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A New Fiber Reinforced Polymer Composite for Infrastructure Applications Using Recycled Carbon Fibers

Mojgan Maadandar

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Mojgan Maadandar

Candidate

Civil Engineering

Department

This thesis is approved, and it is acceptable in quality and form for publication:

Approved by the Thesis Committee:

Dr. Mahmoud Reda Taha , Chairperson

Dr. Fernando Moreu Alonso

Dr. Mehran Tehrani

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By

Mojgan Maadandar

B.S., Textile Engineering, University of Poly technique, Iran, 1997

B.S., Civil Engineering, University of New Mexico, 2015

M.S., Civil Engineering, University of New Mexico, 2017

THESIS

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Dedication

To Arman and Amin for all

their patience throughout this whole

journey

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A New FRP Composite for Infrastructure Applications Using Recycled Carbon Fibers

By

Mojgan Maadandar

B.S., Textile Engineering, University of Poly technique, Iran, 1997
B.S., Civil Engineering, University of New Mexico, 2015
M.S., Civil Engineering, University of New Mexico, 2017

Abstract

Carbon fiber reinforced polymer (CFRP) composites have been attractive materials for many civil and mechanical applications. However, the high cost of carbon fibers has hindered its wide spread in many civil and structural applications. Furthermore, CFRP composites have been mostly disposed of in landfills after their end of service life, creating environmental concerns and potential release of toxic byproducts. A significant interest in recycling composites has appeared in the last decade for obtaining recycled carbon fiber. A process based on wet chemical degradation and thermal treatments of FRP composites showed the ability to produce recycled carbon fibers with 99% purity and properties near those of virgin fibers. The recycled carbon fibers are about 20% of the cost of new carbon fibers.

This Thesis, discusses the use of recycled carbon fibers produced using the above recycling process to produce new CFRP composites. Also, the effect of fiber length and composite volume fraction on the tensile strength of the short fiber reinforced polymer is investigated. Moreover, methods of fabricating a new recycled CFRP composite using recycled carbon fibers is discussed. Two types of recycled composites are tested and compared with neat carbon fiber plain weave composite with similar composite density. Those two types include recycled CFRP produced using neat epoxy and recycled CFRP produced using COOH functionalized multi-wall carbon nanotubes (MWCNTs). The objective of including MWCNTs is to improve the interfacial shear transfer in the recycled CFRP composite. We show that the new recycled CFRP composites have comparable tensile strength to the on-axis strength of plain weave CFRP composite. The significance of fiber volume fraction and MWCNTs content on the behavior of the recycled CFRP composite are also discussed. An analytical model to predict rCFRP strength and stiffness (elastic modulus) is presented and used to explain the experimental observations.

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Chapter 1. Introduction

1.1. Problem statement

The challenge is to fabricate a new rCFRP composite that is low cost and meet the requirements for civil engineering infrastructure application. The problems that are addresses in this thesis include:

- Alignment of chopped recycled carbon fibers as much as possible
- To reach a high-volume fraction of 40 % or higher
- To increase the bonding between fibers and epoxy
- To reach a strength of at least 130 MPa
- Overcome the shear failure of the coupons

These are the challenges we are facing right now in this project. To solve each challenge, a specific method has been used which will be explained in the thesis.

1.2. Motivation

The manufacturing of composites out of recycled carbon fiber has gained a lot of attention. With the increase in CFRP composite usage, there is an important and growing concern of CFRP waste management. In all products, during manufacturing and after the product life cycle some amount of waste is produced. Currently, most composite materials are being thrown away in the landfills. Both environment and economic factors have motivated researchers to develop unique manufacturing method for recycled CFRP composites. Our motivation is that recycled carbon fiber mats can be produced by either wet lay process or by carding of recycled carbon fibers. Also, the regulations set by government regarding waste disposal, motivated scientists to start a research about recycled carbon fiber and its application.

1.3. Outline of the thesis

Chapter 2 of this thesis provides a literature review about recycled carbon fiber which includes carbon fiber reinforced polymer (CFRP), CFRP for civil engineering applications, carbon footprint of CFRP, regulations of FRP landfills, carbon fiber recycling process, and motivation. Chapter 3 covers the experimental method including experimental program, materials, fabrication method, and characterization of recycled – CFRP. Chapter 4 presents the analytical model showing the relationship between fiber length, and orientation with the strength of the composite. The results of all the experimental and analytical investigation are presented and compared. This chapter discusses the experimental behavior and explains those observations using the proposed analytical model. Chapter 6 concludes the research with recommendations for future work.

Chapter 2. Literature review

2.1. Overview

In this chapter, background information on recycled carbon fiber is reviewed. The chapter starts with an outline of fiber reinforced polymer (FRP) definition, and components. Then the application of CFRP's in civil engineering study has pointed out. Later, carbon footprint of CFRP is explained. The chapter continues with regulations of FRP landfills. At the end, the recycling process of carbon fiber and the motivation behind this research is reviewed.

2.2. Carbon Fiber Reinforced Polymer

2.2.1. Definition of fiber reinforced polymer

Carbon Fiber Reinforced Polymer (CFRP) composites are composite materials composed of polymer matrix reinforced with carbon fibers. Fiber reinforced polymer (FRP) composites are characterized by relatively high strength – to – weight ratio, thus suitable for fabricating structural elements used in aircrafts, satellites, submarines, surface ships as well as sophisticated robot components and beyond. Most importantly, CFRP is being utilized for building, strengthening, and repair of structural and non-structural elements in civil infrastructure. Figure 1 illustrates a bridge that the deck is a sandwich construction with carbon fiber (CFRP) laminates.

Figure 1.Bascule FRP Composite Footbridge[1]

2.2.2. Matrix

Polymers used for matrix in producing FRP are called resins and are polymerized using a hardener. The matrix is making usually up to 30 - 40% by volume of a composite. Generally, the matrix sustains composite frame, aligns the fiber reinforcement, functions as a stress transfer medium, and protects the fibers from abrasion, and other environmental damages. Moreover, mechanical properties of the matrix greatly influence the limitations of the composite. For example, the thermal stability, and exposure to hostile environment like aggressive chemical environment or damp environment, may degrade the performance of the matrix, and consequently the composite. Technically, any condition that lowers the glass transition temperature (Tg) of the matrix, can reduce the structural performance of the composite. The glass transition temperature is the temperature at which the material changes its state from a hard state to a viscous (rubbery) state. Below Tg, the resin can transfer load and support the fibers whereas above Tg, the elasticity of the matrix is reduced. Resins are classified to be either thermosets or thermoplastics. Thermoset resins exhibit some irreversible chemical changes when cured by increasing temperature. After curing, if heated, they will not reverse to their pre-curing liquid state; however, they decompose at very high temperatures. While thermoplastics will melt when heated and solidify when cooled down. Thermoplastic composites can be reshaped after forming a composite which is a unique ability allowing recycling, but a drawback for some applications specifically infrastructure applications.

2.2.3. Reinforcement

In general, three types of fibers are used as reinforcement in FRP composites. They are carbon-based fibers, glass-based fibers, and synthetic polymer fibers. The role of fibers is to increase the strength and stiffness or in other words to reinforce the matrix. The basic building block of these fibers are carbon, silicon, oxygen, and nitrogen. Carbon fibers have superior mechanical properties and thermal stability compared with other fibers. High modulus carbon fibers are produced by pyrolysis of a hydrocarbon precursor. Rayon, Polyacrylontrile (PAN) and other petroleum precursors are used to synthesize carbon fibers. Rayon precursors yield about 25% carbon; therefore, it is no longer used to produce carbon. Polyacrylonitrile (PAN) precursors yield up to 50-55% carbon, which currently accounts for most commercial carbon fiber production. Other petroleum precursors yield irregular surface carbon fiber with inferior tensile and compressive strength [2]. It is important to mention that other natural and synthetic fibers are being used too, but for limited applications compared with carbon fibers.

Table 2-1 [3]

Property	PAN			Pitch Type-P4	Rayon
	IM1	HM2	UHM3		
Diamter(µm)	8 -9	7 -12	7 -10	10 -11	6.5
Density(Kg/m3)	1780 -1820	1670 – 1900	1860	2020	1530 -1660
Tensile Modulus (GPa)	228 – 276	331 – 400	517	345	41 -393
Tensile strength (MPa)	2410 –	2070 -2900	1720	1720	620 – 2200
Elongation (%)	2930	0.5	0.3- 0.4	0.4 -0.9	1.5 -2.5
Coeff. Of thermal	1.0				
expansion(x10-6/°C)					
Fiber direction		-0.5 to -1.2	-1.0	-0.9 to -1.6	
Perpendicular to fiber	-0.1 to -0.5	7 – 12	-	7.8	
direction	7 – 12				38
Thermal		70 – 105	140	-	-
conductivity(W/m/°C)	20	925	-	-	
Specific heat (J/Kg/°K)	950				

[1] IM = Intermediate modulus.
[2] HM = high modulus.
[3] UHM = Ultra High Modulus
[4] Mesophase pitch precursor.

2.3. CFRP for Civil Engineering

CFRP composites are important in civil engineering applications. CFRP composites are anisotropic material that have different material properties in different directions due to fiber alignment. This ability to design for anisotropy allows CFRP composites to be used for structural applications in civil infrastructure. CFRP do not corrode, therefore; they are suitable candidate for structures exposed to moisture and wet-dry cycles. CFRP also have excellent fatigue resistance allowing their use in civil structures subjected to cyclic loading.

FRP weight is approximately a quarter that of steel. This reduces transportation cost significantly. Another important reason that civil engineers got interested in CFRP is for electromagnetically sensitive structure such as nuclear power plants, military structures, and airport towers.

2.4. Carbon foot print of CFRP

Carbon fiber is composed mostly of carbon atoms which are connected through covalent bonds and are aligned parallel to the longitudinal axis of the fiber. Thousands of carbon fibers are twisted together to produce a yarn. The fabric then is produced from weaving the yarn together. Finally, epoxy is added to fabric to form composites. The raw material used to make carbon fiber is called precursor. Earlier, carbon fiber was manufactured from heating the strands of rayon precursors until they carbonized. This carbon fiber is about 20% carbon and had relatively low strength and stiffness as shown in. Therefore, polyaclylonitrile (PAN) precursors are used through oxidation as shown in Figure 2. The resultant fiber is 55% carbon with relatively enhanced mechanical properties as shown in Table 2-1.

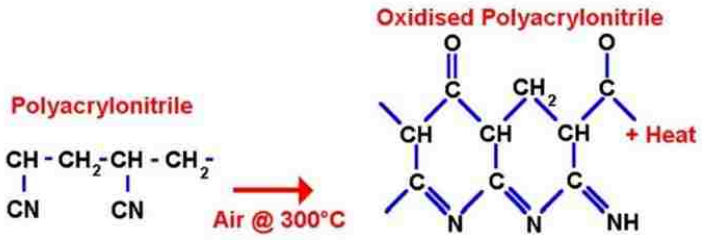


Figure 2. Oxidation of PAN [1]

The manufacturing process of making carbon fibers includes multiple chemical and mechanical processes. The first step is spinning. Acrylonytril plastic powder is mixed with another polymer, like methyl acrylate or methyl methacrylate, to form polyacrylonitrile (PAN) polymer. The plastic then spun into fibers. The fibers then washed and stretched to the desired diameter. The next step is stabilizing. In this step, the linear atomic bond between carbon atoms is altered to a more stable bond by heating the fibers to a temperature of 390°- 590°F (200 – 300 °C) for about 30-120 minutes. The carbonizing step is the next step. In this step the fibers are heated to a temperature of 1830°F – 5500°F (1000 – 3000 °C) for couple of minutes in a furnace which includes no oxygen. The lack of oxygen prevents fibers from burning and all the impurities are expelled. The last two steps include surface treating and sizing. After carbonizing, the surface of the fibers needs to be oxidized so that epoxy bonds with carbon. After surface treating the fibers are coated to protect the fibers from damage. Reaching the high carbonizing temperature of 3000°C requires a lot of energy. The embody energy required to produce carbon fiber is 183- 286 MJ/Kg. Whereas the energy required to produce recycled carbon fiber is 2.03 MJ/Kg [4]. Environmentally and economically, recycling of carbon fiber composites is beneficial to reduce energy consumption, thus greenhouse gases emissions.

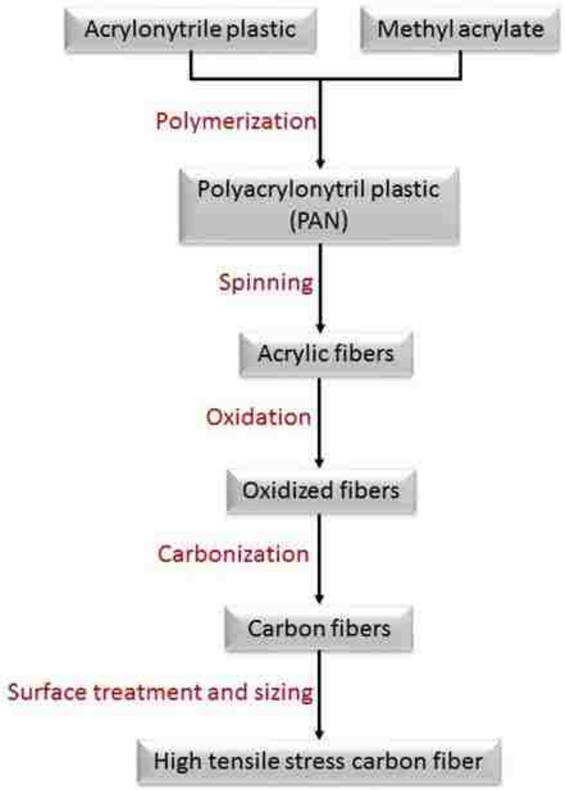


Figure 3.Manufacturing process of PAN carbon fibers

2.5. Regulations of FRP landfills

Over the past couple of decades, recycling has become part of everyday life. Recycling or reusing newspapers, glass, plastic bottles, and aluminum cans has become very common. The recycling of polymer based products started toward the end of 1970's after the oil crisis in 70's which led to considerable increase in raw material costs. The composite products recycling started in mid-80's after the increase in use of composite products in automotive industry which consumes up to a quarter of all composites manufactured. Landfill and incineration have always been the simplest and preferred methods of disposal accounting for

98% of composites waste, while alternative routes such as reuse and mechanical recycling account for the remaining 2%. [5] The high percentage of the CFRP waste is released into the landfills, abandoned graveyards, and airports. However, these solutions are unacceptable for the following reasons:

- Environmental Impact: Concerns about waste disposal and consumption non-renewable resources increases.
- Legislation: Recent European legislation requires new rules for composite disposal. It is required that automotive vehicles after 2015 to be 85% or more recyclable [6]
- Production Cost: Carbon fibers are expensive products, both in terms of energy consumed during manufacturing (up to 165 kWh/kg) and material price (Up to 40 £/kg) [7]
- Economic opportunity: disposing of the CFRP by landfills, can cost approximately 0.20 £/kg. Recycling would convert an expensive waste into a profitable reusable material. [8]

New European waste commands is putting pressure on these traditional disposal routes. Landfill of composite waste has been banned by the end of 2004 by most EU Member States and incineration has limits imposed on the level of energy content.[5] In dealing with waste, the waste legislation emphasizes on waste hierarchy as shown schematically in Figure 4.

Figure 4. The waste hierarchy

In the year 2000, approximately 156,000 tons of composite was produced from the end of life and the production waste. The increase in the amount of waste production has led to the establishment of directives and regulations relating to effective management of composite waste.

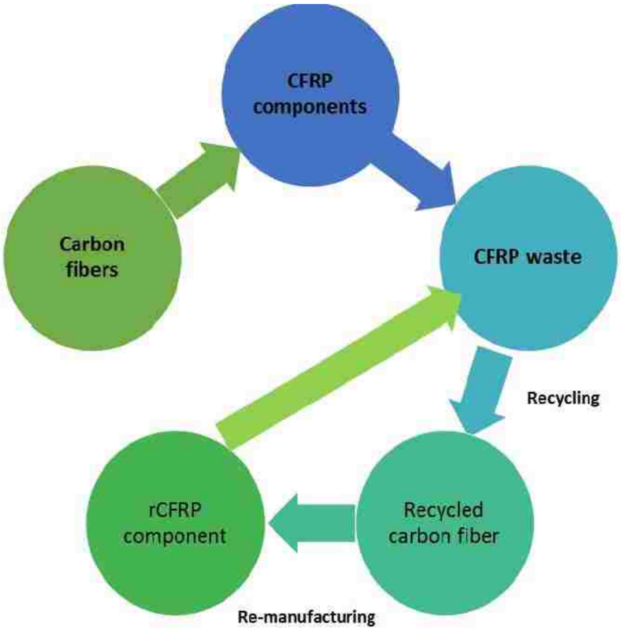


Figure 5.Closed life - cycle for CFRPs

2.6. Carbon fiber recycling processes

Recycling of carbon fibers and CFRP has been investigated for many years. The Boeing Company, Europe Aviation, Rolls-Royce and Adherent Technologies are founding members of the Aircraft Fleet Recycling Association (AFRA). AFRA is an international non-profit association bringing together companies from around the globe to manage the end-of-life aircrafts and engines [9]. Now, the members of AFRA include manufacturers of aircraft parts (Rolls Royce), aircraft manufacturers (The Boeing Company), aircraft salvage yards (Huron Valley Fritz West, Air Salvage International), and recyclers of CFRP waste (Adherent Technologies, Milled Carbon Ltd). The fiber reinforcement has potentially the most recoverable value in a composite [1]. Adherent Technologies, Inc. (ATI) located in Albuquerque, NM and Milled Carbon Ltd. Located in Warwickshire, UK. are two of the main companies specialized in recycling CFRP composites and reclaiming carbon fibers. Adherent Technologies, Inc. has developed two individual technologies for recycling. The first one is vacuum cracking to process polymer-containing waste into raw materials. The second one is the wet chemical breakdown of composite matrix resins to recover fibrous reinforcements [10].

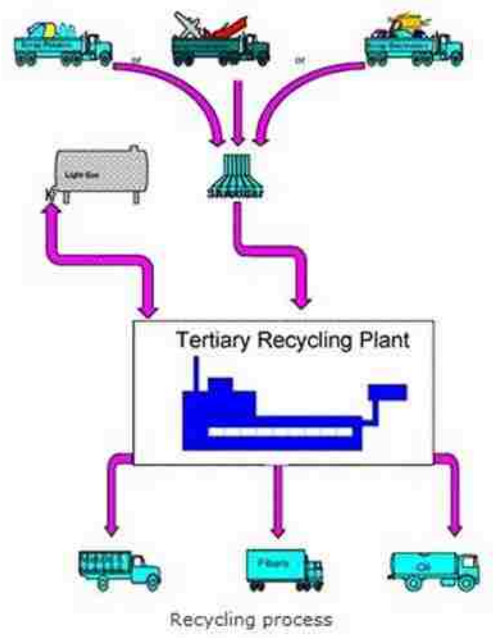


Figure 6.CFRP recycling process concept as suggested by Adherent technologies Inc. [6]

In general, to recycle CFRPs, two technologies exist: Mechanical recycling and fiber reclamation as shown schematically in Figure 6. In mechanical recycling, the size of the waste will be reduced; whereas, in fiber reclamation technique, the waste breaks down into constituent materials.

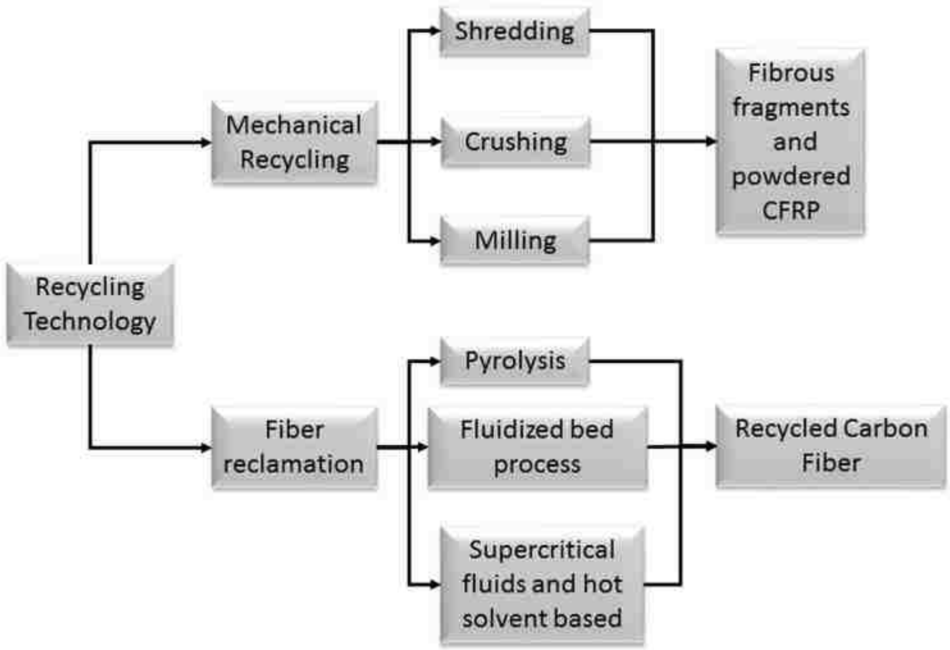


Figure 6. Recycling process

2.6.1. Mechanical recycling

In the mechanical recycling process, CFRP composites are broken down by shredding, crushing, or milling. The resulting mixture then is classified into fibrous fragment and powdered CFRP. Mechanical recycling processes are suitable for scrap composite material which is relatively clean and uncontaminated and from known origin[11]. The powder recyclates have some limitation. They have lower density, so they cannot be used in thermoset compounds they originated from. Moreover, these powders have lower mechanical properties. In addition to having lower density and mechanical properties, their processing is difficult. The fibrous recyclates have some potential as reinforcement materials, but they are not as good as the virgin reinforcement, and there are problems associated with the bonding of the recyclate with polymers and the tendency for the large pieces of recyclate to be stress raisers and act as failure initiation sites[11].

2.6.2. Fiber reclamation

In the fiber reclamation process, fibers are separated from the composite by Pyrolysis, or fluidized bed, or combustion.

2.6.2.1. Pyrolysis

The most common method of reclaiming fibers is pyrolysis. Pyrolysis is the decomposition of compounds in high temperature. Pyrolysis treatment decomposes organic materials by heating in the absence of oxygen. In pyrolysis, the objective is to produce fibers with maximum possible mechanical properties of virgin fibers with defined length and undamaged fiber surface. To do that, production waste or scrap parts are sorted and cut into smaller pieces if necessarily. The predetermined sized pieces will be fed to a pyrolytic decomposition chamber as shown in Figure 7. In this chamber, the thermo-chemical division of the organic compound takes place at temperatures more than 500 °C. The long-chained molecules will be broken into smaller ones through heat without oxygen. The remaining is carbon fiber. The carbon fiber produced this way is undamaged since there is no burning involved and the structure of carbon fiber is unchanged which is very important for the mechanical properties. The limitation of the pyrolysis process is the control of temperature and oxygen content throughout the entire chamber. Usually the fibers are either over oxidized leaving pits in the fiber surface or under oxidized leaving chunks of burned resin attached to the fiber.

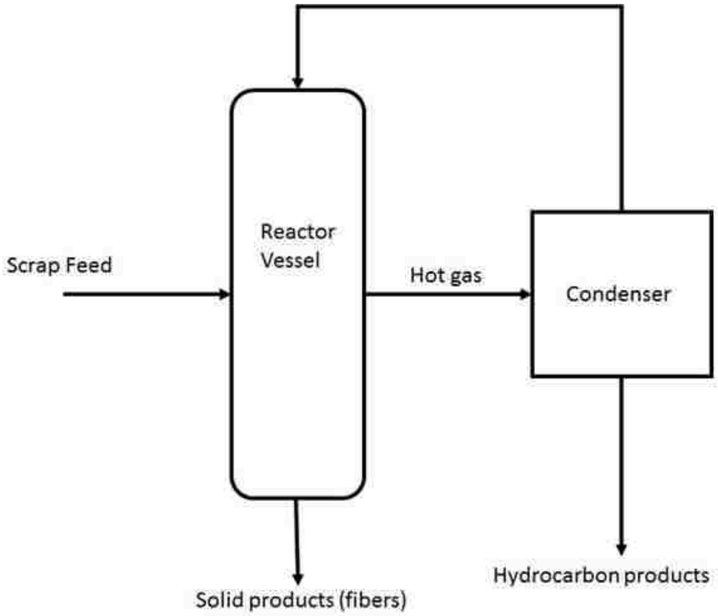


Figure 7.Pyrolysis process

Figure 8.ATI's Phoenix reactor, a pilot scale vacuum pyrolysis unit[8]

2.6.2.2. Wet Chemical treatment

2.6.2.2.1. High temperature, high pressure

ATI developed this method. In this method, a heat transfer fluid along with depolymerization catalyst is used is utilized to produce a recycled carbon fiber with properties close to the virgin fibers. The high purity of 99.9% is observed with 10% reduction in strength compared to virgin fibers[12]. This technique is expensive when high scale products is used. The temperature of 300 °C and the pressure of 500 psi outside of normal change, so the equipment used need to be monitored constantly.

2.6.2.2.2. Low temperature, low pressure

Due to high cost, and demanding maintenance of high temperature, high pressure method, ATI therefore developed a low temperature, low pressure process. The typical temperature of 150°C and pressure of 150 psi is utilized. The catalyst used in high temperature was ineffective in this temperature, so a new catalyst is used. The recycled carbon fiber reclaimed in this method is comparable with the previous one, fig. 9 shows composite recycling reactor.

Figure 9.Low temperature, Low pressure recycling reactor

2.6.2.3. Fluidized bed

This method is a thermal process and composites get exposed to more uniform temperature than pyrolysis. In this process, the waste or scrap of carbon composites is cut into small pieces, usually 25 mm long. The small pieces then fed into a fluidized bed. The fluidized bed is a bed of silica sand. The sand is fluidized with hot air at 450°C - 550°C temperature. The bed material in a fluidized bed stores heat from the hot air blow in and the exotherming of waste material [13]. In the fluidized bed, the fibers and matrix get separated. Then a cyclone separator separates loose carbon fibers from other materials as shown in Figure 10.The limitation of this method is that the fibers come out tangled bunch like a bird nest which makes it extremely difficult to align the fibers.

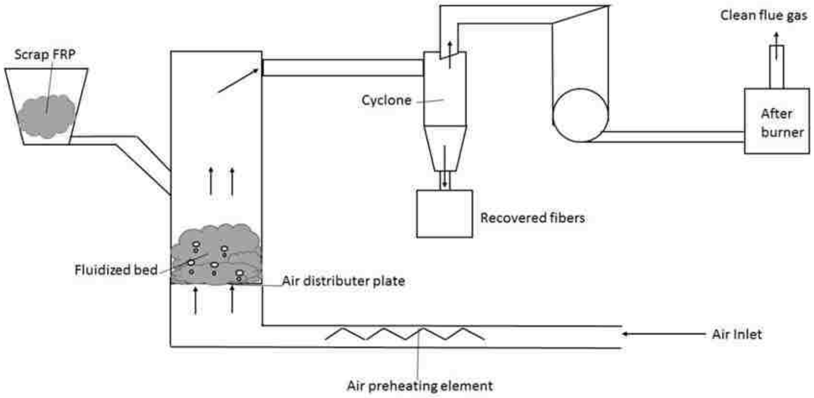


Figure 10.Fluidized bed recycling process[11]

2.6.2.4. Supercritical fluids

Supercritical fluids such as water and alcohol are getting renewed interest in academics. Supercritical fluids recycling technologies have a potential to achieve 100% strength retention in recycled fibers[13].

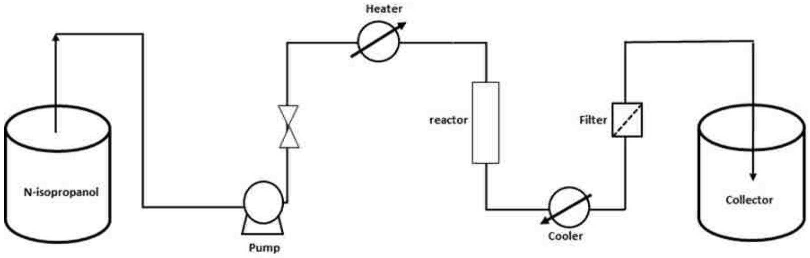


Figure 11.A schematic flow diagram for the recycling process[10]

2.7. Motivation

The manufacturing of composites out of recycled carbon fiber has gained a lot of attention since 2009. With the increase in CFRP composite usage, there is an important and growing concern of CFRP waste management. In all products, during manufacturing and after the product life cycle some amount of waste is produced. Currently, most composite materials are being thrown away in the landfills. Both environment and economic factors have motivated researchers to develop unique manufacturing method for recycled CFRP composites. Our motivation is that recycled carbon fiber mats can be produced by either wet lay process or by carding of recycled carbon fibers.

Chapter 3. Experimental Methods

3.1. Introduction

This chapter describes the experimental methods including materials, fabrication methods, test set-ups, and test procedures. This chapter starts with the material used and fabrication methods. Then the chapter ends with testing procedures.

3.2. Experimental Program

95 coupons were tested. These 95 coupons were classified into 19 groups and the description of all the coupons are given in table 3-1. These FRP samples are categorized according to being fabricated with different methods. For example, some mats are soaked in glycerin and some mats are soaked in water. The results of these two will be compared together. The categories that the rCFRP's are reviewed are:

- Carding machine and no carding machine
- Epoxy and MWCNTs
- (VARTM)Vacuum Assisted Reinforced Transfer Molding and RTM (Resin Transfer Molding) and VAHLM (Vacuum Assisted Hand-layup Molding)
- Glycerin soaked and water soaked
- Sized and un-sized

	Fiber		Condition			Method				Cut		Matrix		Sizing	
	Prepreg	T-800	Water Soaked	Dry	Glycerin Soaked	Carding	VARTM	VAHLM	RTM	Sharp	Curve	Epoxy	MWCNTs	Silane	Epoxy
r-CLD1	✓			✓				✓		✓		✓			
r-CLD2	✓		✓					✓		✓			✓		
r-CLD3	✓		✓					✓		✓		✓			
r-CLD4	✓		✓					✓		✓			✓		
r-CLD5	✓		✓					✓		✓		✓			
r-CLD6	✓		✓				✓			✓		✓			
r-CLD7	✓			✓		✓		✓		✓		✓			
r-CLD8	✓				✓				✓		✓	✓			
r-CLD9		✓			✓				✓		✓				
r-CLD10		✓			✓				✓		✓				
r-CLD11		✓			✓				✓			✓			
r-CLD12		✓	✓						✓		✓	✓			
r-CLD13		✓	✓					✓			✓	✓		✓	
r-CLD14		✓	✓					✓			✓	✓			
r-CLD15		✓	✓					✓			✓	✓		✓	
r-CLD16		✓	✓					✓			✓	✓			✓
r-CLD17		✓	✓					✓			✓		✓		✓
r-CLD18		✓	✓					✓			✓		✓		✓
r-CLD19		✓	✓					✓			✓		✓		✓

Table 3-1. The Specification of tested specimens

3.3. Materials

3.3.1. Recycled Carbon Fibers

Recycled Carbon fibers are chopped fibers which exist in a variety of forms based on the recycling method and/or intended application of the fibers. These varieties include milled, fabric, chopped, and entangled. In this experiment, the chopped recycled carbon fibers produced by Adherent Technologies Inc. (ATI) (Albuquerque, NM) is being used. Pyrolysis method, the most common technology in today's market was used to produce recycled carbon fibers out of composite. As mentioned in section 2.6.2.1, the high temperature in pyrolysis damages the surface of the recycled fibers. The fibers produced in this method are brittle which make them undesirable for structural application; in addition, their strength will decrease significantly due to damage existence in the surface of the fibers. [14] Considering this limitation, Allred el al. [15] proposed combining the catalyst-based wet recycling process with vacuum pyrolysis treatment to reclaim carbon fibers. This proposed method has two steps: First, the 2" x 2" composite plates are fed into a low temperature (150 °C), low pressure tank (150 psi). During this process, polymer bonds are broken in the presence of a catalyst and matrix is dissolved with minimum damage to the fibers. Second, the reclaimed fibers are fed to a vacuum furnace to remove any impurities and to reach the highest level of purity as possible usually between 99% to 99.5%.

Figure 12. Recycling carbon fiber composite at Adherent Technologies. Inc. (a) composite plates prior to recycling (b) reactor of wet chemical composite (c) carbon fiber extracted from the reactor (d) Vacuum furnace (e) recycled carbon fibers

3.3.2. Epoxy

EPOTUF® 37-127 supplied by U.S. Composites Inc. and manufactured by Reichhold chemicals is used in fabricating this recycled carbon fiber composites. The resin is a low viscosity with 100% reactive diluted liquid epoxy resin based on Bisphenol A which contains EPOTUF® 37-058 which is C_{12} – C_{14} glycidel ether. The hardener is EPOTUF® 37-614 which is a aliphatic polyamine curing agent. Resin has a viscosity of 600 cps at 25^{o}C with a density of 1.1348 g/cm^{3} and tensile strength of 9-10 psi (62053 – 68948 Pa). The mixing ratio of resin to hardener is 2:1 with a curing time of 24 hours at 40 °C or 2 hours at 120 °C. The tensile strength of Epoxy is 62 KPa and the flexural strength is 117 KPa as reported by the manufacturer.

3.3.3. Functionalized MWCNTs

Carbon nanotubes are synthesized by the action of catalyst on the gaseous species originating from the thermal decomposition of hydrocarbon[16]. The multi walled carbon Nano-tubes were produced using Catalyzed Chemical Vapor Deposition Technique (CCVD) by Cheap-Tubes. The functionalized MWCNTs (COOH – MWCNTs) used in this work has outer diameter of 20 – 30 nm and inner diameter of 5 – 10 nm, and length of 10 – 30 μm. Functionalization was performed by the manufacturer by means of acid treatment chemistry of H2SO4/HNO3. Carboxyl groups were attached at the ends and side walls of the nanotubes. This process is often performed by adding H_2O_2 to the chemical treatment.

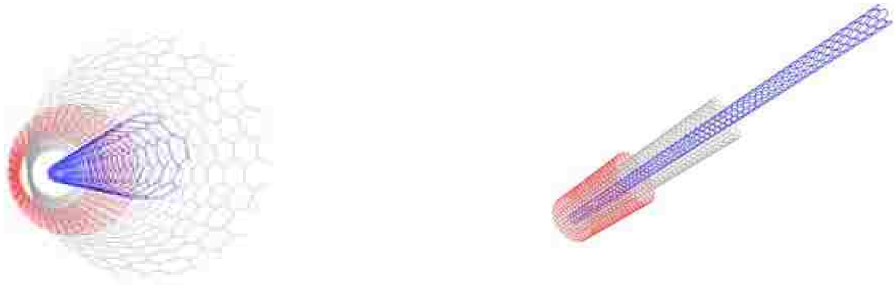


Figure 13.MWCNTs

3.3.4. Silane

(3 – Glycidloxyopropyl) trimethoxysilane supplied by 3- ALDRICH was used in this experiment for surface treatment of recycled carbon fibers in order to reinforce epoxy bonding between fibers and epoxy.

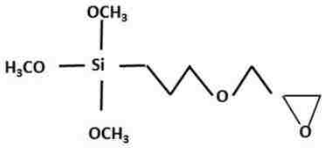


Figure 14. The structure of Silane (3 – Glycidloxyopropyl) trimethoxysilane

3.4. Fabrication Method

In this section, the fabrication method of all the recycled carbon fiber reinforced polymer composites is explained. Majority of composites are fabricated either with vacuum assisted hand lay-up technique, resin transfer molding (RTM), or vacuum assisted resin transfer molding. Occasionally, there has been a few other methods that were examined; however, since either the results were not satisfying or the methods were not repeatable, there has been less emphasis on them. Adherent technology Inc. provided two different types of recycled fibers. The first type was fibers reclaimed from overaged prepregs; little is known about the original carbon fiber they are coming from. The second type was recycled carbon fiber reclaimed from TORAYCA® T800 carbon fiber with known properties.

3.4.1. Recycled carbon fiber mats

3.4.1.1. Water soaked mats

This method of mat making consists of three stages: soaking of recycled fibers in water, dispersing the fibers on a peel ply, pressing the fibers using the heat-press for about 2 hours, drying of the produced veil. Approximately 10 – 15 g of recycled carbon fibers are soaked in water for about 1 hour. Then, the fibers are dispersed on a 7 inch X 7-inch piece of peel ply aligning the short fibers in one direction as much as possible. The semi- aligned fiber's mat is then pressed for two hours. In the last step, the recycled carbon fiber mat is dried in the oven of 104 °C for about 10 hours. Figure 15 illustrates the recycled carbon fiber mat which is produced via water soaking procedure which is explained in 3.4.1.1

Figure 15. Recycled carbon fiber mat

3.4.1.2. Glycerin soaked mats

This method of mat making consists of four stages: fiber dispersion, fiber filtration, fiber bath with Luke-warm water, and mat drying. Small quantities of cut recycled carbon fiber is added to 400 ml of glycerin solution. The solution is stirred using a shear radial impeller as shown in Figure 16.a at a speed of between 850 – 900 r.p.m. for about 10 minutes. The glycerin solution was 96% by weight glycerol to water and contained 0.3 ml per 1 liter liquid anti foaming agent, tribuyl phosphate. Then, the fiber suspension was poured into a filtration system as shown in Figure 16.b where the excess glycerin is drained through a paper filter which was laid inside a round, plastic mesh which is connected to a vacuum. After all the glycerin was filtered, to remove the excess glycerin that is adhered to the fibers, the mat is sprayed with warm water to decrease the viscosity of the glycerin helping the extraction process of glycerin. The round veil is then dried in the oven of 104 °C for about 10 hours.

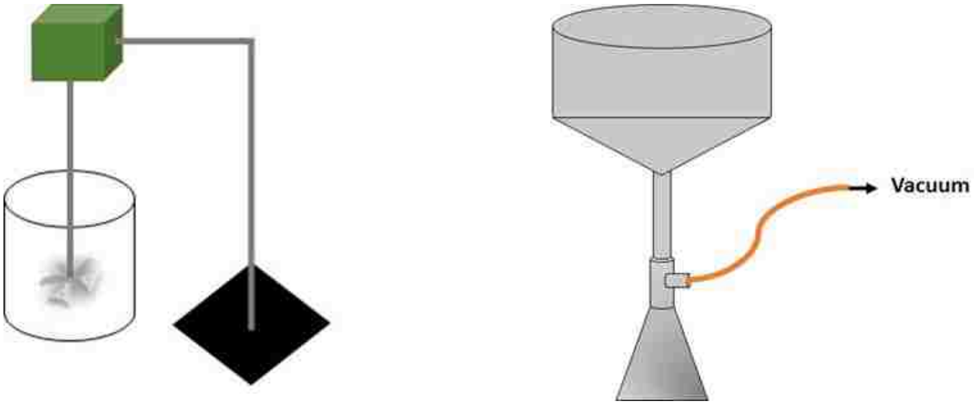


Figure 16.a) Mechanical stirrer b) washing and vacuuming setup

3.4.2. rCFRP coupons using epoxy

3.4.2.1. Vaccum assisted hand lay-up technique

The rCFRP composite plates were fabricated using vacuum assisted hand lay-up technique. Approximately, 5 - 10 g recycled carbon fibers were soaked in the deionized water for about one hour as shown in Figure 17 (a). A hand lay-up technique was used to align the fibers on a 7 X 7 in (177.8 X 177.8 mm) peel ply as shown on Figure 17 (b). Then the semi – aligned wet layer of recycled fibers were put under heat and pressure using a heat-press machine for about 2 hours.

In the next step, the fibers were dried for about 12 hours in the oven of 105 °C. To make a composite, first the non-porous release film was attached over a metal plate; the metallic plate was used to enable the fabrication of a flat composite. Then a peel ply was placed in the middle of the metal plate on top of the release film. The impregnated recycled carbon fibers were then placed on top of the peel ply. Another peel ply was placed over the recycled carbon fiber layer. A porous release film was placed over the peel ply to allow the entrapped air, and excess resins to escape. A breather was be applied over the layers to create even pressure at the same time allowing air to escape. The final layer was a vacuum bag. This film was sealed using seal tape. A nozzle was inserted in the vacuum bag and was connected to a vacuum hose then to the pump. The vacuum nylon bag was checked regularly for any leakage. Then the composite was cured at 40 °C for another 24 hours. Waterjet cutting was applied to create bone shape coupons for tensile testing. Steps of creating a recycled carbon fiber coupon are illustrated in Figure 17:

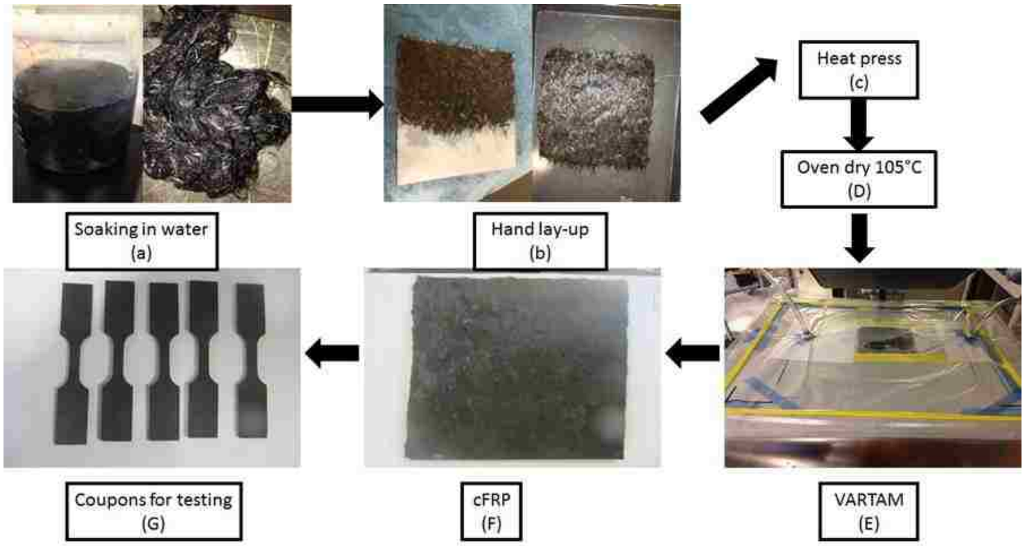


Figure 17.Synthesis of recycled carbon fiber composites

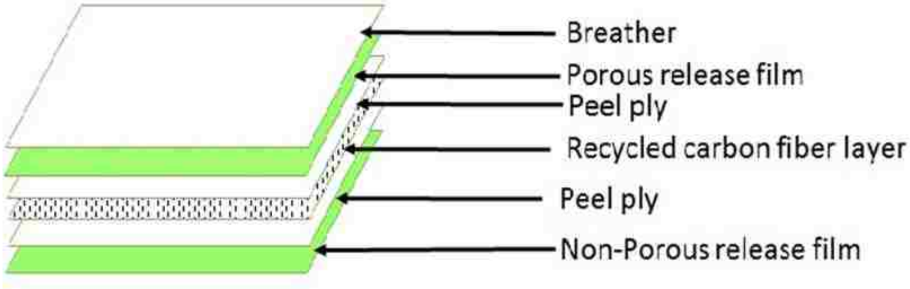


Figure 18.Composite layers

3.4.2.1.1. rCLD1

This specimen was fabricated by randomly dispersing 5 g recycled carbon fibers reclaimed from overaged PrePreg on a 6 X 6-inch (152.4 X 152.4) mm peel ply then dispersing the epoxy over the fibers using a roller. The rest of the process and curing has been thoroughly explained in section 3.4.2.1.

3.4.2.1.2. rCLD3

For fabricating this specimen, 5 g recycled carbon fibers from overaged PrePregs were soaked in water for about an hour. Then they were lined up on a 6

X 6-inch (152.4 X 152.4) mm peel ply and were fabricated using vacuum assisted hand lay-up technique shown in section 3.4.2.1.

3.4.2.1.3. rCLD4

To fabricate specimen rCLD4, first, 15 g of recycled carbon fiber was soaked in water. The purpose of making this specimen was to investigate the influence of weight of the fibers on the mechanical properties of the composites which will be shown in the result section.

3.4.2.1.4. rCLD7

To fabricate this sample, a carding machine was used to align the dry fibers first. In this process, dry recycled carbon fibers were fed into a small drum then a bigger drum in the carding machine. Then by the help of a comb the fibers were aligned.

3.4.2.2. Resin Transfer Molding (RTM)

Resin transfer molding (RTM) was used to fabricate the recycled carbon fiber composites (rCFRP). In this method recycled carbon fibers were first dispersed in the glycerin. Then the solution was mixed with a mixer with velocity of 85 r-p-m. After about 10 minutes of mixing, the content was poured into a funnel and using vacuum the excess glycerin was extracted. After the required thickness of fibers was laid on top of each other, the excess glycerin was then washed using warm water. The dried fibers then transferred into RTM machine and was impregnated via a tube from a piston. When the required amount of epoxy was fed, the machine was stopped and let the composite stay inside RTM for 24 hours, then it was cured for another 24 hours at 40 °C then removed and was prepared for testing. Figure 19 illustrates the RTM machine used for this experiment.

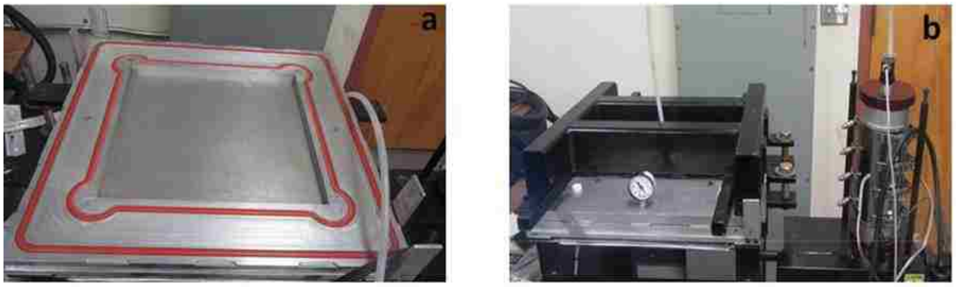


Figure 19. RTM (a) without and (b) with composite inside

3.4.2.3. Vacuum Assisted Resin Transfer Molding (VARTM)

Vacuum Assisted Resin Transfer Molding (VARTM) is a closed mold composite manufacturing system. VARTM is a variation of RTM with some differences in resin flow into the composite. The top part of the system can be replaced with vacuum bag. This allows fabrication of different size composites while in RTM only small sized composites can be produced.

Figure 20.Vacuum Assisted Resin Transfer Molding (VARTM)

3.4.2.3.1. rCLD-8

To fabricate this composite, a different approach was considered. The fibers were first soaked in glycerin. Then a mechanical stirrer was used to separate the fibers by rotating with a speed of 85 rpm for about 10 minutes. A big round funnel was used to filter out the glycerin as shown in Figure 21. The extra glycerin was then washed out by Luke warm water without disturbing the alignment. After drying the semi aligned recycled carbon fibers in the oven, the composite was fabricated using RTM machine as explained in section 3.4.2.2

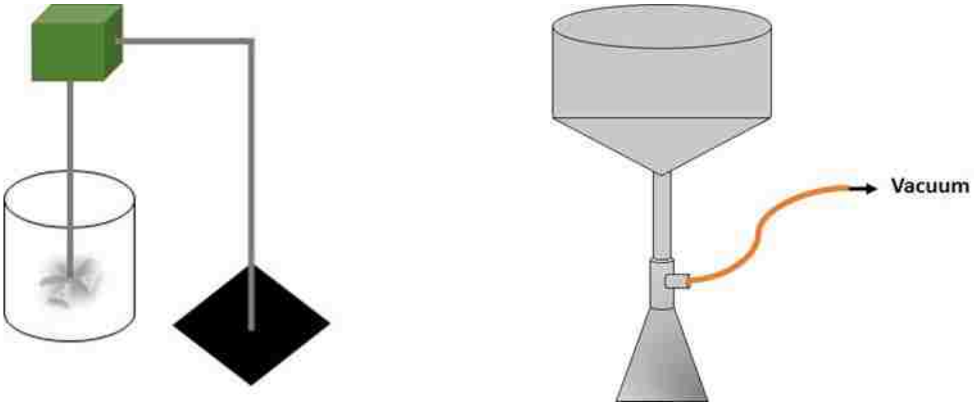


Figure 21.a) Mechanical stirrer b) washing and vacuuming setup

3.4.2.3.2. rCLD11

This composite is fabricated with the same method as in section 3.4.2.3.1. The only small difference is the type of recycled fibers used. For previous sample, a recycled carbon fiber that was reclaimed from an overaged prepreg was used. However, for the sample rCLD11, T-800 was used.

3.4.2.3.3. rCLD12

This composite is fabricated using T-800 reclaimed fibers in RTM. However, the fibers were soaked in water instead of glycerin to see the difference between glycerin soaked and water soaked fibers. The mechanical stirrer and vacuum set up was not used for this sample.

3.4.3. rCFRP composite incorporating MWCNTs

To fabricate a rCFRP incorporating MWCNTs, the desired amount of MWCNTs was dispersed into the epoxy resin prior to fabricate the composite. The mixture was then sonicated to agitate the nanotubes in the mixture for 2 hours at 40°C. Furthermore, using mechanical stirrer the solution was mixed for another 2 hours at 60°C. After the mixture was cooled down to room temperature, the hardener was added and mixed by hand for 2 minutes. The epoxy incorporating MWCNTs is then used to impregnate the recycled carbon fiber mat. The sonication and the stirring procedure machine are shown in Figure 22.

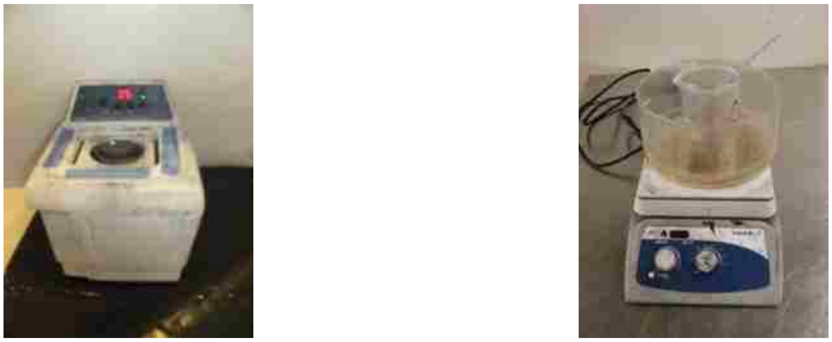


Figure 22.(a) Sonication, (b) Mechanical stirring

For fabricating this specimen, 5 g recycled carbon fibers from overaged prepregs were soaked in water for about an hour. Then they were lined up on a 6 X 6 in (152.4 X 152.4) mm peel ply and were fabricated using vacuum assisted hand lay-up technique and was added 0.5 % MWCNTs according to the method explained in section 3.4.3.

3.4.3.1. rCLD9

This composite is made exactly with the same method explained in section 3.4.2.3.1 but with 0.5 % MWCNTs.

3.4.4. Fiber sizing

The recycled carbon fibers were cleaned using acetone. After waiting for about 15 minutes for the acetone to be evaporated, the fibers were immersed in nitric acid solution for about 2 hours to polarized the fiber surface. Then, deionized water was used to wash the acid completely from the fibers for about 45 minutes. Subsequently, the fibers were dipped in a mixture c with 50% in volume of ethanol aqueous solution at a concentration of 3% by weight for 3 hours. Then, the recycled carbon fibers with saturated absorption of the coupling agent was baked under the vacuum at 120 °C for about 2 hours.

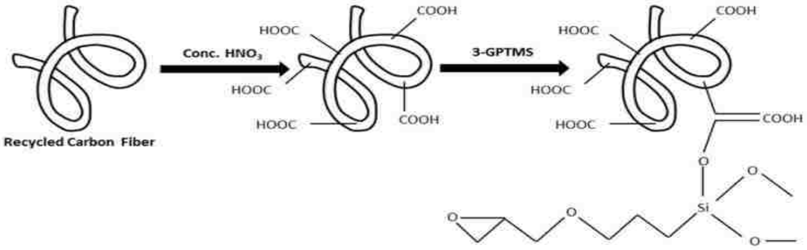


Figure 23.Process of sizing of recycled carbon fibers

3.4.4.1. Sized by Epoxy

3.4.4.1.1. rCLD 16

This composite was fabricated using the T-800 fibers that were sized by adherent company Inc. using epoxy.

3.4.4.1.2. rCLD17

This composite was fabricated using the T-800 fibers that were sized by adherent company Inc. using epoxy. In this sample, 0.5 % functionalized MWCNTs were used.

3.4.4.1.3. rCLD19

This composite was fabricated using the T-800 fibers that were sized by adherent company Inc. using epoxy. In this sample, 2 % functionalized MWCNTs were used.

3.4.4.2. Sized by Silane

3.4.4.2.1. rCLD 13

This composite was fabricated using the T-800 fibers that were sized by the solution of silane (3-glycidoxypropyitrimethoxysilane).

3.4.4.3. Un-sized

3.4.4.3.1. rCLD 14

This composite was fabricated using the T-800 fibers that were not sized to compare it with sized ones.

3.5. Characterization of recycled- CFRP

3.5.1. Mechanical Characterization

3.5.1.1. Tension Testing

The mechanical properties of materials are determined by performing a tension test. This test is a destructive test in which the specimen prepared according to ASTM D3039 and was under tension at a specific rate. The dog bone specimen with the dimensions of 25.4 mm X 152.4mm X 2.5 mm thick were cut using water jet cutting system. For gripping glass epoxy sheets were used as tapered tabs. The specimen geometry is shown in Figure 34. Tapered tabs were also cut and prepared by water jet company. A dog bone shape specimen was gripped at both ends. In a displacement control mode, a force at a rate of 0.25 mm/min was applied to the specimen until the specimen broke down. The displacement was recorded using an extensometer. The specimens were tested in MTS Bionix test machine. The stress of σ and the strain of ε can be computed using the equation 3-1and 3-2 where F is applied force, A is the cross-sectional area, Δl is the change in length, and L is the length of the specimen.

$$\sigma = \frac{F}{A} \tag{3-1}$$

$$\varepsilon = \frac{\Delta l}{L} \tag{3-2}$$

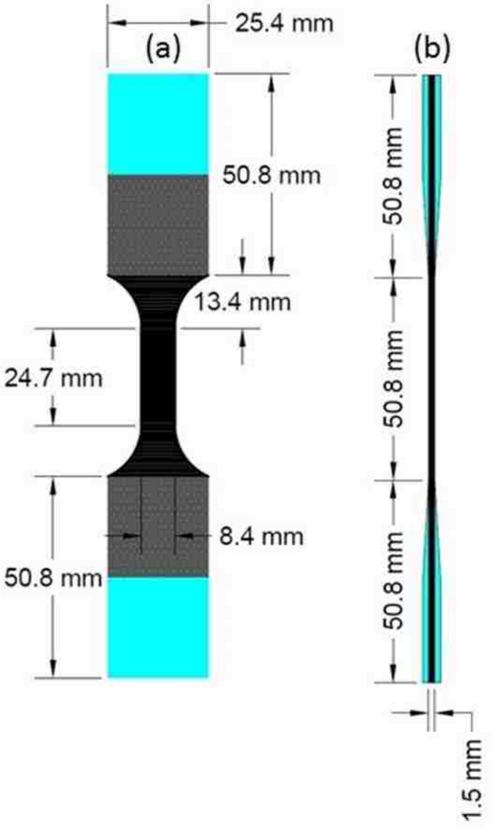


Figure 24. cFRP coupon specification

The linear part of the constructed stress-strain curve was used to calculate the modulus of elasticity E using the 3-3:

$$E = \frac{\sigma}{\varepsilon} \qquad 3\text{-}3$$

3.5.1.2. Fiber Volume fraction

To determine the fiber volume fraction of recycled carbon fiber reinforced polymer, nitric acid was used. In this method, the matrix was physically removed by digesting in hot liquid Nitric acid 69.5% for up to 6 hours under furnace. The remaining residue containing fiber was filtered, washed with distilled water and acetone, dried, cooled, and weighed. The procedure starts with cutting a specific amount of the composite and weighing it to the nearest 0.0001 g. Then using the scale in the lab, the density of each sample was determined. Each sample, usually 4, was placed in a separate beaker containing at least 30 ml of 70% nitric acid. After 4 – 6 hours of constant heating of 80 °C or less, the residue was transferred to the filter, to be washed with distilled water at least three times. An acetone wash was used at the end as a final wash after nitric acid was completely removed from the specimen. The specimen was placed in crucible dish and was dried in the oven of 100 °C for about an hour. After cooling off the specimen, its weight was recorded to the nearest 0.0001 g to be used in calculations. The following equations were used to calculate the fiber volume fraction of the recycled carbon fiber composites.

$$W_r = \frac{M_f}{M_i} * 100 \qquad \text{3-4}$$

Where M_f is the final mass of specimen in grams, M_i is the initial mass of specimen, and W_r is the weight percent of reinforcement in the specimen

$$V_r = W_r * \frac{\rho_c}{\rho_r} * 100 \qquad \text{3-5}$$

ρ_c is the density of the reinforcement in g/cm^3, and ρ_r is the density of the specimen in g/cm^3 and V_r is the volume fraction (volume percent).For the Matrix content, volume fraction, shown in equation 3-6 was used.

$$V_m = \frac{M_i - M_f}{M_i}\frac{\rho_c}{\rho_m} * 100 \qquad 3\text{-}6$$

Finally, the void volume was evaluated using the equation 3-7.

$$V_v = 100 - (V_r + V_m) \qquad 3\text{-}7$$

Chapter 4. Analytical Modelling

4.1. Overview

This chapter presents analytical methods studying the effect of fiber length and fiber orientation distributions for predicting the tensile strength of recycled carbon fiber composites. The effect of mean fiber length, the most probable length (mode length), the critical fiber length(L_c), the mean fiber orientation (φ_{mean}) and the fiber orientation coefficient on the tensile strength of the FRP composite were studied.

4.2. Theoretical background

4.2.1. Composite rule of mixture

Composite stiffness and strength can be calculated using the rule of mixture. To be able to use the rule of mixture, some assumptions need to be made. The first assumption is that fibers should be distributed uniformly throughout the matrix. The second assumption is that there is a perfect bond between fibers and matrix. The third assumption is that the matrix does not have any voids. The fourth assumption is that the applied load is either parallel or normal to the fiber direction. The fifth assumption is that the matrix and fibers are linear elastic. The predicting FRp composite strength and modulus of elasticity is being investigated in the sections 4.2.1.1 and 4.2.1.2:

4.2.1.1. Longitudinal strength and modulus of FRP composite

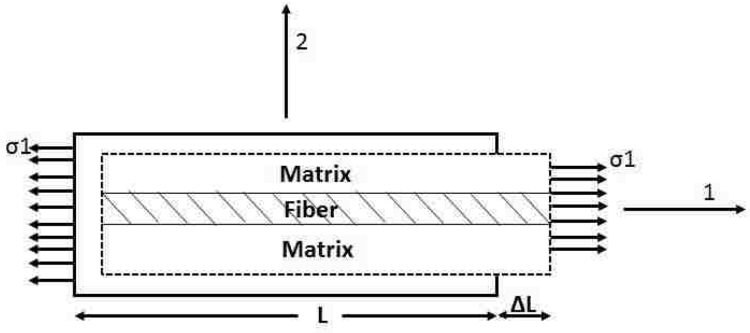


Figure 25. Representative volume element

When the longitudinal forces are applied, the total resultant force equals the sum of the force acting on fibers and matrix as shown in 4-1.

$$F_c = F_m + F_f \tag{4-1}$$

By substituting force with stress multiplied by area, the equation 4-1 can be written as 4-2.

$$\sigma_c * A_c = \sigma_m * A_m + \sigma_f * A_f \tag{4-2}$$

By dividing both sides of the equation by A_c , and substituting V_m, it can be concluded that the composite strength can be written as the rule of mixture as shown in 4-3.

$$\sigma_c = \sigma_m * \frac{A_m}{A_c} + \sigma_f * \frac{A_f}{A_c} \tag{4-3}$$

Since $V_m = \frac{A_m}{A_c}$, and $V_f = \frac{A_f}{A_c}$

$$\sigma_c = \sigma_m * V_m + \sigma_f * V_f \qquad 4\text{-}4$$

Applying hook's law, for linear elastic materials, it can be said that:

$$E_c * \varepsilon_c = E_m \varepsilon_m * V_m + E_f \varepsilon_f * V_f \qquad 4\text{-}5$$

Since $\varepsilon_c = \varepsilon_m = \varepsilon_f$

Then it can be concluded that

$$E_c = E_m * V_m + E_f * V_f \qquad 4\text{-}6$$

And finally:

$$E_c = E_m(1 - V_f) + E_f V_f \qquad 4\text{-}7$$

This equation represents parallel combination rule of mixture for modulus of elasticity. Moreover, the longitudinal tensile strength can also be estimated using the following equation:

$$\sigma_c = \sigma_f V_f + \sigma_m * (1 - V_f) \qquad 4\text{-}8$$

4.2.1.2. Transverse strength and modulus

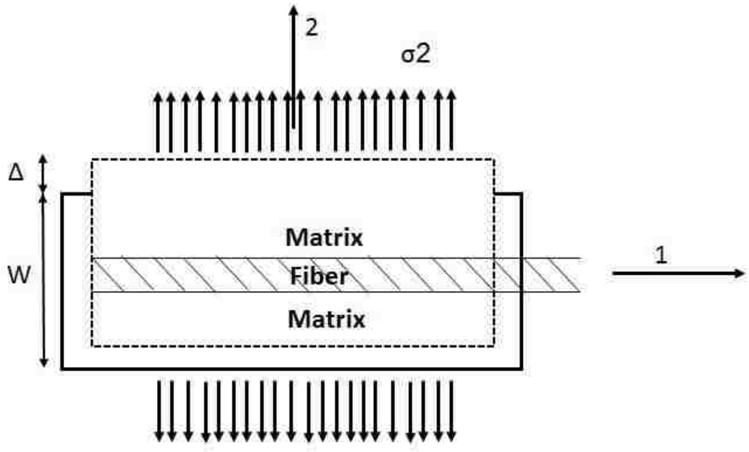


Figure 26. representative volume elemen in transverse direction

In transverse direction, it can be said that

$$\Delta_c = \Delta_m + \Delta_f \quad (4\text{-}9)$$

$$\Delta = w * \varepsilon \quad (4\text{-}10)$$

$$\varepsilon = V_f \varepsilon_f + V_m \varepsilon_m \quad (4\text{-}11)$$

Then

$$\mathrm{w} * \varepsilon = w(\varepsilon_m * V_m + \varepsilon_f * V_f) \tag{4-12}$$

From hook's law: $E * \varepsilon = \sigma$

Considering that $\sigma_c = \sigma_f = \sigma_m$

The equation 4-12 can be written as

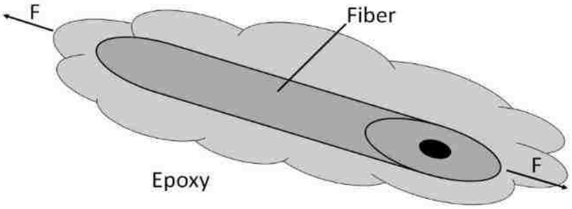
$$\frac{\sigma}{E} = (\frac{\sigma_m}{E_m} * V_m + \frac{\sigma_f}{E_f} * V_f) \tag{4-13}$$

$$\frac{1}{E} = \frac{1}{E_m} * \left(1 - V_f\right) + \frac{1}{E_f} * V_f \tag{4-14}$$

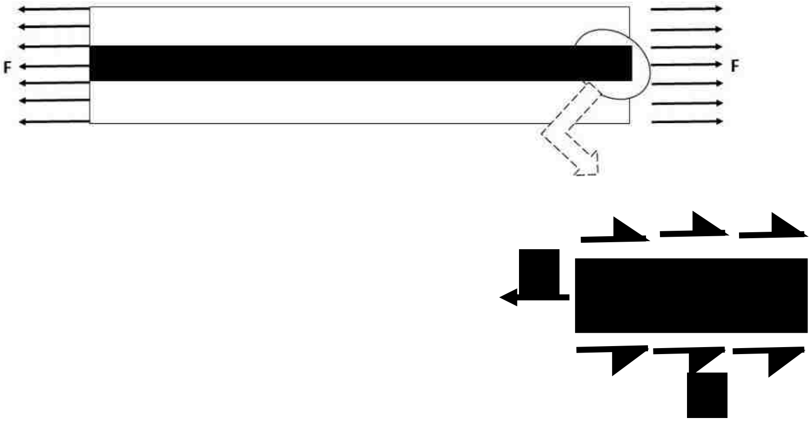
Since the fourth assumption does not apply to the composites made out of recycled carbon fiber, And the applied load makes an angle of between 0 to 90, the above equations needs to be modified to in order to be able to predict modulus and strength of recycled CFRP composites. The detailed modification and calculations is presented in section 4.3.

4.2.2. Shear Transfer

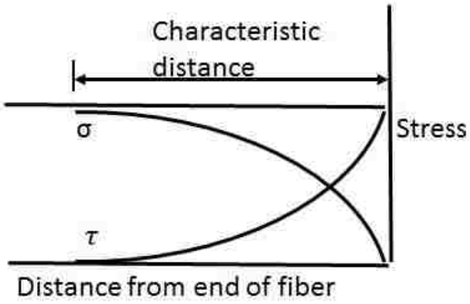
Figure 27 shows the force transfer mechanism into a fiber tow. The fiber tow is bonded with epoxy matrix. The epoxy matrix transfers the load to the fibers through shear stress as shown in Figure 27b. Because of the tension force F, a shear stress act on the outer surface of the fibers. This shear stress causes a tensile stress σ to develop in the fiber. At the end of the fiber the shear stress is high and tensile stress is low as shown in Figure 27c. As the distance from the end of the fiber increases the shear stress decreases and the tensile stress increases [3]



Fiber surrounded by matrix



b. section view



c. stress distribution

Figure 27. Load transfer to fiber: Tension

4.3. Analytical model

Several factors have been reported to affect the mechanical properties of short fiber reinforced polymer composites including fiber length, fiber orientation, fiber matrix adhesion, fiber concentration, and manufacturing technique[17]. In this study, a modified rule of mixture(MROM) is introduced that considers the fiber length and orientation in estimating the composite properties such as strength[18]. For example, equation 4-15 is often used to estimate the tensile strength of the short fiber reinforced polymer composites. Moreover, equation 4-16 is used to estimate the young's modulus of the short fiber reinforced polymer composites. σ_c is the composite strength. χ_1 and χ_2 are respectively the fiber orientation and the fiber length factors. V_f is fiber volume fraction and V_m is the matrix volume fraction? σ_f is the fiber strength and σ_m is the matrix strength in MPa. E_f and E_m are respectively the fiber and matrix modulus of elasticity.

$$\sigma_c = \chi_1 \chi_2 V_f \sigma_f + V_m \sigma_m \qquad 4\text{-}15$$

$$E_c = \chi_1 \chi_2 V_f E_f + V_m E_m \qquad 4\text{-}16$$

The fiber efficiency factors $\chi_1 \chi_2$ depend on the critical length of the fibers in this specific matrix which denotes with L_c. Critical length of the fibers is calculated using the equation 4-17 where r_f is the fiber radius in mm, σ_{cu} is the ultimate strength of the composite in MPa and τ_i is the interfacial shear stress between matrix and fibers in MPa.

$$L_c = r_f \frac{\sigma_{cu}}{\tau_i} \tag{4-17}$$

In the present research, two probability density functions are used for modeling the fiber length and orientation distribution respectively[17].

4.3.1. Fiber length distribution

Mechanical properties of short fiber composites are related to the fiber length distribution and can be calculated using equation 4-18 [19]. a and b are scale and shape parameters.

$$\mathrm{F}(L) = abL^{b-1}e^{-aL^b} \tag{4-18}$$

The average length of the fibers can be calculated using the equation 4-19 where Γ(x) is the gamma function.

$$L_{mean=} \int_0^\infty Lf(L)dL = \ a^{-\frac{1}{b}}\Gamma(\frac{1}{b}+1) \tag{4-19}$$

The most probable length, L_{mod}, can be calculated using the equation 4-20.

$$L_{mod} = (\frac{1}{a} - \frac{1}{ab})^{\frac{1}{b}} \tag{4-20}$$

4.3.2. Fiber orientation distribution

The angle between fiber axis and the loading direction contributes to the composite strength. A fiber orientation distribution function representing the inclination angle must have the property such that the variation of its function's shape parameters can describe a change from a unidirectional distribution to random distribution. FU et al.[20] proposed a two-parameter exponential function to describe the fiber orientation distribution function shown in 4-21. where p and q are the shape parameters which can be used to determine the shape of the distribution curve.

$$g(\theta) = \frac{(\sin\theta)^{2p-1}\,(\cos\theta)^{2q-1}}{\int_{\theta_{min}}^{\theta_{max}}\{(sin\theta^{2p-1})(\cos\theta)^{2q-1}\}\,d\theta} \tag{4-21}$$

Differentiating equation 4.21 and equating it to zero results in the most probable fiber orientation angle, θ mod shown in equation 4-22.

$$\theta_{mod} = \tan^{-1}\sqrt{\frac{2p-1}{2q-1}} \tag{4-22}$$

Moreover, θ mean can be derived from equation4-23.

$$\theta_{mean=}\int_{\theta_{min}}^{\theta_{max}}\theta g(\theta)d\theta \tag{4-23}$$

The fiber orientation coefficient, f(θ), can be obtained using the equation 4-24

$$f_\theta = 2\int_{\theta_{min}}^{\theta_{max}} g(\theta)(\cos\theta)^2 d\theta - 1 \tag{4-24}$$

Finally, θ_{max} is obtained using the equation4-25.

$$\theta_{max} = \tan^{-1}(\frac{1}{A}) \tag{4-25}$$

4.3.3. Strength of short fiber reinforced composite

The strength of the composite is contributed by all the fibers with different length with varying orientation. Composite strength will be evaluated using equation 4-15 in which $\chi_1\chi_2$is obtained from equation

$$\chi_1\chi_2 = \int_{\theta_{min}}^{\theta_{max}} \int_{L_{min}}^{L_{c_0}} f(\mathrm{L}) g(\theta) (\frac{L}{L_{mean}}) \left(\frac{L}{2L_c}\right) e^{\mu\theta} dL d\theta + \int_{\theta_{min}}^{\theta_{max}} \int_{L_{c_o}}^{L_{max}} f(L) g(\theta) \frac{L}{L_{min}} (1 - A\tan\theta)(1 - L_c \left(\frac{1 - A\tan\theta}{2Le^{\mu\theta}}\right)) dL d\theta \qquad 4\text{-}26$$

Chapter 5. Results and discussion

5.1. Introduction

In this chapter, tension, modulus of elasticity, strain, and volume fraction of recycled carbon fiber composites are investigated. In total, six different categories of fabrication were investigated. Two different recycled carbon fibers were available for this research. Fibers which were reclaimed from overaged prepregs, and fibers which were reclaimed from composites that they originated from T-800. Moreover, different mediums were used to help aligning the fibers. The fibers were either dry or wet. And the wet fibers were either soaked in glycerin or in water. The manufacturing of recycled carbon fiber composites was performed using vacuum assisted hand lay-up, vacuum assisted resin transfer mold(VARTM), or resin transfer mold(RTM). In addition, the shape of the coupons was investigated. The coupons were cut either rectangular or dog bone shape. Finally, two approaches of sizing are studied. Sizing by silane (3 – Glycidloxyopropyl) trimethoxysilane at the structural lab, and sizing by epoxy at Adherent company.

5.1.1. Stress-Strain

The first samples that has been looked are rCLD1 and rCLD6: the effect of carding machine on the tensile behavior of cFRP is examined in Figure 28. This graph represents a comparison between stress – strain curve for cFRP's fabricated using carding machine and without a carding machine. Six samples of each specimen were tested. rCLD6 is the coupon made using the carding machine. The average ultimate strength of this specimen was 53 MPa. The rCLD1 cFRP was fabricated with no help from carding machine. As shown in Figure 28, the average ultimate strength of these coupons was 27 MPa. The results show an increase of 96.7 % in strength due to carding machine. The reason lies behind fiber alignment using the carding machine. When the fibers are completely parallel to each other, most of the force that is being applied to the cFRP, transfers to the fibers which are stronger than epoxy as shown in Figure 29 a. Whereas, when the fibers are not completely aligned some of the force transfers to epoxy as shear force and causes a failure in cFRP as shown in Figure 29 b. The Modulus of elasticity of the FRP's were looked at also. Young's modulus of elasticity or stiffness is a fundamental property of every material. It is a very important property because it shows when a material will deform which is important for design criteria. The stiffness of rCLD1 is higher than rCLD6 making it stiffer and more brittle also. It means that the ability of the coupons made with carding machine in transferring the load and bending is higher than rCLD6.

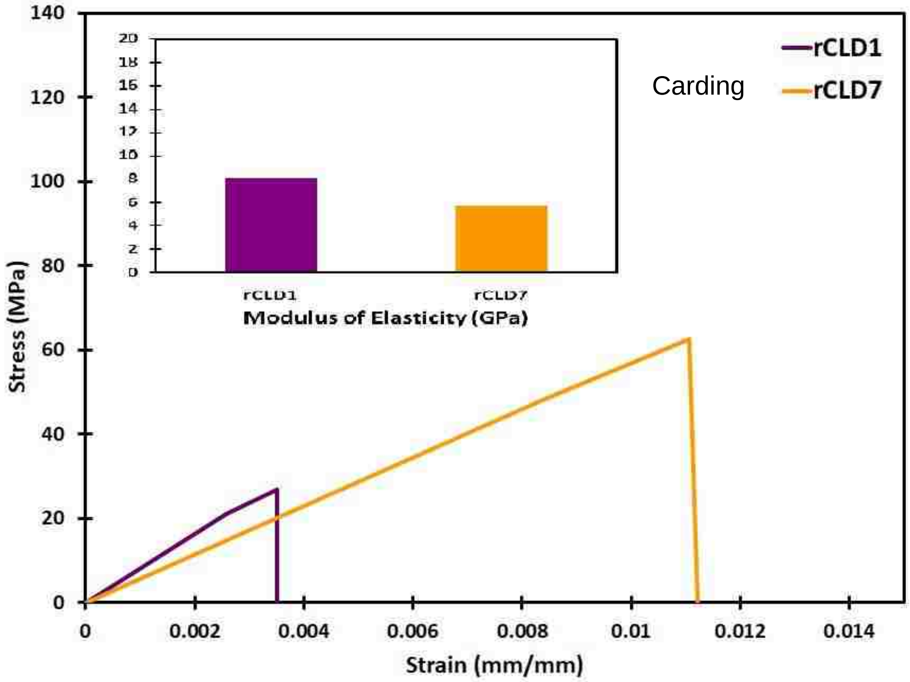


Figure 28. comparison between carding machine and no carding machine

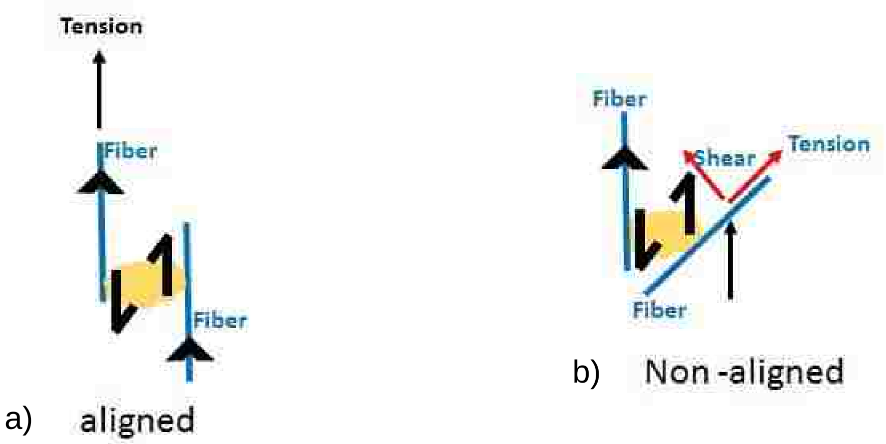


Figure 29. Force transfer Mechanism : a) aligned b) non- aligned

The effect of Vacuum Assisted Resin Transfer Molding (VARTM) and Vacuum Assisted Hand Layup and Resin Transfer Molding (RTM) is illustrated in Figure 30. As explained in section 3.4.2.3. VARTM is a closed mold composite manufacturing process. VARTM is a variation of RTM with a difference in resin flow. The resin is dispersed using a vacuum system. The strength of the cFRP manufactured using RTM is 98.6 MPa which is shown as rCLD12. The strength of the cFRP manufactured using VRTM is 52.7 MPa. The result of this experiment shows that RTM yields stiffer composites than VARTM. The results illustrate that RTM samples are 3 times stiffer making them more resistant to bending and deformation.

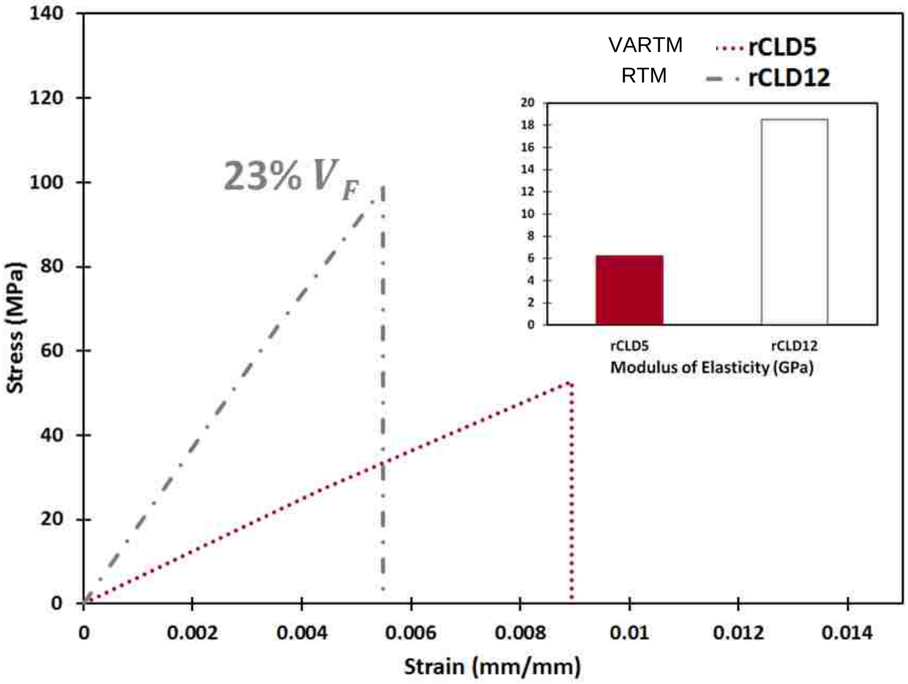


Figure 30. Comparison between VARTM and RTM

Figure 31 represents results for specimens soaked in glycerin and water before making a mat. As mentioned in 3.4.2.2, some composites were made by soaking them in the glycerin subsequently stirring it with a mechanical stirrer to separate the fibers as much as possible. By doing so, as results show, the strength of the composites has decreased. The reasons lie beneath the idea that the glycerin may have not totally been washed and prevented bonding between epoxy and the fibers. The obtained result for the composite's strength fabricated using water soaked fibers is 98.6 MPa and for glycerin soaked fibers is 62.7 MPa. The percent decrease in strength was observed to be 57.3 % which is considerable. According to the results of the stiffness of glycerin soaked and water soaked fibers, water soaked specimens are stiffer than glycerin soaked.

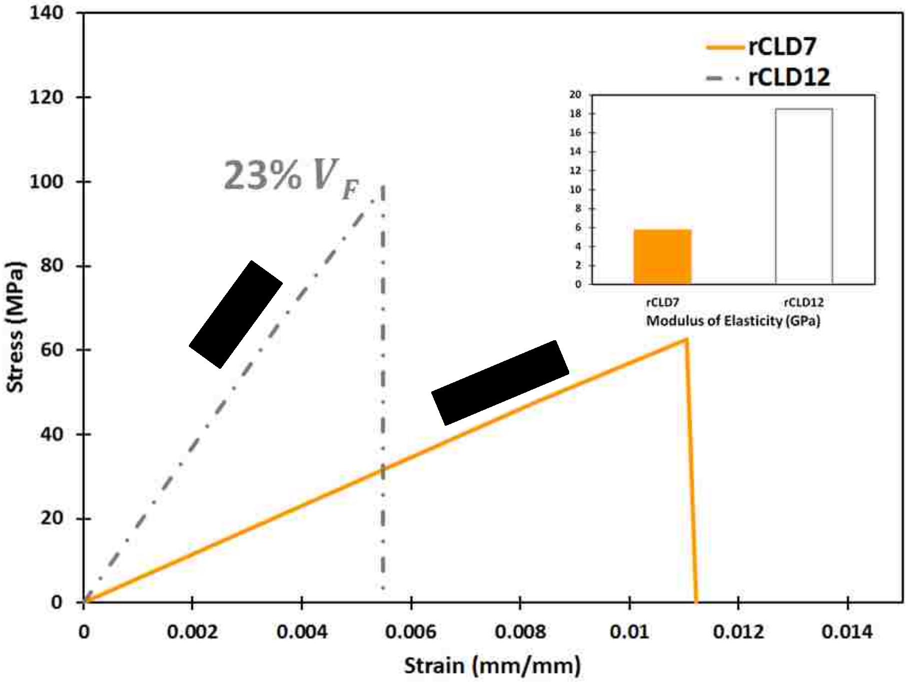


Figure 31. Comparison between cFRP's fabricated using Glycerin or water

Design of mechanical test coupons, especially those using tabs, remains an art rather than science, with no industry consensus on how to approach the engineering of the gripping interface.[21] A specimen is tabbed prior to testing for a couple of reasons. To increase the area of the loading region, and reduce local stress concentration, the material should be protected from the applied load. Different geometries along with multiple gripping were investigated. Specimen rCLD9 is the only sample with tapered glass laminate sheets which is shown to be an excellent tabbing material. The majority of samples were prepared for testing using carbon fiber reinforced composites. The geometry of the rCLD9 differs from the rCLD7. The rCLD7 is rectangular coupon with un- tapered tabs. The rCLD9 with strength of 75.7 MPa has higher strength among other samples with similar constituents which confirms the better load transfer with tapered tabs.

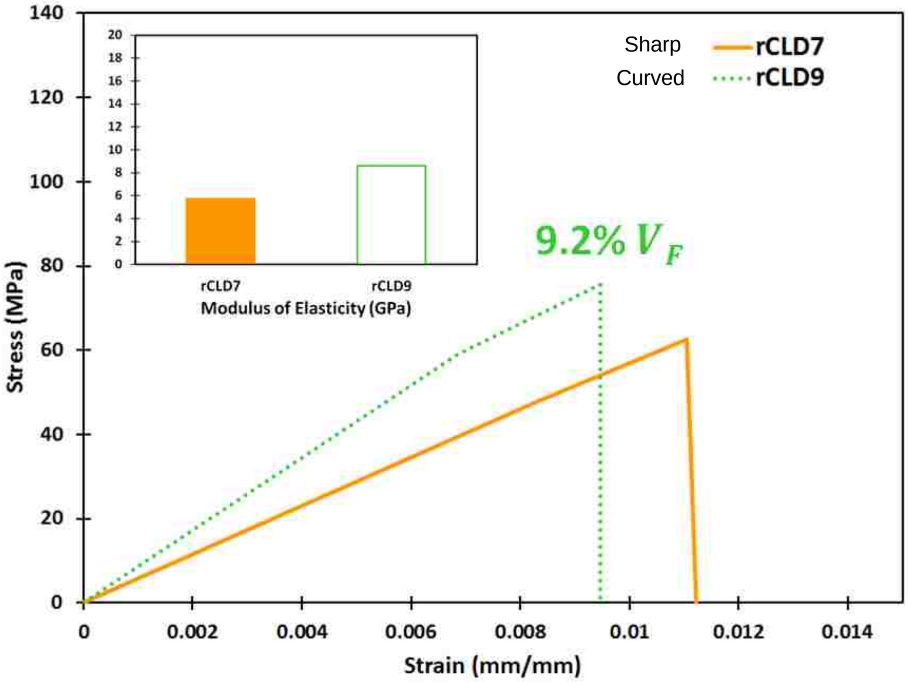


Figure 32.Comparison between different gripping and different geometries

Figure 34. illustrates a comparison between rCLD16 and rCLD17 which are fabricated using epoxy and MWCNTs consecutively. In addition, these recycled fibers are sized with epoxy by the Adherent Company. The Composite with incorporated MWCNTs indicates a strength of approximately 124 MPa and the composite fabricated using epoxy denotes a strength of 106 MPa. Sizing of the fibers improves the bonding between MWCNTs and the fibers and thus leads to a higher strength. The outcome of this experiment proves that nano tubes decrease the stiffness of the material.

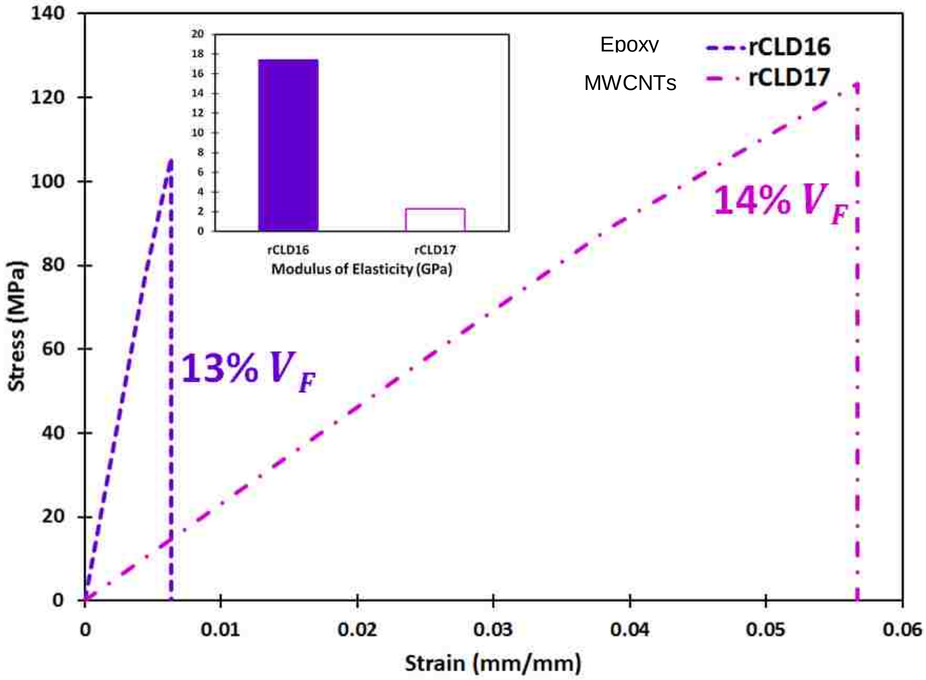


Figure 33.Comparison between Epoxy and MWCNTs

The effect of sizing on recycled carbon fiber is shown in Figure 34. Specimen rCLD15 with strength of 125.7 MPa has a higher strength than those of un-sized. Sizing the fibers improves the mechanical property of composites.

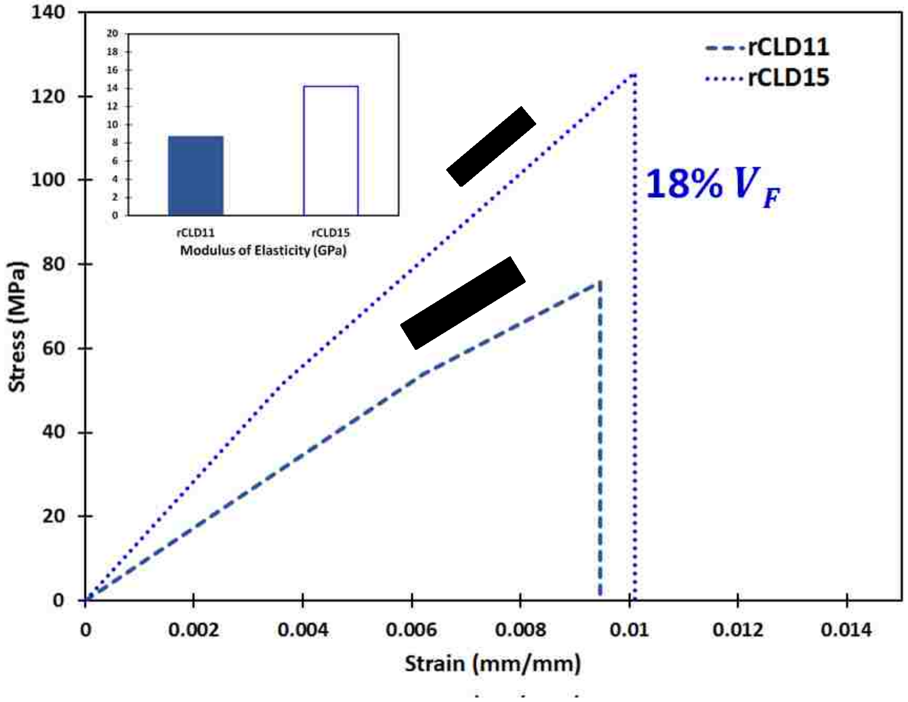


Figure 34. Comparison between composites which are sized and un-sized

Figure 35 illustrates a comparison between sized fibers with MWCNTs and un - sized fibers with MWCNTs. The specimen rCLD 18 has a strength of 106 MPa and rCLD8 has a strength of 59 MPa consecutively. Sizing improves the surface characterization of the fibers, and as a result the bonding between matrix and reinforcement increases. The result for these samples showed that the sizing increases the elasticity of the material.

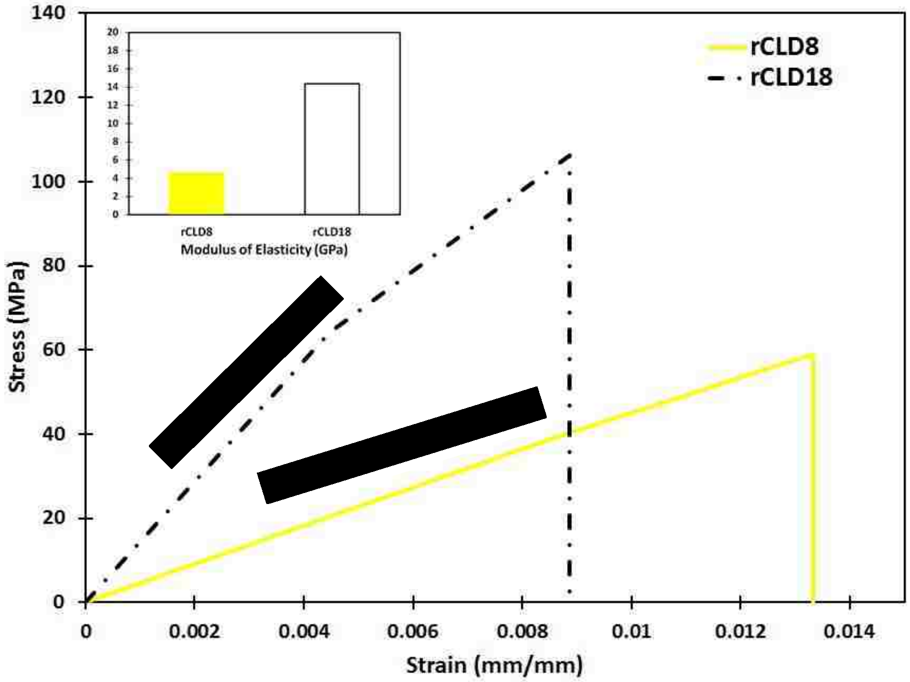


Figure 35. Comparison between sized with MWCNTs and Unsized with MWCNTs

Some of the composites were fabricated using Resin Transfer Molding system and some were fabricated using Vacuum Assisted Hand Layup Molding(VAHLM). Below the comparison between these two methods of fabrication is shown. The rCLD12 is fabricated with VAHLM and the rCLD14 is fabricated with RTM machine. The expectation was that the RTM delivers stronger Composites. However, because of shear failure during the testing it was not possible to know the exact strength. To overcome the shear failure, the high stress concentration area was changed gradually to get the best result. In first stage, any sharp corners were smoothed and curved and the second stage the stem of FRP was narrowed to increase the chance of a tension failure. Figure 36 shows a schematic of decreasing the high stress concentration area.

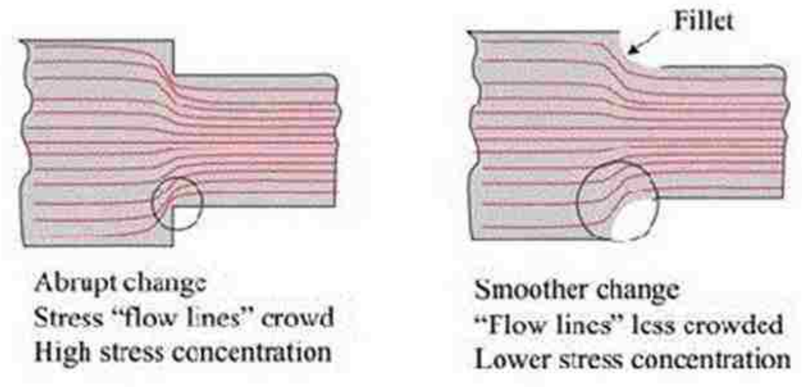


Figure 36.Stress Concentration in FRP(https://www.corrosionpedia.com)

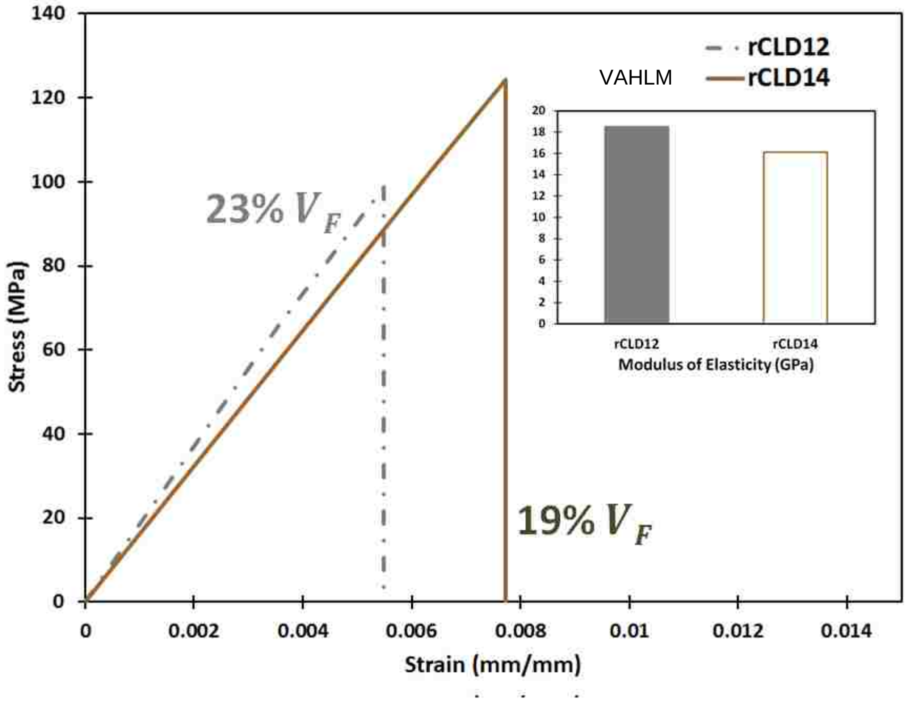


Figure 37. Comparison between Composites made with RTM and VAHLM

5.1.2. Strength

According to test results, which is shown in Table 5 - 1, rCLD15 with 125.67 MPa has the highest strength. This specimen is the mat which is sized by Silane. The rCFRP's with names of 14, 16, 17, and 18 which have higher strengths are sized using either epoxy, or silane. This proves that sizing increases the bond strength between epoxy and fibers.

5-1 Strength at failure

Specimen	Strength at failure (Mpa)
rcld1	26.8
rcld2	62.0
rcld3	45.5
rcld4	76.0
rcld5	52.7
rcld6	52.7
rcld7	62.7
rcld8	59.0
rcld9	75.7
rcld10	59.0
rcld11	75.7
rcld12	98.6
rcld13	88.7
rcld14	124.2
rcld15	125.7
rcld16	105.8
rcld17	123.3
rcld18	106.2
rcld20	96.0

5.1.2.1. Statistical Analysis of tensile strength

Figure 38 shows the result of a Mean Comparison test using R Core Team (2017) which is a language and environment for statistical computing. A Means Comparison test performs multi sample t-tests to determine which sample means are different from each other and reports the value of each samples mean (in the case the fiber's tensile strength). The Tukey's range test method is used to determine which samples are similar and or significantly different. As shown, in the order letter report, each sample is assigned one or multiple letters. Samples that share a letter (s) are not significantly different. rCLD3, rCLD8, rCLD5 and rCLD6 are not significantly different. Moreover, rCLD13, rCLD14, rCLD15, and rCLD20 are not significantly different either. rCLD1 is significantly weaker than all other samples.

Overall Test (F Test)

Table 5-2 F Test result

	Df	**Sum Sq**	**Mean Sq**	**F value**	**Pr(>F)**
factor(sample)	18	41809	2322.7	10.55	3.93E-1
Residuals	48	10572	220.2		

Signif. codes: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1

The F-test shows if two population variances are equal by comparing the ratio of two variances. So, if the ratio of the variances is 1, the variances are equal. If the P – value is small, the null hypothesis can be rejected and one can reject the null hypothesis. For this specific test the P-value is not small, so the null hypothesis can not be rejected meaning that this model does not provide a better fit.

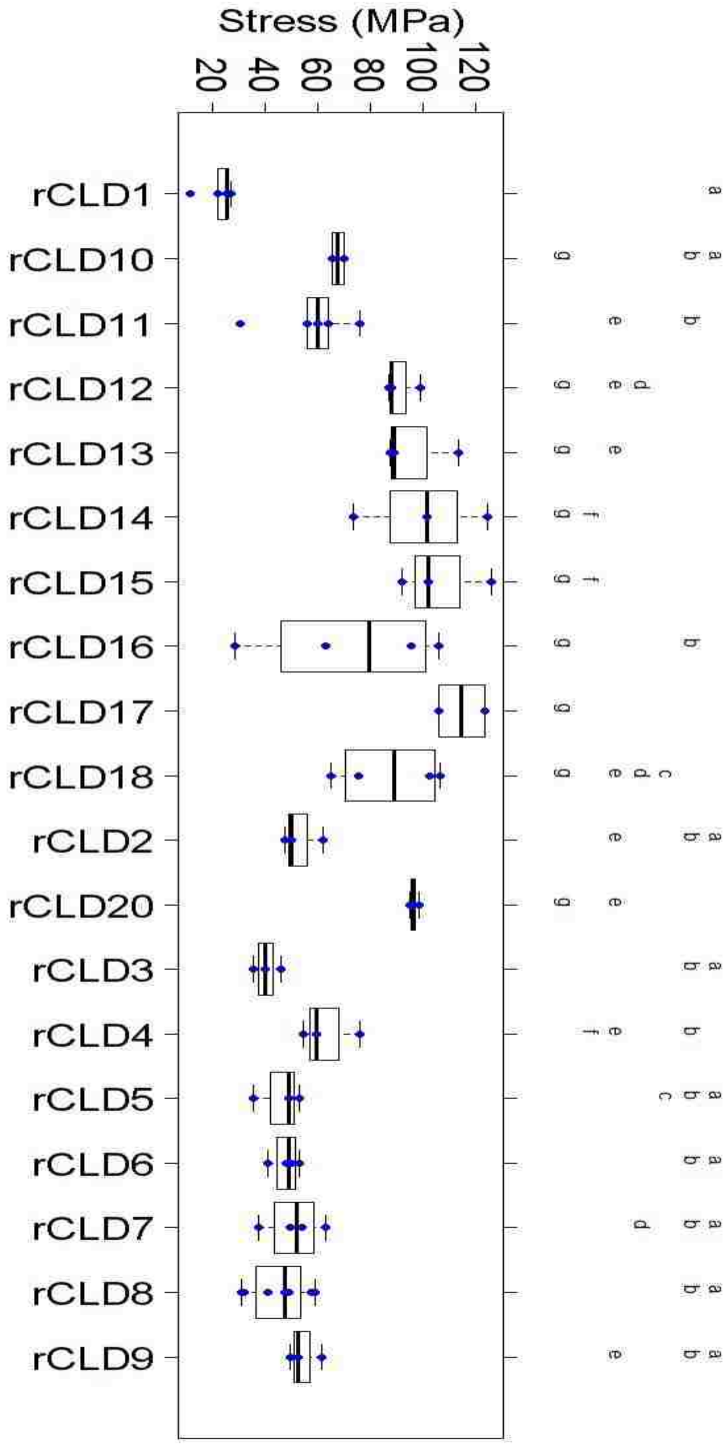


Figure 38.Box plot Analysis of tensile strength

Table 5-3. Ordered letter report using Tukey test

sample	lsmean	SE	df	lower.CL	upper.CL	.group
rCLD1	22.1	6.6	48	8.8	35.4	a
rCLD3	40.3	8.5	48	23.0	57.4	ab
rCLD8	45.2	5.6	48	33.9	56.4	ab
rCLD5	45.6	8.5	48	28.3	62.8	abc
rCLD6	47.8	7.4	48	32.8	62.6	ab
rCLD7	50.9	7.4	48	35.9	65.8	abcd
rCLD2	53.0	8.5	48	35.8	70.2	abcde
rCLD9	54.4	8.5	48	37.1	71.6	abcde
rCLD11	57.1	6.6	48	43.7	70.4	bcde
rCLD4	63.3	8.5	48	46.0	80.5	bcdef
rCLD10	67.4	10.4	48	46.2	88.4	abcdefg
rCLD16	73.1	7.4	48	58.2	88.0	bcdefg
rCLD18	87.1	7.4	48	72.2	102.0	cdefg
rCLD12	91.2	8.5	48	73.9	108.4	defg
rCLD20	96.2	8.5	48	79.0	113.4	efg
rCLD13	96.4	8.5	48	79.2	113.6	efg
rCLD14	99.6	8.5	48	82.2	116.7	fg
rCLD15	106.5	8.5	48	89.2	123.7	fg
rCLD17	114.5	10.4	48	93.4	135.6	g

5.1.3. Tensile Strain at failure

The table 5-1, illustrates the strain at failure of the tested coupons. Samples made with MWCNT's show higher displacement than the ones made with Epoxy.

Table 5-4 strain at failure

Specimen	Strain at Failure
rCLD1	0.35%
rCLD2	1.02%
rCLD3	0.63%
rCLD4	0.65%
rCLD5	0.90%
rCLD6	0.93%
rCLD7	1.12%
rCLD8	1.33%
rCLD9	0.95%
rCLD10	1.33%
rCLD11	0.95%
rCLD12	0.55%
rCLD13	0.55%
rCLD14	0.77%
rCLD15	1.01%
rCLD16	0.64%
rCLD17	5.67%
rCLD18	0.89%
rCLD20	0.58%

5.1.4. Modulus of elasticity GPa

Table 5-5 Modulus of Elastisity

Specimen	Modulus of Elasticity GPa
rCLD1	8.17
rCLD2	6.16
rCLD3	7.55
rCLD4	11.86
rCLD5	6.21
rCLD6	5.78
rCLD7	5.74
rCLD8	4.56
rCLD9	8.6
rCLD10	4.49
rCLD11	8.68
rCLD12	18.52
rCLD13	7.55
rCLD14	16.12
rCLD15	14.23
rCLD16	17.38
rCLD17	2.31
rCLD18	14.35
rCLD20	20.82

Overall Test (F Test) for modulus of elasticity

	Df	Sum Sq	Mean Sq	F value	Pr(>F)
factor(sample)	16	1797.9	112.37	14.04	***
Residuals	42	336.3	8.01		

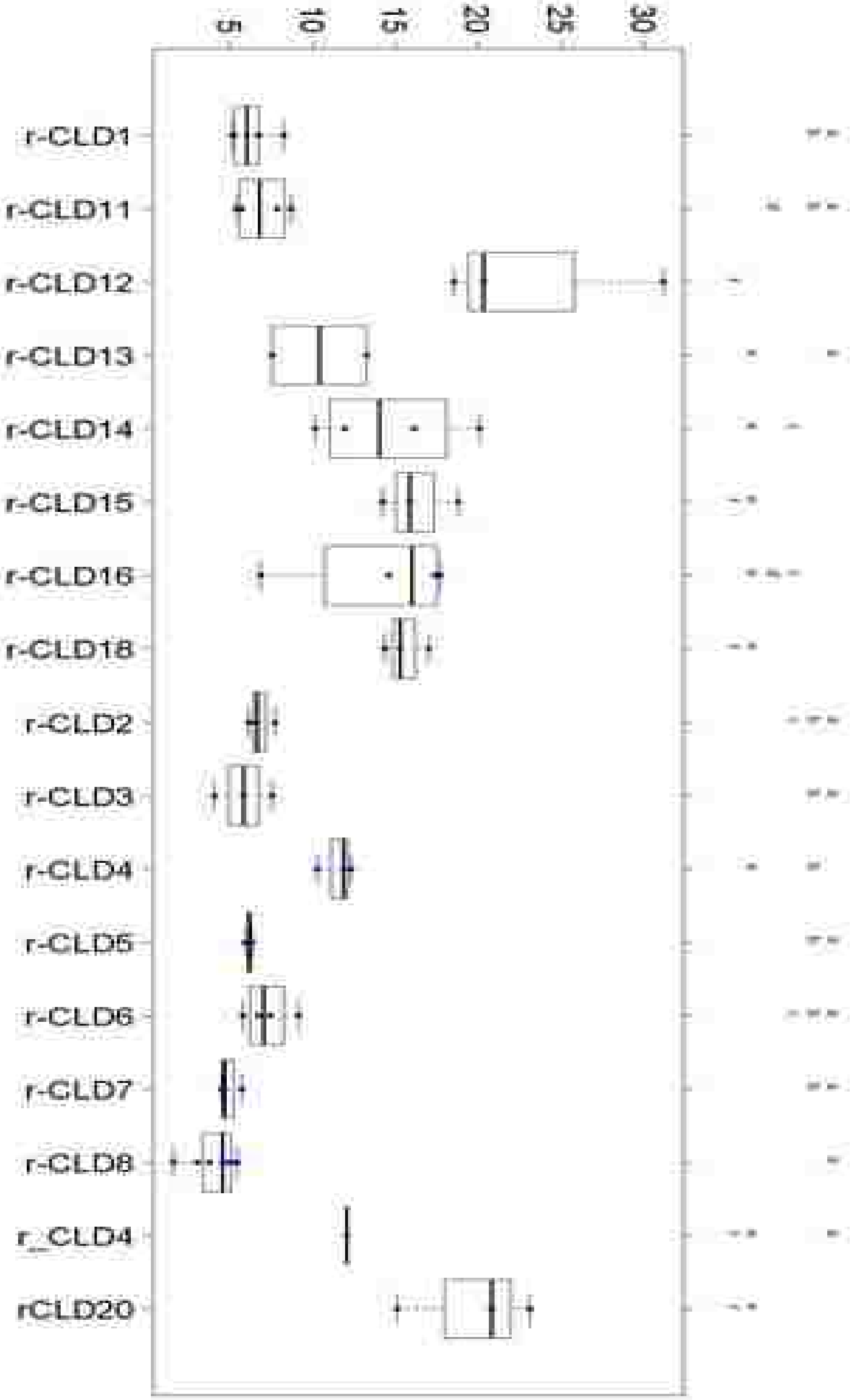


Figure 39 – box plot analysis of elasticity

5.2. Analytical Result

5.2.1. Code verification

The equations developed by Shao -Yun Fu is used for analytical model and is verified by MATLAB coding. The Table 5-6b shows the result from the original paper and the table 5-6a shows the result from the MATLAB code. Using equations 4-22, 4-23, 4-24, and 4-26,θ_{mean}, θ_{mod},f_{θ}, and$\chi_1\chi_2$ have been calculated. Since the two tables are nearly identical, this method is used to predict the recycled carbon fiber behavior.

Table 5-6 a

NO.	P	Q	θ_{mean}	θ_{mod}	f_{θ}	$\chi_1\chi_2$
1	0.5	1	0.5708	0.0000	0.3233	0.5523
2	1	2	0.5890	0.5236	0.3298	0.5729
3	2	4	0.6013	0.5796	0.3330	0.5877
4	4	8	0.6082	0.5993	0.3333	0.5949
5	0.5	0.5	0.7854	NaN	-0.027	0.4166
6	1	1	0.7854	0.7854	-.019	0.4108
7	2	2	0.7854	0.7854	-.009	0.4158
8	4	4	0.7854	0.7854	-.001	0.4172
9	8	4	0.9626	0.9715	-.3447	0.3517
10	4	2	0.9695	0.9912	-.3586	0.3298
11	2	1	0.9817	1.0472	-0.368	0.3086
12	1	1	1.0000	1.5708	-0.368	0.2903

Table 5-6 . b The effect of fiber orientation on the TS of FRP[20]

NO.	**P**	**Q**	θ_{mean}	θ_{mod}	f_θ	$\chi_1\chi_2$
1	0.5	1	0.571	0.0	0.33	0.549
2	1	2	0.589	0.524	0.33	0.569
3	2	4	0.601	0.580	0.33	0.588
4	4	8	0.608	0.599	0.33	0.595
5	0.5	0.5	0.785	NaN	0	0.417
6	1	1	0.785	0.785	0	0.416
7	2	2	0.785	0.785	0	0.415
8	4	4	0.785	0.785	0	0.414
9	8	4	0.962	0.972	-.33	0.351
10	4	2	0.970	0.991	-.33	0.330
11	2	1	0.982	1.047	-0.33	0.308
12	1	1	0.999	1.57	-0.33	0.290

5.2.2. Result

The objective of developing an analytic model is to predict the experimental results. The analytical model is thus verified using the experimental observations. As shown earlier the strength of the composite can be calculated using the Equation 4-15 and 4-26 knowing the fiber length distribution, fiber orientation distribution, the critical fiber length, and the strength of the single fibers. The table below provides the parameters for one of the composites:

Table 5-7. Calculated parameters for rCLD17

Θ_{MIN}	**0 °**	**A**	**0.017**
Θ_{MAX}	90°	b	1.2
Σ_{MATRIX}	68 Pa	L_{min}	0 mm
Σ_{FIBER}	3800 MPa	L_{max}	41.8 mm
VOL_FR_{MATRIX}	60%	A	0.4
VOL_FR_{FIBER}	18%	L_c	8.9 mm
P	0.5	μ	0.1
Q	2		

All the parameters in the Table 5-7 are calculated for the recycled carbon fiber except the constant A which is assumed to be 0.4 and the friction factor is also assumed to be 0.1. Using equations 4-15, 4-26 and the table 4-4, the composite strength can be predicted as 191.3 MPa which is 53% higher than the experimentally observed strength of 125 MPa. This difference can be attributed to the fact that in the analytical model parameters are based on assumed distribution of fiber orientation that most probably is not met experimentally. For example, 100 strands were picked and the length and the angle were measured after taking pictures of the mat before injecting epoxy. The resulted graph resembles the distribution “d” with p=0.5 and q =1.2 as shown on figure 40. The pictures were taken using a 60x camera. Improving picture resolutions to get more accurate measurements to that observed experimentally.

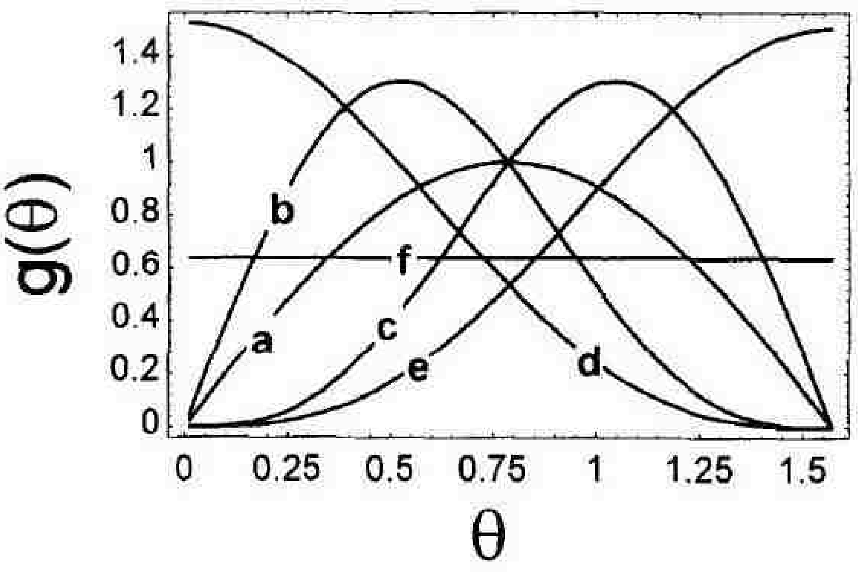


Figure 40.Fiber orientation curves for : (a) p=q=1 ; (b) p=1, q=2 ; (c) p=2, q=1; (d) p=0.5, q=2; (e) p=2,q=1/2;(f) p=q=1/2 [20]

The effect of fiber aspect ratio (l/d) on the strength of the composite has also been studied in rCFRP using the analytical model. short fiber reinforced polymer. The results show that the strength of the composite decreases with the increase in the aspect ratio of the fibers. Around the critical aspect ratio (i.e. critical fiber length L_c), the strength significantly increases with the increase in length as illustrated in figure 41. Moreover, the effect of fiber volume fraction on composite strength has been investigated. As shown in Figure 42, with the increase in the fiber volume fraction the strength increases in the examined recycled carbon fiber composites. The black solid line in Figure 42 illustrates the predicted model and the dotted line shows the results for one of the tested coupons

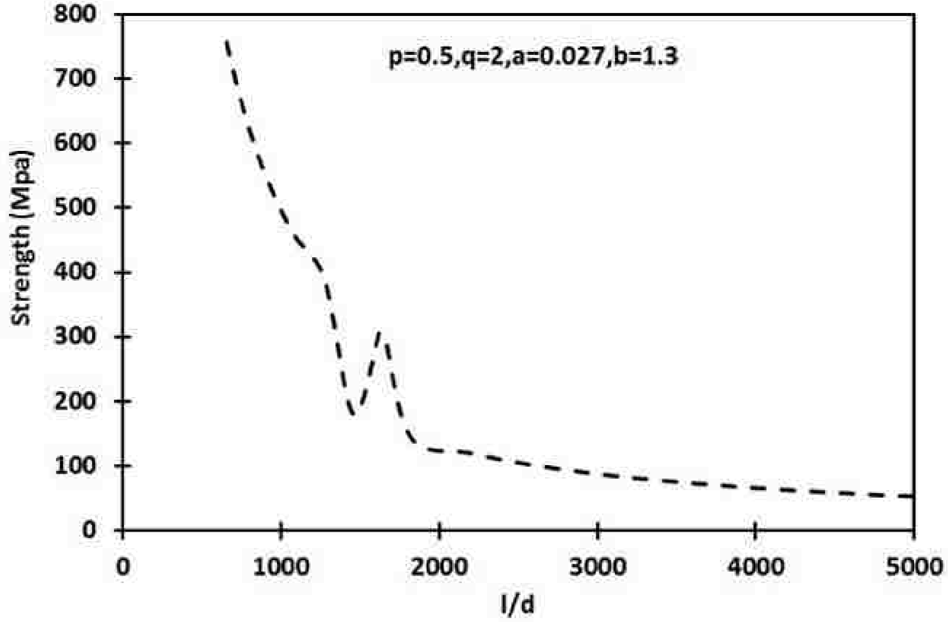


Figure 41. Effect of aspect ratio (l/d) on the strength of the composite

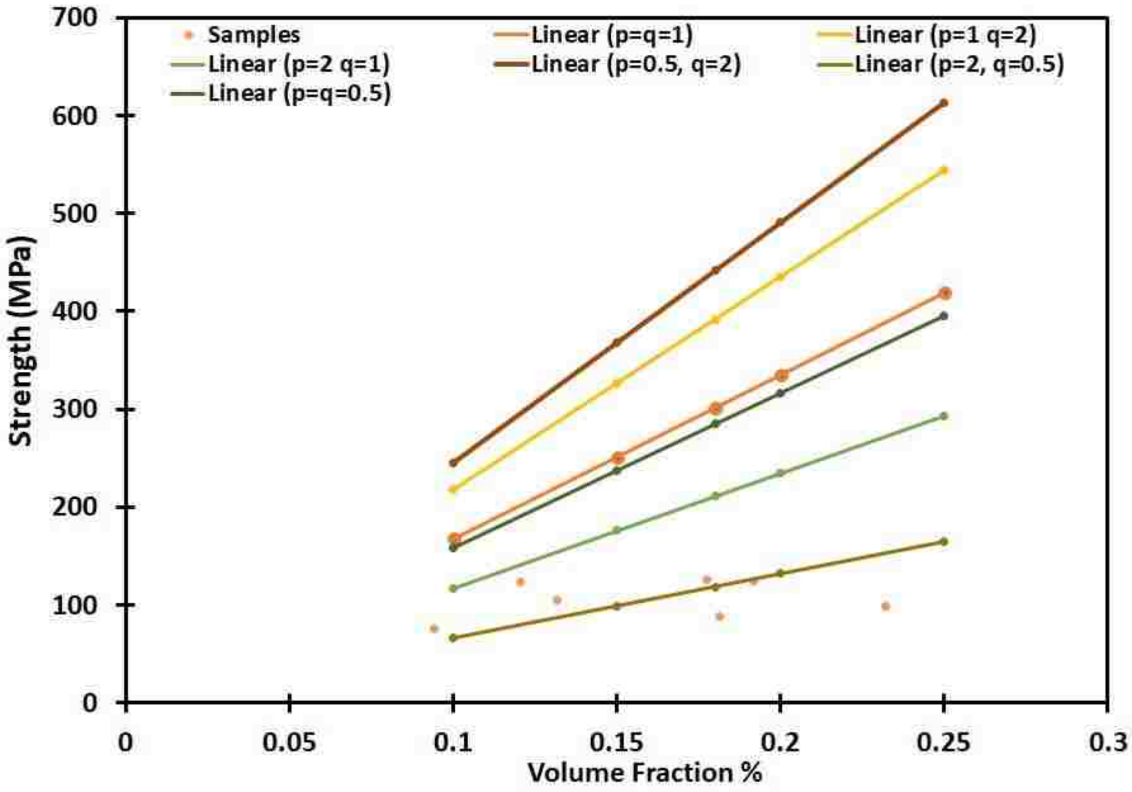


Figure 42.Effect of fiber volume fraction on the strength of the composite

Chapter 6. Conclusion and recommendation

- From the experimental results, it can be concluded that fiber sizing improves the bond between fibers and epoxy. The type of material used for sizing also affects the bond. It can be concluded that Silane bonds and cures better than epoxy bisphenol with recycled carbon fiber. Aligning the fibers can improve the strength of the composite also. The reason is that by aligning the fibers the fiber volume fraction increases leading to composite strength and stiffness.

- MWCNTs are proven to improve the mechanical properties of the FRP; however, it did not change the mechanical properties of the recycled carbon fiber composites.
- It has been shown that it is possible to produce rCFRP composites with approachable strength and stiffness with relatively high fiber volume fraction (about 25%)
- RTM yields a more unique composite because of the type of injection. Moreover, soaking the fibers in glycerin to separate them, blocks recycled carbon fiber from bonding with epoxy.
- Dog-bone shaped coupons prevent shear failure by omitting high stress concentration area and are necessary
- The strength of the composite increases when the mean fiber length decreases and reaches the critical length the composite.

6.1. Future Work and recommendations

Future work shall include in depth investigation of sizing of the recycled carbon fibers with Silane. Fourier Transform Infrared Spectroscopy (FTIR) should be used to confirm the bond existence between silane and recycled carbon fiber. In addition, more research needs to be done on the length of the efficiency of fiber length on composite strength. In the end, a finite element modeling study needs to be done on analytical solution.

Appendix

Matlab coding to calculate X1X2 and the strength of the composite

```
clc
clear
close all
teta_min = 0;
teta_max = pi/2;

p = 0.5
q = 0.5
a = 3.390; b = 1.5;
L_min = eps; %L_Inf =10e-3;
mue = 0.1;
Lc = 0.2;
L_mean = 0.4;
A = 0.4;
L_c0 = 0.1;
L_max = 10;
L_mod = 0.213;
teta_max = atan(1/A);
L_mean = (a^(-1/b))*gamma(1/b + 1);
L_mod = (1/a - 1/(a*b))^(1/b);
%%
denum = @(teta)((sin(teta)).^(2.*p - 1)
.*(cos(teta)).^(2.*q -1));
denum_integ = integral (denum ,teta_min,pi/2);
g_fun = @(teta)((sin(teta)).^(2.*p - 1)
.*(cos(teta)).^(2.*q -1)./denum_integ);
%%
teta_fun = @(teta)(teta.*g_fun(teta));
teta_mean = integral(teta_fun,teta_min,pi/2)
teta_mod = atan(sqrt((2*p - 1)./(2*q - 1)))

% X1X2 = @(L,teta)
%%
f_teta = @(teta) (2.*g_fun(teta).*...
                   (cos(teta).^2));
f_teta_integ = integral(f_teta,teta_min,teta_max);
f_teta_integ = f_teta_integ - 1
```

```
%%
f_L = @(L) (a.*b.*(L.^(b - 1).*exp(-a.*L.^b)));
part1 = @(teta,L) (f_L(L).*...
                        g_fun(teta).*...
                        (L./L_mean).*...
                        (L./(2.*Lc)).*...
                        exp(mue.*teta));
part1_Integ =
integral2(part1,teta_min,teta_max,L_min,L_c0);


part2 = @(teta,L) (f_L(L).*...
                        g_fun(teta).*...
                        (L./L_mean).*...
                        (1 - A.* tan(teta)).*...
                        (1 - Lc.*((1 -
A.*tan(teta))./(2.*L.*exp(mue.*teta))))...
                        );
part2_Integ =
integral2(part2,teta_min,teta_max,L_c0,L_max);
X1X2 = part1_Integ + part2_Integ
%%
X2_part1 = @(L) (((L.^2)./(2.*Lc.*L_mean)).*f_L(L));
X2_part1_integ = integral(X2_part1,L_min,Lc);
X2_part2 = @(L) ((L./L_mean).*(1 -
Lc./(2.*L)).*f_L(L));
X2_part2_integ = integral(X2_part2,Lc,L_max);
X2 = X2_part1_integ + X2_part2_integ;

%%
volf=0.25
sigmaf=3800;
vole = 0.60
sigmae=68e-5;
Sigma = X1X2 * volf * sigmaf + vole * sigmae





function [F] = solve_mojgan(x)
        F(1) = (x(1)^(-1/x(2)))*gamma(1/x(2) + 1) -
0.4;
```

```
        F(2) = (1/x(1) - 1/(x(1)*x(2)))^(1/x(2)) -
0.213;
end




function fig7()
clc
clear all
close all
teta_min = 0;
teta_max = pi/2;

p = 2; q = 1;
% a = 3.390; b = 1.5;
L_min = eps; %L_Inf =10e-3;
mue = 0.1;
Lc_mat = [eps:0.01:0.6];
L_mean = 0.4;
A = 0.4;
L_c0 = 0.1;
L_max = 10;
L_mod = 0.213;
teta_max = atan(1/A);
%L_mean = (a^(-1/b))*gamma(1/b + 1);
%L_mod = (1/a - 1/(a*b))^(1/b);
teta_max = atan(1/A);
[a,b] = calc(1);
for ii = 1:length(Lc_mat)
    Lc = Lc_mat(ii);

    denum = @(teta)((sin(teta)).^(2.*p - 1)
.*(cos(teta)).^(2.*q -1));
    denum_integ = integral (denum ,teta_min,pi/2);
    g_fun = @(teta)((sin(teta)).^(2.*p - 1)
.*(cos(teta)).^(2.*q -1)/denum_integ);
    % X1X2 = @(L,teta)
    %%
    f_teta = @(teta)  (2.*g_fun(teta).*...
        (cos(teta).^2));
    f_teta_integ = integral(f_teta,teta_min,teta_max);
    f_teta_integ = f_teta_integ - 1;
```

```
    %%
    f_L = @(L) (a.*b.*((L.^(b - 1)).*exp(-a.*(L.^b))));
    part1 = @(teta,L) (f_L(L).*...
        g_fun(teta).*...
        (L./L_mean).*...
        (L./(2.*Lc)).*...
        exp(mue.*teta));
    part1_Integ =
integral2(part1,teta_min,teta_max,L_min,L_c0);


    part2 = @(teta,L) (f_L(L).*...
        g_fun(teta).*...
        (L./L_mean).*...
        (1 - A.* tan(teta)).*...
        (1 - Lc.*((1 -
A.*tan(teta))./(2.*L.*exp(mue.*teta))))...
        );
    part2_Integ =
integral2(part2,teta_min,teta_max,L_c0,L_max);
    X1X2(ii) = part1_Integ + part2_Integ;

    X2_part1 = @(L)
(((L.^2)./(2.*Lc.*L_mean)).*f_L(L));
    X2_part1_integ = integral(X2_part1,L_min,Lc);
    X2_part2 = @(L) ((L./L_mean).*(1 -
Lc./(2.*L)).*f_L(L));
    X2_part2_integ = integral(X2_part2,Lc,L_max);
    X2(ii) = X2_part1_integ + X2_part2_integ;
end
plot(Lc_mat,X1X2)
figure
plot(Lc_mat,X2)
end


function [a,b] = calc(ii)
if ii == 1
    fun = @solve_mojgan;
    x0 = [0.4 1.08];
    F = fsolve(fun,x0);
    a = F(1);
    b = F(2);
```

```
end
if ii == 2
    fun = @solve_mojgan4;
    x0 = [0.23 1.04];
    F = fsolve(fun,x0);
    a = F(1);
    b = F(2);
end
if ii == 3
    fun = @solve_mojgan6;
    x0 = [0.15 1.03];
    F = fsolve(fun,x0);
    a = F(1);
    b = F(2);
end
if ii == 4
    fun = @solve_mojgan8;
    x0 = [0.11 1.02];
    F = fsolve(fun,x0);
    a = F(1);
    b = F(2);
end
if ii == 5
    fun = @solve_mojgan10;
    x0 = [0.09 1.01];
    F = fsolve(fun,x0);
    a = F(1);
    b = F(2);
end

end
```

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