# **University of Southern Queensland**

Faculty of Engineering and Surveying

# Investigate the best percentage by weight of glass powder, as fillers in phenolic resins by using tensile tests

A dissertation submitted by

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# Abstract

The aim of the project is investigating the best percentage by weight of glass powder, as fillers, in phenolic resins by testing its mechanical properties of the composite material through the tensile testing. Composites are being increasingly used in a wide range of multitude applications such as aerospace, marine, transportation and civil engineering. In this project, glass powder will be used as filler, it not only reduces the cost but gains the advantage of being lightweight, more corrosion resistant, electrical resistant, heat resistant and stronger.

Phenolic formaldehyde thermosetting resin was mixed with an acidic catalyst at ratios from 30:1 up to ratio of 12:1, as well as adding glass powder by weight as filler. Once initial curing has occurred the specimens were subjected to post-curing in a microwave oven where they were baked at 240W until specimens reaches 100 degree temperatures. (30-40mins) In addition, tensile testing was preformed on the post-cured specimens to test for the yield, tensile and young's modulus of each specimen. This will then allow for an analysis to be conducted, identifying what the effects the glass powder has on the phenol formaldehyde matrix composite. In addition, stereo microscope was used to identify at higher magnification what the surface of the composite looks like as well having a closer look at how much porosity had produced. Furthermore investigations were carried out to identify whether the glass powder was able to blend in with the phenolic resin to enable higher mechanical properties than the neat phenolic resin.

Phenol formaldehyde was filled with glass powder to increase the strength and impact toughness of the composite for structural applications. Research was conducted by the centre in the University of Southern Queensland (USQ). For the forward step, the main target of this project is to reduce the price and test its physical and chemical properties before moving to the industry production. It was found that the best percentage of glass powder by weight which added to the phenolic resin to give an optimum yield and tensile strengths as well as Young modulus and cost is about 10 percent. The contribution of the study was that if tensile properties were the utmost important factors to be considered in the applications of the composites, glass powder is not a suitable filler.

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# **1** Introduction

### **1.1 Introduction**

This chapter will briefly outline the purposes of this research project. This project mainly focuses on investigating the best percentage by weight of glass powder in phenolic resin and using fast post-curing (Microwave oven) through the tensile tests.

# 1.2 Research and Development Work

Glass powder as filler in the phenolic resin composite is not well known and applied to today's society, and there are not many resources about the strength it can add to the phenolic resin mixture. Tensile testing will be conducted to analyze the effects that the glass powder has on the mechanical properties of the phenolic resin composite mixture. The tensile testing will be used as a testing method to find the Young's modulus of elasticity of each specimen as well as the yield and tensile strength. From these test the stress strain graph will be produced where conclusions can obtain the difference that has occurred in the newly formed composite. This paper will go through a series of experiments using different ratios and percentages to the glass powder in the preexisting formulas of the phenolic resin and catalyst mixture to indicate whether glass powder is a viable option to consider when making a phenolic resin composite. Once this is established, more tests will be conducted on the make of the specimen. The phenolic glass composite specimens will be cured and tested. The specimens will consist of three materials, Phenolic Formaldehyde Resin, (Hexion Cellobond J2027L) Phenolic Resin hardener Catalyst (Hexion Phencat 15) and Glass powder (Sphericel Hollow Glass Spheres). This will be mixed at a ratio of 12:1 to 30:1 Resin to Catalyst, depending on the percentage by weight of glass powder. A mixture of 5% intervals of glass powder by weight will be added to the Resin Catalyst mixture. This will be conducted from 0 - 30% glass powder ratio. Once the specimens are made, cured and tested results will be analyzed, to find out the optimum percentage by weight of glass powder to use in the composite mixture to achieve high stronger material properties.

#### **1.3** Composite Materials

Composite materials are a combination of two or more separate substances. A common example of composite materials within the engineering sector is Fibre-glass, concrete, rubber tyres as well as reinforced plastics. A composite material takes advantage of the different material and mechanical strengths and abilities of different materials. The main characteristic of a composite material is it allows materials to be mixed together and cross link each other in a chemical, and still be apparent in the way they sustain their strong material properties as well as captivating the other materials strengths. A general example of composite that originated hundred of years ago is mud and straw bricks. On its own mud characteristics are, a good binding agent but has poor compression force strength, which will allow to bricks to deform. With the addition of another material such as straw,

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which has high compression strength allows to mix and bond with the mud and form a strong bond. The straw acts a reinforcing material similar to how concrete and reinforcement bars work in all buildings being made today.

### **1.4 Phenolic resins**

Phenolic was one of the earliest wholly synthetic polymers to be discovered. It came about the years of study of natural polymers. A polymer is a solid material which contains many chemically bonded parts which form a solid. Some of the earliest records, pre 1800, found that natural polymers for example wool, leather, cotton and rubber can be altered to form a plastic like material. An inventor by the name of Leo Hendrik Bakeland was the first person to form a non-natural polymer (synthetic), which was later called Bakelite in 1909 (Shackelford, 1992). This was the make of phenol and formaldehyde. This later lead scientist across the world to base more experiments using similar non-natural materials to identify other plastic synthetic polymers (Strong, 2000). Phenolic thermoset material is still, among the most highly used material due to its low cost on a cost-per-volume basis (Ku et al, 2007). All synthetic plastics are then divided into two groups' thermoplastics and thermosets, depending on the chemical bonding. Thermoplastics are generally used where softness, flexibility, and resilience are exhibited. Some actual examples are footwear, wire insulation, adhesives, polymer blending Thermosets are either a solid or liquid at room temperature but once heated the resin will set and cure and cannot be melted to be reshaped (Strong, 2000). Some general examples of a thermoset composite being used is in the maritime industry on boats, in air crafts for various assorted devices Student ID: 0050044877

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and most regularly use in components in the electrical industry. The process that creates the thermosets is formed by condensation of polymerization (Smith, 2000). During the progression being carried out via two different conditions, the result in two different intermediate materials being produced, novolacs and the other resoles. The one that occurs depend on the mole ratio of the formaldehyde to phenolic and catalyst mixture.

## 1.5 Glass powder

Glass powder has been used in industry manufactory for a long time since micro balloons or micro spheres have been used as a filler material in many fields. In the construction industry is can be used to fill gaps, which can also dampen sound or vibrations that the building may have as well. Glass micro balloons are also currently being used in the medical area as a source to be used as a skin replacement for burn victims.

Glass powder is used to enhance the performance and reduce viscosity in paints, coatings and as a lightweight additive in plastic parts. As well as having above average material characteristics, glass powder is high heat resistance along with pressure and impact resistance. Accordingly, it is widely used for weight saving of resin molding components for e.g. Automobiles, portable electronic equipment, and buoyancy material for ships, as well as aircraft and in the medical sector for skin replacement (Patent Storm, 2008).

### 1.6 Knowledge relative to this project

The dissertation will allow for future references to be available and allow for this to be presented as previous knowledge on glass powder being used within the phenolic resin mixture as well as providing mechanical testing result to reiterate the findings. This dissertation will draw conclusions on the results obtained from tensile testing of the glass powder phenolic resin composite and show the effects that are had on the mechanical properties of the mixture via tensile strength, yield strength and young's modulus of elasticity.

### 1.7 Summary of Chapters

In Chapter 2, some more journal articles will be reviewed to towards a more in-depth literature review into composite materials (phenolic resin) and some other components used in the final make-up of the phenolic resin glass mixture.

Chapter 3 will mainly explain and develop the methodology of how to prepare the samples, safety issues, testing its properties as well as using method to determine the methods and calculation.

Chapter 4 begins evaluating and discussing the results that were found by the experiments and then recommendations will be made on the viability of the testing. Each of the three mechanical tests conducted will be analyzed individually, then lead into a discussion on what the results mean to industry. Then analysis will be done to compare these results to Student ID: 0050044877 Page Number 13 Student Name: Chaodi, WU other research study results and using excel and mat-lab to analyse the results.

Chapter 5 is the final chapter in this dissertation where the results that have been obtained will be, briefly summarized and recommendations will be drawn so later references can be used for study and experimental purposes.

# **2 Literature Review**

### 2. 1 Introduction

In this literature review chapter, composite materials in particular phenol formaldehyde resin as well as other components that will be used in the final composition of the new glass powder reinforced phenolic resin composite will be analysed and focused. Plastics once modified by natural polymers can form other forms of plastic. A specific example of the modification of the polymers is phenolic resins. It has been used within a wide range of industries for several different applications. Phenolic resins are widely used in manufacturing to form common plastics that make up everyday appliances. Phenolic resins are very versatile and are currently being used in making electronic appliances, floatation devices on ships and as an adhesive agent that is used to join commercially available plywood together. With the ever increasing demand for the phenolic resin, grows the need for a cheaper alternative of phenolic resins to be manufactured. As this demand increases, the need of additional consideration from engineers and technical staff on the possibility of using a filler material increases. The filler could be combined with the phenolic resin to form an innovative glass powder reinforce resin composite that not only is a cheaper alternative but can also have advantages of improved mechanical properties.

#### 2. 2 Introduction to Composite Materials

Composite materials have become a common part of engineering with the need to have a lighter and stronger alternative compared to the more conventional materials. The most primitive composite materials were straw and mud combined to form bricks for building construction. Composites are made up of individual materials referred to as constituent materials. There are two categories of constituent materials: matrix and reinforcement. One common example of composite material is concrete, cement concatenate, sand, water and aggregate all have individual properties and does not a ideal material for support structural but when combined together, it perfectly matching the requirements as a construction material. (Composite material). Other examples of composites within the engineering sector are rubber types, as well as reinforced plastics. Composite materials are defined as a blend or combination of two or more separate materials. The main characteristic of a composite material is it allows materials to be mixed together, yet still be apparent in the way the material will sustain its material properties as well as captivating the other materials strengths. This then leads to a better a composite material than the two materials individually (Smith 2008).

#### 2. 3 Phenolic Resin

Phenol-formaldehyde or phenolic resin thermoset is the make-up of thermoset material of at least two main ingredients, of which are: the resin and its hardener. The fillers, that are included in the phenolic resins, can be reinforcing materials which can be organic or inorganic. Such organic polymers can range from wood flour, glass and cellulose (Smith, 2000). Organic polymer fillers are mostly carbon-based whereas inorganic polymers are glass, silicone, magnesium carbonate, calcium silicate (Kawai et al, 1988).

Phenolic was one of the earliest wholly synthetic polymers to be discovered. This came after years of study of natural polymers. A polymer is a solid material that contains many chemically bonded parts. Some of the earliest records, pre 1800, found that natural polymers for example wool, leather, cotton and rubber can be altered to form a plastic like material. From here the research began to improve and in 1868 John Wesley Hyatt discovered the first plastic. Celluloid was called the first ever plastic as Hyatt had substantially modified the natural polymer and then allowed the new mixture to be cured into a mould forming new shapes (Smith, 2000). Once this development was discovered scientist began to add other natural polymers in the hope to discover a new composite material. An inventor by the name of Leo Hendrik Bakeland was the first person to form a non-natural polymer (synthetic), which was later called Bakelite in 1909. This was the make of phenol and formaldehyde. This led scientists across the world to base more experiments using similar non-natural materials to identify other plastic synthetic polymers (Strong, 2000). Phenolic thermoset material is still, among the most highly used material due to its low cost on a cost-per-volume basis (Ku et al, 2007).

Whilst the resin is subjected to a flame, it will only char rather then catch fire or melt. Phenolic resin is therefore widely applied in situations where a low flammability is required, for e.g. on appliances. The neat resin has darkened pigments which are Student ID: 0050044877 Page Number 17 Student Name: Chaodi, WU ingrained into the resin and limits the use of the resin in certain instances. To overcome this, a dark pigment is then added to homogenize the colour of the resin which also diminishes the UV light sensitivity (Ku, 2009). Once the resin is mixed with another material it is proven that the mechanical properties of the Resin will increase, which properties increase depends on the special properties that the other material contains. For example if graphite fiber is added to the phenolic composite it has proven to increase the tensile strength of the harden material by 10 times of that of the unreinforced phenolic resin (Wang et al, 1997). All synthetic plastics are then divided into two groups' thermoplastics and thermosets, depending on the chemical bonding.

#### 2. 3. 1 Thermoplastic

Thermoplastic elastomers were first introduced in the 1960's. Thermoplastics are commonly used plastic which when the polymers are at room temperature it's a solid but once exposed to high temperatures is able to be melted down to be re-solidified. The main difference between thermoplastic and thermosetting polymers is thermoplastic are not involved in chemical reactions that cross-link materials and become "set", i.e., they can no longer flow or be dissolved. At room temperature, thermoplastic elastomers, crosslinks occur by tying the elastomers chains together in to a three dimensional network. Once heated the strength of the material is lost or dissolved in solvents, when the material cools the solvent evaporates and the integrity of the original network begins to reform and becomes hard again. The addition of a solvent allows the polymer solution to flow easily.

Some common examples of thermoplastic are in areas where high temperatures are not a Student ID: 0050044877 Page Number 18 Student Name: Chaodi, WU major factor in the working conditions of the component, due to the fact that a thermoplastic material can be melted when exposed to high temperatures and then re-solidified. Thermoplastic materials are generally used where softness, flexibility, and resilience are exhibited. Some actual examples are footwear, wire insulation, adhesives, polymer blending (Wiley, 2002).

#### 2. 3. 2 Thermoset

Thermoset plastics can be either solid or liquid at room temperature but once heated the resin will set and cure and cannot be melted to be reshaped (Strong, 2000). The process that creates the thermosets is formed by condensation of polymerization (Smith, 2000). During this process being undertaken under two different conditions, two different intermediate materials are produced, novolacs and the other resoles. Which one occurs, depends on the mole ratio of the formaldehyde to phenolic and catalyst mixture. Resole resin is produced when the mole ratio of formaldehyde to phenol and catalyst is greater than 1. An example of the resole resin used today is Hexion Cellobond J2027 a commercially available resin. The other intermediate material that can form via the condensation of polymerization is novolac. Novolac is the opposite to resole, as a novolac has the composition of excess phenol so the mole ratio of formaldehyde to phenol is less than 1. For example if one mole of phenol was to react with formaldehyde to form a novolac only 0.7 to 0.85 of a mole would be used, where as for a resole the formaldehyde would be increased to above 1 with phenol remaining at 1.

Once the phenolic resin is allowed to be cured a process called chemical crosslinking

occurs. Crosslinking is the process of where the polymer molecules react together and join a three dimensional network. This only occurs by a reaction of the novolac with hexamethylenetetramine (hexa), a basic catalyst. When pressure and heat are applied to the hexa-containing novolac resin, it produces ammonia which then provides methylene. This then starts the crosslinking process and once the hexa material is added they call it a two-stage novolac system. During the formation of the intermediate material results in the development of water a by-product? This causes whilst curing an extensive microvoiding in the phenolic matrix. This has little effect on the composite properties, but due to the high water absorption it can cause structures to delaminate when exposed to high temperatures. Unlike a thermoplastic material once the crosslink of the polymers have occurred in a thermoset material and exposed to high temperatures the material will only soften, but will remain the general shape until much higher temperatures are applied then decomposition will occur (Wiley, 2002; Strong, 2000; Ku et al, 2009 Smith and Hashemir, 2000 Shackelford, 1992).

#### 2. 4 Glass Powder

Glass microspheres have been used in various kinds of polymeric compounds since the mid-1960. Once the idea of using glass was introduced it was found to be unfavorable as results show that it did not have great enough mechanical properties needed for the high shear forces and pressures involved with injection molding. It was not to till the late 1980's that glass spheres were commonly used as filler into the polymeric/ phenolic resin compound mixtures where injection molding was used. Injection molding is a common Student ID: 0050044877 Page Number 20 Student Name: Chaodi, WU 2000

manufacturing process that is used for shaping plastic resins. Most plastic components are formed by this process. The process starts, where the plastic resin is fed into a hopper of a machine then it is forced through an injector barrel where it will be heated. Once it has achieved a certain temperature the heated resin is forced through a nozzle and into a mould, where it later cools and will allowed to cure into desired shape (Chemical Engineering Tools, 2008). Over the period of 20 years it was discovered that the size and shaping of the glass played a large part in the process. Once this was discovered many companies over a variety of industries swapped over to microspheres as they had a more consistent sizing and shapes than what was previously available on the market. Having irregularly shaped particles due to non-uniform shrinkage can lead to misalignment. By contrast, spherical particles respond evenly to shrinkage, so the part does not warp (Potters Industries, 2008). A common method used to produce hollow glass microspheres is by mixing sodium sulfate with sodium borosilicate glass. This mixture is then dropped into a hot flame that melts the powdered glass and sodium sulfate. Once the sodium sulfate is melted the results from the decomposition reaction, is released in a minute amount that will form bubbles within the molten glass droplets. The hollow droplets are then rapidly cooled from the liquid state to form the hollow glass spheres (Patent Storm, 2009).

The glass powder is used in industry 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. With the addition of hollow spheres to fiberglass reinforced plastics (FRP), epoxy, compounds, and urethane castings can provide weight reduction cost savings and improved impact resistance. Insulating features Student ID: 0050044877 Page Number 21 Student Name: Chaodi, WU

of hollow spheres also work to the chemists' advantage in thermal shock and heat transfer areas. (Potters Industries, 2008) According to William Shaker, Market Development of Polymer Additives at Potter Industries, one conventional additive can match the performance benefits of the glass spheres. The advantage of the spherical hollow glass spheres is its low density of between 0.6-1.1g/cc small particle size and lightweight. With particle sizes considerably finer than previously available, SPHERICEL® hollow microspheres can be used in thin film coatings to improve integrity. "Because glass spheres do not absorb resin, more resin is available to create the film," explains Shaker (Potters Industries, 2008). "The result is a tighter and more uniform film with improved durability, even under adverse conditions" (ThomasNet, 2005). Also Potters Industries offer SPHERIGLASS® solid glass spheres. This is a denser alternative to the hollow glass spheres used in this research. In having a higher density than the hollow spheres it allows it to be used where much higher strengths are needed. For the scope of this research only hollow glass spheres will be used during experiments.

#### 2. 5 Materials Selection

In this research the catalyst (Hexion Phencat 15) was chosen as it is a fast action acid catalyst. This will allow for the glass powder and the phenolic resin to react much faster than a slow acting catalyst. Once the chemical reaction has begun the composite compound can start to cure. Using fast acting catalysts will cut down on the time taken to fully cure the specimens. Phencat 15, in most cases used to cure surface pastes, which will be ideal for conducting experiments in the thin moulds that will be used. Also a

surface paste is used when high temperatures are involved. (Super shine, 2008)

#### 2. 5. 1 Catalysts

The reaction between the Phencat 15 catalysts and the phenolic resins can be strongly exothermic (Munoz et al, 2007). The catalyst is toxic, so skin contact of the plane catalyst can cause burns. Munoz found that Phencat 15 catalyst when mixed; whilst the ratio of catalyst to resin is too high (ratio greater than 10:1) experiments had to be halted as the mixture did become exothermic and become quite hot.

#### 2. 5. 2 Phenolic Resin

A general Cellobond J2027L Phenolic resin was chosen to be used in the composite mixture. This resin has a low viscosity which makes it ideal for use in manufacturing where injection molding is used. Due to the low viscosity it will be a good choice as it will be easy to pour and manage in the molding stage.

#### 2. 5. 3 Glass powder

For the glass filler, Potters Industries SPHERICEL® 60P18 (spherical) hollow glass spheres was chosen. The hollow glass spheres were chosen instead of the alternative solid Student ID: 0050044877 Page Number 23 Student Name: Chaodi, WU glass spheres that potters industries also offers. The hollow spheres were a better option as the focus of this research is to investigate into the use of glass as a filler, not only to enhance the mechanical characteristics of the phenolic resin mixture but also allow for a cheaper alternative, than the pre-existing composites currently available. In addition, the hollow spheres have a lower density than the solid spheres which will allow for a lighter but still strong composite to be created (Potters Industries, 2008)

# 2. 5. 4 Application

Composite materials always contain higher properties and lower weight in whatever structural field or new technology applications. For Phenolic Resin and glass powder combination, it will produce a high tensile strength and with few resistant in fire, electrical and water.

Therefore, this kind of marital further can be used as a covering or structural marital as the combination improves or glass powder has changed to other strange marital.

# 2. 5. 5 Cost Analysis

As the glass powder has been used in project as filler, the aim of this is to reduce the total cost of the composites. Glass powder which is lower then other filler materials at a cost of \$3 per kilogram, whereas currently the price of the glass powder which to be used in this study is \$5 per kilogram. For phenolic resin costs approximately \$6.50 per kilogram and Student ID: 0050044877 Page Number 24 Student Name: Chaodi, WU

the catalysts costs \$8.00 per kilogram.

# **3 Methodology**

## 3.1 Introduction

This chapter will identify the working process, important hits, safety issues and different calculations in the research project. Project methodology allows for planning of project as well setting out the requirements of the study in the limited amount of time allocated. When setting out a timeline, adequate time must be given to each task to be conducted. Some task for instance, the making of the specimens will take longer than to post-cure the specimens in the oven. This then needs to be taken into consideration. Firstly an analysis should be conducted on the resources that are required to perform the experiments with, as well as potential quantities that may be required. As the proportion of the phenolic resin and catalyst varies this will require different amounts of the resin so extra resin may have to be prearranged to keep on track with the set out timeline.

### 3.2 Resource Analysis

To carry out this project there are several main resources need be noticed and together with the availability of required laborites and laboratory staff for consultation and direction, about how to use the microwave oven and tensile testing apparatus. The following equipment will be required for this experiment:

Safety equipment (glasses, gloves, full covered footwear, breathing mask)

Plastic molds (six specimens can be made per mold)

Metal bolts with wing nuts (to hold the two layers of the mould tightly together to minimize seepage)

Phenolic Resin

Catalyst

**Glass** Powder

Sheet of glass (prevent material landing on curing specimens)

Cooking oil/wax (to line the mould prior to casting of the composite to allow specimen not to stick to the mould, an easy removal once cured.)

As similar studies have been conducted by the University of Southern Queensland and the Center of Excellence in Engineered Fibre Composites (CEEFC) the phenolic resin and catalyst are already available. The moulds are safety equipment and metal bolts are all reusable items throughout the entirety of the project. These items, equipments and assistant help were supported by the Engineering faculty. The glass powder will have to Student ID: 0050044877 Page Number 26 Student Name: Chaodi, WU

be ordered because it has not been used in previous studies conducted by the University. The cooking oil/wax and the safety gloves (latex) are items required which are not able to be reused; the quantity of these items will have to be monitored, which was throughout the experiment to ensure they do not run out and delay the already rigid timeframe of the project.

The laborites where the practical side of the project will be conducted are:

Laboratory Z106.1 (molds are cast)

Z116.1 (post-curing Microwave oven)

Z104.1 (tensile testing machine)

QUT (Stereo Microscope)

When experiments take place, these three laboratories which are used often and regularly available, laboratory supervisors are notified. The Lab supervisor can also be of assistance especially in the oven and testing labs since they are experienced with how the machines operated. If without lab supervisor's the specimens may be damaged and will have to be remade adding extra time on an already tight time schedule. If the oven or the tensile testing apparatus were to break, this situation would carry out the project to a stand still, as time would have to have been reallocated to allow for the repair of the machine. Now, the university has only one oven and tensile apparatus, that means, time would have to be spent waiting for parts to repair either device if a problem did occur with either machine. For this reason care not only will be taken to prevent the machines from being used outside the limits defined in this project guideline, but also consultation with the Lab supervisor to ensure the machines parameters are not breached. As soon as all the resources have been established a start can be made on the experiments and safety precautions needed.

## 3.3 Safety Analysis

Safety plays a major part in any situation or task being undertaken. The tasks have some difference, so do the risk and safety precautions that need to be taken to ensure everyone's safety. When entering a workplace for the first time, a risk assessment should be carried out to ensure ones safety also the others safety. Entirely through the duration of the experiments the rules and regulations which were followed that are in the Workplace Health and Safety Act 1995, also the recently amended Workplace Health and Safety Regulations 2008. There is a 5 steps risk assessment guidelines to adhere when in the Workplace Health and Safety Act 1995.

Looking for hazards

Evaluate the risks,

Student ID: 0050044877 Student Name: Chaodi, WU Asses' risks,

Implement plan to minimize risks,

Re-examine to classify if the plan works effectively.

A work permit under Workplace Health and Safety Act 1995 was issued for the laboratories required in the experiments, but also material safety data sheets for the three components to be used in the composite mixture. (Queensland Government, 2008) The steps will be evaluated and applied to this study.

#### 3.3.1 Identification of risk factors

There are many risks to be aware of during the execution of this research project. There are three processes which are involved in conducting the experiments for this project. Firstly, the making of the specimens, secondly, post-curing the specimens in a microwave oven and thirdly conducting tensile test on the post-cured specimens. Care needs to be taken when measuring ingredients to ensure accuracy and when handling the materials in creating composites to ensure safety regulations are adhered too.

The specimens post-curing in a microwave oven is another risk that needs to be considered. The oven reaches high enough temperatures, which leads to moderate burns and even possibly severe ones. The third process being conducted is testing the post-cured specimens with the tensile testing apparatus. As with any machine with moving parts, it has the opportunity of being caught. The tensile testing apparatus has moving parts in testing the specimens. Caution needs to be taken in order that the moving parts of the apparatus do not get caught on it. A preventable measure is a surrounding area to be outside whilst machine is operating.

#### **3.3.2 Evaluation**

While having to handle and work with chemicals in and around the laboratory the safety rules and guidelines of personal safety equipment are adhered to, as well as to make sure risks are minimized. The three materials are being mixed together, which is requiring the correct quantities for the specific batch. It may result in the composite becoming unstable and possibly exothermic, leading the mixture to heat up significantly if the quantities is changed or miscalculated.

The three components/materials which used for creating the composite are Phenolic Resin, Catalyst and Glass Powder. The risk connected with each component will be evaluated.

Phenolic Resin "Phenol formaldehyde" Hexion Cellobond J2027L (Chemwatch, 2005a) Risks associated with using the Phenolic Resin

Toxic by inhalation

Toxic in contact with skin and if swallowed

Causes Burns to skin Student ID: 0050044877 Student Name: Chaodi, WU Limited evidence of a carcinogenic effect

Catalyst Hexion Phencat 15\* (Chemwatch, 2005b) (limited evidence) "Phenolic resin hardener catalyst"

Risks associated with using the Catalyst

Harmful by inhalation and if swallowed

Causes burns

Cumulative effects may result following exposure \*

Possible respiratory sensitiser\*

Possible cancer-causing agent following exposure

Glass Powder Spherical Hollow Glass Spheres (Potters Industries INC) "Glass oxide Powder."

Risk associated with using the Glass Powder

Glass Powder dust in excess of recommended exposure limits may result in irritation of the respiratory tract.

Local exhaust fan is recommended for operations that generate nuisance dust in excess Chronic lung conditions may be aggravated by exposure to high concentrations of dust Use NIOSH approved dust mask or respirator where airborne dust is generated to prevent inhaling the dust

NIOSH approved safety glasses or goggles

All three materials appear to have a certain degree of dangerous risk involved. The risks are minimized if care is taken and personal safety equipment is worn. Exposure to the ingredients which is a minimal risk, due to the Exposed period to the materials is about 15-20 minutes whilst the materials are being mixed together, as soon as initial curing in the moulds has commenced the risk of the ingredients begins to decrease.

If the user is not careful when post-curing in the oven commences the possibility of burns to the arms or any other part of the body increases and may result in some degree of burns.

Each specimen will have tensile testing on them using a tensile testing machine, after the specimens have gone through post-curing in the oven. Mistreatment of the tensile machine can cause physical harm. If a body part was caught within the machine, it has safety shields and a kill switch in place in case of an emergency. Before using the

machine, it will have to be studied so the position of the safety switch is known. Until the action it is undergoing has completed, whilst the machine is operating the user should have all safety shields in place and be standing away from the apparatus.

#### **3.3.3 Control Environment**

At first, all laboratories have compulsory protective safety equipment that everyone who enters the laboratory must abide. For instance, wear eye protection, covered footwear. Breathing masks should be worn when making the moulds latex gloves. To avoid any harm occurring to the user literatures are provided focusing on the materials which detail, all the hazards and risks that each ingredient/machine have. Before entering the lab and handling the ingredients, the literature should be read and understood thoroughly. Whether it is a spill on the workbench or on another user, having the literature also allows the user to read safety procedures in case of an emergency. Minimizing and controlling the risks on the oven as well as the tensile testing apparatus warnings signs and marked safe areas for the operator to stand whilst the devices are in use. Laboratory assistant will help the machinist in identifying key safety issues and the warning signs to be aware of whilst operating the devices, to make sure there safety is guaranteed.

### 3.4 Preparation of moulds and ingredients

First task undertaken was to locate the laboratory and equipment needed in the project and to become familiar with safety precautions that each part need. Obtaining literature on the ingredients so good backgrounds on the ingredients which will be used throughout the experiment are obtained. These ingredients are the Catalyst (Hexion Phencat 15) the Student ID: 0050044877 Page Number 33 Student Name: Chaodi, WU Phenolic resin (Hexion Cellobond J2027L) and the Glass powder (60P18 Sphericel Hollow Glass Spheres).



Figure 3-1 Moulds prepared for use

Figure 3-1 is the moulds which used in this research project and once they have been clear of remaining composite, then it is lubricated ready to be used again. To prepare the moulds much consideration and preparation is needed to have the moulds ready for use. Otherwise removal of the specimen could be relatively difficult, when cleaning and preparing the moulds any slight left over resin or groves left on the mould needs to be removed. As soon as the moulds are cleaned they are then sprayed with Canola oil to prevent the composite sticking to the moulds.

#### 3.5 Moulding

Moulding happens once the moulds have been prepared and cleaned thoroughly. The first mould specimens, which were round, were placed at a ratio of 30:1 resin to catalyst. Then, this was allowed to cure at room temperature. This experiment has not been conducted using the glass powder and filler in the phenolic resin mixture, this ratio will be a trial. If Student ID: 0050044877 Page Number 34 Student Name: Chaodi, WU



the specimens did not cure suitably, the ratio may have to be altered at a later stage.

Figure 3-2 illustrates the glass being added to phenolic resin

Once the glass powder have been added to the mixture which as shown in Figure 3-2. During the mixture, it has to been slow and consistent to avoid the mixture getting large amounts of air bubbles. When the specimen cures and is removed from the moulds and is able to continue on to be post-cured then tested the porosity, (air bubbles) it will most likely alter the results found in the tensile tests especially testing for porosity, if too much air is captured in the mixture. It can be removed from the moulds when the specimens have cured and getting hardened in the moulds. It is possible that the specimens may not be fully cured or still plastic, then it may warp or deformed under its weight, when whilst removing the specimens. The deformed shape will be tested at a later stage and the results will possibly give a false reading which could result in lower mechanical properties for the specimen. As it clamps the material before conducting the tensile tests, this will be cause preloading of the specimen by the clamps on the tensile testing apparatus.



Figure 3-3 Homogeneous Mixture


**Figure 3-4 Finished moulds** 

Once the glass powder, phenolic resin and catalysts are blended together and the mixture has a homogenous mixture which it is softly and slowly placed into the molds. It will helps to reduce chance of getting air bubbles. Figure 3-3 indicates what a homogenous mixture looks like. The molds comprise of an upper and lower plate. The plates are illustrated in Figure 3-1. They are clamped together with wing nuts by 9 bolts; all bolts are then tightened to make sure they are tight and hopefully minimize seepage of the composite between the plates. As soon as the composite has been carefully poured into the mold. Figure 3-4 illustrates what the finished mould looks like once the mould has been prepared and all bolts secured the composite is carefully poured in.

It will be placed in the oven to be post-cured hopefully without blemishes or porosity, after the composite mixture has had time to cure in the moulds and are able to be removed. Student ID: 0050044877 Page Number 37 Student Name: Chaodi, WU University of Southern Queensland

When the specimens are placed in the oven they are placed at a series of temperature and time parameters:

## Around 30-40minutes post curing in microwave oven





Figure 3-5 shows the microwave oven that was used to post-cure the phenolic resin, glass powder composite. Once they have been cured in the oven the post-cured specimens will be cooled and then placed through a tensile test to determine the mechanical properties and the strength of each specimen. Issues are the possibility of the specimens warp or deform whilst being baked in the oven, which may occur during the post-curing process.

It is significant that the composite while curing remain straight and flat, when the mechanical testing that will used to test the specimens is tensile testing to identify the Student ID: 0050044877 Page Number 38 Student Name: Chaodi, WU

mechanical characteristics. To make sure that the specimens remain straight as soon as successfully removed from the moulds they will be placed between two pieces of glass. As well as to maintain the cured specimens flat and straight when they are post-cured in the oven the specimens will be subjected to approximately load, which will be applied during the baking of the specimens.



# **3.6 Tensile Tests**

Figure 3-6 Tensile testing machine

Before the tensile test can be conducted all specimens need to be measured and recorded

Student ID: 0050044877 Student Name: Chaodi, WU into the data logger on the computer that runs the program used by the tensile testing machine. Figure 3-6 shows the tensile testing machine that was used to test the glass powder reinforced phenolic resin composites. As soon as the data is logged the computer can test the specimens, and then, interrupt the results into the stress-strain graph. It is clear from the graph given that percentage by weight of glass powder had the greatest improved material/mechanical properties. As well as the Young's Modulus of Elasticity for each of the phenol formaldehyde, glass powder composite specimens, Reading off the stress-strain graph the tensile and yield strength can be determined. Before the specimen can be tested the cross-sectional area needs to be calculated, also taking care in placing the specimen into the machine, setting the distance between the jaws of the apparatus to the same for each specimen (gauge length). This is to eliminate a variable as well as allowing the computer to calculate the yield and tensile strength along with the Young's modulus.

## **3.6.1** Tensile Strength

The most common and sought after property of a material is the tensile strength of a material. Tensile test is easy to resolve as well as being of valuable for the purposes of specifications and quality control for manufactured goods. In tensile tests the force and extension of the specimens are recorded by a data logger. Figure 2 shows a typical example of a stress-strain graph which illustrates the tensile force versus tensile elongation for a specimen undergoing the test.

To calculate the tensile strength, divide the maximum load by the original cross-sectional Student ID: 0050044877 Page Number 40 Student Name: Chaodi, WU area of the specimen. (Wang, 2007)

$$Tensile strength = \frac{Maximum \ load}{Original \ cross - sec \ tional \ area}$$
(1)

or 
$$\sigma = \frac{P_{\max}}{A_o}$$
(2)

where P max is the maximum load in Newton and  $A_0$  is the original cross-sectional area in  $mm^2$ .

For instance, the tensile strength of a sample of how to apply the above equations are illustrated in table 1 in Appendix B

The tensile strength for this example is 796/(5.1\*14.7) = 10.83MPa

#### 3.6.2 Yield Strength

Yield strength is the point at which a definite amount of plastic strain has occurred. This is usually 0.2% of the proof load. For certain materials including phenolic resins a value of 0.05% of the proof load is used to identify an approximate value of the yield point. This is lead to phenolic resin being more brittle than steels and other more ductile Student ID: 0050044877 Page Number 41 Student Name: Chaodi, WU

materials.

By drawing a line in x, y direction the point where it crosses the theoretical values of the stress-strain curve is the proof stress or offset yield strength.

Yield strength is calculated using the relationship below (Wang, 2007)

 $Yield strength = \frac{Yield \ load}{Original \ cross - sec \ tional \ area}$ 

For example, yield strength of the sample illustrated in Figure 1

 $\frac{0.05\% \text{ offset load}}{Original \text{ cross} - \sec tional \text{ area}} = \frac{925}{14.9x5.0} = 12.50(MPa)$ 

The yield strength for this example is 12.50MPa

## 3.6.3 Young's Modulus of Elasticity

Young's Modulus of Elasticity is a measure of how brittle a material is. It is calculated from the previously found stress-strain graph of the material by finding the gradient of the initial linear portion. When the force-extension exhibit a prefect linear relationship, the Young's modulus calculated is the secant modulus at a strain 0.1 percent. To calculate Young's Modulus the following equations are used.

Stress 
$$(\sigma)$$
  $\sigma = \frac{F}{A_o}$ 

Strain (
$$\varepsilon$$
)  $\varepsilon = \frac{\Delta L}{L_o}$ 

Young's Modulus	$F = \frac{stress}{\sigma} = \frac{\sigma}{\sigma}$
Total S Modulus	Σ strain ε
of Elasticity (E)	

$$\mathbf{E} = \frac{\frac{F}{A_o}}{\frac{\Delta L}{L_o}}$$

Where  $\Delta L$  is the change in length of the material, F is the force place on the specimen by the apparatus during testing, subscript, denotes original measurement before testing was conducted. To identify how this is conducted please refer to Figure 2 in Appendix A.

The young's modulus of elasticity for this example is 2.477 GPa

## **3.7 Composite Sample**

Through a range of 0% to 35% by weight in the cured phenol formaldehyde composite / glass powder PF/GP (X %) where X is the percentage by weight of the filler, the filler that was used was SPHERICEL® 60P18 (spherical) hollow glass spheres. When the materials are unrefined the specimens were cast, into moulds to be later tested via tensile testing. The neat resin is a dark brown coloured liquid and once mixed with glass powder the mixture becomes a cream like colour. As the percentage of glass increases in the specimens the whiter the cured specimen becomes, the catalyst is then added it's also a dark coloured liquid. Table 3-1 demonstrates the mass of the resin, catalyst and glass powder that are required to make 150 grams of uncured composite for 10% by weight of glass powder. Six specimens can be obtained from one mould as making the specimens. The amount of mixture required to adequately fill the moulds is approximately a 150 grams. This is to allow for any spills or inconsistency of the mixture. Hopefully by increasing the mixture size to 150 grams instead of the exact amount of 120 grams will minimize or eliminate the possibility of any neat resin, catalyst or glass powder that was not mixed thoroughly being placed in the mixture, if the exact amount the mixing pot would have to be completely clean to have the complete ratio that had been carefully measured previously. It is easy to overlook a small clump of glass powder which would yield an undesirable composite and lead to inconsistent results, this may occur as when dealing with such small amounts. The test pieces must conform to standards, to allow consistency in past, present and future studies.

Parameters	Resin (R)	Catalyst ( C )	R +C	Glass Powder	Composite
Percentage by weight	30	1			
Percentage by weight			10	1	
Weight of Materials in 300g of PF/Glass Powder (10%)	261.2 (g)	8.8 (g)	270 (g)	30 (g)	300(g)

Table 3-1 Required amounts for 10% Glass powder

# 3.8 Concluding Remarks

This chapter has described the methodology that will be used to begin the experimental part of the project to be conducted. The methodology has not only set out the procedures and materials required to complete the experiment in a safe manner but also how to carry out the experiment to make sure the results will be of an acceptable nature, and to permit adequate and precise results to be drawn from the results that have been found. It can be drawn on which percentage of glass powder as filler in phenolic resins is the best selection, from tensile tests preformed on the composite a recommendation.

# **4 Results and Discussion**

# 4.1 Introduction

This chapter provides the results obtained from the tensile test outlined in the previous

chapter as well as explanations of tensile and yield strength and Young's module. In addition, a series of microscopic photos will demonstrate the effects on porosity and its formation. This chapter will analyze the positive and negative effects of using glass powder. Currently the price of the phenolic resin is \$6.50 per kilogram and Phencat catalyst costing \$8.00 per kilogram, previously a study used E-spheres SLG which is fly-ash a by product of burning coal as filler and only incurred a costs \$3 per kilogram, whereas currently the price of the glass powder by weight is \$5 per kilogram. It was a little bit more expensive by using glass powder as filler but this research was not fouce on the comparing of glass powder and fly-ash's filler properties. And the glass microspheres has on the mechanical properties of the phenolic resin has been favorable.

4.2 Summary of Findings



Figure 4-1 Test piece loaded in the tensile testing machine

Figure 4-1illustrates a frontal view of the tensile testing apparatus once a specimen has

been load into it. Student ID: 0050044877 Student Name: Chaodi, WU



Figure 4-2 Test piece that has failed in the tensile testing machine

Figure 4-2 illustrates a specimen that was tested and failed at a particular section. This is a good result as the point at which it failed was due to the weaker mechanical strengths of the material not due to human error or due to machine damage.

Glass powder reinforced phenol formaldehyde matrix composite were made at varying percentages from 0% to 30% glass powder by weight with a 5% intervals. Before slowly adding the glass powder slowly until finally a homogenous mixture is created, the initial process consisted of mixing the phenolic resin and catalysts together.



**Figure 4-3 Measuring of Ingredients** 

This was achieved only after all ingredients had been accurately measured to 0.1 gram of the required quantity. Figure 4-3 shows this being done. As soon as measured the catalyst (Hexion Phencat 15) and Phenolic resin (Hexion Cellobond J2027L) are mixed together to form the basis of the mixture followed by the glass powder (Hollow Glass Spheres) is added slowly as, the mixture is stirred. A chemical reaction will began and the mixture will begin to form a film across the mixture, if there was a delay between adding the glass powder to the phenolic resin and catalyst mixture. It needs removal from the mixture, whilst still stirring as soon as a film has formed. Taking out the film which has formed causes more problems when the ratio mixture is now not correct, as you cannot be make sure how much of phenolic resin and catalyst has been taking out. It was observed that several specimens had not fully cured and broke whilst be removing when the specimens were being removed from the moulding.

Another problem that happened was if any overspill occurred when the uncured mixture was being put in the moulding proved difficult to remove. A revised method once implicated allowed the specimens to be removed easier as this is an increasing problem. This was done by joining paddle pop sticks together on the base side of the moulding and applying pressure down on the paddle sticks. This was done by joining paddle pop sticks together on the base side of the moulding and applying pressure down on the paddle sticks. After trialing, as if appeared to distribute the pressure being applied more efficiently then could be done so without the paddle pop sticks, the method it proved to be much more efficient in removing the specimens. Another theory was that when, which was considered, lubricate is applied to the moulds to allow the specimen to slide out. Instead of using the canola oil wax was implemented and proved to be more successful in applying an even coat of lubricate then the aerosol can of canola oil.

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**Figure 4-4 Deformed Specimen** 

During initial baking of the Specimens it was observed that a number of test specimens become slightly deformed and developed a bow in the middle. Figure 4-4 illustrates one of the worse effected specimens that were deformed. Fortunately, alternative methods were implicated to counteract this occurring in other specimens when this was discovered only a few specimens had been post-cured in the oven. To do so, all test pieces were subjected to a heavier load of approximately 1kg, this load was only applied during the baking of the specimens during the baking session, all test pieces were subjected to a heavier load of approximately 1kg.

When there is limited literature currently available on the use of glass powder as a filler material in phenolic resin mixture an approximation was made based on similar studies Student ID: 0050044877 Page Number 50 Student Name: Chaodi, WU 50 that have been conducted in the past. To commence the experiments a moulding ratio of 30:1 was used, which was phenol formaldehyde resin: catalyst. For the first set of specimens to contain glass powder in the mixture the material once cured was a deep dark colour and appeared to have clumps of glass powder on the surface of the majority of the specimens. Once tested the results yielded reasonable results. An example of this is demonstrated in Figure 4-5



#### Figure 4-5 An example of Glass Clumps

A latter mixture at the same ratio was made and tested with similar results. Also, this ratio was used with an increase of glass powder by weight to 10%; the results that were obtained showed that all three mechanical tests showed an increase in the strength of the composite material. It was observed that the mixture was not as dark as the previously tested 5% mixture when the testing was conducted, also the material when setting up the specimens the tensile machine had to be altered as crushing began to occur and had to be Student ID: 0050044877 Page Number 51 Student Name: Chaodi, WU

aborted. This then leads to belief that material is becoming more brittle as the filler by weight increases.

The next percentage of 15% of glass powder by weight was tried at the current phenolic resin catalysts ratio. After it was allowed to cure for 3-5 days the mixture was still soft and was not curing, to enable the mixture to cure the ratio was increased to 20:1 and later trailed at 15:1, as 20:1 after 5 days was not cured fully and whilst being removed from the moulding the composite specimens deformed or critically failed. Due to the ratio so more care was taken to pour the uncured composite mixture into the moulds, the mould specimens began to cure much faster. Due to this the mixing of the material as it was beginning to cure was becoming difficult at this ratio the process was altered accordingly. The phenolic resin and catalysts were mixed together before the glass powder was added when the materials are being added initially.

Before the catalysts added to the mixture, this process was altered to allow the phenolic resin to be well mixed with the glass powder. As the chemical reaction the catalyst procduces was delayed, this proves to make the mixing process easier. This was evident once the mould had fully cured and was abstracted from the moulding; inspections of the new test specimens appeared to have a better mixture of the phenolic resin and glass mixture than that of the lower ratio's previously used which yielded glass clumps being easily noticeable in the specimens. As soon as the 15% glass powder specimens were loaded into the tensile testing apparatus it was seen that clamp pressure needed to be increased as the clamps were slipping along the test piece. This leads to the assumption

that the material maybe becoming less brittle and more ductile then the 10% by weight of filler composite mixture. The same mixing process was followed for the rest of the experiments.

The proceeding percentage to be cast was 20% glass powder by weight, which was trailed at the current ratio and was left to cure. The specimens were allowed and extra 3 days on top of the 15% specimens. After 5 days the specimens appeared to have a firm top and once beginning to eject the specimens out of the moulds it was discovered that they had in fact not fully cured and were left in the mould for a further 2 days to ensure they were able to be removed without damage.

From here 25% and 30% mixture were made at both 15:1 and later 12:1, at both ratio of phenolic resin to catalyst the composite took up to 2 weeks before the specimens appear to be cured enough to be removed from the moulding. Because this was rather difficult several mixtures were made. It was found that in both cases the 12:1 ratio returned better mechanical strengths then the 15:1 ratios. The reasoning behind using two ratios for both cases was due to the difficulty that occurred whilst mixing the mixture as well placing the uncured mixture into the moulds. Due to the high content of glass powder being 25 and 30% glass powder by weight of the total mixture stirring the mixture proved to be difficult. Get the fast curing mixture into the moulds, and the mixture had to be spooned in instead of previous methods of pouring it in. As placing the mixture into the mould the chance of porosity occurring increase dramatically. Later, if porosity had formed from

this method, once the specimens were closely examined before tensile testing is conducted to identify. Fortunately the specimens came out uniformly with minimal porosity occurring.

Both of the tested glass had to be again reduced, which the percentage began to show sign that the material was becoming more brittle as when being tested the clamping force.

## 4.3 Results of tensile testing

Please Refer to Appendix C

## 4.3.1 Tensile Tests

Tensile testing is a most common and sought after a mechanical test, as it easy to resolve also being of valuable for the purposes of specifications and quality control for manufactured goods. In tensile tests the force and extension of the specimens are recorded by a data logger. The cross-sectional area is calculated, before the specimen is tested. When handling the specimens care is required, placing the specimen into the tensile testing machine when setting the distance between the jaws of the apparatus to the same for each specimen (gauge length). This is to eliminate a variable as well as allowing the computer to calculate the yield and tensile strength along with the Young's modulus.

When attempting to remove the specimens from the moulding took time, which was the specimen, being small, and not fully cured. Several methods were trialed, due to the

difficulty of overspill of the mixture that had cured, incurred problems as by applying force to the ends of the specimens, tended to stick to the edges resulting in cracks at the neck of the specimen. The method was to place two popsicle-sticks on the bottom of the moulded specimen, which proved to have the best result in removing the specimens. From here, minimal pressure was applied to evenly distribution the over the specimen allowing for smoothness of the extraction. It was important to extract the six specimens from the same moulding mixture, and to make sure higher accuracy of results. The temperature and mixing time illustrate the point. This is due as the mixture are made slight variation can occur.

## 4.3.2 Yield Strength

Yield Strength of glass powder reinforced phenolic resin post curing in microwave oven compared to that in a normal oven.



#### **Figure 4-6 Yield Strength**

Student ID: 0050044877 Student Name: Chaodi, WU Once merged to form a reinforced phenol formaldehyde matrix composite, figure 4-6 shows the yield strengths of varying percentages by weight of glass hollow spheres. The neat yield strength of the phenolic resin was 11.5 MPa, which was higher than that of the test pieces that contained a percentage by weight of glass powder. At 5% by weight of filler the yield strength was 8.2 MPa. Then, it was increased to 9.98 MPa with 10% by weight of glass powder. From this percentage of filler by weight, the composite generally was observed that as the percentage by glass powder content improved, which was less than the yield strength became. About 30% filler the composite sample only gave yield strength of 2.12 MPa. At 25% glass powder it appears that the values found could be inaccurate, this could be have been logged into the computer, when preliminary measurements where taken the in corrected value. This is also proven via inspection of Table 4-1, which gives the information about the values of yield strength mentioned above with their standard deviation in brackets as well as showing that the values of glass by weight increase the yield strength drops. It can be argued that a composite with 10% by weight of glass powder is the optimal, if cost and yield strength which are criteria for a project.

Compared to travis's research who did 2008 by post curing in oven, this results are 7-10%less which may effected by posting in a short period with a higher temperature. Moreover, the temperature in microwave may not uniform as the oven does. The higher temperature may burn the chemical structural as well. University of Southern Queensland

percentage by		
weith of Glass	Yield Strength(MPA)oven	Yield Strength(Mpa)Microwave
powder	case	case
0	13	11.5
5	10.14	8.2
10	11.42	9.98
15	8.06	7.2
20	6.74	6.12
25	7.82	6
30	2.82	2.2

Table 4-1 Yield Strength of glass powder reinforced phenolic resin composite

## 4.3.3 Tensile Strength

Tensile Strength of glass powder filled phenolic resin curing in microwave oven compared to that

in a normal oven case.



**Figure 4-7 Tensile Strength** 

Student ID: 0050044877 Student Name: Chaodi, WU Figure 4-7 shows the Tensile Strengths of the varying percentage by weight of glass powder reinforced phenol formaldehyde matrix composite. The tensile strength of the neat increased equated to 12.35 MPa. The glass powder was introduced to the phenolic went up at 5 percent by weight, however, the tensile strength reduced to 9.7 MPa. From this point, it was illustrated that on average the composite mixture increased in value until 10% glass powder by weight, which yielded a figure was 12.08 MPa. The maximum tensile strength that was found from any percentage by weight of the glass powder was at 10% glass powder by weight. After this the glass reinforcement dropped the values of the tensile strength, this is demonstrated in Figure 4-7 Tensile Strength. The tensile strength further lowered to 6.12 MPa having 20% by weight of filler in the test pieces, and then, continued to decrease in strength, which was only 4.1 MPa that was found at 30% glass powder by weight. At 25% glass powder were seem to be an irregularity in the results that were found as seen in the graph. This may be caused to be an irregularity in the results that were found as seen in the graph, for example, surrounding temperatures. The variation of tensile strength with respect to percentage by weight of glass powder is the same as that of yield strength. If cost and tensile strength were considered at the same time, composite with 15% by weight of filler would be selected. Table 4-2 shows the values of tensile strength mentioned above with their standard deviation in brackets.

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Percentage by		
weight og	Tensile	Tensile Strength(Mpa)microwave
Glass Powder	Strength(Mpa)oven case	oven case
0	15	12.35
5	10.14	9.7
10	12.08	12.08
15	11.25	7.1
20	6.98	6.12
25	8.56	6.5
30	4.57	4.1

Table 4-2 Tensile of glass powder reinforced phenolic resin composite

## Standard deviation

Compared to the result of this research, Redjel (1995) found that the tensile strength for neat resin was 27 MPa, which was considerable high. The material that was used was a pure phenolic resin 84055 catalyzed by 3 percent of C 1650 and cured at 80 °C for 8 days. It was produced and prepared by CDF-Chimie, France. The curing time was excessively long and would not be industrially viable and the energy consumption was enormous. And Travis's research which based on curing samples in oven for 40hours in a 240 °C. For instance, the tensile strengths decreased with increasing particle loading (Dekkers and Heikens, 1983). The trend of the tensile strengths of this research was in line with those of glass bead reinforced polystyrene. However, the trend was just the opposite of that of glass beads filled epoxy resin (Amdouni, et al., 1992)

## 4.3.4 Young's Modulus

Young's Modulus of glass powder reinforced phenolic composites curing in microwave oven compared to that curing in a normal oven.



#### Figure 4-8 Young's Modulus

Figure 4-8 illustrates how the Young's modulus changes by weight of glass hollow spheres reinforced into the phenol formaldehyde composite mixture. When the young's modulus of the neat phenolic resin had a reading of 2.41 GPa, the filler material was added the young's modulus decreased to 2.12 GPa when the percentage of glass powder by weight was 5%. Once the 10% filler by weight was tested, it showed slightly higher values than that of the 5% test pieces, which was 2.2 GPa. It dropped to 1.68 GPa when the percentage of filler by weight was 30%. This as well as demonstrated in Table 4-3. As previously, concluded tests showed as well as young's modulus indicated that as the glass quantity in the mixture increases the strength of the composite decreases. Phenolic composite reinforced with glass powder with 10% by weight of filler is optimal, if cost and Young's modulus were to be considered for a specific task.

Percentage by weight	Young's Modulus	Young's Modulus (MPa)microwave
of Glass Powder	(MPa)oven case	oven case
0	2510	2410
5	2253	2120
10	2401	2200
15	2133	1980
20	2230	2170
25	1310	1267
30	1720	1680

#### Table 4-3 Young's modulus of glass powder reinforced phenolic resin composite

## Standard deviation

In comparison to the results of those of phenolic formaldehyde (2.76 – 4.83 GPa) (Callister, 2005). When in another study, using pure phenolic resin the Young's modulus was found to be 2.51 GPa, which is 13.5 % lower than that found by a group of researchers for pure phenolic resin (2.9 GPa). The same team used ICI Fiberite resol-type CMXR-6055 phenolic formaldehyde resin. This research used Chemwatch Borden (Hexion) Cellobond J2027L phenolic formaldehyde resin. When obtaining these results the environmental factors such as working temperatures were not discussed as well as the time allowed for the resin and filler material to mix and cure before continuing on to be post-cured (Ku et al., 2006). Table 2 gives information about the values of Young's modulus mentioned above with their standard deviation in brackets. Compared to the result of this research and that found in literature (Callister, 2005), Redjel (1995) found that the Young's modulus for neat resin was 5.16 GPa, which was considerably higher

# 4.4 Microscopic Analysis



#### Figure 4-9 0% no glass adding 23X

The 0% maintains there is no glass powder as filler which added to the phenolic resins. From the above figure, it is some air bubble shows on the sample (very dark points), it was naturally caused by phenolic resins's evaporate property. There are few scratch displays on the rim which may course by the sample yielding.



Figure 4-10 0% no glass adding 200X

When zoom into 200x scalar, those air bubbles are more clearly shows on the figure. Those small holes reduce the strength of the sample. Some resins are pulling out and was not perfectly mixed, which it is very normal to occur in the lab-testing or industry.



#### Figure 4-11 0% no glass adding 1000X

At 1000x displayed figure, it gets easier to see more information about the micro structural of the Phenol formaldehyde. Not only those air bubbles which appeared, but also a very interesting phenomenon that a "wave" which showing on the figure. This kind of crystal structural is normally occurred in a higher percentage of phenol formaldehydes.



Figure 4-12 20% glass powder by weight adding 25X

In the 20% sample, due to the mixture become harder and harder, the glass powder as filler gets more difficult to mix with resin uniformly. Therefore, more air bubbles will course in this case. Compared to the sample of 0% weight, the surface was not smooth enough and it is easier to see the glass powder structural appear on the surface.



Figure 4-13 20% glass powder by weight adding 200X

Figure 4-13 is the 20% glass powder by weight zoom into 200X, it is clearly shows there is full of glass powder filling in the resin. It is not hard to image the strength will more related to the glass powder than resin itself. And the results of that may led a lower value compared to a lower percentage of glass as filler sample.



Figure 4-14 20% glass powder by weight adding 1000X

Figure 4-14 is the 20% glass powder by weight adding into the phenolic resin, it is quite a lot of glass powder displayed on the figure. There are some broken air bobbles showing the figure and some small glass powder inside. It can be coursed by the hand mixing and some may not mixed very well.

## 4.5 Concluding Discussion

As soon as found by the Yield strength in the chapter the results, Tensile strength and Young's modulus for varying percentages by weight of glass powder reinforced phenolic resin were explained and discussed. The fluidity of the slurry composite was high and could be cast easily into moulds if where there was less than 25% glass powder by weight present in the mixture. The values once reviewed with those found by another study, which contained no filler material. However, they had varying results between the different studies. It is possible that when the fusion between phenolic resin (matrix) and glass powder (reinforce) increases by adding some other fillers and resins to the composite, may lead to an increase in the tensile strength. The best percentage of glass powder by weight, that when added to the phenolic resin to give an optimum Yield and Tensile strengths, Young modulus and cost is approximately 10 percent. These figures are illustrated, which was using glass powder by weight as filler and reinforcing agent within the existing phenol formaldehyde matrix composite is a viable option. To view the raw data from each set of samples that were post-cured by the conventional oven and had tensile testing conducted on them please find the Appendix B attached.

# **5** Conclusion and Recommendations

## **5.1 Introduction**

This chapter will summarize the findings found in this study, also drawing a final conclusion from chapter 4. A recommendation will be recognized on which percentage gives the best percentage of glass powder by weight. It was able to been seen how the different glass powder percentages affect the final mechanical properties of the phenolic resin mixture, after the results have been reviewed and analysis in chapter 4. The majority of the data gathered appears to be accurate with only a few results outside the estimated range.

## **5.2 Summary**

Throughout chapter 2 a detail literature review was conducted on several journal articles relating to the chosen topic. As industry is ever improving in the sense that their always the need to redesigning equipment for efficiency as well as to be more cost effective from this review numerous authors concluded that the need for more research on phenolic resin is required This places more emphasis on the need to conduct similar studies on adding filler material to the existing phenolic resin in the hope that a stronger more specific composite can be created for a specific purpose.

Not long after the methods had to be altered to keep the project on the all ready tight timeframe, after a methodology setting out the procedures was conducted the experiment commenced. These consisted of using a different type of lubricate on the preparation of the moulds. As soon as this was solved there were still several specimens that would become stuck in the mould and would break once more pressure was applied, doing this allow for a higher percentage of the specimens came out without any fractures or deformation. The amount of specimens breaking or having stress fracture deteriorated down to only a few after the paddle pop concept was implied.

It was found what effects occurred when glass powder is added to the phenolic resin mixture at varying percentages by weight, after the results had been found form the tensile testing. The fluidity of the slurry composite was high and enable to be cast easily, in cases where there was less than 25% glass powder by weight present in the mixture. Results showed that when no filler material is used found that it differed to other studies that had been reviewed previously. It also is possible that by additional materials to the mixture as well as another filler or resin that a stronger more specific composite can be discovered.

Results concluded that the best percentage of glass powder by weight to add to the phenolic resin to give the best Yield and Tensile strengths, Young modulus and still be a cheaper alternative was approximately 10 percent. Tensile and young's modulus was highest at approximately 10% glass powder by weight, for the full tabulated data please refer to Appendix D, which was evident in chapter 4 where the mean results illustrated that for yield.

## **5.3 Recommendation for Future Work**

Some future work can be more focusing on a different type of phenolic or glass powder. It may be easy to find a better combination by trying a few more materials. For this phenolic resins and glass powder, the future work which includes some further analysis focusing on the mixing, maybe to adjust how the composite is mixed by using mechanical devices to mix the uncured composite. The mixing process consists of placing the glass powder and phenolic resin together and stirring still the two ingredients have blended without a chemical reaction. As the catalyst is added to the mixture a chemical reaction is triggered and the mixture begins to cure. It could possible allow for a more homogeneous mixture to be formed prior to pouring into the moulds if a mechanical device was to be used for mixing the materials instead of manual stirring. A comparison can then be obtained whether using the mechanical devices to mix the uncured composite will add strength to the moulds. If a mechanical device was used there is a possibility that the unmixed (clumped) glass powder that was found microscope would be eliminated. With a mechanical device, a set timeframe to mix the uncured composite mixture could be established where a homogeneous mixture are obtained.

Compared with the results, fast curing process (microwave oven) will reduce the strength around 10% compared to a normal curing process. When glass powder has been identified as a viable option to consider for filler in a phenolic resin composite, this option needs to be reinforced with future studies focusing on the strength, feasibility density of glass powder as a filler option. When previously stated solid glass spheres are also available, and a similar study can be conducted to show what characteristics the solid spheres have on the phenolic resin and review the results to this study on the differences of the two materials.

Investigation could be done on focusing a certain percentage of glass, 10-15%, which brings a quite reasonable results compared to research a very wide range. Additional materials would have to be added to the mixture to overcome the issues of occurred when mixing the high percentage of glass powder if this was conducted.

# **6** References

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### Appendix A Project Specification

#### University of Southern Queensland Faculty of Engineering and Surveying

#### ENG 4111/4112 Research Project PROJECT SPECIFICATION

Project title:

Investigate the best percentage by weight of glass powder, as fillers, in phenolic resin using tensile tests post-cured in microwaves

Student:

Chao di, Wu - 0050044877

Supervisor: Co-Supervisor:

Sponsorship:

#### **Project Synopsis:**

The project is about using glass powder as fillers to reduce the total cost and increase the influence on the final structure properties. This project involves the production of a range of phenolie resin specimens with different percentage by weight of filers. After preliminary curing of heating the samples to 100 degree and cool them in the facility.

Dr. Harry Ku

Tensile tests will be used to evaluate its tensile properties. The findings will have to be analysed in detail in order to establish behavior trends and formulas that can be used for theoretical prediction of filled polymer behavior.

#### Timelines:

1. Calculated different weighting percentage of materials in each mixing.

Begin	: 16 <sup>th</sup> November 2008
Completion	: 16 <sup>th</sup> November 2008
Approx. Hours	: 2 hours

2. Mixing simples and preliminary curing. (Part One upper to 20%)

Begin	: 16 <sup>st</sup> November 2008
Completion	: 8 <sup>th</sup> December 2008
Approx. Hours	: 60 hours

3. Mixing simples and preliminary curing. (Part Two upper to 30%)

Begin	: 15 <sup>st</sup> February 2009
Completion	: 1 <sup>st</sup> April 2009
Approx. Hours	: 30 hours

4. Post-curing samples in microwave

Begin	: 2 <sup>nd</sup> April 2009
Completion	: 10 <sup>th</sup> April 2009
Approx. Hours	: 30 hours

5. Tensile tests

Begin	: 11 <sup>th</sup> April 2009
Completion	: 17 <sup>th</sup> April 2009
Approx. Hours	: 20 hours

6. Analysis of results.

Begin	: 20 <sup>th</sup> April 2009
Completion	: 25 <sup>th</sup> April 2009
Approx. Hours	: 30 hours

7. Draw up conclusions.

Begin	: 26 <sup>th</sup> April 2009
Completion	: 30 <sup>th</sup> April 2009
Approx. Hours	: 10 hours

8. Discussion for the thesis outline with supervisors.

Begin	: 4 <sup>th</sup> May 2009
Completion	: 10 <sup>th</sup> May 2009
Approx. Hours	: 10 hours

9. Thesis initial drafting - each chapter in draft form and shown to supervisors

Begin	: After the semester1 exam 2009
Completion	: 1 <sup>st</sup> August 2009
Approx. Hours	: 70 hours

10. Final draft of thesis, to incorporate modifications suggested by supervisor.

Begin	: 10 <sup>th</sup> August 2009
Completion	: 10 <sup>th</sup> September 2009
Approx. Hours	: 20 hours

9. Complete other forms or tests if need.

Begin	: 13 <sup>th</sup> September 2009
Completion	: 2 <sup>nd</sup> November 2009
Approx. Hours	: 30 hours

AGREED:

\_\_\_\_\_ (student)

\_\_\_\_\_ (Supervisor)

(Date)\_\_\_/\_\_/

FACULTY OF ENGINEERING & SURVEYING WORK PERMIT
Permit No: 1029
This form is to be used where a Standard Work or Operating Procedure (SWP/SOP) indicates that a permit is required to use Engineering and Surveying facilities and equipment.
APPLICATION
Name of Applicant: Chao di Wu
I wish to apply for approval to use the Faculty of Engineering and Surveying equipment and facilities:
Work Area / Location: 7106,7113 (Work area staff must be consulted BEFORE using any facilities)
Equipment / Process: Sample pays, marourne cur, testing
Relevant SWPs:For Unit / Project:
From (Start):AM/PM Date:
To (Permit Expires):AM/PM Date:O
I certify that I have read and understand the requirements of the Standard Work Procedure applicable to this permit. I agree to comply with those requirements and any cracial precautions/instructions listed below.
Signature: <u>R</u> <u>R</u> <u>Date</u> : <u>114109</u>
(To be completed by Work Area Manager/Supervisor)
Special Precautions/Instructions: Use msDS bylow zoning any channe
covered cootestar Respirator Dust mask, eye project
and other splity equ. as permined
ALL WORK AREAS AND EQUIPMENT MUST BE CLEANED AFTER USE.
The above applicant has shown to me that he/she is competent to carry out the procedure and/or operate the equipment specified in this work permit. The Permit is granted for the period stated above.
Name: MOHAN TRADA Date: 114109
Position: Sr. Trech. Mitry Signature: MZ

**SWP 001** 

THIS PERMIT MAY BE REVOKED AT ANY TIME.

# **Appendix B How to calculate tensile tests**

Student ID: 0050044877 Student Name: Chaodi, WU



Load against extension of phenolic resin Reinforced with Glass powder (10%)

Figure 1: Load against extension of a sample showing the 0.05% proof load

To calculate the tensile strength, divide the maximum load by the original cross-sectional are of the specimen. (Wang, 2007)

where  $P_{max}$  is the maximum load in Newton and  $A_0$  is the original cross-sectional area in mm2. For example, the tensile strength of a sample of how to apply the above equations are illustrated in Figure 1 in Appendix

Yield strength is calculated using the relationship below (Wang, 2007)

For example, yield strength of the sample illustrated in Figure 1



Figure 2: Graph showing how to obtain data to use when calculating the Young's modulus for phenolic composite

Student ID: 0050044877 Student Name: Chaodi, WU To calculate Young's modulus the following equations are used.

Where  $\Delta L$  is the change in length of the material, F is the force place on the specimen by the apparatus during testing, subscript.' denotes original measurement before testing was conducted. To identify how this is conducted please refer to Figure 2 in Appendix A.

# Appendix C Actual data logged

wu-0%R30- RC 30:1 G

#### **Specimen Results:**

Test Date : 24/04/2009 Method : MMT Tensile Test with return.msm

#### **Specimen Results:**

Specimen	Thickness	Width	Area	Peak Load	Peak	Break	Break
#	mm	mm	mm^2	Ν	Stress	Load	Stress
					MPa	Ν	MPa
1	5.880	14.550	86	708	8.28	708	8.28
2	5.570	14.620	81	675	8.29	675	8.29
3	5.770	14.690	85	796	9.39	796	9.39
4	5.910	14.760	87	1014	11.62	1014	11.62
5	5.810	14.830	86	685	7.95	685	7.95
6	5.590	14.770	83	514	6.22	514	6.22
7	5.550	14.720	82	507	6.20	507	6.20
8	5.100	14.700	75	796	10.61	796	10.61
9	5.050	14.700	74	728	9.81	728	9.81
Mean	5.581	14.704	82	714	8.71	714	8.71
Std Dev	0.316	0.083	5	154	1.84	154	1.84

Student ID: 0050044877 Student Name: Chaodi, WU Page Number 81

Specimen	Elongation	Stress At	Load At	
#	At Break	Offset	Offset	
	mm	Yield	Yield	
		MPa	Ν	
1	0.366	7.926	678.121	
2	0.390	6.678	543.840	
3	0.446	7.406	627.766	
4	0.618	7.851	684.835	
5	0.371	6.585	567.339	
6	0.305	0.699	57.741	
7	0.326	6.205	506.912	
8	0.527	5.866	439.772	
9	0.483	7.009	520.341	
Mean	0.426	6.247	514.074	
Std Dev	0.102	2.195	189.265	



wu-5%G- RC 30:1

### **Specimen Results:**

Test Date : 24/04/2009 Method : MMT Tensile Test with return.msm

Specimen	Thickness	Width	Area	Peak Load	Peak	Break	Break
#	mm	mm	mm^2	Ν	Stress	Load	Stress
					MPa	Ν	MPa
1	5.290	14.700	78	342	4.40	342	4.40
2	6.010	14.400	87	624	7.21	624	7.21
3	5.200	14.460	75	110	1.46	110	1.46
4	5.500	14.740	81	238	2.94	238	2.94
5	5.450	14.570	79	547	6.89	547	6.89
6	5.160	14.600	75	366	4.86	364	4.83
7	5.270	14.600	77	440	5.72	440	5.72
8	5.220	14.660	77	396	5.18	396	5.18
9	5.220	14.600	76	469	6.16	469	6.16
10	5.420	14.600	79	382	4.83	382	4.83
11	5.320	14.650	78	261	3.35	261	3.35
Mean	5.369	14.598	78	380	4.82	380	4.82
Std Dev	0.239	0.098	3	144	1.73	144	1.73

Specimen	Elongation	Stress At	Load At		
#	At Break	Offset	Offset		
	mm	Yield	Yield		
		MPa	Ν		
1	0.230	2.644	205.618		
2	0.353	4.383	379.345		
3	0.076	1.072	80.569		
4	0.198	1.325	107.425		
5	0.395	4.777	379.345		
6	0.363	2.781	209.479		
7	0.445	2.627	202.094		
8	0.346	2.825	216.193		
9	0.504	2.687	204.779		
10	0.326	2.444	193.365		
11	0.229	1.568	122.196		
Mean	0.315	2.648	209.128		
Std Dev	0.122	1.142	96.309		



wu-10%G

# **RC 15:1 Specimen 1 deleted** due to irregularity

#### **Specimen Results:**

Test Date : 24/04/2009

Method : MMT Tensile Test with return.msm

Specimen	Thickness	Width	Area	Peak Load	Peak	Break	Break
#	mm	mm	mm^2	Ν	Stress	Load	Stress
					MPa	Ν	MPa
1	5.760	14.590	84	798	9.50	798	9.50
2	5.730	14.550	83	1000	12.00	****	****
3	5.810	14.510	84	715	8.49	715	8.49
4	5.490	14.570	80	977	12.21	975	12.19
5	5.630	14.510	82	910	11.14	910	11.14
6	5.400	14.580	79	1165	14.80	1165	14.80
7	5.620	14.530	82	1196	14.65	1196	14.65
8	5.640	14.460	82	748	9.17	748	9.17
9	5.950	14.660	87	871	9.99	871	9.99
Mean	5.670	14.551	83	931	11.33	922	11.24
Std Dev	0.166	0.058	3	171	2.30	181	2.44

Specimen	Elongation	Stress At	Load At		
#	At Break	Offset	Offset		
	mm	Yield	Yield		
		MPa	Ν		
1	0.458	5.233	439.772		
2	****	5.959	496.841		
3	0.380	5.844	492.645		
4	0.662	6.211	496.841		
5	0.519	5.837	476.867		
6	3.765	7.129	561.296		
7	0.691	7.285	594.867		
8	0.458	5.219	425.672		
9	0.489	5.157	449.843		
Mean	0.928	5.986	492.738		
Std Dev	1.151	0.785	55.407		



RC 15:1

wu-15%G

### **Specimen Results:**

Test Date : 24/04/2009 Method : MMT Tensile Test with return.msm

Specimen	Thickness	Width	Area	Peak Load	Peak	Break	Break
#	mm	mm	mm^2	Ν	Stress	Load	Stress
					MPa	Ν	MPa
1	5.160	14.470	75	480	6.43	477	6.39
2	5.200	14.560	76	648	8.56	648	8.56
3	4.500	14.390	65	498	7.68	498	7.68
4	5.000	14.500	72	396	5.46	396	5.46
Mean	4.965	14.480	72	505	7.03	505	7.02
Std Dev	0.322	0.071	5	105	1.36	105	1.37

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	
1	0.365	2.203	164.495	
2	0.438	3.458	261.849	
3	0.338	2.717	175.909	
4	0.319	1.528	110.782	
Mean	0.365	2.477	178.259	
Std Dev	0.052	0.816	62.544	



wu-20%R12- RC 12:1 G

# **Specimen Results:**

Test Date : 24/04/2009 Method : MMT Tensile Test with return.msm

Specimen	Thickness	Width	Area	Peak Load	Peak	Break	Break
#	mm	mm	mm^2	Ν	Stress	Load	Stress
					MPa	Ν	MPa
1	5.410	14.660	79	207	2.62	205	2.58
2	5.030	14.561	73	131	1.79	131	1.79
3	4.880	14.470	71	406	5.74	404	5.72
4	5.250	14.520	76	519	6.81	519	6.81
5	5.000	14.660	73	312	4.26	312	4.26
6	5.210	14.510	76	453	5.99	451	5.97
Mean	5.130	14.564	75	338	4.53	337	4.52
Std Dev	0.194	0.080	3	149	2.00	149	2.00

Specimen	Elongation	Stress At	Load At		
#	At Break	Offset	Offset		
	mm	Yield	Yield		
		MPa	Ν		
1	0.209	1.058	83.926		
2	0.123	0.798	58.412		
3	0.366	2.073	146.367		
4	0.395	2.907	221.564		
5	0.267	1.026	75.198		
6	0.358	2.798	211.493		
Mean	0.286	1.776	132.827		
Std Dev	0.106	0.943	71.409		



25% and 30% was analysis in the excel