University of Southern Queensland

Faculty of Engineering and Surveying

Investigation of the Best Percentage, by Weight, of Glass Powder as Filler in Phenolic Resin using Three Point Bending Test

A dissertation submitted by

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In fulfilment of the requirements of

Courses ENG4111 and 4112 Research Project

Towards the degree of

Bachelor of Mechanical Engineering

Submitted: October, 2008

Abstract

Phenolic Resin is a useful but expensive engineering material used in a wide range of structural applications. Fortunately the nature of Phenolic Resin means that fillers can be easily added to it to produce a Phenolic Composite with a lower cost per unit volume than the original resin. The addition of the filler also means the composite has different physical properties. Previous research has been conducted into which materials can be used as fillers and Glass Powder was found to be one such material. This project aims to investigate the Best Percentage, by weight, of glass powder as Filler in Phenolic Resin using the Three Point Bending Test. The percentages tested were 5%, 10%, 20%, 25% and 30%. A number of phenolic composite specimens of each composition were created by hand and poured into moulds for preliminary curing. After preliminary curing they were removed from the moulds and placed in a conventional oven for post curing. Post Curing took place at 50°C for 4hrs, 80°C for 4hrs and 100°C for 2hrs. At the completion of post curing the specimens were subjected to the Three Point Bending Test according to International Standard ISO 14125:1998(E) which applies tensile, compressive and shear stresses to test specimen. The testing machine produced a plot of load versus elongation from which Flexural Strength, Flexural Strain and Flexural Modulus were calculated and plotted for analysis in order to determine the best percentage of glass filler by weight.

Certification

I certify that the ideas, designs and experimental work, results, analyses and conclusions set out in this dissertation are entirely my own effort, except where otherwise indicated and acknowledged.

I further certify that the work is original and has not been previously submitted for assessment in any other course or institution, except where specifically stated.

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Signature

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Date

Acknowledgements

Firstly, I would like to thank my project supervisor Dr Harry Ku of the University of Southern Queensland for providing invaluable information and assistance throughout the undertaking of this project.

I would also like to thank the University of Southern Queensland's research technologist Mohan Trada for his technical advice during specimen creation, testing and analysis particularly since Harry had many other project students to supervise this year.

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1 INTRODUCTION

Topic: "Investigation of the Best Percentage, by Weight, of Glass Powder as Filler in Phenolic Resin using Three Point Bending Test"

1.1 Introduction

This chapter will outline the purpose and aims of this research project.

1.2 Purpose of the Research

When the Phenolic Resin and Glass Powder mentioned in the title are mixed together they form a Phenolic Composite. Phenolic's themselves have been used in engineering for about 100 years, mainly for ergonomic applications. More recently composites have been increasingly used for structural applications in aerospace, marine, transport and civil engineering hence there is a need for research into their mechanical properties. The three point bending test used in this research is a useful test because the behavior of specimen under the test is very good indication of the basic characteristics of the material.

1.3 Aim

The aim of the research was to produce a number of phenolic composite test specimens post cured by conventional oven with different percentages of glass filler by weight and use the three point bending test to determine the best percentage depending on cost and performance. The main measures of performance under the three point bending test are flexural strength, flexural strain and flexural modulus.

1.4 Conclusions: Chapter 1

The purpose and aim of this research project have been outlined. The following chapter is Literature Review of the topic which provides the appropriate background knowledge.

2 LITERATURE REVIEW

2.1 Introduction to Literature Review

This chapter will define the term composite, investigate the materials required to a make a phenolic composite test specimen and describe the three point bending test.

2.2 Composites

By general definition a composite is produced when two or more materials or phases are used together to give a combination of properties that cannot be obtained by other means. Composites have a wide range of applications because the combination properties of a composite can be altered by changing the materials used or by using the same materials but adjusting their respective compositions. Composite materials and compositions are chosen to give unusual combinations of stiffness, strength, weight, high temperature performance, corrosion resistance, hardness, conductivity and cost. This project looks into achieving a balance between the cost of the composite and its mechanical performance by increasing the amount cheaper component, the glass powder. A phenolic composite is classed as a particulate composite because of the glass powder particles.

2.3 Background of Phenolic Resin

The main component in the phenolic composite is the Phenolic Resin. Phenolic Resins, also called phenol-formaldehyde polymers, were the first completely synthetic polymer to be commercialized in 1907.

Phenolic Resin is a phenol-formaldehyde based resin where the polymer is created by condensation polymerization reaction between the phenol and formaldehyde. This reaction can result in two intermediate forms resole or novalac. In this study resole resin was used. The final Phenolic Polymer is a type of plastic under the category of a thermosetting polymer. Thermosetting Polymers (thermosets) consist of highly cross-linked polymer chains that form a three dimensional network structure. The cross links mean the chains cannot rotate or slide and so thermosets possess good strength, stiffness and hardness but it

is also means they have poor ductility and impact properties. Askeland and Phule(2006) state that in a tensile test thermosets display properties similar to a brittle metal or ceramic.

Thermosetting Polymers often begin as linear chains. The initial polymer maybe either a solid or liquid resin; and various methods can be used to initiate the cross-linking process. In this case a catalyst and heat are used to initiate and sustain the reaction. Cross-linking is not reversible so once formed thermosets can't be reused or easily recycled.

The previously mentioned characteristics of a thermoset make the Phenolic Resin an ideal candidate for use in producing a composite suitable for structural applications. The strength, stiffness, hardness, heat and fire resistance and chemical inertness are all desirable final properties while the fact that the initial polymer is a liquid resin makes adding a filler to improve its other properties a simple mixing process. Initially being a liquid also means that the final composite can be produced in many different shapes as they are needed.

2.4   Background of Fillers

The properties of composite material are heavily dependent on the properties of the base material but often there may be one property or several properties of the base material are undesirable or make it unsuitable for a particular application. A filler can be added to address this situation and allow the composite material properties to be adjusted to suit a wide range of applications. According to Osswald and Menges (1995) Fillers can be divided into two categories. The first being fillers that reinforce the polymer and improve its mechanical performance; and the second includes those that are used to take-up space and so reduce the amount of actual resin to produce a part. Fillers in the first category may improve the polymers mechanical performance by increasing its strength and fracture toughness or by reducing its brittleness while not affecting the desirable properties of the original polymer. The fillers in the second category allow a certain volume of composite to be produced with lesser amount of resin. These fillers are chosen to have lower density than the resin and/or be cheaper than the resin so that for a given volume the composite is lighter and cheaper than the neat resin. Some fillers may fit into both categories. They may be added for one reason but have beneficial or adverse affects in another area.

2.5 Composite Materials used in this Research

2.5.1 Phenolic Resin

The Phenolic Resin used in this research was Hexion Cellond J2027L a commercial resole phenolic resin produced by Hexion Specialty chemicals Pty Ltd [Chemwatch, 4601-85].

2.5.2 Catalyst

The Catalyst used in this research was Hexion Phencat 15 also produced by Hexion Specialty Chemicals. Phencat 15 is a phenolic resin hardener catalyst with the active ingredient xylenesulfonic acid [Chemwatch, 4601-93].

2.5.3 Glass Powder Filler

The Glass Powder is a powder of spherical hollow glass spheres called SPHERICEL® 60P18 produced by Potters Industries. The glass spheres are composed of a fused inorganic oxide and the spheres have a diameter from 6-32µm giving an appearance of a white powder like icing sugar. The glass is often used to enhance performance and reduce viscosity in paints and coatings and as lightweight additives in plastic parts. They are chemically inert, non-porous, and have very low oil absorption. They also do not absorb the resin to provide maximum filler function [Eager Plastic Inc, 2006]. The density of the hollow glass powder used in this research is 0.6 g/cc. This low density means that on a per volume basis the glass powder is far lighter and cheaper than the phenolic resin.

2.6 Resin and Catalyst Ratio

For this project the phenolic composite is produced by mixing the phenolic resin, glass powder and catalyst pouring them into moulds and then allowing time for preliminary curing before post curing in an oven. To get a successful reaction you need the right ratio of catalyst to resin; a reasonable range of glass composition and suitable range of oven settings. A study by Ku (2007) into phenolic composites found that the catalyst to resin ratio may have to be adjusted depending on the manufacturer of resin since different manufacturers produce resins with different viscosities and densities due to a different

water content. In previous a study a resin to catalyst ratio of 20:1 was used but it was found to be too vigorous for a Hexion supplied resin and so a ratio of 50:1 was used. Both of these studies used Slg filler, which proved to affect curing differently to the glass used in this research so the ratio was adjusted as is explained methodology section of this dissertation, however 50:1 was used as a starting point.

2.7 Glass Powder Composition

The glass powder compositions in the phenolic composites tested and compared in this research were 5%, 10%, 15%, 20%, 25% and 30% by weight. The composition of 0% glass powder, in other words neat resin, did not need to be tested because the results for neat resin were already available. In previous studies at University of Southern Queensland composites wouldn't cure properly for filler compositions above 30%.

2.8 Three Point Bending Test

The three point bending test is an engineering materials test that produces tensile, compressive and shear stresses in the specimen making it a useful test because the behavior of specimen under the test is very good indication of the basic characteristics of the material in the specimen. The performance of a specimen in the three point bending test is used to obtain the modulus of elasticity in bending, the flexural stress, the flexural strain and the flexural stress-strain response of the material.

For the three point bending test the testing apparatus makes contact with the test specimen at three points to apply force in a way that will induce bending in the test specimen. More specifically the three point bending test involves supporting a test specimen at two points, one at each end, and then applying a load in the opposite direction to one point in the middle of specimen until it breaks. The standard used for the test was the International Standard ISO 14125:1998(E) an expansion of ISO 178. This standard specified the span for the test, the distance from support to support, to be 64mm (ISO 14125, 1998).
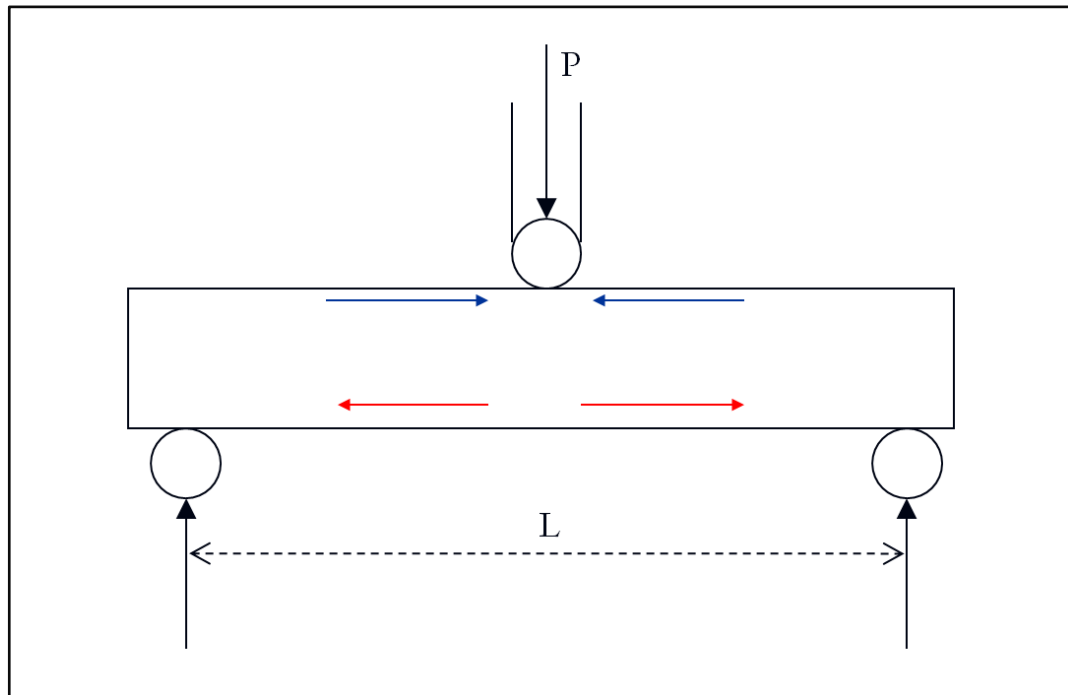
Figure 2.1: The Three Point Bending Test

The testing machine provides the load and deflection of the centre of the beam at the point the specimen fails. This maximum load and deflection are then used to calculate the flexural strength, flexural strain and the flexural modulus which is Young's Modulus for bending or the Modulus of Elasticity for bending.

2.8.1 Flexural Strength

The flexural strength, often referred to as the modulus of rupture, bend strength or fracture strength is the strength of a material in bending. The flexural strength is determined by the highest stress in the material at the time of rupture. The highest stress in the material occurs at the outermost fibres, the fibres at the surface, on the tensile side of the test specimen. The flexural strength is therefore calculated as the stress in the surface of the test specimen at the instant of failure. In plastics the flexural strength is generally higher than straight tensile strength (Redjel, 1995). In SI units flexural strength is measured in Pascals and expressed in Megapascals.

The equation used for calculating the flexural stress,

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

2.8.2 Flexural Strain

The flexural strain is a measure of the deformation of a material due to bending. Flexural Strain is determined by the maximum deformation of the material which is the deformation at the time of failure. Flexural Strain is calculated using the maximum deflection of the centre of the beam which is the deflection of the centre of the specimen at the time of rupture. As with normal strain flexural strain is dimensionless property expressed as a percentage.

The equation used for calculating the flexural strain,

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

2.8.3 Flexural Modulus

The flexural modulus, often referred to as modulus of elasticity in bending, the elastic modulus in bending or Young's Modulus in bending, is the ratio of flexural stress to flexural strain. If the flexural modulus of material is known it can be used to determine the deflection of a beam for a given load. Since the flexural modulus is calculated by dividing flexural strength in Pascals by flexural strain which is dimensionaless the SI unit for the flexural modulus is Pascal and is expressed in Megapascals or Gigapascals.

The equation used for calculating the Flexural Modulus

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

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2.8.4 Flexure Calculations Symbols

σ_f: stress in outer fibre at midpoint, MPa;

ε_f: strain in the outer surface, %;

E_B: modulus of elasticity in bending, MPa;

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

L: span from support to support, mm;

b: width of test beam, mm;

h: depth of test beam, mm;

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

m: slope of the tangent to the initial straight line portion of the load deflection

curve, N/mm.

2.8.5 Testing Machine

The testing machine used to perform the three point bending test for this research was the University of Southern Queensland's universal testing machine an MTS 810 Material Test System. The machine is computer controlled and servo-hydraulically operated which makes it sufficiently accurate to perform the test on the composites but not perfect because the machine is rated to a 100kN load far above the load at which the test specimens fail. The software used with the testing machine is Test Works 4.

2.8.6 Test Specimen Requirements

To meet the conditions specified by the Standard ISO 14125:1998(E) using the Unviersity of South Queensland testing machine the test specimens need to have a rectangular cross section and have dimensions of approximately 100mm long, 10mm wide and 5mm thick. The length is a less critical dimension as long as it's sufficiently greater than the specified 64mm span. The other dimensions need to be as accurate as possible although the actual

value of width and thickness of each specimen is measured and put into the testing machine computer.

The number of test specimens required for each composition is at least five. This is due to the non uniformity of the material. In the case of a uniform material like steel only one test would be needed.

2.9   Curing

2.9.1 Initial Curing

Previous research by Ku et al (2006) found that the specimens should be allowed at least 24hrs for initial curing in the moulds at room temperature. This was for Slg filler in the resin however and glass powder proved to be different as described later in the methodology section of this dissertation.

2.9.2 Post Curing by Conventional Oven

Oven Post Curing is the process of providing the heat which assists the cross linking reaction of the phenolic resin. A conventional oven is used because it provides and the heat can be easily and accurately be controlled and maintained. From previous studies at USQ it was determined that the specimens be post cured for: 4 hours at 50°C, 4 hours at 80°C and 2 hours a 100°C. The specimens are removed from their moulds before post curing but need to be constrained by some means to prevent warping as the temperature increases.

2.10 Conclusion: Chapter 2

The Literature Review in this chapter has provided background and technical information necessary to understand the phenolic composite and the three point bending test. The next chapter will describe the methodology for the research.

3 RESEARCH DESIGN AND METHODOLOGY

3.1 Introduction to Research Design and Methodology

This chapter covers everything required to produce the results for this research. It contains a Resource Analysis, Risk Management and an Assessment of Consequential Affects as well as Moulding, Mixing, Testing Information

3.2 Resource Analysis

This research was sponsored by the Faculty of Engineering and Surveying at the University of Southern Queensland and so all the resources were supplied by the faculty.

- **Phenolic Resin:** The Phenolic Resin used is Hexion Cellobond J2027L sourced by the faculty from Hexion Specialty Chemicals. The Resin can only be stored for a short period so it should be ordered to arrive as it is needed. If other people are using the resin it will need to be ordered early because it can take some time to be delivered. The cost of resin is $7/kg

- **Catalyst:** The phenolic resin hardener catalyst (acid catalyst) used is Hexion Phencat 15 sourced by the faculty from Hexion Specialty Chemicals. The catalyst should also be ordered early to avoid delays. The cost of the catalyst is $8/kg.

- **Glass Powder Filler:** The glass powder filler used is Spherical Hollow Glass Spheres sourced by the faculty from Potter's Industries Inc. Since there are no storage limitations with the glass powder a large quantity can be stored in Laboratory so it is unlikely to run out. The glass powder cost $5/kg. (See Figure 5 in Appendix)

- **Laboratories:** Access to several rooms of the Engineering and Surveying Building was required for this research. Materials Preparation room Z106.1 was used for mixing the materials moulding the specimens since it has appropriate ventilation facilities; Heat

Treatment Room Z116.1 for use of the convential oven; and Non-Destructive Testing Room Z104.1 for use of the Tensile Testing Machine.

- **PVC Moulds:** PVC Moulds are needed to contain the phenolic composite for preliminary curing.

- **Metal Screws:** Metal Screws with wing nuts are used to clamp the mould pieces tightly together

- **Lubricant:** Lubricant is essential for the easy removal of the test specimens from the moulds. At present a non-stick kitchen cooking spray is being used as well as a special wax.

- **Spoons:** Simple Disposable plastic food spoons are used in measuring out the chemicals, mixing the chemicals and pouring into the moulds

- **Containers:** Ice cream containers are used to measure the chemicals into and also to mix the chemicals in.

- **Measuring Scales:** Measuring Scales are used to measure out chemicals to achieve the correct composition.

- **Convential Oven:** Faculty of Engineering and Surveying's conventional oven was used for post curing

- **Testing Machine:** The Faculty of Engineering and Surveying's Universal Testing Machine was required to perform the three point bending test.

3.3   Risk Management

3.3.1 Risk Identification

To produce the phenolic composite test specimens this project involves handling industrial chemicals. The properties of these chemicals provide the largest dangers to a person undertaking this work. Both the resin and the catalyst have hazard rating of moderate to high for both toxicity and body contact. The resin is toxic by inhalation, causes burns and poses a risk of serious damage to the eyes, it is therefore classified as, Harmful: Serious damage to health by prolonged exposure through inhalation, skin contact or if swallowed.

The catalyst poses similar risks to the resin and it's a corrosive liquid since it contains xylensulfonic acid it is also a possible cancer causing agent following repeated inhalation.

Mixing of the resin and the catalyst also poses another risk. If not controlled, mixing the resin and catalyst directly together may be strongly exothermic giving off much heat and may even be violent.

Other risks include the wax lubricant which is also harmful to inhale and the fact that there is no natural ventilation in the room if the artificial ventilation system is not turned on.

Some less obvious risks are oil spray and glass powder getting on the floor making the floor slippery, danger of cutting a finger when scraping moulds clean and risk of flying particles striking eyes when cleaning moulds.

3.3.2 Risk Evaluation

The risk of inhaling the fumes from the chemicals or coming into physical contact with them is quite high since they are poured and mixed by hand. The fact that the resin is viscous and has adhesive properties making it awkward to pour cleanly also adds to the risks. Since the laboratory is small and not naturally ventilated there is also a risk to other

people who may be working at the same time. All people in the laboratory would be at risk to the fumes as well as splashes and spillages caused by an unsteady hand.

A slippery floor due to the oil spray also poses a considerable risk to everyone in the lab. A fall could cause direct physical injury and could also cause a spillage if a person slips while handling chemicals. The oil is particularly dangerous to those who are not aware of it because the oil can't be seen on the floor.

The risk of impaling oneself with the tools required to clean and prepare moulds is a possibility but not a considerable risk since none of the tools required are that sharp and the forces that need to be applied are not that extreme. Flying composite particles are a bigger risk with a high chance of hitting someone in the eye.

3.3.3 Risk Control

The first step in risk control is to read the Material Safety Data Sheets for each of the materials before undertaking a workshop induction. Protective equipment is worn to prevent inhalation and physical contact with the materials. The protective equipment includes two layers of rubber gloves, a face mask and safety glasses. Clothes should be washed after handling. The laboratory also has an artificial ventilation system which must be turned on to extract harmful fumes. Any spillages of chemicals should be cleaned up immediately and care should be taken when cleaning moulds.

3.4 Assessment of Consequential Affects

3.4.1 Sustainability

Since phenolic composites are already widely used in industry it is unlikely that this project will cause any issues with sustainability apart from those that already exist. The biggest problem for sustainability with phenolic composites is that the curing process is not reversible so they cannot simply be recycled. Waste from phenolic components which are no longer required is a serious issue. If this study was to produce useful findings then it could encourage an even broader use of phenolic composites which would further add to sustainability issues. Whether these problems are worse than those related to other

engineering materials that could be used in place of phenolic composites would have to be investigated. Another factor to consider when discussing sustainability is that once the catalyst is added to the other materials the polymer will begin to set and therefore any excess product instantly becomes waste and so it is important that only the amount material is mixed.

3.4.2 Ethical Responsibility

Should the findings of this study encourage the use of phenolic composites as structural materials in certain applications then it is important that results are accurately obtained and recorded. Unexpected failure of a structural component due to poor material properties would have disastrous consequences possibly endangering human life and also resulting in a large financial cost. Another Safety and Ethical concern is the toxicity of the formaldehyde based resin during processing and in the residual traces of formaldehyde in the final composite.

3.5 Moulding

The test specimens for this research were created using moulds. Before any of the composite materials were mixed or even removed from their respective storage containers it was essential that the moulds were organised, assembled and prepared.

3.5.1 Moulds Used

The moulds used for this research were available from the Faculty of Engineering and Surveying at USQ since their manufacture was required for previous Faculty studies. The moulds consist of two PVC (poly vinyl chloride) plates which are clamped together tightly using screws and wing nuts. The bottom plate is just a smooth flat plate while the top plate has the mould shapes cut into it. This configuration allows the specimens to be removed from the moulds. Each mould set is capable of producing six specimens at one time.

The moulds were intended to be used in a three piece configuration where three flat plates are fastened together using screws and wingnuts. The plates are the same thickness as the specimen so the middle plate has the specimen shapes cut into it and provides the side walls for the moulds. The top and bottom plates are normal flat plates that act as the flat floor and

flat ceiling of the mould. In this research problems with curing meant that the top plate was left off for ease of monitoring the progress of the cure, hence in this research the moulds are used in a two piece configuration.

Figure 3.1: The Two PVC Plates that make up a Mould Set

3.5.2 Mould Preparation

The moulds needed to be thoroughly cleaned and lubricated before they were assembled each time to avoid contamination of the test specimen and allow its easy removal.

Cleaning usually just involved removing composite material residue from a previous sample. This was best achieved using a standard paint scraper. Once the scraper got underneath the residue resin it would fairly easily flake off. If the moulds were not thoroughly cleaned foreign material could have ended up in test specimens. This could have affected the curing process or geometry of the test specimens and the results from the test would have been inaccurate.

Lubrication of the moulds required some consideration. Initially a standard kitchen "non-stick" cooking spray was used to lubricate the moulds due to the unavailability at the of the specially designed wax lubricant that was later used.

The problem with the cooking spray is that it produces a froth and droplets in the mould which means the lubricant spread is not even. The spray also tends to pool as liquid in the

bottom of the moulds as more is applied to cover the entire surface of the moulds. These properties of the spray could cause it to mix into the composite possibly creating an entirely different chemical. Trying to wipe off excess residue from the spray with a rag or paper towel would again result in insufficient lubrication.

Once the specially designed wax for polymers was available it was then used for all further lubrication. The wax could be applied more evenly than the cooking spray but it needed considerable downward pressure to "smear" it on to the mold surfaces. After the initial application there were often small clumps of wax left behind that had to be brushed off. Due to the properties of the wax and the small mould size the best way to lubricate the moulds thoroughly was to do it while the pieces of the mould were separated. This allowed the wax sponge to be pushed through the moulds to lubricate the side walls and also meant no clumps of wax were left in the corners of the mould. The top surface of the mould was also lubricated to make removal of residue easier for the next sample.

Figure 3.2: Wax Lubricant with Sponge and Paint Scraper used for Cleaning

3.5.3 Mould Assembly

When both pieces of the mould had been thoroughly lubricated the mould could then be assembled. The two mould plates were clamped together using screws and wingnuts. There could not be any gap between the two plates or the composite would leak out affecting the

geometry of the specimen and making removal more difficult. To avoid gaps as many screws as possible were used and they were tightened firmly and evenly. The screws were tightened evenly to prevent warping of the plate. If each screw was fully tightened in one go while others are still completely loose there was a chance of minor warping which would have been enough for the composite to leak.
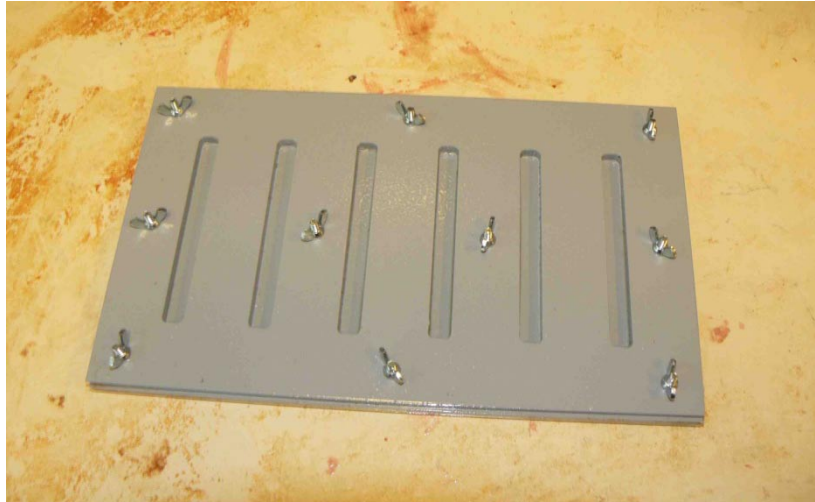
Figure 3.3: The Assembled Mould

3.6 Mixing of Phenolic Composite Materials

It was found that 100g of the phenolic composite with any composition was enough to fill two sets of mould with minimal wastage. This was beneficial as it kept the mixing ratio simple and meant there were 12 specimens from which to gain five suitable specimens. The composite materials were therefore mixed to make 100g batches.

3.6.1 Mixing Ratios

Two mixing ratios determine the mass of each material required to produce a batch of phenolic composite for a given composition.

- Resin + Catalyst : Glass
- Resin : Catalyst

Therefore for 5% glass by weight, Resin + Catalyst : Glass = 95:5

Since the total batch mass is 100g the required mass of the Resin + Catalyst is 95g while the mass of glass powder is 5g. For Resin : Catalyst = 30 : 1 the mass of catalyst is equal to 95g divided by 31 parts, (95/31)g which works out to be 3.064g of catalyst. The mass of the resin is then simply 30 times the mass of the catalyst which is 91.935g of Resin. These masses were rounded to the nearest 0.1g the maximum accuracy of the measuring scales. See Table 3.1 for masses for batches of all compositions.

Originally the resin to catalyst ratio was intended to remain at 50:1 no matter what the composition of the glass however the composite was not curing properly so this ratio was eventually dropped to 12:1. It seemed that the higher the composition of glass the more catalyst was needed. Due to time constraints the samples could not be remade to have all the same resin to catalyst ratio and this could have affected the results however the samples with lower compositions of glass set faster than the higher compositions did with the more catalyst anyway. Increasing the amount of catalyst had to be done with care because its Material Safety Data Sheet states that if the resin and catalyst are not mixed according to the manufacturer's directions the reaction could be violent and exothermic giving off much heat. This would be dangerous and would also damage the specimen.

3.6.2 Material Masses required for a Batch

%Filler (by weight)	Resin:Catalyst	Resin (g)	Catalyst (g)	Glass (g)
5	30:1	91.9	3.1	5
10	20:1	85.7	4.3	10
15	20:1	81	4	15
15	15:1	79.7	5.3	15
20	12:1	73.8	6.2	20
25	12.1	69.2	5.8	25
30	12:1	64.6	5.4	30

Table 3.1: Masses of Materials required to produce a 100g batch of phenolic composite

3.6.3 Measuring and Mixing

In order to ensure that the specimens had the composition that they were labelled with it was important to measure the masses of the materials accurately.

Before any of the materials were measured out, the container they were being added to was placed on the scales and the scales were re-zeroed. The resin was measured out first into one container and then glass powder was measured out into a separate container. The measured amount of glass powder was then carefully added to the resin container making sure that all the glass was added and that none was spilt or left in its original container. The glass was then stirred meticulously into the resin.

Figure 3.4: Measuring Resin

Figure 3.5: Mixing Glass and Resin

It was found that mixing the glass into resin before the catalyst was added produced a better result. A more even mixture was achieved and it took less time. It was important that the glass was stirred in well enough to ensure an even mixture and no glass clumps

however if stirring was too vigorous air would get into the mixture and result in air bubbles in the final specimen. The air bubbles would weaken the material by reducing the actual cross-sectional area of the specimen and hence the area over which the load is applied.

Figure 3.6: Glass clump in Final Specimen due to poor mixing

Once the resin and glass were mixed the mixing container (containing the glass and resin) was placed on the scales and the scales were re-zeroed again. The catalyst was then measured directly into resin/glass mixture. This was done because the amount of catalyst was very small and so a small change in mass would have been a large in the catalyst ratio. When the catalyst was measured into a separate container first there was often at least about a gram of catalyst still left in the container after the catalyst had been added to the resin/glass mixture therefore the amount catalyst in the composite would not have be equal to the measured amount. Once the catalyst was added it was also carefully stirred in making sure that no air got into the composite.

Figure 3.7: Mixed Phenlic Composite ready for Pouring

3.7 Initial Curing

3.7.1 Pouring into Moulds

After the materials are completely mixed and the mixture is consistent the phenolic composite was poured into the already prepared and assembled moulds where they remained for initial curing at room temperature.

For ease of removal of the specimen after initial curing and to ensure a high quality test specimen care had to be taken when the mixture was being poured into the moulds. The mixture was filled right to the top of the mould so that the specimen would have neat rectangular cross section. In order to have the mould filled to the top it initially had to be overfilled and then as the mixture settled it would end up level with the top.

When slightly overfilling it was important to make sure that the mixture didn't spill over top of the mould sides as this made removal of the solid specimen difficult particularly if there was any leakage at all at the bottom mould because the specimen would become locked in. It was also easier to pour neatly in the first place than it was to fix it up afterwards. As the glass percentage increased the mixture became more difficult to pour neatly.

Figure 3.8: Pouring Composite Mixture into Moulds

3.7.2 Specimen Removal from Moulds

Previous studies suggested that initial curing of the composite would take 24hrs but these studies used Slg filler as opposed to the glass filler used in this research. The glass filled composite took more than a week at the least to have hardened sufficiently to be removed from the moulds. After initial curing the composite is still quite weak and many specimens were broken during the removal process. Even with great care taken in lubricating and pouring, considerable force was still required to remove the specimens. The technique eventually used to remove the specimens without breaking them involved cutting several paddle pop sticks to the length of the specimen and taping them together one on top the other to create a block that could be used to push the specimen out. It was convenient that the width of the paddle pop sticks was about the same width as the specimen. Taped together the paddle pop sticks are rigid enough to apply the pressure evenly across the entire surface of the specimen and thick enough to push all the way through the mould. When pushing out a specimen the mould plate was supported so that a downwards force could be applied and the specimen could fall out underneath. This method of supporting the mould plate also helped keep even pressures on the specimens so they weren't destroyed or damaged.

Figure 3.9: Single Paddle Pop Stick Neatly fitting over Specimen
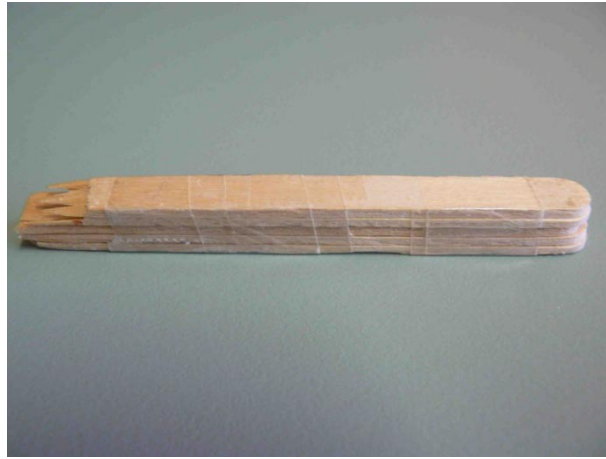
Figure 3.10: Paddle Pop Stick Specimen Remover

3.7.3 Specimen Identification

Upon removal the specimens were clearly labelled so each specimen could be identified later. The label included information such as the owner's initials, the glass composition and the resin to catalyst ratio.

3.8   Conventional Oven Post Curing

Post curing was performed using the Faculty of Engineering and Surveying's conventional oven. The specimens were cured for 4 hours at 50°C followed by 4 hours at 80°C and 2 hours a 100°C. During the post cure the specimens were constrained by 2kg weights to prevent warping.

Figure 3.11: Specimen Support for Post Curing (Side View)
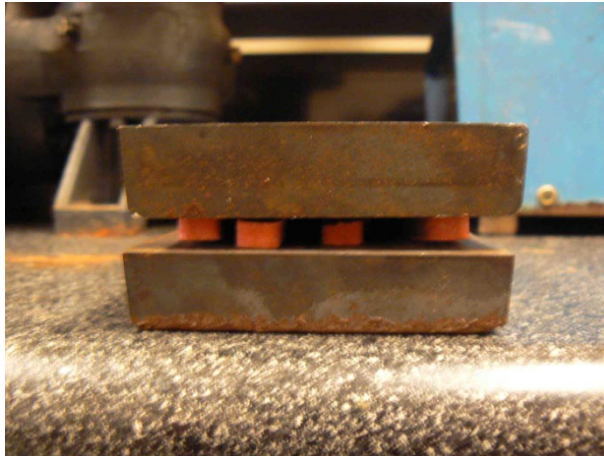
Figure 3.12: Specimen Support for Post Curing (End View)

3.9   Testing

Once the specimens were post cured they were ready for testing. The University of Southern Queenslands Universal Testing Machine was first prepared with the previously manufactured Three Point Bending Test attachments and the three point bending test was conducted according to International Standard ISO 14125:1998(E). The computer software Test Works 4 was then used to control the testing machine to conduct the tests.
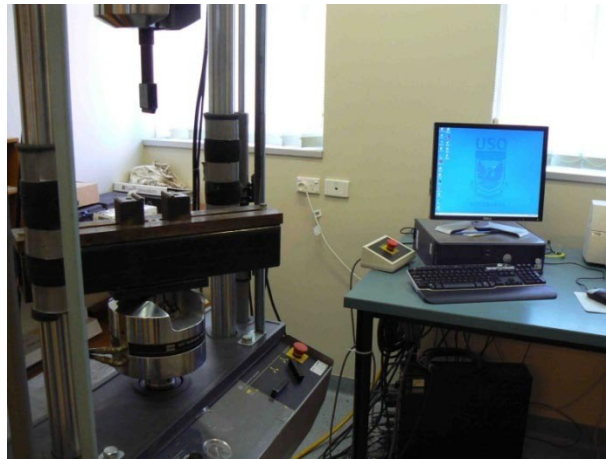
Figure 3.13: Testing Machine and Computer

Five Specimens of each composition of filler were tested. The computer required that the width and height of each specimen be entered for each test. These where measured using a set of digital vernier callipers. The machine slowly increases the load on the specimen until it fails. The computer provides the peak load and elongation. The elongation is the deflection of the centre of the beam.

Figure 3.14: Specimen during Test

The computer also produces a plot of the elongation against the load for each test. The plot contains a lot of noise because of the machine is not really design to apply such small loads accurately. The overall slope of the plot is better indication of how the specimen performed during the test.
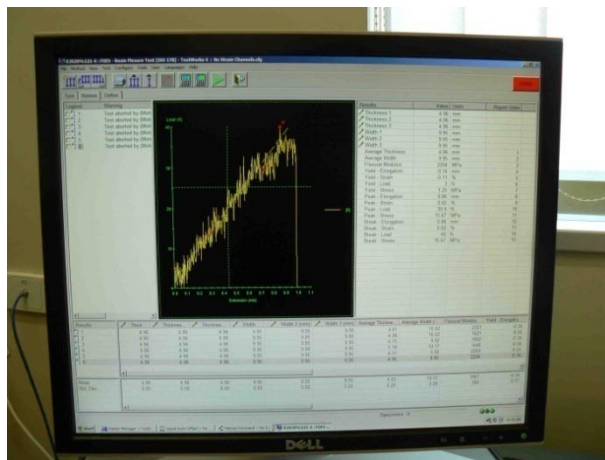
Figure 3.15: Load and Elongation Plot produced during the Test

3.10 Data Collection and Manipulation

Once the testing was completed the data was then collected, reviewed and screened for any anomalies. Any data that was implausible (most likely due to an error in the running of the test) was discarded along with any data that included an extremely low failure load (most likely due to a specimen fault). The average peak load and elongation for each composition were then calculated from their five respective specimens. The flexural strength, flexural strain and flexural modulus for each composition were then calculated and plotted

4 RESULTS AND ANALYSIS

4.1 Introduction to Results

This chapter contains the results for the Three Point Bending Test. The Flexural Strength, Flexural Strain and Flexural Modulus are all presented as plots against the phenolic composites composition, by weight, of glass powder filler. An analysis of each of the plots is also included as well a comparison to results found by a study involving the same resin but SLG Filler.

4.1.1 Flexural Strength Results
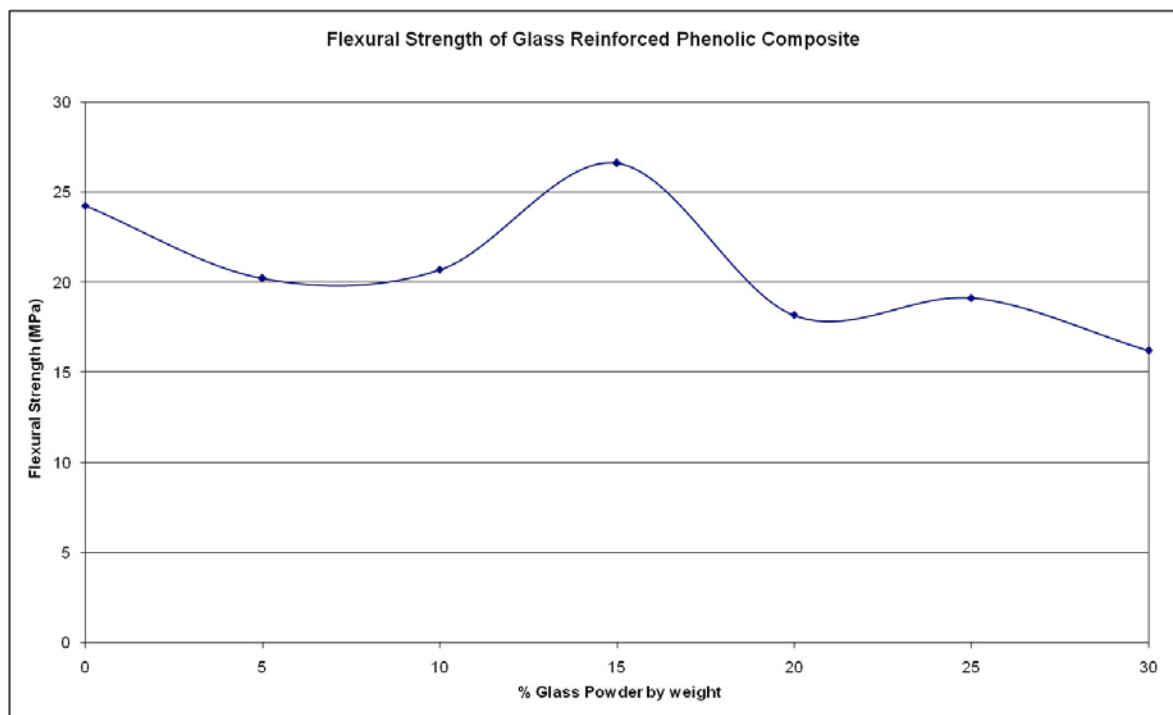
Figure 4.1: Flexural Strength of Glass Filled Phenolic Composite

Figure 4.1 shows the changes in average flexural strength as the composition, by weight, of glass filler changes. The flexural strength of the phenolic composite at 0% filler is that of the neat resin which was 24.26MPa (Ku, 2007); for 5% the flexural strength of the composite dropped to 20.2MPa; then were was a gentle rise for 10% to 20.7MPa; for 15% there was a large spike to 26.6MPa before falling back to 18.2 MPa at 20%; the flexural

strength rose again at 25% to 19.1MPa before finishing up at 16.2MPa for 30%. These results compare reasonable with the study by Ku for the same resin but with SLG Filler which found Flexural Strengths ranged from a peak of 37.2MPa at 25% to a low of 10.95MPa at 30%

The general trend for the flexural strength of the phenolic composite appears to be that it decreases as the glass filler composition increases. Most of rises in flexural strength as the glass composition increases are only slight with 15% being the exception. Although the flexural strength results for 15% seem to be inconsistent with the general trend it would be reasonable to assume it is accurate because it was calculated using two different samples. The composite for each sample contained 15% glass but had different resin to catalyst ratios. One sample had a resin to catalyst ratio of 20 to 1 while the other was 15 to 1. The samples were combined because due curing problems early in the research there was not enough specimens in either sample alone. Both of their results were nearly identical so the calculations were just based on one of the samples.

4.1.2 Flexural Strain Results
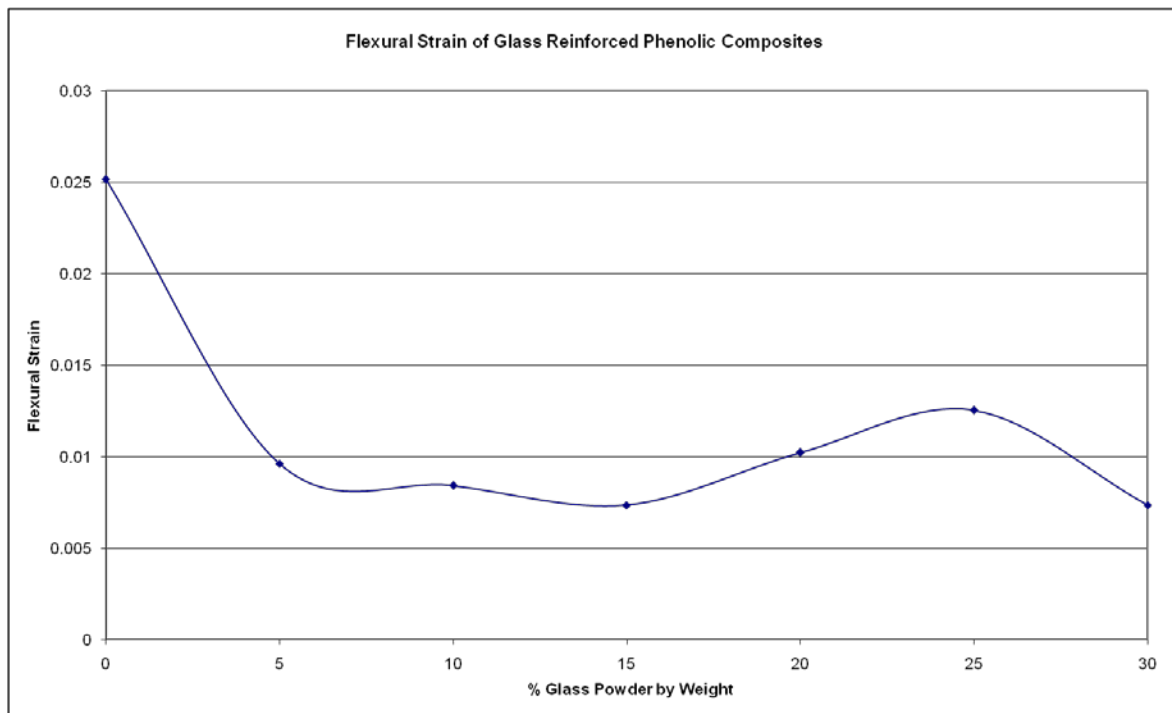
Figure 4.2: Flexural Strain of Glass Reinforced Phenolic Composite

Figure 4.2 shows the changes in the average flexural strain of the glass filled phenolic composite as the glass composition by weight increases. The flexural strain of the phenolic composite is the highest at 0.025 for 0% glass it then drops rapidly to 0.009 for 5% glass; from then on it remains fairly constant with values of 0.008 for 10% and 0.007 for 15%; a slight rise begins at 20% with 0.010 and there is a minor peak at 25% with 0.013 before dropping back to 0.007 at 30%. These results compare well with what was found for flexural strain in the SLG filled resin in the study by Ku. The previous study showed the flexural strain of the SLG filled composite is nearly same as the flexural strain for the glass filled composite for compositions between 10% filler and 30% filler. The plots for both fillers start at about 0.008 for 10% and rise gradually to 0.013 for glass and 0.017 for SLG at 25% before dropping back to 0.007 at 30%.

4.1.3 Flexural Modulus (Young's Modulus in Bending) Results
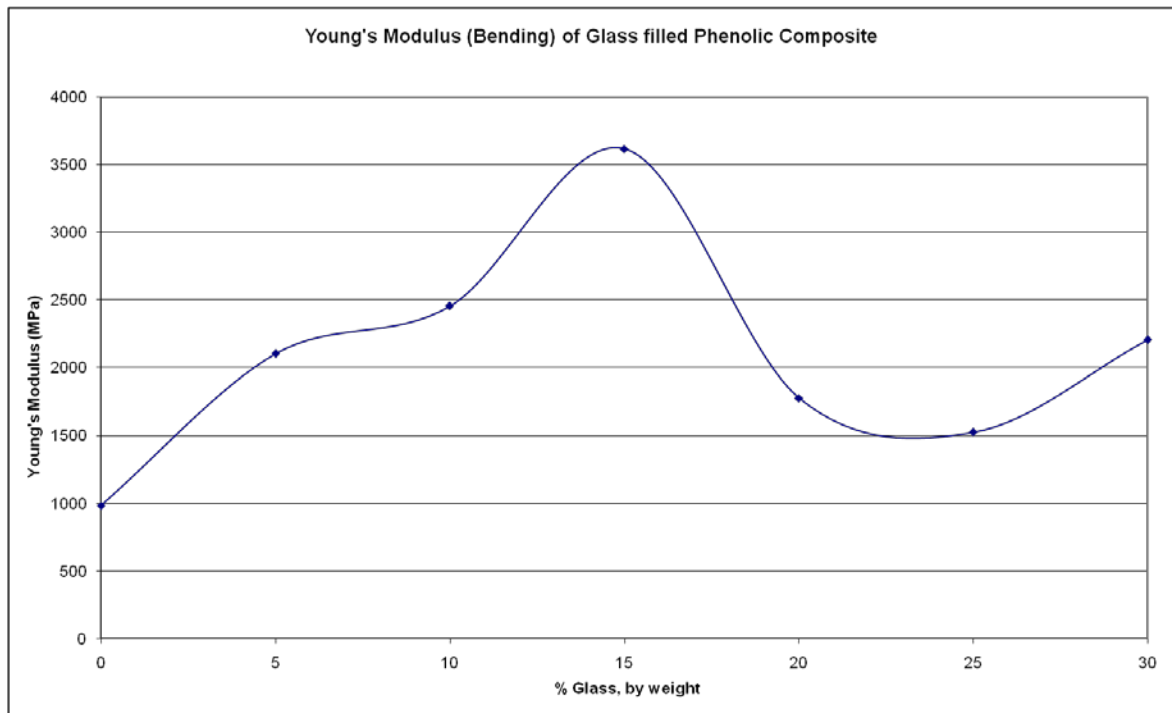
Figure 4.3: Flexural Modulus (Young's Modulus in Bending) of Glass filled Phenolic Composite

Figure 4.3 shows the changes in the average Flexural Modulus of the glass filled phenolic composite as the composition of the glass, by weight, changes. The Flexural Modulus for the neat resin is 982MPa; for a composite with 5% glass it climbs to 2103MPa; for 10% glass it rises further to 2455MPa; at 15% the flexural modulus is at its highest of 3616MPa before dropping away to 1777MPa at 20%; and then on down to 1525MPa for 25%; It then rises to finish at 2205MPa for 30%. The peak in the flexural modulus at 15% glass is due to the peak in the flexural strength at 15%. The low flexural modulus for the neat resin, 0% glass, is due to the high flexural strain in the neat resin. The flexural modulus values here compare well with the study by Ku although the values at same compositions do not match up the general flexural modulus values for both are all just about in the range of 1000MPa to 3000MPa.

5 CONCLUSIONS

5.1 Introduction to Conclusions

This chapter will conclude the dissertation.

5.2 Final Conclusions

From the results obtained in this research it can be concluded that 15% is the best percentage, by weight, of glass filler in phenolic resin according to Three Point Bending Test. The composite with 15% glass is cheaper than the neat resin and has a higher Flexural Strength and Higher Flexural modulus.

5.3 Future Work and Recommendations

Throughout this research the main problems were caused by the fact that often specimens wouldn't cure. Those specimens that did cure usually took at least a week if not several weeks, which is a serious contradiction to the 24hrs or even 72hrs that was successful with previous studies. Most of the previous research has focused on SLG Filler where as this research involved glass powder filler. The glass powder seemed to mix poorly compared to the SLG and also seemed to inhibit the curing process. The resin to catalyst ratios recommended by the SLG research had no chance of curing a glass filled composite. During this research the resin to catalyst ratio was lowered to 12:1 to achieve a cure at 30% but more detailed research into the curing of glass filled specimens is needed.

5.4 Conclusion

This concludes the dissertation the final conclusions have been made and the future work and recommendations have been proposed.

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APPENDIX A: Project Specification

University of Southern Queensland

FACULTY OF ENGINEERING AND SURVEYING

ENG4111/4112 Research Project

PROJECT SPECIFICATION

FOR: ROBERT NIXON

TOPIC: INVESTIGATE THE BEST PERCENTAGE, BY WEIGHT, OF GLASS POWDER AS FILLERS IN PHENOLIC RESIN USING 3 POINT BENDING TEST

SUPERVISOR: Dr. Harry Ku

SPONSORHSIP: USQ, Faculty of Engineering and Surveying

PROJECT AIM: To determine the best percentage, by weight, of glass powder as fillers in phenolic resin using the Three point bending test where the final structural properties of the phenolic resin specimens are tested by subjecting them to tensile, compressive and shear stresses. Each composition will be post-cured for various lengths of time and at numerous temperatures to check for consistency.

PROGRAMME: (**Issue A, 19th March 2008**)

1. Literature review into phenolic resins. Inspect Material Safety Data Sheets to determine safety issues involved with handling the phenolic resin, catalyst and glass powder required to produce test specimen.

2. Familiarisation with resin mixing process and equipment.

3. Mix glass powder filler at different percentages by weight with resin and catalyst and pour into moulds for curing.

4. Post-cure specimens of each composition at various temperatures and for different lengths of time in oven.

5. Test specimens with three point bending test and record results.

6. Analyse results to establish behavioural trends and formulas that can be used for theoretical prediction of filled polymer behaviour.

7. Compare findings with previous research on fillers for phenolic resins.

8. Submit an academic dissertation on the research.

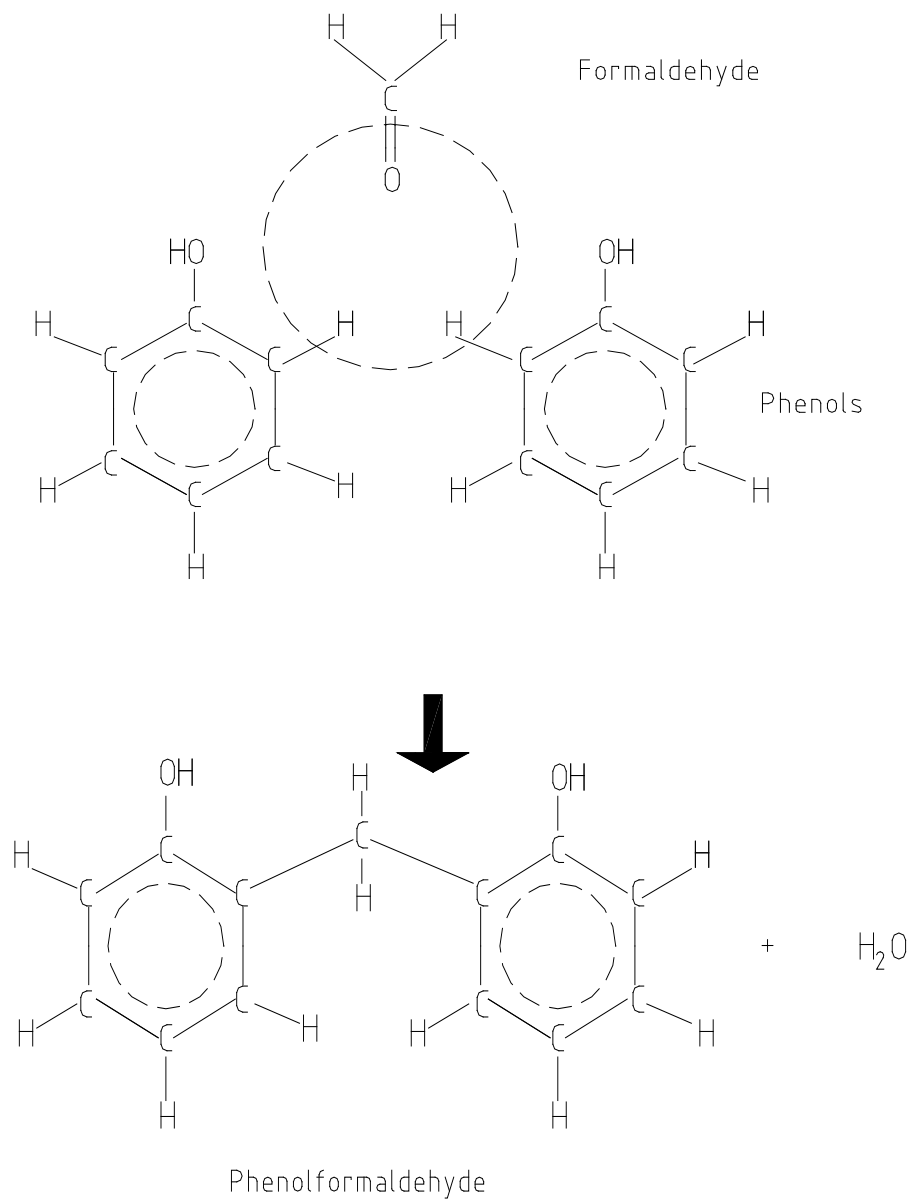AGREED   (student)
(supervisor)

_____   _____

            Date: /   / 2008   Date: /   / 2008

Co-examiner:_____

APPENDIX B: Formation of Phenolformaldehyde

Formaldehyde

Phenols

Phenolformaldehyde

$+$ H_2O

APPENDIX C: Spread Sheet Data

Peak Load							
Percentage	0	5	10	15	20	25	30
specimen 1	52.7	53.2	86.6	67.1	73.9	42.1	33.6
specimen 2	34.2	53.5	79.2	38.1	73.2	23.5	33.6
specimen 3	33.2	57.1	83.9	43.6	73.9	53.7	40.1
specimen 4	25.2	67.1	60.1		59.4	50.2	38.8
specimen 5	34.4	60.4	36.9		67.1	46.7	46.5
specimen 6			87.3		90.6	30.2	39.9
mean load (N)	35.94	20.24	72.3	49.6	73	41.1	38.7
Std	10.11375	5.751782	20.06815	15.40292	10.30717	11.85321	4.821929
Thickness							
Percentage	0	5	10	15	20	25	30
specimen 1		5.28	5.38	6.3	6.41	4.9	4.61
specimen 2		5.45	5.9	5.82	6.66	4.62	4.96
specimen 3		5.26	6.09	4.9	6.14	4.68	4.75
specimen 4		5.3	5.27		6.11	4.5	5.18
specimen 5		5.36	6.1		5.57	4.73	4.51
specimen 6			5.73		5.51	4.88	4.96
Width							
Percentage	0	5	10	15	20	25	30
specimen 1		9.94	9.69	6.3	9.7	9.9	10.02
specimen 2		9.73	10.12	5.82	9.68	9.75	10.02
specimen 3		9.75	9.9	4.9	9.7	9.88	9.92
specimen 4		9.75	10.08		9.68	10	10.17
specimen 5		9.56	9.75		9.61	9.9	9.98
specimen 6			9.95		9.61	9.75	9.95
Flexural stress							
Percentage	0	5	10	15	20	25	30
specimen 1		18.43014	29.64154	25.76156	17.80032	17.00302	15.1475
specimen 2		17.7713	21.583	18.55356	16.3666	10.84053	13.08517
specimen 3		20.32035	21.9363	35.57701	19.40024	23.82302	17.19954
specimen 4		23.52	20.60933		15.77974	23.79852	13.64968
specimen 5		21.11158	9.764125		21.60528	20.24094	21.99078
specimen 6			25.65389		29.81075	12.4863	15.64796
Mean fstress	24.26	20.23067	20.70686	26.63071	18.19044	19.14121	16.21453
Std		2.285507	6.667157	8.544943	5.195019	5.571335	3.228394
D							

Percentage	0	5	10	15	20	25	30
specimen 1		1.06	1.43	0.89	1.41	1.76	0.85
specimen 2		0.97	0.9	0.71	1.14	1.76	0.96
specimen 3		1	0.94	1.09	1.14	2.23	1.03
specimen 4		1.57	1.04		0.8	1.89	0.98
specimen 5		1.56	0.75		1.15	1.51	1.43
specimen 6			1.23		2.05	1.52	0.86
Flexural Strain							
Percentage	0	5	10	15	20	25	30
specimen 1		0.008198	0.01127	0.008213	0.013239	0.012633	0.00574
specimen 2		0.007744	0.007778	0.006053	0.011122	0.011911	0.006975
specimen 3		0.007705	0.008386	0.007824	0.010253	0.015288	0.007167
specimen 4		0.012189	0.008029		0.00716	0.012458	0.007436
specimen 5		0.012248	0.006702		0.009383	0.010462	0.009447
specimen 6			0.010324		0.016546	0.010866	0.006248
Mean fstrain	0.02515	0.009617	0.008433	0.007363	0.010232	0.01255	0.007353
Std		0.010581	0.00171	0.001151	0.003263	0.001711	0.00128
Flexural Modulus							
Percentage	0	5	10	15	20	25	30
	982.1862	2103.645	2455.528	3616.643	1777.882	1525.14	2205.154

APPENDIX D: Load and Elongation Plot from Testing