4mm) of the test pieces. Creating the test specimens using the ISO14125 standard yielded results that could be used and compared with other future studies into fillers and their effects on strength in phenolic resins.

3.9.2 Curing of test pieces

Initial Curing

Initial curing was the minimum time allowed before the test specimens were removed from the mould. 24 hours was given to ensure that the specimens would be fully dried. This ensured that no specimens were wet, therefore reducing waste.

Baking of test pieces

After initial curing when the test pieces were removed from the mould, they needed to be post cured. This was achieved by baking the pieces in an oven.

Oven temperatures and times were as follows:

- 4 hours at 50°C
- 4 hours at 80°C
- 2 hours at 100°C

During the initial baking process of 4 hours at 50°C, it was observed that a number of test pieces were developing a bow in middle. This bowing was between 1mm and 4mm in the middle of the piece and seemed to be exacerbated by the higher temperature baking processes. It was also noted that bowing was all in the same orientation; bowed around the "upper" (in relation to moulding) face of the test piece.

To counteract this, after the test pieces were removed from each baking session, all pieces were subject to an approximate 2kg load while between two pieces of toughened glass. The time for this weighting was approximately 16 hours as they cured overnight.

The reason for this bowing is not immediately apparent. A number of possibilities are feasible though this is more suited to further study. Some supposed possibilities are as follows:

- When the test pieces were oven cured, the resin expanded. With porosity in the top surface, there was less material to support this expansion, therefore the top face contracted, pulling the ends of the pieces up.
- An internal stress was developed while drying in the mould.
- During removal from the mould, the upper surface of the test specimen became flawed.

3.9.3 Removal of porosity

When the test pieces were removed from the mould, they were 6mm in height. As the required height (by ISO14125) is 4mm, 2mm needed to be taken off the top of the piece (see Figure 14). The extra height was allowed for porosity forming within the upper surface of the resin during the initial drying process. Porosity is the formation of air bubbles during the curing process.

The extra 2mm and included porosity was removed using a belt sander attachment to a bench grinder head. Using the belt sander allowed for easy consistent removal of the top 2mm of the material without the need for machining of any further preparation and gave good dimensional accuracy for the finished test piece.

All measurements were carried out using Kincrome Digital Vernier Calipers. These callipers had an accuracy of 2 decimal places, sufficient for this research.
Figure 14: Removal of porosity



3.10 Three-point bending test machine

The three-point bending test is a test used to measure the flexural strength of a material or component. It uses a vertical force applied to a long thin member to force the member to deflect and then finally fail, at which point the peak load sustained by the member is recorded and from this, material strength can be ascertained.

It is suitable not only as a method of testing material qualities and properties but also as a quality control method. Figure 15 depicts the machine used to conduct the threepoint bending test used in this research.

Figure 15: Three-point bending test machine



The three-point bending test machine used was a hydraulic type with vertical rams. For this particular (three-point) test, the bottom ram was actuated and allowed to move vertically, moving the test piece supported on the lower two points to contact the upper point, therefore acting on the centre of the specimen (See Figure 18).

Using this machine allowed for the measurement of certain parameters while the test was taking place. For this research, the load and deflection were measured throughout the full extent of the test, allowing the values of flexural strength/stress and strain to be calculated.

The upper and lower supports were held in place by the jaws of the rams (MTS 647 Hydraulic Wedge Grip) (see Figure 18 and Figure 20). These were a sliding chuck type that could be used to grip cylinders of different sizes.

Figure 16: MTS 647 Hydraulic Wedge Grip



Figure 17: Detail of hydraulic jaws



3.10.1 Test rig supports

The dimensions for the supports of the three point bending test were given in ISO14125. In addition to these dimensions, the ISO14125 standard specified loading rates, which were to be 2mm/min with an accuracy of 2% (see Appendix C, Figure 34 for dimensions and detail). These supports were already constructed, and only minor adjustments had to be made to dimensions.

The lower support was made from mild steel consisting of two uprights (which held the diameter 10mm steel rod) welded to a base of 10mm mild steel. A pin made of 100mm rod was inserted into a corresponding central hole on the base of the steel to locate the test supports. The hydraulic jaws held this pin (see Figure 18).



Figure 18: Upper and lower supports of three-point testing machine

The upper support was also constructed of a mild steel base block and was held in place by a threaded rod welded to this. The lower 10mm diameter rod was welded onto the base block (see Figure 22 and Figure 20). The hydraulic chuck of the test machine gripped onto this 10mm rod.



Figure 19: Upper point of the three-point testing machine

3.10.2 Conducting the Three-Point bending test

The procedure for conducting this test is defined in ISO14125: 1998 Fibre-reinforced plastic composites – Determination of flexural properties. Firstly, test specimens were measured and ensured that they complied within the required dimensional accuracy. This was done immediately after specimen preparation with vernier calipers.

The test system (see Figure 16) was then set up for the appropriate loading rate of 2mm per minute on the control-center computer attached. Specimens were then placed central on the supporting cylinders (see Appendix C, Figure 34 and Figure 16 for more information) and any details noticed regarding the test specimens was noted. The test was then commenced. At this time the computer attached to the test system recorded real time data about load, deflection and time. Figure 18 and Figure 20 illustrate the load applied to a test specimen and the resulting flexure in the center of the specimen.

Figure 20: Testing in progress



The data recording was stopped approximately 4 seconds after the specimens broke and a print out was acquired detailing peak load, deflection, deflection at failure, time and a graphical readout of these results (see Appendix E for examples of results from testing).

3.10.3 Data collection

After testing was completed, all data was gathered together and reviewed. In this review process, any test pieces that did not produce reasonable results (for example, did not hold any load or failed under very low loads) were discarded. This left 6 specimens per percentage weight data set for analysis.

Mean failure loads and deflections at failure were calculated from this data (see Appendix D, Table 2 for failure loads from the refined data set) and from this, values of flexural stress/strength, strain and Young's modulus of Elasticity were obtained for each percentage of slg-filler. Chapter 4 and Chapter 5 both contain records and discussion of results. Appendix D describes the refined data and further calculated values and Appendix F depicts sample calculations.

3.11 Viscosity of Resin

The viscosity of the Phenol formaldehyde resin mixture must be measured to ensure that the viscosity is low enough at different percentage levels of slg-filler to still allow pouring into a mould.

Viscosity was measured using the Brookfield RDVD-II+ viscosity-testing machine at the Centre for Excellence in Engineered Fibre Composites (CEEFC) at the University of Southern Queensland. This testing system is shown in Figure 21.

Testing was conducted at room temperature on each percentage mix (0%, 5%, 10%, 15%, 20%, 25%, 30%) of phenol formaldehyde resin and slg-filler mixture. Please note that for this viscosity testing, the catalyst Hexion Phencat 15 was not introduced, therefore yielding results for the pure resin and slg mixture, rather than the cured mixture.



Figure 21: Brookfield RDVD-II+ viscosity testing machine

Throughout the testing two different spindles were used to measure the viscosity. Number 6 was used for the first two percentages of 0% and 5% filler. Number 7 spindle was then used for testing of 10% to 30% mixtures. Figure 22 shows an example of some spindles used in this particular machine. Please note that the smaller the disc on the spindle, the larger the Spindle Number.





3.12 Conclusions – Chapter 3

This chapter has provided the methodology used in this study in accordance with the requirements of ISO14125. It has outlined the manufacture of moulds, test specimens, testing and data acquisition. The next chapter will outline the results recorded from the three point bending test.

Chapter 4 – Test Results

4.1 Introduction

This chapter provides the results obtained from the three-point bending test outlined in chapter 3. It gives full explanations of peak load, material flexural stress/strength, strain and the modulus of elasticity for each percentage of slg-filled phenol formaldehyde composite mixture. The final section gives results for viscosity testing of percentage values of 0%, 5%, 10%, 15%, 20%, 25% and 30% filler by weight.

Please refer to Appendix D for the tables of results and data obtained during testing.

4.2 Peak Load

The following graph (Figure 23) shows the means of the peak loads supported (at material failure) by the varying mixes of slg-filled phenol formaldehyde resins against the corresponding percentage of filler in the resin, by weight. This graph was calculated from results given in Table 3 of Appendix D, while Table 3 was calculated from the mean values of loads from Table 2 of Appendix D, which depicted peak load results by percentage of filler.





It can be seen directly from Figure 23 that the specimens containing 5% of slg-filler were the strongest, sustaining approximately 74N (Newtons) of load. The specimens containing 25% of filler by weight were the next strongest, with a peak load of 45N, followed by 30% with a peak load of 37N, 10% with a peak load of 32N, 15% with a peak load of 31N and lastly 20% sustaining a peak load of 28N.

4.3 Flexural stress/strength

The following graph Figure 24, shows the mean flexural strength of each percentage, plotted against the percentage of filler by weight. The flexural strength is given in megapascals (MPa). These results can be seen in Table 3 of Appendix D.





Figure 24 represents the direct strengths of the different percentages of resin. It is essentially a representation of the previous graph Figure 23, expressed in megapascals.

It can be seen that the specimens containing 5% of slg-filler were the strongest, with a flexural strength of 44MPa. The specimens containing 25% of filler by weight were the next strongest, with a flexural strength of 27MPa, followed by 30% with a strength of 22MPa, 10% with a strength of 19MPa, 15% with a strength of 18MPa and lastly 20% with the lowest flexural strength of 17MPa.

4.4 Flexural strain

Figure 25 depicts results of the flexural strain, which were plotted from values in Table 4 from Appendix D.



Figure 25: Flexural strain of test specimens

From Figure 25 it can be seen that the flexural strain varies from one percentage to the next, though it can be observed that there is a general downward trend from the lower values of filler (left hand side) to the higher values of filler (right hand side).

The highest strain experienced was by the 10% filler data set with a value of 0.017, while the next highest was 0.016 from the 20% filler. 5% filler experienced a strain of 0.014 while both 15% and 30% experienced a strain of 0.013, while the lowest was 25% with a strain of 0.012. It should be noted that the strain values were taken from calculations using data from the mean values of Table 3 in Appendix D.

4.5 Modulus of elasticity

Figure 26 depicts the values of modulus of flexure (given in MPa) for each of the percentage weights of slg-filled phenol formaldehyde composite, plotted against these corresponding percentage weights. These results are also shown in Table 5 of Appendix D.





It can be seen from the previous Figure 26, that once again, the 5% slg-filled resin has the highest modulus of elasticity with a value of 3232MPa. 25% has the next highest modulus with 2286MPa, followed by 30% with 1666MPa, 10% with 1158MPa and 15% with a value of 1421MPa. The lowest modulus of elasticity was held by 20% with 1106MPa.

4.6 Viscosity of Phenol formaldehyde resin and slg filler

It can be seen from Figure 27, that the lowest viscosity is held by the resin with no slg filler mixed in. This is a reasonable result, as one would expect the resin to have a lower viscosity with no filler mixed in; confirmed by the viscosity of 2790cP. The graphs results then increase as the amount of filler is added. The raw data in Table 8 of Appendix D, shows that the viscosity of 5% slg-filled PF resin is 3650cP, 10% slg-filled is 5120cP, 15% slg-filled is 6750cP, 20% slg-filled is 9400, 25% slg-filled is 13000cP and the final tested mixture of 30% slg-filled PF resin composite is 19000cP.



Figure 27: Viscosity of Phenol formaldehyde resin and slg-filler composite

4.7 Conclusions – Chapter 4

This chapter has outlined results found from the three-point bending test at varying percentages by weight of slg-filler. The following chapter will provide a discussion of these results and their implications.

Chapter 5 – Conclusions, discussions and implications

5.1 Introduction

This chapter will provide a detailed discussion of results obtained and shown in Chapter 4. Discussed results include; flexural strength/stress, strain, Young's modulus of elasticity and viscosity of the different percentages of slg-filler tested. Discussions will be dealt with in light of the aims and objectives of this dissertation; to ascertain the best percentage by weight of microspheres as fillers in phenolic resins using a three point bending test.

5.2 Discussion of results

5.2.1 Flexural Strength

Figure 24 shows the varying mean strengths of the specimens corresponding to their percentage mixture of slg filler by weight. It can be instantly observed from this graph that the 5% by weight has a far greater strength than all other amounts with 44MPa, while the next strongest (25%) is just over half of this amount at 27MPa. The mixture of 30% by weight has strength of 22MPa; while after this 10%, 15% and 20%, show values of strength decreasing from 19MPa to 17MPa.

Therefore the best flexural strength is observed to be at a mixture of 5% by weight. At this low percentage of reinforcement, the filler has the least impact on the flexural strength of the resin.

Figure 28: Extrapolated Flexural Strength (MPa)



It can be seen from Figure 28 that when the data line on the left is extrapolated from 5% down to no filler, the strength increases to approximately 72MPa. This amount is nearly twice as strong as the 5% filler by weight, which is a significant increase.

The fact that unfilled resin has a higher flexural strength than filled resin, was confirmed by Wang, Adanur & Jang. (1997). In this study, Wang, Adanur & Jang (1997) discovered that the flexural strength of unfilled phenolic resin was 71.3MPa, and the strength of 20% by weight ceramic powder reinforced phenolic formaldehyde matrix composite was 10.5MPa.

This research found that the strength of the 20% of filler by weight phenolic resin was 17.2MPa. The notable difference, in strength, can be attributed to the resin used in this research being different to that used by Wang, Adanur & Jang (1997). Wang, Adanur & Jang (1997) used IBI Fiberite resol-type CMXR-6055 phenolic formaldehyde resin while this research used Chemwatch Hexion Cellobond J2027L phenolic formaldehyde resin.

This result shows that although the phenol formaldehyde resin mixture with slg-filler has the highest flexural strength when there is no filler added, there is a jump in strength at 25% filler. At this point there is a significant lowering of material costs as 25% of the weight is filler while it still maintains a reasonable flexural strength.

5.2.2 Flexural strain

Figure 25 shows the calculated values of strain experienced by the different amounts of slg-filler reinforced phenol formaldehyde composite. The largest value of strain experienced was by the 10% slg-filled PF resin. The 20% filled resin experienced a strain of 0.016 while the other slg percentage weights were very close; between 0.012 and 0.014.

The values obtained in this study are considered reasonable when compared to other studies of glass ($\varepsilon = 0.009$) and aluminium oxide ($\varepsilon = 0.007$) (Callister 2005).

It was found by Redjel (1995) that the flexural strain of pure unfilled phenolic resin is 0.00143. The specific resin used for Redjel's (1995) research was 84055; manufactured by CDF-Chimie in France. The catalyst used was C1650 mixed to 3% and cured for 8 days at a temperature of 80°C. This study found a flexural strain of 0.014 (Figure 25 and Table 4) for a percentage weight of filled PF resin of 5%. Though this is approximately 10 times greater than that found by Redjel (1995), the difference may be attributed not only to the reinforcing particles in this particular mixture but also to the newer resin used in this study and the superior post-curing method of 4 hours at 50°C, 2 hours at 80°C and 2 hours at 100°C rather than that of curing at 80°C for 8 days.

5.2.3 Young's modulus of elasticity

From the graph of Figure 26, we can see that for the increasing values of slg- filler, the modulus of elasticity varies though it does follow the general trend of Figure 24: Flexural stress/strength of test specimens. Please see Table 5 in Appendix D for exact values.

The stiffest specimens were the 5% slg-filled set, with a modulus of 3232MPa. This was followed by the 25% slg-filled sample with 2286MPa. As the 25% slg-filled sample is the strongest value after the 5% sample, this result for Young's modulus of elasticity is a significant result, showing that the 25% slg-filled resin composite has a useful strength, while still having a significant reduction in cost due to the 25% weight of slg E-Spheres in the mixture.

5.2.4 Viscosity of Resin and Filler mixture

It can be seen from Figure 29, that as the amount of filler increases, so does the corresponding viscosity. In light of this research, at the optimum strength percentage of 25% slg, the viscosity is 13000 cP, which is still a low enough viscosity that it is suitable for pouring into a mould. This means that this 25% mixture of resin is suitable as a commercially used resin.



Figure 29: Viscosity of phenol formaldehyde and slg filler mixture

5.3 Final material strength conclusions

The strongest value of flexural strength was held by the 5% by weight slg-filled phenolic resin with a mean peak load sustained of 74N, flexural strength of 44MPa, strain of 0.014, and modulus of elasticity of 3232MPa. This correlates with research carried out by Wang, Adanur & Jang (1997). As one of the major reasons for conducting this study was the reduction in costs of phenol-formaldehyde resin composites this low amount of only 5% slg-filler by weight is not suitable as a final recommendation.

The next strongest percentage weight of filler was 25% by weight with a peak load sustained of 45N, a flexural strength of 27MPa, strain of 0.016 and modulus of elasticity of 2286MPa. Though this is not as strong as the 5% filled resin however, it still maintains reasonable strength in flexure and also provides a significant cost reduction as 25% of the final composite by weight if filler, a much cheaper material than the phenol formaldehyde resin. A viscosity of 13000cP is still usable for pouring into moulds at this percentage.

In light of the main objective of this research; to find the best percentage weight of filled phenol formaldehyde resin using the three-point bending test, a mixture of 25% slg-filler by weight is suitable.

5.4 Limitation of Results

Limitations to consider when reviewing the previous research are:

- All measurements (weights, lengths and volumes) were conducted by hand and though all steps were taken to remain accurate to the limit of the equipment, inaccuracies may still result.
- The three point bending test system used had the potential to measure loads up to 100kN, therefore the loads experienced by test specimens during this research were extremely small. The test system may have had difficulty reading results due to the sensitivity of the load cell. This can be viewed in Appendix E.

5.5 Fulfillment of Objectives and Further Research

Although time constraints were present, all project objectives were fulfilled with the exception of electron microscope reviewing of the fracture surfaces of the broken test specimens. This was acceptable, as this objective was deemed in the Project Specification as only possible if time permitted. Electron microscope reviewing is a potential area for further research as the data gained from the fracture surfaces of the test specimens can expand upon the knowledge gained during this study. Another area of possible consideration for future research is to ascertain why the test specimens bowed during the oven curing process and how to minimize this in future.

5.6 Conclusions – Chapter 5

This chapter has provided a discussion of results and their relevant meanings for flexural strength/stress, strain, modulus of elasticity and viscosity. The discussion was undertaken in light of material mechanics and the objectives of this dissertation of ascertaining the best percentage weight of microspheres as fillers in phenolic (phenol formaldehyde) resins using a three point bending test.

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Figure 30: Lower mould piece detail



Figure 31: Middle mould piece detail



Figure 32: Upper mould piece detail



Figure 33: Test specimen detail - Refer ISO14125

Appendix B – Belt sander specifications

Belt sander:	Multitool Belt and Disc Grinding Attachment. Minimum 1/3 hp to run
Belt specifications:	915mm x 50mm. Continuous Industrial rating.
Belt grit:	180
Belt Speed:	1280m/min



Figure 34: ISO14125 3-Point bending test specimen support detail

Appendix C – ISO14125 specimen support dimensions

Appendix D – Tables of results

	Percentage (%) of Filler						
	5	10	15	20	25	30	
Specimen 1	74.7	36.1	27.5	22.2	47	43.8	
Specimen 2	76	30.5	27.2	38.2	44.3	28.2	
Specimen 3	66.3	28.5	27.5	30.2	41.1	37.8	
Specimen 4	79.7	32.4	36.9	32.6	47	36.9	
Specimen 5	76	33.6	38.6	28.5	44.3	39.1	
Specimen 6	70.5	31.7	31.4	20.1	48.3	34.9	
Mean Load	73.87	32.13	31.52	28.63	45.33	36.78	
Standard Deviation	4.74	2.61	5.10	6.69	2.62	5.15	

Table 2: Peak load results by percentage of filler

Table 3: Mean flexural stress/strength by percentage of filler

		Percentage (%) of Filler				
	5	10	15	20	25	30
Mean Load (N)	73.87	32.13	31.52	28.63	45.33	36.78
Flexural Strength (MPa)	44.322	19.278	18.912	17.178	27.198	22.068

Table 4: Flexural strain of test specimens by percentage of filler

		Percentage (%) of Filler						
	5	10	15	20	25	30		
Mean Load (N)	73.87	32.13	31.52	28.63	45.33	36.78		
Flexural Strength (MPa)	44.322	19.278	18.912	17.178	27.198	22.068		
Mean Deflections (mm)	2.34	2.84	2.27	2.65	2.03	2.26		
Flexural Strain	0.014	0.017	0.013	0.016	0.012	0.013		

Table 5: Young's modulus of elasticity by percentage of filler

		Percentage (%) of Filler					
	5	10	15	20	25	30	
Mean Load (N)	73.87	32.13	31.52	28.63	45.33	36.78	
Flexural Strength (MPa)	44.322	19.278	18.912	17.178	27.198	22.068	
Mean Deflections (mm)	2.34	2.84	2.27	2.65	2.03	2.26	
Flexural Strain	0.014	0.017	0.013	0.016	0.012	0.013	
Young's Modulus (E)	3232.602	1158.49	1421.871	1106.306	2286.597	1666.492	

Table 0. Mean mean at su ess menuality standard deviations
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	Percentage of filler (%)						
	5	10	15	20	25	30	
Mean Stress	44.32	19.28	18.91	17.18	27.2	22.07	
Standard Deviation	2.84	1.56	3.06	4.02	1.57	3.09	

Table 7: Weights of materials required to make 1000g of Phenol formaldehyde +E-Sphere filler

Materials	Resin (R)	Catalyst (C)	R + C	Slg (Filler)	Composite
Parameters					
Percentage by weight	20	1			
Percentage by weight			7	3	
Weight of materials in 300g of PF/SLG (10%)	667(g)	33 (g)	700 (g)	300 (g)	1000 (g)

Table 8: Viscosity of phenol formaldehyde resin and slg filler

Percentage Filler	Viscosity (cP)	Spindle Number	Temperature (°C)
0	2790	6 (27.7%)	24.7
5	3650	6 (36.8%)	24.9
10	5120	7 (13.4%)	25.6
15	6750	7 (18.7%)	24.9
20	9400	7 (25.4%)	26
25	13000	7 (30.1%)	26
30	19000	7 (45%)	25.4

Appendix E – Example Specimen Graphs





Figure 36: 10% slg-filler by weight Specimen 3



Figure 37: 15% slg-filler by weight Specimen 7



Figure 38: 20% slg-filler by weight Specimen 2



Figure 39: 25% slg-filler by weight Specimen 1


Figure 40: 30% slg-filler by weight Specimen 3



Appendix F: Sample calculations

This Appendix shows sample calculations used for stress, strain and modulus of elasticity.

Example results: Slg filler by weight 5%: Specimen #4 (See Figure 33)

F = 76N = Peak Load D = 2.50mm = Elongation at peak load b = 10mm h = 4mmL = 64mm

The following equations are used:

Stress:

Strain: $\sigma_{f} = (3FL) / (2bh^{2})$ $\epsilon_{f} = (6Dh) / (L^{2})$ Modulus of Elasticity (E): $E = \sigma_{f} / \epsilon_{f}$

Using the results for 5%, #4:

Stress:

$$\sigma_{f} = (3FL) / (2bh^{2})$$

 $\sigma_{f} = (3*76*64) / (2*10*4^{2})$
 $\sigma_{f} = 45.6MPa$

Strain:

$$\epsilon_{f} = (6Dh) / (L^{2})$$

 $\epsilon_{f} = (6*2.5*4) / (64^{2})$
 $\epsilon_{f} = 0.014$

Modulus of Elasticity:

$$E = \sigma_f / \epsilon_f$$

E = 45.6 / 0.014
E = 3112MPa

Appendix G: Project Specification

University of Southern Queensland Faculty of Engineering & Surveying

ENG 4111 & 4112 Research Project PROJECT SPECIFICATION

FOR:	RICHARD GURNEY
TOPIC:	Best percentage weight of microspheres as fillers in resin and
	three-point bending test.
SUPERVISOR:	Dr. Harry Ku
SPONSORSHIP:	University of Southern Queensland
PROJECT AIM:	This project seeks to find the best percentage by weight of microspheres as fillers in resin using a three-point bending test to determine material properties. Further investigation into
	formulas that could be for theoretical predictions of behaviour
	and surface fractures of test specimens may be conducted.

PROGRAMME: Issue A, 24th March 2006

- 1. Research background information for three point bending tests and standard testing procedures as well as research phenolic resins and their uses.
- 2. Design mould for test specimens and have this fabricated.
- 3. Use constructed mould to create specimens then test these using a three point bending test and record data.
- 4. Analyse collected data and calculate stress, strain and modulus of elasticity in the material. From these results draw conclusions as to the best percentage of filler.
- 5. These results will then be applied to determine trends and formulas for use with theoretical predictions relating to filled composites and their behaviour.

As time permits:

6. View broken samples under scanning electron microscope at Queensland University of Technology (to be arranged)

AGREED:

_____(Student)

____/___/____

_____(Supervisor) ____/___/