University of Southern Queensland Faculty of Engineering and Surveying

Synthesis of Modified Phenolic Resins using Renewable Materials for Advanced Composites in Civil Engineering Structures.

A dissertation submitted by

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Abstract

In this project the synthesis and characterisation of Phenolic formaldehyde resins (PF) and Phenolic formaldehyde resins modified with Cardanol (CPF) resin and natural renewable materials as thermoset fillers is investigated. The project also investigates the effect of percentage of filler by weight of renewable materials (Sawdust fibres) on the ultimate mechanical and thermal properties of the composites with PF and CPF resins.

Phenol Formaldehyde (PF) resins are made from Benzene a petroleum product. Petrochemical supplies are finite and the economics of their use by enhancing and extending their use is of considerable interest from both an economic and resource basis. PF resins are among the most widely used thermosets because of their economics on a cost-per-volume basis. Environmental and cost concerns have lead to the search for alternative chemicals obtained from renewable raw materials to complement and /or replace PF resins.

The natural substitute for PF resins used in this study is Cardanol, a natural Phenolic resin distilled from cashew nut shell liquid (CNSL). CNSL itself is derived from waste cashew nut shell. Cardanol can be blended with PF resins to make Cardanol Phenolic Formaldehyde (CPF) resins, extending and complementing the PF resins. In this study fillers in the form of Sawdust are also added to extend these PF and CPF resins as they are characterised or formed.

This project has prepared, tested and characterised the synthesised samples based on PF resins and CPF resins. It has been found that Cardanol can effectively substitute commercial phenol in the synthesis of Phenolic resins. The Cardanol in the synthesised CPF resins has had a positive effect where the samples have been found to be less brittle and more flexible than PF resins. The sawdust fibres have also been found to be suitable fillers for composites with PF and CPF/PF resins. The composites with CPF and sawdust fibres have shown high flexibility and strength with a high glass transition temperature. These attributes make the composites suitable for potential civil engineering structural applications.

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Nomenclature

- CEEFC Centre of Excellence in Engineering Fibre Composites
- DMA Dynamic Mechanical Analysis
- CPF Cardanol Phenolic Formaldehyde Resin
- NaOH Sodium Hydroxide
- PF Phenolic Formaldehyde Resin
- PPE Personal Protective Equipment
- Tg Glass Transition Temperature
- USQ- University of Southern Queensland
- SWP Standard Work Procedures

Glossary of terms

Feedstock - Raw material required for an industrial process.

SWP procedures - Standard Work Procedures; a document distributed at the initial on site induction at the Centre for Excellence in Engineering Fibre Composites (CEEFC)

1 Introduction

In this project the synthesis and characterisation of Phenolic formaldehyde resins (PF) and Phenolic formaldehyde resins modified with Cardanol (CPF) resin and natural renewable materials as thermoset fillers is investigated. The project also investigates the effect of percentage of filler by weight of renewable materials (Sawdust fibres) on the ultimate mechanical and thermal properties of the composites with PF and CPF resins.

This chapter describes the project topic, the project background, the research aims and objectives, the justification for the project, its scope and conclusions gained from the project.

1.1 Project Topic

Synthesis of Modified Phenolic Resins using Renewable Materials for Advanced Composites in Civil Engineering Structures.

1.2 Project Background

Phenol Formaldehyde (PF) resins are made from Benzene a petroleum product. Petrochemical supplies are finite and the economics of their use by enhancing and extending their use is of considerable interest from both an economic and resource basis. 'The synthesis of polymers starting from renewable resources is object of significant research efforts due to the increasing prices of petro chemical products associated with growing environmental concerns' (Maffezzoli et al 2004, p.1).

PF resins are among the most widely used thermosets because of their economics on a cost-per-volume basis (Ku et al 2008, p 2). Phenol Formaldehyde thermosetting resins (or cross-linked polymers) are made from petrochemicals in the form of Benzene. 'Environmental concerns and cost have lead to the search for alternative chemicals from renewable raw materials' (Mwaikambo 2009, p.3).

Cardanol is a natural Phenolic resin distilled from cashew nut shell liquid (CNSL). This natural Phenolic can be blended with PF to make CPF resins and used to replace or

complement PF resins. Fillers in the form of sawdust are also added to extend and/ or complement these PF and CPF resins.

In this project the synthesis and characterisation of modified Phenolic formaldehyde PF and CPF resins and the use of natural renewable materials as thermoset fillers is presented. The project investigates the effect of different percentages of Cardanol and sawdust fibres as thermoset filler by weight mixed with Phenolic resin.

The Tests completed to assess the samples properties were:

- Flexural (Three point bending),
- Dynamic Mechanical Analysis (DMA)

Tensile tests were to be carried out but the properties of the resin samples from the enclosed condensation reaction made the samples defective, therefore these tests were not carried out.

1.3 The Research Project Aims and Objectives

The projects aims and objectives are taken from the Project Specification (Appendix A), where the research project is in four parts:

- Literature review of Phenolic resins and modifying mechanisms and techniques
- Synthesis of PF resins modified with different amounts of Cardanol (CPF)
- Testing and characterisation of the synthesised CPF resins
- Preparation and testing of composites using the modified CPF resins and different amounts of the renewable materials (sawdust).

1.4 Justification

The purpose for this project is to find cost efficient renewable materials to complement and extend petrochemical based Phenolic resins. This is with the aim of eventually finding natural alternatives that can be sourced from sustainable feedstock. As Vilaplana et al (2010, p.12) succinctly describe 'only by the conscious integration of sustainable crop growth, raw material extraction, synthetic and modification steps, material processing and product manufacture, safe service life in the intended application, and suitable waste management will we be able to achieve the true goal of sustainable development in material and product design'.

1.5 Scope

The scope of the project is taken from the Project Specifications aims and objectives (Appendix A). To complete this aim a literature review was completed to give an outline of previous work and these used as a background to the project. This information then was used to complement the research and methodology and the characterisation and testing of the Phenolic formaldehyde (PF) resins and Cardanol Phenolic formaldehyde (CPF) resins modified with sawdust fibres. The results have been compiled with conclusions drawn from the results and recommendations made as to further work.

1.6 Conclusion

Cardanol can effectively substitute commercial phenol in the synthesis of Phenolic resins. The Cardanol in the Synthesised CPF resins has had a positive effect where the samples have been found to be less brittle and more flexible than PF resins. The PF samples have generally an overall higher flexural modulus, stress values and Glass transition temperature.

The sawdust fibres have also been found to be suitable fillers for composites with CPF and CPF/PF resins. Effects due to wood fibre have been generally that Storage modulus, Flexural modulus, Stress and Glass Transition Temperature for all Composite blends fall with the increase of wood fibre percentage. Wood fibre at a filler percentage of 5% gives in most samples the highest modulus showing Sawdust fibres are suitable fillers for composites with CPF and CPF/PF resins. There is no percentage of wood fibre with better strain qualities than any other.

The CPF 50%– PF 50% resin mix was the most successful as it gave stable results with the brittleness decreasing in a linear manner with the higher percentage of filler.

The values of the resin samples CPF 100% peak at C-20% and fall to a lower value at C-30% in the majority of test results (Including strain). The samples of CPF 50% decrease in values at C-20% and increase of values at C-30% (Including strain).

The composites with CPF and Sawdust fibres have shown high flexibility, strength with a high glass transition temperature. These attributes make the composites suitable for potential civil engineering structural applications.

2 Literature Review

2.1 Introduction

This chapter will give the background of Phenolic composite resins, thermoset fillers/or reinforcement and resins made from renewably derived materials. The synthesis, curing and characterisation of Phenolic Formaldehyde (PF) and Cardanol Phenolic Formaldehyde (CPF) resins with the thermoset fillers is discussed. The testing equipment used and the results obtained are outlined, as well as safety and social effects of this research project.

2.2 Definition of composite materials

A composite material according to Mwaikambo (2009, p.1) is a solid material consisting of two or more materials, in which the individual components retain their separate identities. An example of this is the mud brick (Figure 1) which was used in the time of the Pharaoh (Mwaikambo 2009, p.1) and is still used as a building material today. A mud brick is a simple structural material made of a clay/silt matrix with reinforcement from a cellulose fibre such as grass or straw.



Figure 1: Mud Brick; a simple composite material

The composite resin matrix as Manthey (2009 p.7) found is to transfer load, and hold the reinforcement in place, but also protect the reinforcing material. The matrix reinforcement may be either continuous or discrete particles (Mwaikambo 2009, p.1). The reinforcement is put in the composite matrix to give the composite material characteristics that are superior to the matrix material on its own. (Manthey 2009 p.6).

2.3 Background of Composite Building Materials

Material compositions of building materials changed as better materials were found from trial and error. The composite material components dependent on the available materials. Some ancient mortars and concretes were made from natural or artificial aggregates and with organic and pozzolanic additives in the matrix with vegetable and/or animal materials as reinforcement (Moropoulou et al, 2005, p.296 -7).

Today composites are made for the same reasons as ancient people; the sourcing of economic materials to create building structures. The composite resin composite is essentially similar to the mud brick composite with a matrix and reinforcement with only the properties of the materials differing.

Currently the use of polymer matrix composites containing fibres is increasing at 10% per year Mwaikambo (2009, p.1). The main driving forces are the continuing high prices for petroleum-based raw materials and the potential environmental advantages of using renewable resources (Bicerano & Associates Consulting Inc 2009).

2.4 Background of Phenolic Resin and its Composites

Pira was the first to use Phenolic resin in 1843, but it was not until 1907 that Dr Leo Baekeland registered a patent for producing a synthetic resin he called Bakelite (Painter, Coleman 2000, p.1). Bakelite was created by reacting phenol and formaldehyde under heat and pressure (Simpson 1995, p.74). This new product was originally extracted from coal tar and came to be called the material of a thousand uses (EngineersHandbook.com).

A large number of products were displaced by Phenolic resins due to economics. An example is billiard balls which were up until the invention of Bakelite, were made from ivory. The high production costs of billiard balls had led to a search for a substitute (Border Billiards 2009). It can be argued that the Bakelite radio is one of the most iconic products made from Bakelite.



Figure 2: Bakelite radio (tuberadioland) and Bakelite billiard balls (flickr 2010)

Henry Ford produced a Phenolic resin car in 1941 (Figure 3). The car was a Phenolic composite with 70 percent cellulose hemp fibre with soybean meal as an extender. The composite car was put aside with the mechanisation for WW2 and largely forgotten. With oil's abundance, low price and good chemistries, petrochemicals were a less expensive feedstock than the renewable alternatives (Composites Technology 2008).



Figure 3: The 1941 Ford which was made from Phenolic resin and hemp fibre

In commercial manufacture Phenolic resin is produced mainly from benzene. Its additive formaldehyde is made on a commercial scale from methanol (Simpson 1995, p.65). Phenolic resins are available in flake, powder, and liquid form dependent on the applications in which they are used (ASM International 2003, p.27).

2.5 Phenolic Resin derived from renewable materials

There are a number of polymer resins being developed from renewable materials. This project has used Cardanol but there are a large number of other types covered in Section 2.13.

2.5.1 Cashew Nut Shell Liquid

Cashew Nut Shell Liquid (CNSL) is an alkylphenolic oil by-product made from the mesocarp of the cashew nut (Anacardium occidentalis) waste shell (Maffezzolia et al 2004). Yadav & Srivastava (2007) found it to be one of the few major and economic sources of naturally occurring phenols and regarded CNSL as a versatile and valuable raw material for polymer production.



Figure 4: Cashew nut (seaexport 2009)

The produced alkylphelonic oil is mainly composed of anacardic acid and lesser amounts of Cardanol and Cardol. The structure of the CNSL constituents is in Figure 5.



Figure 5: Constituents of crude CNSL (Chuayjuljit et al, 2006, p.2)

The makeup of these constituents is dependent on the extraction process used, there are two main processes:

- The cold -solvent process $CNSL \rightarrow Natural CNSL$
- The hot-oil and/or roasting process $CNSL \rightarrow Technical CNSL$

The varied composition of the two types of CNSL is given in Table 1:

Table 1: Composition of Phenolic components in natural and technical CNSL (Mwaikambo2009, p.50, Menon et al, 1985, Gedam and Sampathkumaran, 1986)

Components	Natural CNSL %	Technical CNSL %
Anacardic acid	77.02	-
Cardanol	2.37	82.15
Cardol	16.77	13.71
2-Methyl cardol	2.83	4.1
Polymers by difference	1.01	0.04

The Anacardic acid in natural CNSL is further decarboxylated to produce anacardol and then with hydrogenation yields cardanol (Mwaikambo 2009, p.50, Pansare, 1964). The diagram of this process is in Figure 6.



Figure 6: Conversion of Anacardic acid into anacardol and then cardanol (Chuayjuljit, Rattanametangkool & Potiyaraj, 2006, p.2)

2.5.2 Cardanol

Cardanol is distilled from CNSL and its chemistry is unusual, as Attanasi et al (2009, p.71) noted, that it is difficult to make phenols with a long unsaturated chain such as the cardanol fifteen carbon atom chain. Additionally it was found to be a 'peculiar, simple and easily available starting material for the synthesis of various derivatives'.

The aliphatic side chains of cardanol usually carry one, two, or three double bonds, and are a mixture of four components varying in the degree of side-chain unsaturation (Chuayjuljit et al 2006, p.1). Because of its Phenolic nature, cardanol can react with

formaldehyde in the same way as oil based Phenolics. This reaction through condensation polymerisation produces Cardanol-Phenol Formaldehyde (CPF) resins (Chuayjuljit et al 2006, p.1).

2.6 Thermoset Resins

All synthetic plastics are divided into two groups; thermoplastics and thermosets, these are dependent on the chemical bonding (Cecil 2008, p.15). PF and CPF resins are thermosets meaning they cannot be reshaped when reheated. The resin types are determined by the makeup of the catalyst during the synthesis of the resin.

2.6.1 Types of Phenolic Thermoset Resin

The two types of Phenolic resins are Resol and Novolac. The type being made is dependent on the pH of the catalyst and the ratio of phenol to formaldehyde. Resols are made under alkaline conditions with a ratio of phenol to formaldehyde from 1:1 to 1:3 (Tyberg 2000, p.1). An outline is given in Figure 7.



Figure 7: Synthesis pathway of Resol and Novolac (Adapted from Painter and Coleman 2000, p.1).

2.7 Synthesis of Phenolic Formaldehyde resin

Phenolic formaldehyde resins (resols) are the resins used in this project. The phenol formaldehyde reaction is a base catalysed electrophilic aromatic substitution to form hydroxymethylphenols as in Figure 8 (Tyberg 2000, p.4).





Figure 8: Reaction of phenol with formaldehyde (Adapted from Tyberg 2000, p.4)

The hydroxymethylphenols form the prepolymers and depending on pH and temperature lead to dihydroxydibenzylether or dihydroxydiphenylmethane (Tyberg 2000, p.5). These are in Figure 9.



Figure 9: Resol prepolymer formation (Adapted from Tyberg 2000, p.4)

The reactive methylol groups in the resols molecules condense when heated, forming a fused-larger molecule or crosslinked resin (Painter and Coleman 2000, p.1). The prepolymers are heated to promote crosslinking of the resin from further reaction of hydroxymethylphenols. Water and formaldehyde are produced and volatilize during curing. This release of volatiles results in networks with a significant amount of voids that can detract from the mechanical properties (Tyberg 2000, p.5).

2.8 Synthesis of Cardanol Phenolic Formaldehyde resin

The synthesis of Cardanol Phenolic Formaldehyde (CPF) resin is essentially the same process as with PF resins but with some minor differences. Firstly the Cardanol must be bonded with the PF resin in an oil bath at an elevated temperature.

2.8.1 Bonding of Cardanol and Phenolic Formaldehyde

CPF resins are prepared from the reactions between formaldehyde and cardanol. They are dependent on the formaldehyde-to-cardanol ratio and the reaction conditions employed (Chuayjuljit et al, 2006, p.2).

The chemicals Phenol, Cardanol and Formaldehyde are reacted together in the presence of Sodium Hydroxide (NaOH) Catalyst, this solution is then put into a glass flask and then rotated in an oil bath within a fume cupboard (see Appendix E for apparatus). The diagram of this process is in Figure 10.



Figure 10: Phenol and Cardanol Bonding to create CPF resin (Cardona 2009)

The solution is in the oil bath at:

- 60°C for 1 hour,
- 80°C 1 hour and
- Then 60°C 1 hour.

From this a new modified Phenolic resin (CPF) with two types of bonds is created in Figure 11.





Figure 11: Bonded Liquid Cardanol Resole Phenolic Resin (Cardona 2009)

2.8.2 Synthesis of CPF resins

The CPF resins and their composites are then reacted in the presence of an acid catalyst using the same method as for PF resin. The samples are cured at room temperature for 24 hours, followed by post-curing at 80°C for 4 hours. The completed CPF thermoset matrix is outline in Figure 12.



Figure 12: Cardanol Phenolic Formaldehyde Thermoset (Cardona 2009)

2.9 Phenolic Resin Characteristics

The cured Phenolic polymer resin is a giant single molecule whose shape cannot be changed except by breaking bonds between atoms (Bédard and Riedl, 1990, p1). On being heated a thermosetting plastic first flows and then becomes crosslinked (cured) and will not soften significantly if reheated. Scrap from the process cannot be reused (Simpson 1995, p.27). Phenolics can be moulded by compression, transfer, and injection mould to close tolerances (ASM International 2003, p.27). Thermosetting matrix materials such as Phenolics are brittle due to the high crosslink densities (Tyberg 2000, p. 55). The main characteristics of Phenolic resin are summed in Table 2.

Advantages	Source
Dimensional and thermal stability	(ASM International 2003, p.27)
Exceptional load bearing characteristics at elevated	(ASM International 2003, p.27)
temperatures	(1511 International 2000, p.27)

Table 2: Phenolic Resin Characteristics

Range of use at approximately 120° to 260° C	(ASM International 2003, p.25)
Good chemical resistance	(ASM International 2003, p.25)
Low water absorption	(ASM International 2003, p.25)
Good electric strength	(ASM International 2003, p.25)
High char yield, low smoke volume and low smoke toxicity	(Simpson 1995, p.65)
Superior oxidative resistance relative to steel	(Tyberg 2000, p.1)
Better freeze-thaw durability relative to concrete	(Tyberg 2000, p.1)
High chemical resistance, hard facing and are relatively low cost	(Bédard and Riedl, 1990, p1).
Excellent flame retardance	(Tyberg 2000, p.8)
Self-ignition temperature of 482°C	(Tyberg 2000, p.8)
Superior thermal resistance and chemical resistance as well as adhesive properties	(Sumitomo Bakelite 2009, p.3).
Disadvantages	
Fairly good insulation resistance but not as good as other resins	(Simpson 1995, p.65)
They can be fairly brittle	(Simpson 1995, p.65)
Use of organic materials in construction is limited by their high combustibility	(Tyberg 2000, p.1)

2.10 Cardanol Phenolic Formaldehyde Resin Characteristics

Cardanol has a Phenolic structure substituted in *meta* position (Figure 13) with a long alkyl chain not easily obtainable by synthetic routes (Attanasi et al 2009, p.76). The long side chains impart flexibility because of internal plasticizing resulting in the formation of soft resins at elevated temperatures. This is unlike PF resins, which are hard and brittle (Chuayjuljit et al 2006, p.2). The side chain also imparts a hydrophobic nature making the resin water repellent and resistant to weathering (Raquez et al 2010, p.3).



cardanols

Figure 13: Cardanol; n = 0, 2, 4 & 6 (Mwaikambo 2009, p.51)

Chuayjuljit et al (2006, p.1) note that 'CPF resins are special Phenolic materials having properties such as high-temperature resistance, modulus retention at elevated temperatures, resistance to chemicals and detergents, high surface hardness, and low cost'. NEC has found CPF resin composites have high durability and malleability with characteristics better than other natural polymers such as Polylactic Acid (PLA) and Cellulose Acetate (CA) resin (ringier plastics, 2010).

2.11 Phenolic Resin uses

Phenolics low smoke volume and low smoke toxicity has had them specified in applications where escape from fire may be restricted, such as passenger transport applications and public buildings (Simpson 1995, p.65).







Figure 14: Applications of Phenolic resin (Value to Wood 2009)

Applications include:

- Impregnation of timber under pressure to increase hardness; I beam LHS of Figure 14.
- As a water and insect proof coating; centre of Figure 14, and
- As an adhesive to make laminates; RHS of Figure 14. (Value to Wood 2009)

Phenolic resin has a very broad number of uses, some of these uses are in Table 3.

Phenolic resin uses	Source	
nylon and polyester fibres	(Bédard and Riedl, 1990, p1)	
rubbers	" "	
polyethylene	" "	
polypropylene		
plexiglass	دد در	
adhesives	دد دد	

Table 3: Table of Phenolic resin uses

Since Phenolic products cannot be melted by heating once they have been cured, they can be reused as fuel (thermal recycling) or reused as filler after being crushed into fine particles (material recycling) (Sumitomo Bakelite 2009, p.3).

2.12 Cardanol Phenolic Composite Resin Uses

Cardanol uses have been generally in industrial applications such as brake pads due to high wear resistance. But now more high end electronic equipment such as Laptops, computer accessories, and mobile phones are being manufactured using CPF composites. This is in part for marketing a green product (see Figure 15) but also for production economics and the plastics unique characteristics.



Figure 15: NEC mobile phone made from CPF resin and cellulose fibre

2.13 Development of other resins and composites

Renewable resin polymer development in the United States has been largely focused on the use of soybean oil and corn oil, mainly due to the facilities and equipment being in place to produce oil from these feedstock (Composites Technology 2008).

Biopolymers, such as polyhydroxyalkanoates and cellulose, are obtained directly from living organisms such as plants and/or bacteria. They also include polymers prepared in the laboratory by modifying biopolymers (such as cellulose triacetate) or by polymerizing bio-based monomers e.g. polylactic acid or PLA (Bicerano & Associates Consulting Inc 2009). Plant oil (triglycerides) resins are obtained by thermal or cationic copolymerization, and lignin a Phenolic has been considered as a promising substitute for phenol in phenol–formaldehyde-based resins (Raquez et al 2010, p.2) A list of renewable polymers is in Table 4.

Renewable polymers	
Chitin	Lignin
Cellulose	Non Starch Polysaccharides
Hemicellulose	Proteins
Starch	Polyhydroxyalkanoates
triglycerides	Tannin compounds

Table 4: Renewable polymers (Vilapana et al 2010, p.4), (Bicerano & AssociatesConsulting Inc 2009) & (Raquez et al 2010)

There is concern that renewable sources such as plant resources from feed grain that is used in plastics such as PLA, may lead to the possibility of future food shortages. This has emphasized the importance of using non-edible plant resources (ringier blog spot 2010).

2.14 Thermoset Resin Fillers and Reinforcement

2.14.1 Introduction

The main advantage to using renewable or waste materials as fillers is economics; industry is starting to look seriously at using renewable or waste materials in their products. This is in part to community expectations, government regulation, economics and marketing. 'In recent years, (bio) plastics composed from plant resources attracted much attention as an effective measure to reduce the depletion of petroleum resources and alleviate global warming' (ringier, 2010).

Fillers or extenders are used as a bulking agent and /or reinforcement in thermoset resins and can be discrete or continuous particles in the composite matrix (Mwaikambo 2009, p.1). Thermoset fillers are added to the polymer matrix for two main reasons, one to improve the properties of the resin, such as mechanical and thermal properties

(Marcovich et al 2000, p.1) and secondly fillers are used for reasons of economy as an extender making a greater volume and therefore lowering the cost of production.

Vilaplana et al (2010, p.4) propose that as fillers /reinforcements, a wide range of materials from various origins can be used in the manufacture of sustainable biocomposites. Rowell et al (1997, p.3) proposed that the use of waste materials from agriculture could have a tremendous impact in lowering the usage of petroleum based plastics.

Vilaplana et al (2010, p.4) have grouped the fillers /reinforcements as follows:

(i) Lignocellulosic materials, including wood flour, natural fibres.

(ii) Inorganic nano-fillers/ reinforcements that may contribute to the enhanced biodegradability of the resulting nano- biocomposites.

(iii) Different synthetic waste materials that can be recycled and incorporated into new products as fillers/ reinforcements.

Because this list was to discuss bio-composites it does not include synthetic materials that are manufactured to purpose as fillers. These are added to the list for this study of thermoset fillers. Synthetic fillers are still an option where a particular characteristic is required in a composite resin. This gives a fourth origin of materials for thermoset resins: (iv) Man made synthetic materials

2.14.2 Lignocellulosic Thermoset Resin Fillers

Lignocellulosic products need to be processed to extract them from their natural origin and modified to achieve the required characteristics (Vilapana et al 2010, p.4). Some newer examples of these products are cellulose whiskers and starch nanocrystals.

2.14.2.1 Lignocellulosic Filler Advantages

The cost of natural fibres are in general less than that of the thermoset plastic and a high fibre loading can result in significant material cost savings. Marcovich et al (2000, p.1) found natural fillers are utilized because of their low density, but also because of good mechanical properties and the plus that they are able to be disposed of at the end of their use life by biodegradation and/or combustion.

Rowell et al (1997, p.49) found Cellulose fibres to be soft and non-abrasive with high possible filling levels. The reduced equipment abrasion reduced re-tooling costs through the use of agricultural based fibres.

2.14.2.2 Lignocellulosic Filler Disadvantages

The use of natural fibres is complicated as fibres can vary in chemical composition and physical properties, generic characteristics, place of growth and preparation conditions (Mwaikambo 2009, p.3, Gassan et al, 1996).

The presence of hydroxyl groups in plant fibres also cause reactivity with water, causing a lack of adhesion between the hydrophilic cellulose fibres and the hydrophobic synthetic resins due to their incompatibility (Mwaikambo 2009, p.3 & 4). It is difficult to entirely eliminate the absorption of moisture without using expensive surface barriers created from chemical modification of some of the hydroxyl groups present (Rowell et al 1997, p.4, Rowell *et al*, 1986).

Dispersion levels of the fibres in the resin matrix are also negatives. Manthey (2009, p.49) using hemp fibres found difficulty in achieving uniform fibre dispersion in the resin matrix and that this could cause variations of strength in the composite.

Rowell et al (1997, p.4) found the primary drawback of the use of lignocellulosic fibres was found by to be the lower processing temperatures required in manufacture (about 200°C), this was due to the possibility of degradation of the fibres and/or the possibility of volatile emissions effecting composite properties.

Vilaplana et al (2010, p.4) have expressed concern that production of Biomass feedstock must be suitably selected so that the food supply availability and food costs are not interfered by the production of biomass.

2.14.3 Inorganic Thermoset Resin Fillers

Kaolinite, Talcum are two of the older filler types that are readily available. Carbon nanotubes (clay platelets) are example of inorganic thermoset resin fillers that are biodegradable (Bicerano & Associates Consulting Inc 2009).

2.14.3.1 Inorganic Filler Advantages

Talcum is used as it imparts even texture and softness. This is used in automotive body applications as it is able to be sanded after curing.

2.14.3.2 Inorganic Filler Disadvantages

The main disadvantage with inorganic fillers, is that as they are sourced from mining their resources are finite.

2.14.4 Synthetic Waste Material Thermoset Resin Fillers

Fillers made from by-products of industry are at the present moment highly desirable as the use of waste materials as filler has both an economic and an environmental benefit. An example is fly ash which is used as filler in resin, fly ash is a finely divided residue that resulting from the combustion of pulverized coal. Fly ash consists of silt-sized particles which are generally spherical, typically ranging in size between 10 and 100 micron (US department of Transportation 2006). Other recently developed synthetic fillers are glass microspheres RHS of Figure 16. These small discrete particles of Fly ash and glass microspheres are called Nanofillers.



Figure 16: Fly ash particles LHS (US department of Transportation 2006) and glass microspheres RHS (concrete canoe 2010)

Some other products are made from rubber tyres, recycled polyethylene terephthalate (PET) and post-industrial polystyrene (McConnell 2009).

2.14.4.1 Synthetic Waste Material Filler Advantages

Hollow inorganic microspheres have low bulk density, raw material cost savings, reducing weight and improve impact properties according to a number of websites that sell these materials. Some of these fillers are made to handle heat, have high strength
and are chemically inert. Gurney (2006, p.1) found a cost reduction in using microspheres as fillers as weight is reduced and cost is reduced because fillers are cheaper than the resin that they replace.

2.14.4.2 Synthetic Waste Material Filler Disadvantages

Degradation of the resin matrix or polymer involves several physical and/or chemical processes which lead to significant deterioration of the quality of the polymeric materials. However, the environmental and toxicological implications of such nano-sized fillers and the derived nanocomposites are not fully understood yet (Vilaplana et al 2010 p.7). There is also the possibility of mercury off-gassing from fly ash (Ecological Building Network 2010).

2.14.5 Synthetic Man Made thermoset resin fillers

Some of the most common synthetic man made thermoset resin fillers are glass fibres, carbon fibres, boron fibres, and aramid fibres (Mwaikambo 2009, p.1). Phenolic resin matrix systems such as prepregs are used with these type of fibres. When the themoset resin is cured the prepreg 'transforms into a solid structural material that is highly durable, temperature resistant, exceptionally stiff and lightweight' (Hexcel 2010).

E-glass reinforced Phenolic prepregs have also been used in interior and exterior components aboard high speed trains such as the AVE S103 in Spain and the Combino Plus in Portugal (McConnell 2008).

2.14.5.1 Synthetic Man Made Filler advantages

Synthetic fibres can be produced with properties engineered for particular applications, (Mwaikambo 2009, p.1), this means that the integrity and quality of the filler can be rigidly controlled resulting in uniform products. The Phenolics have a good surface finish, curing is in short cycles, 10 minutes at 160° C.

2.14.5.2 Synthetic Man Made Filler disadvantages

The main disadvantages of manmade synthetic fillers is cost, as petroleum prices increase the costs to produce synthetics increase. Today costs are recognised as being environmental as well as economic costs.

2.15 Testing of Modified Phenolic Resin Samples

2.15.1 Introduction

This section outlines the methods of testing, the requirements for each method and the testing machinery required.

2.15.2 Flexural Testing

The flexural test results are in three parts:

- Flexural modulus (Youngs Modulus) in MPa
- Strain as a percentage
- Stress in MPa

2.15.3 DMA Testing

Dynamic Mechanical Analysis (DMA) is a testing technique used to measure the mechanical properties of materials. DMA measures the viscoelastic properties using either transient or dynamic oscillatory tests. Dynamic oscillatory tests have been carried out on these samples. The dynamic oscillatory test is where a stress or strain is applied to the material and the resultant strain or stress is measured (see LHS of Figure 17).



Figure 17: Dynamic Oscillatory test details (TA Instruments 2008)

Also measured is the phase difference (δ) between the two sine waves.

From RHS of Figure 17:

Youngs Modulus E* = stress/strain

The storage modulus, E' =
$$E^* \cos \delta$$

The loss modulus, E'' = $E^* \sin \delta$
Tan delta (δ) = loss modulus/ storage modulus = E''/ E'

The storage modulus is the elastic component and related to the sample's stiffness. The loss modulus is the viscous component and is related to the sample's ability to dissipate mechanical energy through molecular motion. Tan delta (δ) or tangent of phase difference provides information on the relationship between the elastic and inelastic components. Glass transition temperature (Tg) is also measured using DMA testing (Figure 18).



Figure 18: An example of the attributes measurable using DMA testing

Storage modulus and Glass Transition Temperature (Tg) are the properties used for comparison between the various composite sample combinations in this project. The DMA testing machine is in Figure 19, it has a sample ready to be tested in the clamps. The blue top of the machine automatically swivels around to cover the sample when the testing sequence if initiated.



Figure 19: DMA test machine with sample for testing

2.16 Risk Management of Modified Phenolic Resin Composites

2.16.1 Introduction

The risk management process should be an integral part of management (AS/NZS ISO 31000 2009, p.13). The need for risk management is backed by legislative requirements of the Qld Workplace Health and Safety Act 1995. The risk management process at the Centre of Excellence in Engineered Fibre Composites (CEEFC) begins with an on-site safety induction.

2.16.2 Safety Induction

The on-site safety induction is given before starting work at the CEEFC. The layout, emergency exits, location of fire extinguishers, and assembly points are given. The safe operating procedure, possible hazards and personal protective equipment (PPE) for each machine to be used is given. At the completion of the instruction for each machine each machine is signed off by the person giving the induction. Only when the induction is completed, access to the machinery is allowed.



Figure 20: Safety signage in the CEEFC workshop

2.16.3 Risk Assessment

Risk assessment is the overall process of risk identification, risk analysis and risk evaluation (AS/NZS ISO 31000 2009, p.17). The on-site induction has carried out this process for the workplace at the CEEFC. A risk assessment is now completed for each task that I will perform in carrying out my project.

Project tasks are:

- Preparation of composite resin samples
- Preparation and shaping of composite resin samples for testing
- Testing of composite resin samples
- Disposal of composite resin samples

These tasks are put into a risk assessment table (Appendix F: Risk Assessment Table) and these tasks and related subtasks are rated against risk identification, risk analysis and risk evaluation.

2.16.4 Actions against Risk

The actions against the identified risks have resulted in all final risks having a low to very low likelihood of incidence. These actions are in the table of Risk Assessment (Appendix F).

2.16.5 Monitoring and review of Risk

As part of the risk assessment process both monitoring and review should planned as part of the risk management process. This involves checking or surveillance of activities. As part of this process the risk assessment table (Appendix F).

2.17 Consequential Effects

2.17.1 Introduction

The intended effect of my research is to contribute to existing knowledge on using renewable products to extend and eventually replace Phenolic resins made from petrochemicals. Using the Institution of Engineers initiative; *Towards Sustainable Engineering Practice: Engineering Frameworks for Sustainability*, I have created an analysis of aspects of sustainability of my project as a Table (Appendix G). This Table has been used to rate possible implications and effects due to my project work.

2.17.2 Consequences

I have compared my project work against the criteria of ten aspects of sustainability (Appendix G) and have given a rating of sustainability of 78%. The objectives of my project are broad but from this research it may be possible to partially or fully replace of petrochemical products with renewable products. It is difficult to see a difference being made to part 8 and part 10 of aspects of sustainability. These are:

- The identifying a potential impact of my project work on eradication of poverty, the reduction in differences in living standards and the full participation of women, youth and indigenous people.
- How my project work, and/or its outcomes, contribute to international understanding.

The main detraction is that it is difficult to see a difference made to these problems by shifting the focus of Phenolic thermoset production from petrochemicals to renewable feedstock.

2.18 Ethical responsibility

The ethical responsibilities of this project have been compared to the Engineers Australia 2000 Code of Ethics. The project has been compared against the criteria of the nine tenets under this code in Appendix H.

The tenets express the shared commitment of the members of Engineers Australia to act in a manner to uphold these principles and to regulate working habits and relationships (The Institution of Engineers Australia 2000). This project has been found to uphold these tenets and therefore is ethically responsible.

2.19 Timelines

The timelines for the various aspects of the Research Project are in Table 5 below, the final date for submission is October 28th 2010.

	0	Task Name	Duration	Start	Finish	Predecessors
1		Research Project Planning	196 days	Thu 28/01/10	Thu 28/10/10	
2		Project Selection	30 days	Thu 28/01/10	Wed 10/03/10	
3		Project specification	10 days	Wed 10/03/10	Tue 23/03/10	2
4		Project appreciation	50 days	Thu 11/03/10	Wed 19/05/10	3
5		Submit Extended Extract	20 days	Thu 20/05/10	Wed 16/06/10	4
6		Submit Draft Dissertation to Supervisor	7 days	Thu 17/06/10	Fri 25/06/10	5
7		Complete Presentation & continue Dissertation	44 days	Mon 28/06/10	Thu 26/08/10	6
8		Ready to Present Paper	0 days	Fri 27/08/10	Fri 27/08/10	7
9		Complete Dissertation	15 days	Fri 27/08/10	Thu 16/09/10	8
10		Review of Dissertation	28 days	Fri 17/09/10	Tue 26/10/10	9
11		Submit Dissertation	2 days	Wed 27/10/10	Thu 28/10/10	10

Table 5: Timelines for project completion



3 Research & Methodology

3.1 Introduction

In this chapter the research and methodology are applied to synthesise the PF and CPF composite resin samples for characterisation and testing. The results will then be compared to results obtained in this project and others identified in the literature review. The synthesis, testing and characterisation of the PF and CPF composite resins is to be carried out in the laboratory at the Centre of Excellence in Engineering Fibre Composites (CEEFC) at the University of Southern Queensland.

3.2 Materials

3.2.1 Phenolic Resin Used in Synthesis

The Phenolic resin used is J2027L (Hexion Cellobond) a commercial resin manufactured by Hexion Specialty Chemicals Pty Ltd.

3.2.2 Catalyst Used in Synthesis

The catalyst was manufactured in the lab at the Centre of Excellence in Engineering Fibre Composites (CEEFC) at the University of Southern Queensland (USQ) and is of similar makeup to the commercial catalyst Phencat 10 also manufactured by Hexion Specialty Chemicals Pty Ltd. The proportions of the catalyst were:

Chemical Name	Composition by Weight
P-Toluene sulfonic Acid (PTSA)	50%
Water	30%
Phosphoric Acid	20%
Total	100%

 Table 6: Catalyst proportions

3.2.3 Cardanol used in Synthesis

The Cardanol used in the project is produced by the company Satya PTY LTD.

3.2.4 Thermoset Resin Filler Used in Synthesis

The Thermoset resin filler was sawdust sourced from a local sawmill. The sawdust was passed through a sieve to 300 micron. These fibres were chosen for the lost cost, ease of repeatability of the tests and the fact that sawdust is a waste material. It was anticipated that the modulus of the fibres due to their small size would not have a large effect on the overall modulus of the sample and may only act as filler.

3.3 Preparation and Synthesis of Test Samples



Figure 21: Phenolic resin samples with the different volumes of wood fibre

Figure 21 above shows the variation in the synthesised samples. From left to right the samples are 25% and down to 5% wood fibre.

3.3.1 Preparation for Synthesis of Test Samples

During the preparation stage the chemicals for use in the project are sourced, the inductions to use the testing machinery are carried out, and sample moulds prepared. The samples are manufactured according to the type of test required.

3.3.2 Equipment for Synthesis of Test Samples

Equipment used is; scales, disposable plastic containers (round), pipettes, and oven. The scales were the main piece of equipment used as the various materials of the composite resins were measured by weight.

3.3.3 Determination of PF and CPF Composite Sample Composition

Early in the project it was determined that the samples would follow from a pattern of synthesis and characterisation of samples and then study these results to meet the requirements of the project.

- Synthesis of PF resins modified with different amounts of Cardanol (CPF)
- Preparation and testing of composites using the modified CPF resins and different amounts of the renewable materials (sawdust).

To meet these requirements these steps were indentified to begin sample preparation for testing, in summary these are:

- Part 1; preparation of sawdust wood fibre
- Part 2; preparation of a suitable catalyst
- Part 3; preparation of a trial 100% Phenolic Formaldehyde (PF) resin sample
- Part 4; preparation of CPF resin
- Part 5; preparation of PF and CPF samples with varying amounts of wood fibre
- Part 6; preparation of CPF samples with varying amounts of blended CPF resin and unblended PF resin and wood fibre

3.3.4 Steps in Sample Preparation for synthesis

The steps are outlined here for the PF and CPF composite sample preparation.

3.3.4.1 Part 1; preparation of sawdust wood fibre

The wood fibres were produced from sieving sawdust passing 300 micron and strored in a dry container.

3.3.4.2 Part 2; preparation of a suitable catalyst

From research a catalyst was determined for Phenolic resins, this is similar to the commercial product Phencat 10. The initial amount of catalyst trialled with the 100 gram sample of PF resin was 6% (6 g).

3.3.4.3 Part 3; preparation of a trial 100% Phenolic Formaldehyde (PF) resin sample.

A PF resin sample is prepared as a baseline for the PF composite samples and CPF composite samples to be compared against. The PF resin was measured into a plastic

container and the catalyst added. Within half an hour the sample was extremely hot due to the rapidity of the reaction. The cured sample is in Figure 22. It was determined the amount of catalyst should be reduced to 5% and a fresh batch of PTSA used in the preparation of more catalyst. This new supply of catalyst was trialled again and found to produce a stable reaction.



Figure 23: PF resin sample produced during study showing excessive voids from excess catalyst in reaction.

3.3.4.4 Part 4; preparation of CPF resin

CPF resin is prepared by bonding the resin at elevated temperatures. The chemicals Phenol, Cardanol and Formaldehyde are reacted together in the presence of Sodium Hydroxide (NaOH) Catalyst in the CEEFC laboratory. This solution is then put into a glass flask and then rotated in an oil bath within a fume cupboard, see Figure 24.

The solution is in the oil bath at:

- 60°C for 1 hour,
- 80°C 1 hour and
- Then 60°C 1 hour.

From this process a Cardanol Phenolic Formaldehyde resin is created.



Figure 24: Oil bath in a fume cupboard (LHS) and the CEEFC laboratory RHS.

3.3.4.5 Part 5; preparation of PF and CPF samples with varying amounts of wood fibre

The PF and CPF resin samples were mixed with sawdust wood fibre in a range from 0% to 25% by weight. The steps were:

Step 1. Mix the 100g of resin thoroughly with the 5g of catalyst for the PF samples and 6% for the CPF samples

Step 2. Mix in the sawdust wood fibre at 0% to 25% by weight.

Step 3. Let stand to cure for 24 hours

Step 4. Into the oven at 80° for 4 hours for post curing

Note. In step 1 the CPF resin sample had a much lower viscosity and an increase of 1% was used to enable the resin to react and crosslink adequately.

3.3.4.6 Part 6; preparation of CPF samples with varying amounts of blended Cardanol and PF resin and unblended PF resin and wood fibre

Two different blends were used, these were:

1. Cardanol blended at 20% with PF resin and added 50-50 to make a Phenolic mix.

2. Cardanol blended at 30% with PF resin and added 50-50 to make a Phenolic mix.

To both of these mixes were added sawdust wood fibre at 0% to 25% by weight.

3.3.5 Curing of Test Samples

The test samples are cured in their plastic containers at:

- Room temperature for 24 hours,
- Followed by Post-curing in an industrial oven at 80°C for 4 hours.

3.4 The Cutting and Shaping of Test Samples

Both the flexural test samples and the Dynamic Mechanical Analysis (DMA) samples were cut from the same 90mm diameter cured resin samples. This is for efficiency in production of the samples, but more importantly allows for comparison between the two different types of testing.



Figure 25: Machinery for cutting and shaping of samples; LHS tile cutter and RHS face grinder.

3.4.1 Flexural Testing Sample Production

Test pieces are cut with a tile cutter (Figure 25) from each of the 90mm diameter cured resin samples into three test samples 10mm thick, these are ground into shape with a surface grinder (Figure 25) until of uniform thickness and width. Exact measurements are taken before testing for entry into the computer. The samples are now ready for testing. The machine used is a MTS Alliance RT/10 universal testing machine.

3.4.2 DMA Testing Sample Production

The test pieces are cut at the same time as the pieces for the flexural test and using the same method. There is only one sample cut with the tile cutter 4mm thick and this is ground using a surface grinder until of uniform thickness and width. The samples are now ready for testing; an example is in Figure 26.



Figure 26: Sample ready for testing in the DMA machine

3.4.3 Tensile Testing Sample Production

The moulds were cleaned and waxed and the prepared 100% PF resins were poured into the mould and left to cure. The samples unfortunately were not successful, Figure 27 has the image of the sample after curing. None of the samples were able to be removed from the mould without breakage along the stem of the samples.



Figure 27: Tensile samples after curing

The enclosed nature of the mould did not have sufficient surface area for the dissipation of water and formaldehyde vapour in the condensation reaction. This created a boiling effect where the moisture from the reaction created voids within the resin samples. The voids were retained into the cured samples and undermined the test samples integrity. The 100% PF with 0% wood fibre samples were the only type of samples trialled, it was

not expected that the CPF composites would be any better so these were not trialled. Therefore there are no tensile samples to test.

3.4.4 Costs of Materials

There were 6 batches with 28 samples produced. Each sample was 100g and is rounded off for the purpose of simplifying the cost as it is low.

Material	Cost per L	Amount (L)		Total
Phenol Formaldehyde	\$6.00	2.80	(28 x 100 ml)	\$16.80
Cardanol	\$2.50	0.840	(28 x 30 ml)	\$ 2.10
PTSA	\$15.00	0.140	(28 x 5ml)	\$ 2.10
Sawdust	\$0.00	28 x 75g		\$ 0
				\$21.00

The cost of materials is summarised in the following table:

3.5 Concluding Remarks

This chapter has demonstrated the methodology to prepare and synthesise the PF and CPF composite resin samples for characterisation and testing. There are no tensile test results due to the tensile samples poor integrity.

4 Characterisation and Testing of Modified Composite Resins

4.1 Introduction

This chapter provides the characterisation and testing of modified PF and CPF resins. The characterisation includes the identification of the composites characteristics and discussion of the testing results using flexural tests and DMA tests. Characterisation will include:

- Graphing of the test results to clarify the variations within the samples and the different composite blends.
- Comparison to the stated objectives of the project

4.2 Testing

The test samples are named from their composition, some sample names may have an suffix on the end of the names such as S1or S8. This means sample 1 or sample 8. These are:

PF - 100	100% Phenolic formaldehyde resin matrix
CPF - 100 - C - 20	Cardanol Phenolic formaldehyde resin matrix with 20%
	Cardanol in mix.
CPF - 50 - C - 20	Cardanol Phenolic formaldehyde resin matrix with 20%
	Cardanol in mix is 50% of resin with the balance (the
	other 50%) being Phenolic formaldehyde resin.
CPF - 100 - C - 30	Cardanol Phenolic formaldehyde resin matrix with 30%
	Cardanol in mix.
CPF - 50 - C - 30	Cardanol Phenolic formaldehyde resin matrix with 30%
	Cardanol in mix is 50% of resin with the balance (the
	other 50%) being Phenolic formaldehyde resin.

Table 7:	Naming	of sa	mples	against	composition
		~ ~ ~ ~ ~ ~			

4.2.1 Flexural testing

The flexural testing or three point bending test (Figure 28) is carried out using the MTS Alliance RT/10 with the flexural testing attachments. A gap of 64mm is required between the two lower points with the weight descending at 2mm per minute on to the sample.

The flexural test results are in three parts:

- Flexural modulus (Youngs Modulus) in MPa
- Strain as a percentage
- Stress in MPa



Figure 28: Flexural testing of CPF resin sample

4.2.2 DMA Testing

Dynamic Mechanical Analysis is used to find the mechanical and thermal properties of the tested composite samples. Storage modulus is the mechanical test and Glass Transition Temperature (Tg) is the thermal test. The properties from the tests are used for comparison between the various composite sample combinations in this project and other projects. The machine used is the TA instruments Q800.

4.3 Flexural Testing Results

The full tables and graphs are in Appendix B Flexural test results and Appendix C DMA test results. There are no results for tensile testing due to the poor integrity of the produced samples (see tensile sample production, page 50).

4.3.1 Flexural Modulus results

4.3.1.1 Wood Fibre flexural results

The wood fibre results in the graph in Figure 29 are taken from Table 8. Observations of the effect of Flexural modulus from filler percentage are:

- The 100% Phenolic formaldehyde sample (PF 100%) demonstrates the stiffness of PF resin. It has an overall higher flexural modulus than the other tested samples, except for CPF 50 C-20 at 0% wood fibre.
- Wood fibre at 5% gives PF 100% gives the peak value at 2590 Pa.
- The other CPF samples have had a lessening of flexural modulus with the increasing percentage of wood fibre.
- The CPF sample with CPF -50% C- 30% (50% PF + 50% CPF with 30% Cardanol) appears to be the most stable over all of the wood fibre percentages. This blend and mix has had a more linear result with the lessening flexural modulus.
- Wood fibre at a filler percentage of 5% gives in most samples (3 of the 5) the highest modulus.



Figure 29: Graph of all Flexural Modulus vs. Filler % Results

Wood	PF - 100%	CPF - 50%	CPF - 100% -	CPF - 100%	CPF-50%
Fibre %		- C - 20%	C - 20%	- C - 30%	- C-30%
WF - 0	1768	2112	698	1383	1413
WF - 5	2590	1633	889	813	1499
WF - 10	2339	227	800	331	972
WF - 15	1845	6	697	56	591
WF - 20	626	-	127	-	424
WF - 25	601	-8	-	-	-

Table 8: All Flexural Modulus results (Pa) against wood fibre percentage

4.3.1.2 Cardanol Flexural Modulus Results - CPF 100%

The effects on flexural modulus by Cardanol percentage for CPF 100% are:

- The increase in the percentage of Cardanol results in the decrease of flexural modulus.
- Wood fibre in the range 5%, 10% and 15% gives a higher flexural modulus than 0% wood fibre for C-0% and C-20%.
- Within the samples, the flexural modulus falls with the increase of Cardanol percentage.
- The modulus across all samples falls with the increase in wood fibre.



Figure 30: Graph of Flexural Modulus vs Cardanol % - CPF 100 %

4.3.1.3 Cardanol Flexural Modulus Results- CPF 50%

The effects on flexural modulus by Cardanol percentage for CPF 50% are:

- An increase in the percentage of Cardanol results in a decrease of flexural modulus for 0% and 5% wood fibre.
- The increase in the percentage of Cardanol at 10% and above of wood fibre results in a flexural modulus low at C-20% to a medium value at C-30%.
- The flexural modulus across all samples falls with the increase in wood fibre.



Figure 31: Graph of Flexural Modulus vs Cardanol % - CPF 50%

4.3.1.1 Summary of Flexural Modulus Results

The sample results have shown:

- A high modulus at a wood filler percentage of 5% for all percentages of Cardanol and both composite blends.
- The CPF sample with CPF -50% C- 30% appears to be the most stable for the varying percentages of wood fibre from the graph in Figure 29.
- The flexural modulus across all samples falls with the increase in wood fibre.
- CPF 100% generally falls in modulus with the increase in Cardanol percentage.
- CPF 50% generally falls in modulus to a low at 20% Cardanol and then increase to a secondary high (See Figure 29).

The relationship between the composite resins is shown in the next figure where the different CPF resins are compared with the same percentage of wood fibre (The full set is on page 89).





4.3.2 Flexural Strain results

4.3.2.1 Wood Fibre Flexural Strain Results

From the graph of strain vs filler in Figure 33, the effect of the Cardanol and wood fibres on the base PF resin can be seen. The observations for flexural strain effects from the wood fibre filler percentage are:

- The strain result for most of the samples is low, from 0% 5%.
- Above 5% the samples CPF 50% C 20% and CPF 100% C 30% increase in strain due to increased flexibility.
- Of the samples; PF 100%, CPF 50% C 30% and CPF 100% C 20% appear to be the most stable from all amounts of wood fibre.
- There does not appear to be a percentage of wood fibre that has given better qualities than any other amount.



Figure 33: Graph of all Strain vs Filler % Results

The results graphed in Figure 33 are taken from Table 9.

Wood Fibre %	PF - 100%	CPF - 50% - C - 20%	CPF - 100% - C - 20%	CPF - 100% - C - 30%	CPF-50% - C-30%
WF - 0	0.78	1.27	4.16	2.99	0.57
WF - 5	1.84	1.13	1.59	2.14	1.6
WF - 10	1.59	8.58	1.6	7.77	1.67
WF - 15	1.73	22.31	1.96	21.15	1.56
WF - 20	2.15	21.57	2.72	23.02	424
WF - 25	1.7	21.75	-	-	-

 Table 9: All Strain results (%) against wood fibre percentage

4.3.2.2 Cardanol Flexural Strain Results- CPF 100%

The effects on flexural strain by Cardanol percentage for CPF 100% are:

- 0% wood fibre has a peak at C-20%
- 5% wood fibre strain is almost flat and has no effect for the three values of Cardanol.
- Wood fibre above 5% has a large increase in strain values at C-30%.
- The strain values for wood fibre of 15% and 20% are very high with the combination of a higher percentage of both wood fibre and Cardanol.



Figure 34: Graph of Flexural Strain vs Cardanol % - CPF 100%

4.3.2.3 Cardanol Flexural Strain Results- CPF 50%

The effects on flexural strain by Cardanol percentage for CPF 50% are:

- The lower percentages of wood fibre, 0% and 5% a stable and have had no effect due to Cardanol percentage.
- C-20% gives a peak in values above 5% wood fibre
- The peak values for 15, 20 and 25% wood fibre are very similar.



4.3.2.1 Summary of Flexural Strain Results

The sample results have shown:

- Low flexural strain values with the low percentages of wood fibre.
- Above 5% wood fibre both of the composite resins (CPF) the samples increase in strain due to the fibres better flexibility than the resin matrix.
- There does not appear to be a percentage of wood fibre that has given better qualities than any other amount.
- The peak values of strain are similar for both of the CPF resins above 10% wood fibre.
- The peak values for CPF 50% are at a Cardanol percentage of C-20% and CPF 100% gives a peak at a Cardanol percentage of C-30% (See Figure 36 and page 102 for the set of graphs)



Figure 36: Flexural Strain vs Cardanol % - Wood Fibre 10% Result Comparison

4.3.3 Flexural Stress Results

4.3.3.1 Wood Fibre Flexural Stress Results

The effects of stress from filler percentage are:

• The PF resin (PF-100%) has a higher overall stress values with a peak around 5% of wood fibre.

• The CPF samples have had a lessening in the stress due to increasing filler percentage and are more flexible.



• CPF -50% - C- 30% is the most stable over all the percentages of wood fibre.

Figure 37: Graph of all Stress vs Filler Results

The results in the graph in Figure 37 are taken from Table 10.

Wood Fibre %	PF - 100%	CPF - 50%	CPF - 100%	CPF - 100%	CPF-50%
		- C - 20%	- C - 20%	- C - 30%	- C-30%
WF - 0	14.54	28.74	22.7	44.53	8.34
WF - 5	48.46	18.37	14.74	18.25	24.44
WF - 10	39.06	6.71	12.3	12.61	16.08
WF - 15	31.26	0.47	12.76	3.06	9.49
WF - 20	14.2	0.83	2.38	0.47	11.67
WF - 25	10.79	0.65	_	-	-

Table 10: All stress results (MPa) against wood fibre percentage

4.3.3.2 Cardanol Flexural Stress Results- CPF 100%

The effects on flexural stress by Cardanol percentage for CPF 100% are:

- An increase of flexural stress for 0% of wood fibre with the increase of Cardanol percentage.
- There is a general decrease of flexural stress from a peak at C-0% to a low at C-20% and a rise at C-30%.

• Looking at the wood fibre sets in Figure 38 they descend in an order of lessening flexural stress according to increasing fibre percentage.



Figure 38: Graph of Flexural stress vs Cardanol % - CPF 100%

4.3.3.3 Cardanol Flexural Stress Results - CPF 50%

The effects on flexural stress by Cardanol percentage for CPF 50% are:

- A peak of flexural stress for 0% of wood fibre at C-20% and a low at C-30%.
- A decrease of flexural stress from a peak at C-0% to a low at C-20% and a rise at C-30%. This is a similar result to the CPF 100% samples.
- The wood fibre sets descend in an order of lessening flexural stress according to increasing fibre percentage (See Figure 39).



Figure 39: Graph of Flexural stress vs Cardanol % - CPF 50%

4.3.3.1 Summary of Flexural Stress Results

The sample results have shown:

- PF resin has a higher overall stress values and has a peak value around 5% of wood fibre.
- The CPF samples have had a lessening in the stress due to increasing wood fibre filler percentage.
- CPF -50% C- 30% is the most stable over the varying percentages of wood fibre.
- CPF 100% has a low in stress at C-20% for fibre percentages below 15%, above 15% the stress values decrease to a low at C-30.
- CPF 50% has a low in stress values at C-20% for all fibre percentages except 0%. C-30% give the secondary high value (see Figure 40 and page 95 for the full set of comparison graphs).



Figure 40: Graph of Flexural Stress vs Cardanol % Wood Fibre 10% for Comparison

4.4 Dynamic Mechanical Analysis (DMA) Test Results

4.4.1 Storage Modulus

4.4.1.1 Wood Fibre Storage Modulus Results

The effects of wood fibre percentage on storage modulus results are:

- A peak storage modulus value for PF resin at 10% wood fibre and for CPF 50% C-20 at 0% wood fibre.
- The CPF resin blends generally show a decrease in storage modulus with the increase of wood fibre.



Figure 41: Graph of all DMA Storage Modulus (MPa) results for all resin types

Wood Fibre %	PF - 100%	CPF - 50% - C - 20%	CPF - 100% - C - 20%	CPF - 100% - C - 30%	CPF-50% - C-30%
WF - 0	1654	2144	1277	1301	1649
WF - 5	1748	1859	1111	1135	1478
WF - 10	2043	815.9	877	748.4	1170
WF - 15	1627	257.4	1693	364	916.1
WF - 20	931.7	224	216.7	95.78	-
WF - 25	577.8	193.3	-	-	-

Table 11: All DMA Storage Modulus (MPa) results for all resin types

4.4.1.2 Cardanol Storage Modulus Results - CPF 100%

The 0% Cardanol and 0% wood fibre sample is the 100% PF sample. Comparison of the effect of Cardanol and wood fibre will be against this and between the samples. The effects due to Cardanol percentage increase are:

- The increase of Cardanol percentage on CPF 100% has had the effect of decreasing Storage Modulus from C- 0 to C-20 and C-30.
- The samples are generally concave with a decrease in values, only the 15% wood sample is convex.
- The 15% sample (C-20) may be an anomaly, if it was rotated on its axis down, the values at 20% Cardanol would appear correct. This sample value at C-20 will be left out of further analysis.

- At 0% of Cardanol both a wood fibre of 5% and 10% have a higher Storage Modulus than the PF resin. The increases are 6% and 24% respectively.
- Looking at the samples as discrete lines (or layers), they are almost in an order descending in Storage Modulus according increasing to fibre percentage.



Figure 42: Graph of DMA results of Storage modulus vs Cardanol% - CPF 100% Table 12: DMA results of Storage modulus vs Cardanol% - CPF 100%

Cardanol %	WF - 0%	WF - 5%	WF - 10%	WF - 15%	WF - 20%	WF - 25%
C - 0	1654	1748	2043	1627	931.7	577.8
C - 20	1277	1111	877	1693	216.7	-
C - 30	1301	1135	748.4	364	95.78	-

4.4.1.3 Cardanol Storage Modulus Results - CPF 50%

The sample that is C-0% and WF-0% wood fibre is 100% PF sample. Comparison of the effect of Cardanol and wood fibre will be against this and between the samples. The effects due to Cardanol percentage increase are:

- The samples from 0% 5% wood fibre are concave, they have increased in modulus from 0% Cardanol to a peak at 20% Cardanol and a fall in modulus at 30% Cardanol.
- The rest of the samples are convex with a high at C-0% and a decrease in Storage modulus at C 20%, to an increase of modulus at C-30%.
- The samples in a similar manner to the CPF 100% samples are descending in an order of lessening Storage Modulus according to increase in fibre percentage.



Figure 43: Graph of DMA results of Storage modulus vs Cardanol% - CPF 50% Table 13: DMA results of Storage modulus vs Cardanol% - CPF 50%

Cardanol %	WF - 0%	WF - 5%	WF - 10%	WF - 15%	WF - 20%	WF - 25%
C - 0	1654	1748	2043	1627	931.7	577.8
C - 20	2144	1859	815.9	257.4	224	193.3
C - 30	1649	1478	1170	916.1	-	-

4.4.1.4 Summary of Storage Modulus Results

The sample results show:

- PF resins have the higher Storage Modulus; the 5% wood fibre generally gives the composites highest storage modulus.
- CPF 100% Storage Modulus value falls with the increase in Cardanol percentage (see Figure 44).
- CPF 50% a low in values at C-20% and an increase of values at C-30%.

The full set of comparisons between CPF 100% and CPF 50% for Storage Modulus are on page 113.



Figure 44: DMA Results - Cardanol comparison 5% WF - CPF 100% & CPF 50%

4.4.2 Glass Transition Temperature (Tg)

4.4.2.1 Wood Fibre Glass Transition Temperature Results

The effect on glass transition temperature by wood filler percentage for the different blends is:

- PF 100% resin is the most stable for all wood fibre percentages, this is expected as temperature tolerance is one of PF resins advantages that was identified during the literature review.
- The most stable of the CPF composite resins is the CPF -50% C- 30% (50% PF + 50% CPF with 30% Cardanol). This sample has a higher Tg and is more stable throughout the different percentages of wood fibre.
- The other CPF resin samples have a lessening in temperature tolerance with the increasing percentage of wood fibre.
- The blend CPF 50% C-30% has superior Tg for 0%, 5% and 10% of higher than the PF resin by 8%, 8% and 6%.

There is unfortunately no results at 20 and 25% due to poor sample integrity.



Figure 45: DMA Glass Transition Temperature (Tg) results for all resin types

Wood Fibre %	PF - 100%	CPF - 50% - C - 20%	CPF - 100% - C - 20%	CPF - 100% - C - 30%	CPF-50% - C-30%
WF - 0	128.64	115.47	85.26	131.29	139.21
WF - 5	132.95	107.06	146.18	107.92	144.31
WF - 10	133.39	117.19	128.5	76.94	141.66
WF - 15	139.43	91.35	131.14	91.38	119.91
WF - 20	129.93	90	123.38	83.82	_
WF - 25	119.58	88.14	_	_	-

Table 14: Glass Transition Temperature results against wood fibre percentage

4.4.2.2 Cardanol Glass Transition Temperature Results – CPF 100%

The DMA Glass transition (Tg) result information in Table 15 has been expanded into the graph in Figure 46 for CPF samples of 100% Cardanol. The sample in the table of (C-0, WF-0%) is the PF resin used as a base for comparison against for the PF and CPF composites. The following observations were made:

- Wood fibre up to 20% added to the CPF resins has had a beneficial effect giving a higher glass transition temperature (Tg).
- A wood fibre of 25% (WF-25%) is only able to carry temperature with 0% Cardanol (C - 0)
- For 0% wood fibre C 0; Cardanol at 30% has 2% higher Tg than the base sample and has a 35% Tg than C 20%.
- For the wood fibre samples at 5% there is a higher Tg except at C- 0% where wood fibre at 15% is 5% higher.

- The wood fibre samples Tg in these results is from highest to lowest:
 - o 5 % wood Fibre
 - \circ 15% wood fibre
 - \circ 10% wood fibre
 - \circ 20% wood fibre
 - $\circ \quad 25\% \ wood \ fibre$
- The percentage difference within the CPF 100% samples are 2%, 16% and 22% for the three rates of Cardanol. The 25% sample is not included as the lowest, due to non-result of 0 for 20% and 30% Cardanol.



Figure 46: DMA - CPF 100% Tg vs Cardanol percentage

Table 15: DMA result - CPF 100% Tg vs Cardanol percentage

Cardanol %	WF - 0%	WF - 5%	WF - 10%	WF - 15%	WF - 20%	WF - 25%
C - 0	128.64	132.95	133.39	139.43	129.93	119.58
C - 20	85.26	146.18	128.5	131.14	123.38	0
C - 30	131.29	107.92	76.94	91.38	83.82	0

4.4.2.3 Cardanol Glass Transition Temperature Results – CPF 50%

The DMA Glass transition (Tg) result information in Table 16 has been expanded into the graph in Figure 47 for CPF samples of 50% Cardanol. The sample in the table of (C-0, WF-0%) is the PF resin used as a base for comparison against for the PF and CPF composites. The following observations were made:

- At a Cardanol percentage of 30% wood fibre values of 20 and 25% have no temperature tolerance.
- The 15% wood fibre sample has the highest Tg at 0% of Cardanol but its performance at C- 20% and C- 30% is 15% less than the nearest result. At C-20 the result is also very similar to the 20 and 25% wood fibre results.
- Temperature tolerance at 0% Cardanol with wood fibre from 5 20% have better Tg than the 100% PF sample.
- Cardanol at 30% with a wood fibre percentage of 15% and below gives a substantially higher Tg than 20% Cardanol.
- The wood fibre samples Tg in these results from highest to lowest are:
 - \circ 10 % wood Fibre
 - 0% wood fibre
 - 5% wood fibre
 - \circ 15% wood fibre
 - o and 20% marginally better than 25% wood fibre



Figure 47: DMA - CPF 50% Tg vs Cardanol percentage

Table 16: DMA result - CPF 50% Tg vs Cardanol percentage

Cardanol	WF -					
%	0%	5%	10%	15%	20%	25%
C - 0	128.64	132.95	133.39	139.43	129.93	119.58

C - 20	115.47	107.06	117.19	91.35	90	88.14
C - 30	139.21	144.31	141.66	119.91	0	0

4.4.2.4 Summary of DMA Glass Transition Temperature Results

The sample results show:

- The graphs of the CPF 100% samples are generally convex and the CPF 50% are concave (See Figure 48).
- CPF 100% has consistently higher values for Cardanol at 20%
- CPF 50% has consistently higher values for Cardanol at 30%.
- At 0% Cardanol there is little difference between the two blends.
- The wood fibre is optimum at 5% for CPF 100%
- The wood fibre is optimum at 10% for CPF 50%



Figure 48: DMA Results: Comparison of CPF 100% and CPF 50% highest values

4.5 Discussion of Testing Results

All of the PF and CPF test results are brought together for discussion and comparison.

4.5.1 Flexural Modulus Result Discussion

The PF - 100% sample has generally an overall higher flexural modulus than the other tested samples. The exception is CPF 50 C-20 at 0% wood fibre. The CPF samples have
had an overall lessening of flexural modulus with the increasing percentage of wood fibre. The CPF sample; CPF -50% - C- 30% appears to be the most stable (graph in Figure 29). Wood fibre at a filler percentage of 5% gives in most samples the highest flexural modulus. CPF 100% generally falls in modulus with the increase in Cardanol percentage. CPF 50% generally falls in modulus to a low at 20% Cardanol and then increase to a secondary high (See Figure 29).

4.5.2 Flexural Strain Result Discussion

The strain result for most of the composite samples is low, from 0% - 5%. Above 5% wood fibre both of the composite resins samples increase in strain due to increased flexibility. There does not appear to be a percentage of wood fibre that has given better qualities than any other amount. The peak values of strain are similar for both of the CPF resins above 10% wood fibre. The peak values for CPF 50% are at a Cardanol percentage of C-20% and CPF 100% gives a peak at a Cardanol percentage of C-30% (See Figure 36)

4.5.3 Flexural Stress Result Discussion

PF resin has a higher overall stress values and has a peak value around 5% of wood fibre. The CPF samples have had a lessening in the stress due to increasing filler percentage and are more flexible. CPF -50% - C- 30% is the most stable for all the percentages of wood fibre. CPF 100% has a low in stress at C-20% for fibre percentages below 15%, above 15% the stress values decrease to a low at C-30. CPF 50% has a low in stress values at C-20% for all fibre percentages except 0%. C-30% gives the secondary high value (see Figure 40)

4.5.4 DMA - Storage Modulus Result Discussion

PF resins have the higher Storage Modulus; the 5% wood fibre generally gives the composites highest storage modulus. CPF 100% Storage Modulus value falls with the increase in Cardanol percentage (see Figure 44). The samples are generally concave with a decrease in values. The samples of CPF 50% are low in values at C-20% and an increase of values at C-30%. The highest values are at C-0% Cardanol with a low in

values at C-20% and an increase of values at C-30%. The order of Storage modulus results of both blends falls with the increase of fibre percentage.

The composite that had the highest storage modulus was CPF 50% C-20% with 5% wood fibre.

4.5.5 DMA - Glass Transition Temperature Result Discussion

PF 100% resin is the most stable for all wood fibre percentages, with the most stable of the CPF composite resins is the CPF -50% - C- 30% with a generally higher Tg than PF resin up to 10%. All CPF resin samples have a lessening in temperature tolerance with the increasing percentage of wood fibre. The graphs of the CPF 100% samples are generally convex and the CPF 50% are concave (See Figure 48). CPF 100% has consistently higher values for Cardanol at 20%. CPF 50% has consistently higher values for Cardanol at 20%. CPF 50% has consistently higher values. The wood fibre is optimum at 5% for CPF 100% and at 10% for CPF 50%.

4.6 Summary of All Test Results

The PF - 100% samples have generally an overall higher flexural modulus and stress values, and a low strain. PF 100% resin was the most stable for all wood fibre percentages for Glass Transition Temperature.

4.6.1 Wood Fibre effects on composite samples

Effects due to wood fibre have been generally that Storage modulus, Flexural modulus, Stress and Glass Transition Temperature for all Composite blends fall with the increase of wood fibre percentage. Wood fibre at a filler percentage of 5% gives in most samples the highest modulus. There is no percentage of wood fibre with better strain qualities than any other. CPF -50% - C- 30% is the most stable blend for the applied tests.

4.6.2 Cardanol effects on composite samples

The values of the resin samples CPF 100% peak at C-20% and fall to a lower value at C-30% in the majority of test results. The samples of CPF 50% decrease in values at C-20% and increase of values at C-30%. This includes the strain values.

4.6.3 External Comparison to Results

4.6.3.1 Filler

Gurney (2006, p.39) who found the best flexural strength for mixture of slg filler by weight in Phenolic resin was 5%, followed by 25%, and 30%, and Nixon (2008, p.28) who found 15% glass filler (spheres) to have the optimum strength for the amount of filler. In this project 5% was the optimum for 100% PF resin (Figure 29 and Table 8)

Table 17: Properties of natural fibre reinforced Phenolics

Flexural mechanical properties of natural short fibre reinforced composites. The matrix is the resole–epoxy resin without acid catalyst

Fibre type	Fibre content (wt.%)	Flexural modulus (MPa)	
Hemp	13	1750 ± 150	
Ramiè	15	1790 ± 115	
Flax	15	420 ± 25	

Comparing to the results obtained from 15% wood fibre Phenolic resin, the result obtained in this project is PF WF - 15 1845 MPa. This is very similar to the hemp and ramie fibres but well above flax fibre in the study by Maffezzoli et al (2004, p.6).

Cardanol comparisons were unable to be sourced due commercial confidentially, it is hoped that the results in this project will open research in this product.

5 Conclusions

5.1 Introduction

In this section the conclusions are presented, and related back to the projects objectives. The research project objectives were:

- 1. A literature review of Phenolic resins and modifying mechanisms and techniques.
- 2. The synthesis of PF resins modified with different amounts of Cardanol (CPF)
- 3. The preparation and testing of composites using the modified CPF resins and different amounts of the renewable material (sawdust).
- 4. The testing and characterisation of the synthesised CPF resins

5.1.1 Objective 1: Literature review

The literature review was a study of Phenolic resins and modifying mechanisms and techniques to PF resins as a background to the project. The review:

- Defined composite resins
- Researched the background of composites as building materials
- Researched the background of Phenolic resins and its composites
- Researched Phenolic resins derived from renewable materials, with the main focus on CNSL and Cardanol.
- Defined thermoset resins and researched the types of Phenolic resin.
- Presented the synthesis of PF resin and bonding and synthesis of CPF resin
- Researched the Characteristics of PF and CPF resins
- Presented the uses of PF and CPF resins
- Researched the development of other resins and composites
- Researched thermoset resin fillers and reinforcement, with the four main types of filler/ reinforcement.
- Researched the testing of composite Phenolic resin samples for flexural testing, Dynamic mechanical Analysis and tensile testing.
- Presented risk management of composite Phenolic resins, including safety induction, risk assessment, actions against risk and monitoring and review of risk.

- Researched consequential effects of my actions in researching this project. This included looking at the consequences and ethical responsibilities of this work.
- Timelines for project completion are given.

5.1.2 Objectives 2 and 3

Objectives 2 and 3 are:

- The Synthesis of PF Resins Modified with Different Amounts of Cardanol (CPF) and,
- The Preparation of Composites using Modified CPF resins and Different Amounts for testing of the Renewable Material; Sawdust.

Both of the objectives are discussed in Section 3; Research and Methodology. Section 3 covers the synthesis and preparation of modified CPF resins with different amounts of sawdust. The discussion and methodology included:

- The materials used in synthesis
- Preparation and synthesis of test samples
- The cutting and shaping of test samples

5.1.3 Objective 4: The Testing and Characterisation of the Synthesised CPF Resins

The testing and characterisation of the synthesised CPF resins is in Section 4; Characterisation and testing of Modified Composite Resins. The testing methods are discussed, the results for flexural testing and DMA testing is given. The flexural testing is divided into three sections testing and characterisation; these are:

- Flexural Modulus
- Flexural Strain
- Flexural Stress

Dynamic Mechanical Analysis consists of two main tests used for the testing and characterisation of the composite Phenolic samples; these are:

- Storage Modulus and
- Glass Transition Temperature

The test results are collated and discussed in their sections, the results are summarised to compare the characteristics of the different blends of Cardanol and wood fibre compositions.

5.2 Conclusion

I have found in this project that:

- Cardanol was able to substitute commercial phenol in the synthesis of Phenolic resins (CPF).
- The synthesised CPF resins are less brittle and more flexible than PF resins as the PF samples have generally an overall higher flexural modulus, stress values and Glass transition temperature.
- Effects due to wood fibre have been generally that Storage modulus, Flexural modulus, Stress and Glass Transition Temperature for all Composite blends fall with the increase of wood fibre percentage.
- Wood fibre at a filler percentage of 5% gives in most samples the highest modulus showing Sawdust fibres are suitable fillers for composites with CPF and CPF/PF resins.
- There is no percentage of wood fibre with better strain qualities than any other.
- The CPF 50%– PF 50% resin mix was the most successful as it gave stable results with the brittleness decreasing in a linear manner with the higher percentage of filler.
- The values of the resin samples CPF 100% peak at C-20% and fall to a lower value at C-30% in the majority of test results (Including strain).
- The samples of CPF 50% decrease in values at C-20% and increase of values at C-30% (Including strain).

6 Recommendations

6.1 Introduction

The results have shown the positive effects of Cardanol and wood fibre on phenol formaldehyde resins. This demonstrates the required modulus and temperature tolerance of these thermoset resins can be met with different types of natural composite blends. Composites with CPF and sawdust fibres showed high flexibility and strength that make the composites suitable for potential civil engineering structural applications.

6.2 Restrictions and Challenges

Restrictions in the project were being able to generate sufficient samples to confirm the results of all the test samples. Factors such as variability that can occur within in the samples cut from the same piece and differences in in preparation can make the samples integrity questionable.

6.3 Recommendations for Future Work

There is the potential for future work with the large variability and range of potential tests to trial with varying percentages of Cardanol and wood fibre. Cardanol can be trialled as a blend at higher and lower percentages than 20% and 30%. The Cardanol blend and mix can be trialled at other percentages than 50-50. In particular since the 50-50 % mix was successful, there may be a range at 40-60% or 60-40% that may have better properties.

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Appendix A: Project Specification

University of Southern Queensland FACULTY OF ENGINEERING AND SURVEYING ENG 4111/4112 Research Project **PROJECT SPECIFICATION** FOR: NIGEL POLA Synthesis of modified phenolic resins using TOPIC: for advanced compositor enewable materials in engineering structures eivil CARDONA CEEFC RANCISCO SUPERVISORS: ENG 4111 14112 ENROLMENT: PROJECT AIM: This project seeks to investigate the synthesis and characterization (thermal and mechanical fied phenolic resins using natural renewable right. Composites using these PF resins will be SPONSORSHIP: PROGRAMME: Keview of phenolic Literature resins and mechanisms & techniques ynthesis Cardan 2. with ensi and 3. Testing and CharaiTerization of the synthesised rosins hig of composites using reportion of and renewable materials 105Ins AGREED: , Head Carling (Supervisors) 1/ola (student) 19 1 1910212010. 10212010 1 Examiner/Co-examiner:

Appendix B: Flexural Test results

 Table 18: Table of flexural test results

	Stress	Strain	Flex Modulus
TEST ID	(Mpa)	%	(Mpa)
PF - 100 - WF - 0 - S1	18.15	0.85	1901
PF - 100 - S3			
PF - 100 - WF - 0 - S2	14.54	0.78	1768
PF - 100 - WF - 5 - S3	48.46	1.84	2590
PF - 100 - WF - 10 - S3	39.06	1.59	2339
PF - 100 - WF - 15 - S3	31.26	1.73	1845
PF - 100 - WF - 20 - S3	14.2	2.15	626
PF - 100 - WF - 25 - S3	10.79	1.7	601
CPF - 50 - C - 20 - S4			
CPF - 50 - C - 20 - WF - 0 - S4	28.74	1.27	2112
CPF - 50 - C - 20 - WF - 5 - S4	18.37	1.13	1633
CPF - 50 - C - 20 - WF - 10 - S4	6.71	8.58	227
CPF - 50 - C - 20 - WF - 15 - S4	0.47	22.31	6
CPF - 50 - C - 20 - WF - 20 - S4	0.83	21.57	-
CPF - 50 - C - 20 - WF - 25 - S4	0.65	21.75	-8
CPF - 100 - C - 20 - WF - 0 - S5	22.7	4.16	698
CPF - 100 - C - 20 - WF - 5 - S5	13.3	2.64	534
CPF - 100 - C - 20 - S6			
CPF - 100 - C - 20 - WF - 0 - S5	22.7	4.16	698
CPF - 100 - C - 20 - WF - 5 - S6	14.74	1.59	889
CPF - 100 - C - 20 - WF - 10 - S6	12.3	1.6	800
CPF - 100 - C - 20 - WF - 15 - S6	12.76	1.96	697
CPF - 100 - C - 20 - WF - 20 - S6	2.38	2.72	127
CPF - 100 - C - 30 - S7			
CPF-100-C-30-WF-0-S7	44.53	2.99	1383
CPF-100-C-30-WF-5-S7	18.25	2.14	813
CPF-100-C-30-WF-10-S7	12.61	7.77	331
CPF-100-C-30-WF-15-S7	3.06	21.15	56
CPF-100-C-30-WF-20-S7	0.47	23.02	-
CPF-50-C-30-S8		1	
CPF-50-C-30-WF-0-S8	8.34	0.57	1413
CPF-50-C-30-WF-5-S8	24.44	1.6	1499
CPF-50-C-30-WF-10-S8	16.08	1.67	972
CPF-50-C-30-WF-15-S8	9.49	1.56	591
CPF-50-C-30-WF-20-S8	11.67	424	424

Note: Explanation of naming of samples is in Table 7: Naming of samples against composition on page 25.

Flexural Modulus vs Wood Fibre Filler



Figure 49: Flexural Modulus vs Filler – All results



Figure 50: Flexural modulus vs wood fibre filler - 100% PF



Figure 51: Flexural modulus vs wood fibre filler - 50% PF - 50% CPF (C - 20%)



Figure 52: Flexural modulus vs wood fibre filler - 50% PF - 50% CPF (C - 30%)



Figure 53: Flexural modulus vs wood fibre filler - 100% CPF (C - 20%)



Figure 54: Flexural modulus vs wood fibre filler - 100% CPF (C - 30%)

Flexural Modulus vs Cardanol %



Figure 55: Graph of Flexural Modulus vs Cardanol (CPF 100%)



Figure 56: Graph of Flexural Modulus vs Cardanol (CPF 50%)



Figure 57: Graph of Flexural Modulus vs Cardanol % - Wood Fibre 0% Result Comparison



Figure 58: Graph of Flexural Modulus vs Cardanol % - Wood Fibre 5% Result Comparison



Figure 59: Graph of Flexural Modulus vs Cardanol % - Wood Fibre 10% Result Comparison



Figure 60: Graph of Flexural Modulus vs Cardanol % - Wood Fibre 15% Result Comparison



Figure 61: Graph of Flexural Modulus vs Cardanol % - Wood Fibre 20% Result Comparison

Flexural Stress vs Wood Fibre Filler



Figure 62: Graph of Flexural Stress vs Filler % - All Results



Figure 63: Stress vs wood fibre filler - 100% PF



Figure 64: Stress vs wood fibre filler - $50\%\,$ PF - $50\%\,$ CPF (C- 20%)







Figure 66: Stress vs wood fibre filler - 100% CPF (C - 20%)



Figure 67: Stress vs wood fibre filler - 100% CPF (C - 30%)

Flexural Stress vs Cardanol %



Figure 68: Stress vs Cardanol % All CPF 100% Results



Figure 69: Stress vs Cardanol % All CPF 50% Results



Figure 70: Graph of Flexural Stress vs Cardanol % Wood Fibre 0% for Comparison



Figure 71: Graph of Flexural Stress vs Cardanol % Wood Fibre 5% for Comparison



Figure 72: Graph of Flexural Stress vs Cardanol % Wood Fibre 10% for Comparison



Figure 73: Graph of Flexural Stress vs Cardanol % Wood Fibre 15% for Comparison



Figure 74: Graph of Flexural Stress vs Cardanol % Wood Fibre 20% for Comparison

Flexural Strain vs Wood Fibre Filler %



Figure 75: Flexural Strain vs Filler % - All Results



Figure 76: Strain vs wood fibre filler - 100% PF



Figure 77: Strain vs wood fibre filler - 50% PF - 50% CPF (C - 20%)



Figure 78: Strain vs wood fibre filler - 50% PF - 50% CPF (C - 30%)



Figure 79: Strain vs wood fibre filler - 100% CPF (C - 20%)



Figure 80: Strain vs wood fibre filler - 100% CPF (C - 30%)

Flexural Strain vs Cardanol %



Figure 81: Flexural Strain vs Cardanol % - All CPF 100% Results



Figure 82: Flexural Strain vs Cardanol % - All CPF 50% Results



Figure 83: Graph of Flexural Strain vs Cardanol % - Wood Fibre 0% Result Comparison



Figure 84: Flexural Strain vs Cardanol % - Wood Fibre 5% Result Comparison



Figure 85: Flexural Strain vs Cardanol % - Wood Fibre 10% Result Comparison



Figure 86: Graph of Flexural Strain vs Cardanol % - Wood Fibre 15% Result Comparison



Figure 87: Graph of Flexural Strain vs Cardanol % - Wood Fibre 20% Result Comparison

Appendix C: Dynamic Mechanical Analysis Test

Results

Table 19: DMA Test Results

TEST ID	Tg (C°)	Storage modulus (MPa)
PF - 100 - WF - 0 - S1	128.64	1654
PF - 100 - S3		
PF - 100 - WF - 0 - S1	128.64	1654
PF - 100 - WF - 5 - S3	132.95	1748
PF - 100 - WF - 10 - S3	133.39	2043
PF - 100 - WF - 15 - S3	139.43	1627
PF - 100 - WF - 20 - S3	129.93	931.7
PF - 100 - WF - 25 - S3	119.58	577.8
CPF - 50 - C - 20 - S4		
CPF - 50 - C - 20 - WF - 0 - S4	115.47	2144
CPF - 50 - C - 20 - WF - 5 - S4	107.06	1859
CPF - 50 - C - 20 - WF - 10 - S4	117.19	815.9
CPF - 50 - C - 20 - WF - 15 - S4	91.35	257.4
CPF - 50 - C - 20 - WF - 20 - S4	90	224
CPF - 50 - C - 20 - WF - 25 - S4	88.14	193.3
CPF - 100 - C - 20 - S6		
CPF - 100 - C - 20 - WF - 0 - S5	85.26	1277
CPF - 100 - C - 20 - WF - 5 - S6	146.18	1111
CPF - 100 - C - 20 - WF - 10 - S6	128.5	877
CPF - 100 - C - 20 - WF - 15 - S6	131.14	1693
CPF - 100 - C - 20 - WF - 20 - S6	123.38	216.7
CPF - 100 - C - 30 - S7		
CPF-100-C-30-WF-0-S7	131.29	1301
CPF-100-C-30-WF-5-S7	107.92	1135
CPF-100-C-30-WF-10-S7	76.94	748.4
CPF-100-C-30-WF-15-S7	91.38	364
CPF-100-C-30-WF-20-S7	83.82	95.78
CPF-50-C-30-S8		
CPF-50-C-30-WF-0-S8	139.21	1649
CPF-50-C-30-WF-5-S8	144.31	1478
CPF-50-C-30-WF-10-S8	141.66	1170
CPF-50-C-30-WF-15-S8	119.91	916.1
CPF-50-C-30-WF-20-S8		

Note: results in **blue** were averaged, as the sample was of poor integrity.

Tg vs Wood Fibre %



Figure 88: Graph of DMA Glass Transition Temperature vs Filler % All Results



Figure 89: Graph of DMA - Tg vs. Filler % All CPF 100% comparison with PF resin



Figure 90: Graph of DMA - Tg vs. Filler % All CPF 50% comparison with PF resin

Tg vs Cardanol %



Figure 91: Comparison of highest and lowest values of Tg results



Figure 92: DMA Glass Transition Temperature vs Cardanol %, CPF 100%



Figure 93: DMA Glass Transition Temperature vs Cardanol %, CPF 50%



Figure 94: DMA result Tg vs. Cardanol % - 0% Wood Fibre



Figure 95: DMA result Tg vs. Cardanol % - 5% wood fibre


Figure 96: DMA Glass transition Temperature vs Cardanol %, - 10% Wood Fibre



Figure 97: DMA Glass transition Temperature vs Cardanol %,- 15% Wood Fibre



Figure 98: DMA Glass transition Temperature vs Cardanol % - 20% Wood Fibre



Figure 99: DMA Glass transition Temperature vs Cardanol% - 25 % Wood Fibre

Storage Modulus vs Wood Fibre % Results



Figure 100: Graph of DMA – Storage Modulus vs. Filler % All CPF 100% comparison with PF resin



Figure 101: Graph of DMA – Storage Modulus vs Filler % All CPF 50% comparison with PF resin

Wood	PF -	CPF - 50%	CPF - 100% -	CPF - 100%	CPF-50%
Fibre %	100%	- C - 20%	C - 20%	- C - 30%	- C-30%
WF - 0	1654	2144	1277	1301	1649
WF - 5	1748	1859	1111	1135	1478
WF - 10	2043	815.9	877	748.4	1170
WF - 15	1627	257.4	1693	364	916.1
WF - 20	931.7	224	216.7	95.78	-
WF - 25	577.8	193.3	-	-	-

Table 20: Storage Modulus vs. Cardanol %



Figure 102: DMA result CPF-100%; Storage Modulus vs. Cardanol %



Figure 103: DMA result CPF-50%; Storage Modulus vs. Cardanol %



Figure 104: Graph of Storage Modulus vs Cardanol % - Wood Fibre 0% Result Comparison



Figure 105: Graph of Storage Modulus vs Cardanol % - Wood Fibre 5% Result Comparison



Figure 106: Graph of Storage Modulus vs Cardanol % - Wood Fibre 10% Result Comparison



Figure 107: Graph of Storage Modulus vs Cardanol % - Wood Fibre 15% Result Comparison



Figure 108: Graph of Storage Modulus vs Cardanol % - Wood Fibre 20% Result Comparison

Appendix D: DMA Machine Graphed Results

DMA graphs showing storage modulus and glass transition temperature (Tg).



Figure 109: 100% PF resin - Wood fibre 0%



Figure 110: 100% PF resin - Wood fibre 5%







Figure 112: 100% PF resin - Wood fibre 15%







Figure 114: 100% PF resin - Wood fibre 25%



Figure 115: PF 50% CPF 50% resin - (Cardanol 20% of CPF) Wood fibre 0%



Figure 116: PF 50% CPF 50% resin - (Cardanol 20% of CPF) Wood fibre 5%



Figure 117: PF 50% CPF 50% resin - (Cardanol 20% of CPF) Wood fibre 10%



Figure 118: PF 50% CPF 50% resin - (Cardanol 20% of CPF) Wood fibre 15%





DMA Graphs of CPF 100% and Wood Fibre



Figure 120: CPF 100% resin - (Cardanol 20% of CPF) Wood fibre 0%



Figure 121: CPF 100% resin - (Cardanol 20% of CPF) Wood fibre 5%



Figure 122: CPF 100% resin - (Cardanol 20% of CPF) Wood fibre 10%



Figure 123: CPF 100% resin - (Cardanol 20% of CPF) Wood fibre 15%



Figure 124: CPF 100% resin - (Cardanol 20% of CPF) Wood fibre 20%



Figure 125: CPF 100% resin - (Cardanol 30% of CPF) Wood fibre 0%



Figure 126: CPF 100% resin - (Cardanol 30% of CPF) Wood fibre 5%



Figure 127: CPF 100% resin - (Cardanol 30% of CPF) Wood fibre 10%



Figure 128: CPF 100% resin - (Cardanol 30% of CPF) Wood fibre 15%



Figure 129: CPF 100% resin - (Cardanol 30% of CPF) Wood fibre 20%



Figure 130: PF 50% CPF 50% resin - (Cardanol 30% of CPF) Wood fibre 0%



Figure 131: PF 50% CPF 50% resin - (Cardanol 30% of CPF) Wood fibre 5%



Figure 132: PF 50% CPF 50% resin - (Cardanol 30% of CPF) Wood fibre 10%



Figure 133: PF 50% CPF 50% resin - (Cardanol 30% of CPF) Wood fibre 15%

Appendix E: Preparation, Shaping and Cutting Equipment



Figure 134: Hafco Tile cutting machine used for cutting of samples



Figure 135: Surface Grinder for shaping samples



Figure 136: Fume cabinet holding oil bath for bonding Phenol and Cardanol



Figure 137: Measuring Scales (LHS) and Flexural Test moulds (RHS)



Figure 138: DMA machine – TA Q800

Table 21: TA Q800 Technical specifications

TECHNICAL SPECIFICATIONS

Maximum Force	18 N
Minimum Force	0.0001 N
Force Resolution	0.00001 N
Strain Resolution	1 nanometer
Modulus Range	10³ to 3x1012 Pa
Modulus Precision	± 1%
Tan δ Sensitivity	0.0001
Tan δ Resolution	0.00001
Frequency Range	0.01 to 200 Hz
Dynamic Sample Deformation Range	± 0.5 to 10,000 μm
Temperature Ra nge	-150 to 600 °C
Heating Rate	0.1 to 20 °C/min
Cooling Rate	0.1 to 10 °C/min
Isothermal Stability	± 0.1 °C
Time/Temperature Superposition	Yes

OUTPUT VALUES

Storage Modulus	Complex/Dynamic Viscosity	Time
Loss Modulus	Creep Compliance	Stress/Strain
Storage/Loss C ompliance	Relaxation Modulus	Frequency
Tan Delta (δ)	Static/Dynamic Force	Sample Stiffness
Complex Modulus	Temperature	Displacement

Main Task	Individual Task	Risk identification	Risk analysis	Risk evaluation	Action	Final Risk
Preparation	Bonding of	Handling of chemicals	Some of the chemicals are	High risk - burns	Wear PPE and	Low
of samples	resin		Spillage	High risk - slip & fall	procedures	Very Low
	Preparation of catalyst	Handling of chemicals	Some of the chemicals are extremely toxic Spillage	High risk - burns High risk - slip & fall	Wear PPE and follow SWP procedures	Low Very Low
	Reaction of PF or CPF resin with catalyst	Reaction may become very hot	Possible burns to person or property	Moderate risk	Wear PPE and follow SWP procedures	Low
Preparation	Sample cutting with tile cutter	Cutting injury to person. Injury from fragments	Tile cutter very sharp Sample fragments may gain	High risk with tile cutter Moderate risk with	Wear PPE and follow SWP	Low
testing		flying off cutter. High noise levels Dust particles inhaled	velocity from cutter. Noise levels Dust is given off from	fragments in eyes High risk with hearing High risk with breathing	procedures Use dust	Low Low Low
			samples.	& skin irritation	extraction machine & water	
	Sample shaping with Surface Grinder	Cutting injury to person. Injury from pieces flying off cutter.	Surface grinder very sharp Sample fragments may gain velocity from cutter.	High risk with cutting person High risk with fragments	Wear PPE and follow SWP procedures	Low Low
		Hot work pieces High noise levels Dust particles inhaled	Burns from work piece Noise levels Dust is given off from samples.	In eyes High risk of burns High risk with hearing High risk with dust & skin irritation	Use dust extraction machine	Low Low Low
Testing of	Flexural testing	Injury from pieces flying	Sample fragments may gain	Moderate with fragments	Keep guards in	Very Low
samples		Crush injury from test	Hand or body part caught in test	Moderate when adjusting sample position	and follow SWP procedures	Very Low

Appendix F: Risk Assessment Table

	Impact testing	Injury from pieces flying	Sample fragments may gain	Moderate with fragments	Keep guards in	Very Low
		off test piece.	velocity from test	in eyes	place. Wear PPE	
		Crush injury from test	Hand or body part caught in	Low when adjusting	and follow SWP	
			test	sample position	procedures	
	Tensile testing	Injury from pieces flying	Sample fragments may gain	Moderate with fragments	Keep guards in	Very Low
		off test piece.	velocity from test	in eyes	place. Wear PPE	
		Crush injury from test	Hand or body part caught in	Low when adjusting	and follow SWP	
			test	sample position	procedures	
	DMA testing	Burn injury from pieces	Sample may be hot when	Low if sample allowed	Wear PPE and	Very Low
			test is completed	to cool	follow SWP	
		Crush injury from	Hand or body part caught in	Low if allowing	procedures	
		machine closing	test	computer to close		
Disposal of	Disposal of	Possible risk to the	CPF and PF resins may be	CPF and PF resins are	Normal disposal	None
		environment	toxic.	inert.	of rubbish.	
samples	tested CPF & PF					
	resin samples					

SWP procedures - Standard Work Procedures; a document distributed at the initial on site induction

Appendix G: Aspects of Sustainability

Effects	Yes/	Comments	Score
	No		/10
Will development today undermine the development		Development of CPF resins today should be the building blocks of	10
and environmental needs of future generations	No	development for future generations extending the life of petrochemical	
		supplies with the aim of replacing them with a more sustainable	
		feedstock.	
Environmental protection shall constitute an integral	Yes	Using waste products in new products is partly contributing to	7
part of the development process.		environmental protection by removing waste products from landfill.	
Global environmental impacts of local actions and	Yes	Globally the environmental impact of using waste products and lessening	9
policies taken into account		dependence on oil to produce the same products should create a positive	
		impact.	
Scientific uncertainty used to postpone measures to	No	There appears to be no uncertainty, and no postponement involved in	10
prevent environmental degradation.		producing goods from these waste products.	
Environmental issues should be handled with the	Yes	Any participation on environmental issues is encouraged through the	9
participation of all concerned citizens.		offering of the projects to students, and open days.	
The community has a right of access to, and an	Yes	The publication of the results from these investigations provides	10
understanding of, environmental information.		information to the community.	
The polluter should bear the cost of pollution and so	Yes	The environmental cost is lessened by using waste products from other	7

environmental costs should be internalised by adding		industries. There is still a cost in using a petrochemically derived product.	
them to the cost of production.			
The eradication of poverty, the reduction in		Production of these thermoset products in non oil producing countries	3
differences in living standards and the full	No	may lead to a positive effect that will lessen these problems. But there is	
participation of women, youth and indigenous		no hard evidence of this and that these aims will be met.	
people to achieve sustainability.			
Responsibility to assist in the achievement of	Yes	This is an effort to achieve an sustainable feedstock for producing	10
sustainability.		thermoset products	
Warfare is inherently destructive of sustainability,		If the longer term aim of replacing petrochemical supplies with renewable	3
and, in contrast, peace, development and	No	products is successful there may be a lessening of global tensions. But	
environmental protection are interdependent and		again there is no evidence of this.	
indivisible. Will project contribute to international			
understanding?			
	•	Total %	78

Appendix H: Ethical Responsibility

Effects	Yes/ No	Comments	Applied
Members shall place their responsibility for the welfare,		This project has no responsibility for the welfare, health and safety to	
health and safety of the community before their responsibility	Yes	sectional or private interests, or to other members	\checkmark
to sectional or private interests, or to other members			
Members shall act with honour, integrity and dignity in order		Yes any results from the project will be reported honestly and properly	
to merit the trust of the community and the profession	Yes		
Members shall act only in areas of their competence		Thermoset resins were not a field I had previously studied, but with my	
and in a careful and diligent manner	Yes	supervisor I have been able to work competently in this field.	
Members shall act with honesty, good faith and equity and		I have acted with honesty, good faith and equity and without	
without discrimination towards all in the community;	Yes	discrimination towards all in the community as well as my workplace	\checkmark
		while completing my project.	
Members shall apply their skill and knowledge in the interest		I have applied my skill and knowledge in the interest of the University of	
of their employer or client for whom they shall act with	Yes	Southern Queensland with integrity without compromising any other	\checkmark
integrity without compromising any other obligation to these		obligation to these Tenets;	
Tenets;			
Members shall, where relevant, take reasonable steps to		Reasonable steps have been taken in the literature review of this project	
inform themselves, their clients and employers, of the social,	Yes	and a review of the possible consequences of this project.	\checkmark

environmental, economic and other possible consequences			
which may arise from their actions;			
Members shall express opinions, make statements or give		The Appreciation and Dissertation of this project will only give	
evidence with fairness and honesty and only on the basis of	Yes	information based on well researched evidence.	\checkmark
adequate knowledge;			
Members shall continue to develop relevant knowledge, skill		This project and final Dissertation will continue my development, and	
and expertise throughout their careers and shall actively assist	Yes	fulfil the requirements of my engineering degree.	\checkmark
and encourage those with whom they are associated, to do			
likewise;			
Members shall not assist in or induce a breach of these Tenets		These tenets will not be breached and support to uphold them will be	
and shall support those who seek to uphold them if called	Yes	given if required.	\checkmark
upon or in a position to do so.			