TO STUDY THE MECHANICAL PROPERTIES OF SURFACE TREATED NATURAL FIBER-PLA BASED GREEN COMPOSITES

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By

ISHAN SAXENA

(11310274)

Under the guidance of

MR. JAIINDER PREET SINGH

(14740)



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PUNJAB



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| Supervisor Name: | Jaiinder Preet Singh | UID: | 14740 | | Designations: | Assistant Professor |
| Qualification: | | | | Research Experience | : | |

| SR.NO. | NAME OF STUDENT | REGISTRATION NO | ВАТСН | SECTION | CONTACT NUMBER |
|--------|-----------------|-----------------|-------|---------|----------------|
| 1 | Ishan Saxena | 11310274 | 2013 | M1326 | 9592669274 |

SPECIALIZATION AREA: CAD/CAM & Mechatronics

Supervisor Signature:

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| PAC Committee Members | | | |
|-----------------------------------------|------------|------------------------|--|
| PAC Member 1 Name: Jaiinder Preet Singh | UID: 14740 | Recommended (Y/N): NA | |
| PAC Member 2 Name: Piyush Gulati | UID: 14775 | Recommended (Y/N): NA | |
| PAC Member 3 Name: Dr. Manpreet Singh | UID: 20360 | Recommended (Y/N): NA | |
| DRD Nominee Name: Dr. Sumit Sharma | UID: 18724 | Recommended (Y/N): Yes | |
| DAA Nominee Name: Kamal Hassan | UID: 17469 | Recommended (Y/N): Yes | |

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PAC CHAIRPERSON Name: 12174::Gurpreet Singh Phull

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CERTIFICATE

I hereby certify that the work being presented in the dissertation entitled "To study the mechanical properties of surface treated natural fiber-PLA based green composites" in partial fulfillment of the requirement of the award of the Degree of Master of Technology and submitted to the Department of Mechanical Engineering of Lovely Professional University, Phagwara, is an authentic record of my own work carried out under the supervision of (Name Of Supervisor, Designation) Department of Mechanical Engineering, Lovely Professional University. The matter embodied in this dissertation has not been submitted in part or full to any other University or Institute for the award of any degree.

(Date)

Ishan Saxena (11310274)

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

(Date)

Mr. Jaiinder Preet Singh (14740)

COD (ME)

The external viva-voce examination of the student was held on successfully _____

Signature of Examiner

DECLARATION

We hereby declare that the project work entitled 'To study the mechanical properties of surface treated natural fiber-PLA based green composites' is an authentic record of our own work carried out as requirements of Thesis Project for the award of Degree of Master of Technology in the Department of Mechanical Engineering from Lovely Professional University, Phagwara, under the guidance of Mr. Jaiinder Preet Singh, during August to December 2017.

All the information furnished in this capstone project report is based on our own intensive work and is genuine.

Project/Subject Code: MEC604

Name of Student: Ishan Saxena

Registration Number: 11310274

ABSTRACT

With increase in the global warming, fuel prices, depletion of fossil fuels, leads to the sustainable development. Because of these concerns, there is increase in the demand of eco friendly material which does not have a negative impact on the environment. Nowadays, lot of research is going on in the field of green composites. Green composites are the material in which matrix material is a biodegradable developed from the nature and natural fibers are used as the reinforcement material. In this study, Natural fibers are used as the reinforcement and polylactic acid (PLA) as the matrix material which is derived from the corn starch. Green composites have develop with the help of metallic molds and compression molding machine. All the composites would be develop at particular curing temperature and at fiber volume fraction ranging from 30% to 55% fiber volume by weight. Different surface treatments would be proposed on the fibers with concentrations ranging from 1%, 3%, 5%, 7% and 9%. The aim of using the surface treatment is to study the percentage improvement the mechanical properties of the developed composites. The study also proposed the different ways to improve the mechanical properties of the developed composites. Furthermore failure mechanism of the tested specimens will be analyze with the help of SEM.

| СНА | PTER-1 | : INTRODUCTION | 1 |
|-----|--------|----------------------------------------------------------|----|
| 1 | . CON | MPOSITE MATERIALS | 1 |
| | 1.1. | Types of Composites Based On Reinforcements | 3 |
| | 1.2. | Types of Composites Based on Matrix Materials | 5 |
| | 1.3. | Advantages of Composite Material | 6 |
| | 1.4. | Disadvantages of Composite Material | 7 |
| 2 | . GRE | EN COMPOSITES | 7 |
| | 2.1. | Components of Green Composites | 8 |
| | 2.2. | Advantages of Green Composites | 9 |
| | 2.3. | Disadvantages of Green Composites | 9 |
| 3 | . FAB | RICATION METHODS OF COMPOSITE MATERIALS | 10 |
| | 3.1. | Hand Layup Method | 10 |
| | 3.2. | Spray Lay-up method | 11 |
| | 3.3. | Compression Molding | 11 |
| | 3.4. | Resin Transfer Molding (RTM) | 12 |
| | 3.5. | Injection Molding Process | 13 |
| | 3.6. | Pultrusion | 14 |
| 4 | . SUR | RFACE TREATMENT METHODS OF FIBERS | 15 |
| | 4.1. | Silane Treatment | 15 |
| | 4.2. | Alkaline Treatment | 16 |
| | 4.3. | Benzoylation Treatment | 16 |
| | 4.4. | Acetylation Treatment | 17 |
| | 4.5. | Permanganate Treatment | 17 |
| | 4.6. | Peroxide Treatment | 17 |
| | 4.7. | Isocyanate Treatment | |
| СНА | PTER-2 | SCOPE OF THE STUDY | 18 |
| СНА | PTER-3 | : LITERATURE REVIEW | 20 |
| 1 | . REV | IEWS BASED ON GREEN COMPOSITES WITH SURFACE TREATMENT | 20 |
| 2 | . REV | IEWS BASED ON GREEN COMPOSITES WITHOUT SURFACE TREATMENT | 22 |
| СНА | PTER-4 | : OBJECTIVE OF THE STUDY | 27 |
| СНА | PTER-5 | : EXPERIMENTAL SETUP | |

Contents

| 1. OV | ERALL SETUP |
|----------|----------------------------------|
| 1.1. | Fabrication Setup |
| CHAPTER- | 6: RESEARCH METHODOLOGY |
| 1. MA | ATERIALS USED |
| 1.1. | Reinforcement Material |
| 1.2. | Matrix Material |
| 1.3. | Surface Treatment Material35 |
| 2. CA | LCULATION OF VOLUME FRACTION |
| 3. ME | CHANICAL CHARACTERISATION |
| 3.1. | Tensile Test |
| 3.2. | Flexural Test |
| 3.3. | Impact Test |
| 3.4. | Hardness |
| 3.5. | Morphological Characterization37 |
| CHAPTER- | 7: REFERENCES |

TABLE OF FIGURES

| Figure 1- Flow chart of classification of composites based on matrix materials [14] | 2 |
|-------------------------------------------------------------------------------------|----|
| Figure 2- Flow chart of classification of composites based on reinforcements [14] | 3 |
| Figure 3- Hand layup method [26] | 10 |
| Figure 4- Spray layup method [26] | 11 |
| Figure 5- Compression molding [26] | 12 |
| Figure 6- Resin transfer molding [26] | 13 |
| Figure 7- Injection molding process [26] | 14 |
| Figure 8- Pultrusion [26] | 15 |
| Figure 9- Complete view of setup | |
| Figure 10- Compression testing machine | 29 |
| Figure 11- Upper and lower dies | |
| Figure 12- Heating rods | |
| Figure 13- Teflon sheet [26] | |
| Figure 14- J-type thermocouple | |
| Figure 15- Controllers | |
| Figure 16- Jute fiber | |
| Figure 17- PLA | |
| Figure 18- Sodium hydroxide | |
| Figure 19- Barcol hardener [26] | 37 |
| | |

CHAPTER-1: INTRODUCTION

1. COMPOSITE MATERIALS

A composite material or composition material having superior characteristics from the individual components, with which it is made up of. It may consist of two or more constituents with more significantly different chemical or physical properties.

Composite tends to bind constituent's materials with a certain amount of significant proportion in order to produce a better combination of properties. For modern-day's best example are the fibers or particles indulged in matrix of a different material, which is extremely structural. Though, composites are eventually distinguishable from one another, no naked determination can be strictly made. For often facilitate definition, the product is shifted to levels at which considerable differentiation take place through microscopic or macroscopic.

Composites cannot be formed by components with divergent linear expansion characteristics. The area of contact between the components of composite materials is the interface, and it is always variable, which give composites a very unique property.

The two most important factors that deals with every specific composites are:

- Matrix materials
- Reinforcements

Matrix materials are such materials which provide strong bonds for the solids which accommodate stress to other constituents. The two paramount purposes are, under an applied force, distribution of stresses among the particles of reinforcements and also the adhesion between the reinforcement's particles.

Commonly used matrix materials are generally organic and inorganic. The organic compounds such as polyester resin, vinyl ester resin, epoxy resin, PLA (poly lactic acid), shape memory

polymer resin etc. The inorganic compounds include cement (concrete), ceramics, metals, glass etc.

Whereas, reinforcing materials or reinforcements generally adhere maximum load and they also serve desirable properties. Reinforcements usually maintain rigidity and protect the material from crack propagation.

Some of the examples of reinforcements are glass fibers, cellulose, jute fibers, sisal fibers, banana leaves, silicon carbide fibers, steel bars, steel mesh or wires etc.

Matrix materials support and surround the reinforcement materials by keeping their relative positions intact. Reinforcements reflect the special physical and mechanical properties to improve matrix properties.

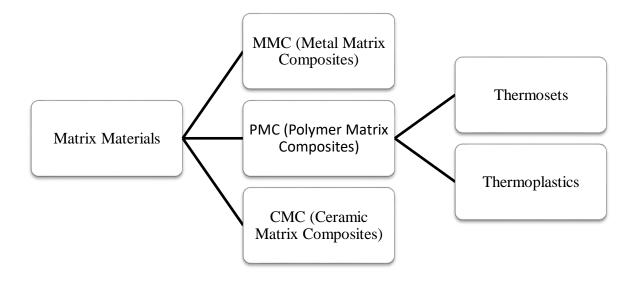


Figure 1- Flow chart of classification of composites based on matrix materials [14]

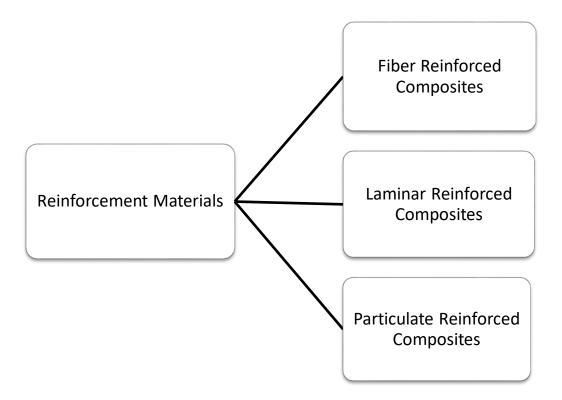


Figure 2- Flow chart of classification of composites based on reinforcements [14]

1.1.Types of Composites Based On Reinforcements

- Fiber Reinforced Composites
- Laminar Reinforced Composites
- Particulate Reinforced Composites

1.1.1. Fiber Reinforced Composites

The fibrous composites mainly consist of fiber as reinforcement in the matrix. This fiber can be continuous fiber or discontinuous fiber. Long fibers in various forms are inherently much stiffer and stronger than the same material in the bulk form. For example, ordinary plate glass fractures at stresses of only a few thousand pounds per square inch, in material that have dislocations the fiber form has less dislocation than the bulk form.

1.1.2. Laminar Reinforced Composite

Laminated composite is mainly made up of at least two or more layer of different materials that are stuck together. The single layer is called Lamina the best example is plywood in this we can easily identify the laminas are arranged layer by layer to create lamina. The properties that can be emphasized by lamination are strength, stiffness, low weight, corrosion resistance, wear resistance, beauty, thermal insulation and acoustical insulation etc. These are mainly classified into three categories: Bimetals, Clad metals and fiberglass

1.1.3. Particulate Composite

It contains particles of metals or non-metals suspended in a matrix of another material which can be metallic or non-metallic depends on the particles and matrix which we used in the matrix. This is mainly classified as: (i) metallic into metallic; (ii) metallic into non-metallic; (iii) nonmetallic into non-metallic; (iv) non-metallic into metallic; (v) cremates

Examples:

- Concrete in which sand and gravel particles are combined with mixture of cement and water.
- Aluminum paint in which Al-particles are suspended in polymer resin when aluminum paint is applied on a surface the Al-particles align themselves giving a good surface finish.
- Lead particles used in copper alloys and steel to improve machinability.
- Tool steels are generally made from a combination of oxide particles on metal matrix.
- Cremates such as tungsten carbide, boron carbide, titanium oxide etc. are used in a metallic matrix such as cobalt, nickel and copper is often used in high temperature applications such as turbine parts.

1.2.Types of Composites Based on Matrix Materials

- Metal Matrix Composites
- Polymer Matrix Composites
- Ceramic Matrix Composites

1.2.1. Metal Matrix Composites

Metal matrixes are nothing but the metals which are used as matrixes. Metal matrixes are used where the conducting properties are required. The common types of metal matrixes are Aluminum, titanium and nickel-chromium alloys. These are heavier than that of polymer matrixes and they are expensive.

1.2.2. Polymer Matrix Composites

The Polymer is defined as the substance which has a molecular structure made from a large number of similar units bonded together. These are mainly classified into two types:

1.2.2.1.Thermoplastics Polymers

Thermoplastics are the material which has a linear and branch chain and this can be given different shape by reheating. These polymers can easily soften repeatedly when heated and hardened when cooled with little change in their properties. Especially they can be reheated number of times. For e.g. Nylon, Polypropylene, Polythene, Polyvinylchloride, Teflon, etc.

1.2.2.2.Thermosetting Polymers

Thermosetting plastics are crossed linked chain polymers which has a property that once heated it forms a permanent shape by destroys their cross-linked structure. These are the polymers which undergoes permanent change on heating. For e.g. epoxy, polyimides, phenolic, Bakelite, melamine formaldehyde, etc.

1.2.3. Ceramic Matrix Composites

Ceramic matrixes are the one which uses ceramics as matrix material. These are used where the heat resistant properties are required. Common types of ceramic matrices are silicon carbide, boron carbide, tungsten carbide etc. These can be used at temperature above 1000°C where polymers and metals cannot be used.

1.3.Advantages of Composite Material

- Composites are light in weight, compared to most metals. Their lightness is important in aircraft, where less weight means better fuel efficiency.
- Strength-to-weight ratio is a material's strength in relation to how much it weighs. Some
 materials are very strong and heavy, such as steel. Composite materials can be designed
 to be both strong and light. This property is why composites are used to build airplanes,
 which need a very high strength material at the lowest possible weight.
- Composites resist damage from the weather and from harsh chemicals that can eat away at other materials. Outdoors, they stand up to severe weather and wide changes in temperature.
- Composites can be molded into complicated shapes more easily than most other materials. This gives designers the freedom to create almost any shape or form.
- A single piece made of composite materials can replace an entire assembly of metal parts. Reducing the number of parts in a machine or a structure saves time and cuts down on the maintenance needed over the life of the item.
- Composites retain their shape and size when they are hot or cool, wet or dry. They are used in aircraft wings, for example, so that the wing shape and size do not change as the plane gains or losses altitude.

- Radar signals pass right through composites, a property that makes composites ideal materials for use anywhere radar equipment is operating, whether on the ground or in the air. Composites play a key role in stealth aircraft, such as the U.S. Air Force's B-2 stealth bomber, which is nearly invisible to radar.
- Structures made of composites have a long life and need little maintenance. We do not know how long composites last, because we have not come to the end of the life of many original composites. Many composites have been in service for half a century.

1.4.Disadvantages of Composite Material

- Since composites are often constructed of different ply layers into a laminate structure, they can "delaminate" between layers where they are weaker.
- They are relatively of higher cost.
- The fabrication process is usually labor intensive and complex, which further increases cost.
- Delamination and cracks in composites are mostly internal and hence require complicated inspection techniques for detection.
- Metals expand and contract more on variations in temperature as compared to composites. This may cause an imbalance at joinery and may lead to failure.

2. GREEN COMPOSITES

A green composite is a special type of composite material which is made up of green materials in the sense environment-friendly. In this case, it can be bio-degradable or non-bio degradable according to the matrix we are using. Green composites are those composites that are made from natural occurring resin and fiber matric interface. There are various fiber matrix are obtain from the nature that are jute, flax, hemp, cotton, kenaf, coir, abaca, wool, silk, pineapple, coconut, banana and sisal. Natural fibers are grouped into the three type's seed hair (cotton, coir), basalt fibers (hemp, flax, jute, kenaf) and leaf fibers (sisal, abaca). The properties of natural fiber have low cost, high specific mechanical property, good thermal, acoustic insulation and bio degradability.

Fiber reinforced polymers are very common especially in the engineering applications. But most of the normal composites are hazardous to the environment. There comes the application of green composite, in this the matrix as well as the fibers also bio-degradable. Most common fibers are plant fibers used to make a green composite. Because not only it is environmental friendly but also no negative health hazard made this as very interesting now a days for the researchers. The beauty of green composite is it is very easy to fit into any size and shape according to the requirement and wide range applications. In the meantime, it is very difficult to find bio degradable resins and epoxy materials. This made a great interest in PLA (poly lactic acid), about this material it a fully bio degradable material and eco-friendly. The research has been going on this material and researchers have not found any health hazard about PLA .It is produced from lactic acid by fermentation process. PLA can be made from two methods, first is condensation and second is ring opening polymerization.

2.1.Components of Green Composites

2.1.1. Biodegradable Matrices

With the increasing use of polymers/matrix, the problems of disposal of waste of these polymers are also posing alarming causes regarding environment and health issues. So are main concerns to go the biodegradable polymers which can be broken rapidly by soil micro-organisms and do not cause any serious effects on the environment. Biopolymers degrade mainly by enzymatic hydrolysis and to some extent by oxidation. The common examples of biodegradable polymers are poly-hydroxy butyrate (PHB), poly-hydroxy butyrate-co- β -hydroxy valerate (PHBV), poly-glycolic acid (PGA), poly-lactic acid (PLA) etc.

2.1.2. Biodegradable Fibers

In the case of fibers, we have many options but they are mainly classified into two types natural and synthetic a classification is given below fig 1.3. Talking about the fibers there are vast varieties are already available in the market as well as in some of the papers researchers tried to extract the fibers from the plants. This made an interest to think about a new direction to extract fiber from the Castor Plant. The methods which we used different molar values of varied temperature this has given a positive feedback for the further processing. The jute fiber also has taken for consideration.

2.2. Advantages of Green Composites

- These are very less costly and affordable.
- These are lighter in weight and easy to handle.
- They have a better flexibility.
- They can be renewed and recycled easily.
- They can also be thermal recycled.
- They are user-friendly and produce no skin irritation

2.3.Disadvantages of Green Composites

- They are comparatively less in magnitude in mechanical properties.
- They swell easily as there rate of water absorption is comparatively more.
- They are very less durable.
- They are poorly resistive to fire.

3. FABRICATION METHODS OF COMPOSITE MATERIALS

The composite materials are made by certain fabrication methods which also include the mixing and pre-treatment processes of matrix and reinforcements.

3.1.Hand Layup Method

Hand lay-up is the oldest open molding method of the composite fabrication processes. It is a low volume and labor intensive method suited especially for large components, such as boat hulls etc. Glass or other reinforcing mat or woven fabric or roving is positioned manually in the open mold, and resin is poured, brushed, or sprayed over and into the glass plies. Entrapped air is removed manually with squeegees or rollers to complete the laminates structure. Room temperature curing polyesters and epoxies are the most commonly used matrix resins. Curing is initiated by a catalyst in the resin system, which hardens the fiber reinforced resin composite without external heat. For a high quality part surface, a pigmented gel coat is first applied to the mold surface.

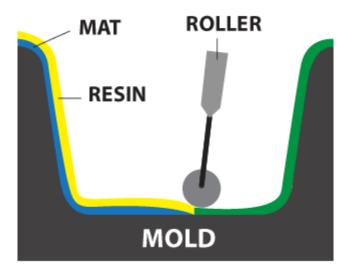


Figure 3- Hand layup method [26]

3.2.Spray Lay-up method

Spray-up method similar to hand lay-up in its suitability for making boats, tanks, transportation components and tub/shower units in a large variety of shapes and sizes. A chopped laminate has good conformability and is sometimes faster to produce than a part made with hand lay-up when molding complex shapes. As with hand lay-up, gel coat is first applied to the mold and allowed to cure. Continuous strand glass roving and initiated resin are then fed through a chopper gun, which deposits the resin-saturated "chop" on the mold. The laminate is then rolled to thoroughly saturate the glass strands and compact the chop. Additional layers of chop laminate are added as required for thickness. Roll stock reinforcements, such as woven roving or knitted fabrics, can be used in conjunction with the chopped laminates. Core materials of the same variety as used in hand lay-up are easily incorporated.

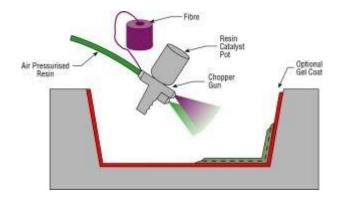


Figure 4- Spray layup method [26]

3.3.Compression Molding

It is a high-volume thermoset molding process that employs expensive but very durable metal dies. It is an appropriate choice when production quantities exceed so many parts. As many as parts can be turned out on a set of forged steel dies, using sheet molding compound, a composite sheet material made by sandwiching chopped fiberglass between two layers of thick resin paste. To form the sheet, the resin paste transfers from a metering device onto a moving film carrier. Chopped glass fibers drop onto the paste, and a second film carrier places another layer of resin on top of the glass. Rollers compact the sheet to saturate the glass with resin and squeeze out entrapped air.

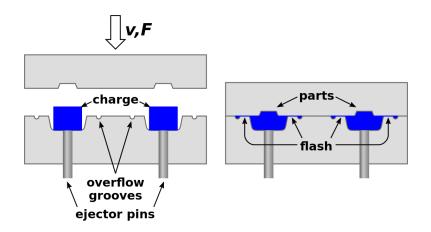


Figure 5- Compression molding [26]

3.4. Resin Transfer Molding (RTM)

RTM is a vacuum-assisted, resin transfer process with a flexible solid counter tool for the other side surface compression. This process yields increased laminate compression, a high glass-to-resin ratio, and outstanding strength-to-weight characteristics. RTM parts have two finished surfaces. Reinforcement mat or woven roving is placed in the mold, which is then closed and clamped. Catalyzed, low-viscosity resin is pumped in under pressure, displacing the air and venting it at the edges, until the mold is filled. Molds for this low-pressure system are usually made from composite or nickel shell-faced composite construction.

Resin Transfer Molding

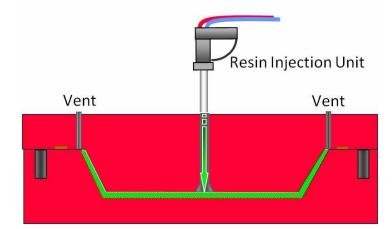


Figure 6- Resin transfer molding [26]

Similar to RTM, Vacuum Assisted Resin Transfer Molding (VARTM) is typically an open-top mold composite part fabricating method that attaches a vacuum bag to the top of the mold tool and applies vacuum to assist the continuous flow of low-pressure infused resin from one side of the mold to the other. Vacuum removes air from the perform and assists the VARTM machine's on-ratio metered and mixed flow of degassed air-free resin through the compressed composite fiber performs below the vacuum bag.

3.5.Injection Molding Process

Natural fibers are randomly oriented or short fibers were used as reinforcement and composites were developed with the help of Injection molding process. It is a fast, high-volume; low-pressure, closed process using, most commonly, filled thermoplastics, such as nylon with chopped glass fiber. Injection molding is the most commonly used manufacturing process for the fabrication of plastic parts. The injection molding process requires the use of an injection molding machine, raw plastic material, and a mold. The plastic is melted in the injection molding machine and then injected into the mold, where it cools and solidifies into the final part.

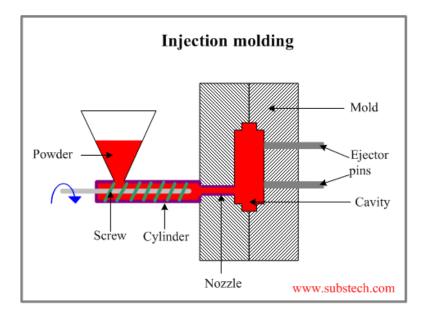


Figure 7- Injection molding process [26]

3.6.Pultrusion

Pultrusion process is like RTM, has been used for decades with glass fiber and polyester resins, but in the last years the process also has found application in advanced composites applications. In this relatively simple, low-cost, continuous process, the reinforcing fiber is typically pulled through a heated resin bath and then formed into specific shapes as it passes through one or more forming guides or bushings. The material then moves through a heated die, where it takes its net shape and cures. Further downstream, after cooling, the resulting profile is cut to desired length. Pultrusion yields smooth finished parts that typically do not require post processing. A wide range of continuous, consistent, solid and hollow profiles are pultruded, and the process can be custom-tailored to fit specific applications.

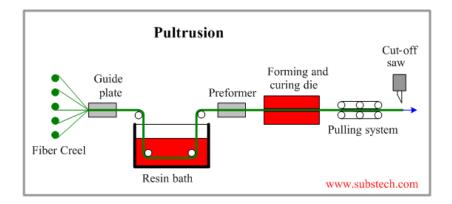


Figure 8- Pultrusion [26]

4. SURFACE TREATMENT METHODS OF FIBERS

Surface treatment includes metals, polymers, plastics etc. that are treated to improve their properties such as hardness, wear resistance, corrosion resistance, better decoration, adhesion etc. Certain processes are used in surface treatment as blanching, case hardening, painting, dipping, ceramic glazing, cladding, galvanizing, electroplating, knurling, coating (bluing, anodizing and chrome plating), electro polishing, chemical-mechanical planarization, flame polishing, chemical treatment methods etc. Chemical surface treatment processes are referred to as post processing methods for fibers. They play a vital role in the function, appearance and life of the product. These kinds of processes affect either a thin layer of the surface or add a thin layer on the surface of the fiber. For better chemical treatment of the fiber based composites, the post processing methods used are as follows:

4.1.Silane Treatment

This treatment methods includes a chemical compound i.e. Silane which has a chemical formula, SiH4. They are used as coupling agents for specially glass fibers to adhere with polymers. It reduces number of cellulose hydroxyl groups in the interface of fiber-matrix, which leads to formation of silanols. This silanols reacts with hydroxyl groups of fiber, which forms stable covalent bonds to the wall. Therefore, it creates a cross-linked network. Thermal stability of composites gets improved after such treatment. Among the alkaline treatment and silane treatment, the coupling between fiber and matrix, silane treatment provides better tensile strength to the composites.

4.2.Alkaline Treatment

For reinforcing thermosets and thermoplastics, the highly used chemical treatment is the alkaline treatment. The alkaline treatment helps in removing a specific amount of wax, lignin and oils that are covering the outer surface of the fiber walls. This further helps in depolymerizing the cellulose and exposing the crystallites. Addition of sodium hydroxide (NaOH) to the fibers regulates the ionization of hydroxyl group to alkoxide. In this treatment, fibers are dipped in NaOH solution for a directed period of time. This leads to two effects:

- Increasing the surface roughness of fibers, this result in effective mechanical interlocking.
- Increasing the cellulose quantity exposed on the surface of fibers, which results in increase of possible reaction sites.

It also improves the impact fatigue and dynamic behavior of fiber-based composites. If the concentration of NaOH is not optimum, the tensile strength of composites decreases abruptly.

4.3.Benzoylation Treatment

For organic synthesis, benzoylation is the best chemical treatment method. In fiber treatment, benzoyl chloride is often used. Benzoyl chloride consists of benzoyl which is progressed to hydrophilic nature of fiber. The chemical reaction is shown as: The benzoylation of fiber improves:

- Strength of the composite materials.
- Fiber-matrix adhesion is more progressed.
- Thermal stability is thoroughly improved.
- Decreasing the rate of water absorption.

4.4.Acetylation Treatment

This type of treatment includes an acetyl functional group (CH3COO-). It causes plasticization of cellulose fibers. This chemical treatment is also known as esterification method. Chemical modification with (CH3-C (=O)-O-C(=O)-CH3) replaces the hydroxyl group of polymer with acetyl groups. The acetylation treatment helps in:

- Reducing the fiber's hygroscopic nature.
- Increasing the dimensional stability of composite materials.
- Improving the fiber-matrix adhesion.
- Improving the covalent bond formation

Acryl nitrite (AN), having a chemical formula (CH2=CH-C N), is also used to modify the fibers.

4.5.Permanganate Treatment

This treatment contains a compound which is permanganate, having a permanganate group. It leads to formation of cellulose radical by MnO3- formation. Due to this treatment, the hydrophilic tendency gets reduced. Thus, the absorption, of water reduces, in the composite materials. This reduction in hydrophilic tendency is because of increase in KMnO4 concentration. The permanganate methods have certain benefits:

- Adhesion between fiber and matrix improves.
- Thermal stability increases effectively.
- Water absorption is very less.

4.6.Peroxide Treatment

Peroxide is a special molecule, having a functional group, which is represented by ROOR. It also contains a divalent ion, O-O. For freeing the radicals, peroxides decompose very easily. This RO group then reacts with hydrogen group of fibers and matrix. In this type of treatment, fibers are

coated with DCP or BP, dipped in acetone solution for around 30 minutes, after alkaline pretreatment. The peroxide treatment helps in:

- Decreasing the hydrophilicity.
- Increasing the tensile properties.

4.7.Isocyanate Treatment

This treatment includes isocyanate compound, containing the isocyanate functional group, as -N=C=O. The isocyanate group helps fibers reinforced composites, as a coupling agent. Isocyanate treatment helps composites materials in:

- Especially for sisal fibers, hydrophilic nature and tensile properties improves.
- The fiber-matrix interfacial adhesion improves.
- Comparing the silane and acrylic treatment, the Young's modulus is much more increased.

CHAPTER-2: SCOPE OF THE STUDY

As an aspiring mechanical engineer with a passion in green composites, this topic gave me glimpse into something I could be working with in the near future. The importance of green composites and what role they can play in our lives in the future when they become much more accessible is well understood. Engineers improvises the technology to make it more effective while achieving the needs of society, I think that green composites can create strides in the composite materials that our everyday things are made out of.

Common issues caused because of non-biodegradable materials:

- Changes in the climate.
- Degradation of natural elements in the environment.
- Physical and mental illness of human beings.
- Harmful effects due to air, soil and water pollution.

Considering all these problems we need something instead of non-biodegradables. This is the point where the green composites come into the picture. The use of green composite is the best alternative for the problems created by the non-biodegradable materials Green composites for aircrafts and aero planes will aid in the travel industry, nano-technology using green composite materials in the medical industry, body material for automobiles etc. after such composites are finalized and standardized. Engineers, along with other scientists, are the main cause for making such technology a reality and they are almost in reach. If engineers can develop such materials for common uses, it would be a much greater step in the appropriate direction. I believe that if we have the right information and knowledge of these materials to make something better, we should. Using composite materials is an example of such thing. I chose this topic because it is very pertinent to my corresponding field. I find green composites very exciting and is something I believe could help in improving different materials or processes. I believe that continued work in this field is essential.

CHAPTER-3: LITERATURE REVIEW

Researchers have been working under green composites for a long time. We have gone through a number of research papers for the identification of development problem formulation and a complete understanding for the further processing of researches.

1. REVIEWS BASED ON GREEN COMPOSITES WITH SURFACE TREATMENT

- John O. Akindoyoet al (2017) [1] studied about hydroxyapatite and PLA matrix based on extrusion and injection molding. For better results, they used a modifier i.e. fabulase 361, which is phosphate based & it helped in modifying the surface of hydroxyapatite (HA). 10 %wt of HA was used for analysis and it is appropriate amount for better effectiveness and adhesion, mechanical and thermal properties and dispersion of HA.
- Zeren Ying et al (2017) [2] studied the mechanical properties of Pristine's Basalt fibers as reinforcement to PLA based composites. They used 20 %wt that will be able to increase strength and modulus by an appropriate amount. The surface treatment of BF was done with silane that helps in increasing the affinity to PLA matrix, increasing both compact and tensile strength. It was done by the cold crystallization of PLA and further solid annealing. Due to solid annealing, mechanical strengths were improved.
- Romana Nasrin et al (2017) [3] study involves the manufacturing of PLA based composites, including available waste prawn shell derived chitin as reinforcement. It was produced by hot press method at 160 °C. After an effective surface treatment, the laminated composite material had higher tensile strength and elongation break.
- **Panayiotis Georgiopoulos et al (2016)** [4] include unidirectional flax fiber composites, which is exposed to different dynamic mechanical analysis. It was fabricated by film stacking method. There were different treatment processes used, named as plasticization,

silanization and treatment with maleic anhydride. Due to these surface treatments, a good adhesion between matrix and fibers were observed. Improvement in crystallinity, Youngs' modulus, storage modulus and flexural modulus were also detected.

- Paweena Saenghirunwattanaa et al (2014) [5] studied the mechanical properties of green composites (soy protin) with modified cornhusk fiber. Using alkali solution fibers were obtained from cornhusk. Due to alkali treatment non cellulosic substance and wax were removed. Using bio plastic constructed from soy protin, defatted soy flour and polylactic acid the fibers were surface modified along with silane. Noteworthy improvements of mechanical properties at 5wt% were observed.
- **B K. Goriparthi, K.N.S. Suman, Nalluri Mohan Rao** (2012) [6] worked to improve the adhesion with PLA on jute fiber. Surface treatment lead to decline of Izod impact strength and enhancement of flexural and tensile properties. DMMA result indicated that they have lower tangent delta and higher storage modulus. It was also found that composite's wear resistance is sensitive to matrix/fiber adhesion (abrasive wear test result) and they have better thermal stability for composite treated with silane.
- C. Sareena et al (2013) [7] studied about the natural rubber reinforced with natural fillers like coconut shell and peanut shell powder. They were buried under for about three/six months. The biodegradation test was implemented and also the tensile strength and hardness measurements were calculated. The chemical treatment, filler content and filler size helps in improving the durability. The composite containing treated fillers were resistant to soil erosion. The results progressed that after the soil burial testing, the hardness and tensile strength were decreased.

• V. Fiore, G. Di Bella, A. Valenza (2014) [8] studied about kenaf fibers, which were pretreated with alkaline i.e. NaOH solution at a 6%wt. The chemically treated kenaf fibers were allowed to furnish their surface for 48 hours. The treated and untreated kenaf fibers were reinforced with epoxy resin composites. The composites detected higher moduli when the effect of stacking sequence on the mechanical properties was observed. The compatibility of fiber and matrix helped in increasing the mechanical properties of composites.

2. REVIEWS BASED ON GREEN COMPOSITES WITHOUT SURFACE TREATMENT

- Marius Marariu, Philippe Dubois (2016) [9] studied about biodegradable polymers, and restrict its use with high carbon footprint from petrochemical origin such as packaging, automotive etc. It was found that the composite was brittle and have 10% or less, and have low toughness. These polymers from non-fossil natural resources by fermentation of sugar. Due to this, using different fibers with PLA, the tensile strength, dimensional stability and barrier properties are improved.
- Lin Xin Zhong et al (2010) [10] studied to improve phenolic resin / cellulose fiber interfacial adhesion in composites comprised of aramid fiber and sisal fiber by surface micro fibrillation on cellulose fiber. Sisal fiber's surface micro fibrillation to 24°SR surge the internal bonding strength, wear resistance and tensile strength values by 124%, 31%, 93% respectively.
- K. Kanny, V. Paul, G.G. Redhi (2014) [11] studied thermal, morphological and mechanical properties of a bio composite originated from banana plant. Resin generated from banana sap were introduced with banana fiber was used as bio-based composite. At high temperature there was a decrease in moduli. With increase of glass transition

temperature thermal stability was improved. Electron microscopy exposed an improved compatibility amid banana sap and fiber. The degradation test revealed improved microbial activity on the composite. This indicates improved biodegradation rates. The mechanical test revealed increase of tensile strength, tensile modules and flexural modulus by 15%, 12% and 25% respectively.

- Harekrishna Deka, Manjusri Misra, Amar Mohanty (2012) [12] explored the physico-mechanical and inter-phasic properties of "all green composites" acquired from poly bio resin and kenaf natural fiber. The optimal properties of composites were accomplished by 20wt% which results in major increment in flexural strength (48%), tensile strength (310%) and storage modulus (123%). After exposing the composite to boiling water adequate retention were absorbed up to 89% for flexural, 82% for impact and 83% for tensile strength.
- M. Terano et al (2013) [13] studied to substitute non-biodegradable fibre (synthetic) with ecofriendly bio composites. The plain WJF reinforced PLLA composites were made by molding method (hot press method). Morphological studies demonstrate improved adhesion between PLLA and treated fabric was achieved. It was also established the modulus of composites and strengths in weft direction is less than in warp direction. The average tensile modulus, flexural modulus, flexural strength, impact strength and tensile strength were improved about 211%, 42.4%, 95.2%, 85.9% and 103% respectively.
- M.J.A. van den Oever, B. Beck, J. Müssig (2010) [14] studied consequences of moisture on mechanical properties and degradation of agrofibre composites reinforced with PLA. The fibre evaluated are flax, cotton and ramine containing moisture in dried state (0.2-0.4 mass %) and undried state (6-9 mass %). Here they found out the result of water content on PLA degradation (semi crystalline grade) during mechanical

performance and processing flexural strength, Charpy impact and stiffness are not considerably affected by water existing in the undried state.

- P.E. Bourban et al (2014) [15] have compared the damping and mechanical properties of unidirectional and 2/2 flax fibre containing thermoplastic (PP), PLA and thermoset (epoxy) with glass (GF) and carbon fibre (CF) epoxy composites. The best compromise amid damping and stiffness was found with FF reinforced PLA. The composite with FF showed better damping with respect to CF and GF reinforced composite.
- Yihu Song, Qiang Zheng (2008) [16] studied the properties and structure of green composite. These were made by blending of methylcellulose as filler, glycerol as plasticizer and wheat gluten followed by cross linking the matrix at 127°C by compression molding. It was discovered the presence of MC fibre can substantially improve E and σ_b and also there is increase of glass transition temperature of WG matrix.
- Changduk Kong, Hyunbum Park, Joungwhan Lee (2014) [17] study involves vinyl ester/flax natural fiber composites. The manufacturing method used was VARTM (Vacuum Assisted Resin Transfer Molding) method. The mechanical properties of the composites were compared with data cited from certain reference. After specific analysis, it was detected that designed chemical storage tank is appropriate for stability and safety.
- Yao Peng et al (2014) [18] studied about the effects of UV weathering on the mechanical properties of polypropylene reinforced with lignin, wood flour and cellulose at different levels. The surface was differentiated by ATR-FTIR and SEM. The results shown that (1) the composites which involved lignin shown less cracks, less loss of modulus and flexural strength and better hydrophobicity; (2) Due to presence of lignin, the discoloration was accelerated.

- H. Ku et al (2011) [19] studied about the natural fibers reinforced with polymer and its tensile properties. Natural fibers have lower cost and due to this, they can be used as a replacement of conventional fiber, as they have mechanical properties, non-abrasive, biodegradability, high specific strength and eco-friendly characteristics. Natural fibers are effective in many ways, than the other fibers such as carbon, glass and aramid fibers. Natural fibers provide a better interfacial adhesion between fiber and matrix materials.
- E. Trujillo et al (2014) [20] study involves a long bamboo fibers and its high end uses as a reinforcement for composite materials. At different gauge lengths, Weibull distribution, which requires only three parameters, explained accurately the fibre strength of fibers. With increasing gauge lengths, the fiber strength decreases. While comparing with natural fibers, Weibull shape parameter had shown low strength variability, indicating high quality.
- V. Fombuena et al (2013) [21] worked to use the calcium carbonate on sea shell combined with epoxy matrices (eco-friendly) to move a highly renewable material. The shells were washed and then grinded. The obtained powder and resin were later tested. The addition of calcium carbonate is an effective way to enhance mechanical properties and to decrease the residue of shells. The test showed that adding 30wt% of shells improves hardness, flexural modulus and thermal properties.
- Xiaosong Huang, Anil Netravali (2007) [22] studied characterization of green composites (soy protein concentrate altered by nano-clay particle. The modified SPC was prepared by blending SPC alongside nano-clay particles and then glutaraldehyde is used for cross-linking. For reinforcement, flax fibers were used in both fabric and yarn farms. The unidirectional flax, SPC composites displayed 298 MPa and 4.3 GPa of tensile failure stress and Youngs' modulus respectively.

- Nurfatimah Bakar et al (2014) [23] studied the effect of ungrafted and grafted kenaf fibers on polyvinyl ethylene/chloride vinyl acetate composites. Fibers were made by graft co-polymerization reaction and as a monomer methyl methacrylate is used. About 0% to 30% variation was used for fiber loading. Tensile strength and impact strength started to decline when kenaf fiber is added on EVA/PVC composite. The company of PMMA has improved the effectiveness of stress transfer (matrix to fiber).
- Ahmed Fotouh, John D. Wolodko, Michael G. Lipsett (2014) [24] studied fatigue behavior of high density polyethylene (high fiber reinforced) composite. It is explored with the help of fatigue life curve at separate volume fraction. A new revised stress level is suggested to normalize S-N curve. A simplified model is formed to simulate composite's fatigue life response. The developed model can foresee the fatigue behavior of different fatigue stress ratio and fiber fraction.
- Marie-Joo Le Guen et al (2014) [25] found that adding polyglycerol and glycerol increased coefficient by 21-25 % and 10-13 % respectively. The coefficient of non-treated flax was smaller than polyol treated composites. This is all because of stick slip mechanism which involves reformation and cyclic breaking of hydrogen bonds between lamellae and polyols of microfibrilis.

CHAPTER-4: OBJECTIVE OF THE STUDY

- Investigation on effect of different surface treatments of natural fibers on mechanical properties of developed composites.
- Morphological study to find out the failure mechanism of developed composites using SEM.
- Find the new ways to improve the mechanical strength of developed composites.

CHAPTER-5: EXPERIMENTAL SETUP

1. OVERALL SETUP

A setup had been made for the development of Green Composite. The complete setup of fabrication of green composites as gives below.



Figure 9- Complete view of setup

1.1.Fabrication Setup

The complete setup consists of following components:

- Compression Molding Machine
- Compression Molding Dies
- Heaters
- Releasing Agent

• Electrical Control Panel

1.1.1. Compression Molding Machine (CMM)

For such kind of testing, HEICO's machine is used. This machine consists of loading unit as well as pumping unit. At the base of the loading unit, a hydraulic jack is fitted and a load display unit is attached to the upper end of loading unit which is used to operate the pumping unit and controls the load. Specification: motorized pump (200V, single phase AC), maximum load (1000KN), and Hydraulic oil servo system ENKL 68.



Figure 10- Compression testing machine

1.1.2. Compression Molding Dies

It is a specialized tool used for the fabrication of composite by pressing it. Basically, it consists of an upper part (punch plate) and the lower part (die block) as shown in figure 11. There are

different types of strips used for making the different samples. The die is made of EN31 steel material which achieves very high compressive strength with high hardness value.



Figure 11- Upper and lower dies

1.1.3. Heaters

The setup was heated by commercial heating rod elements as shown in figure 12. Total nine heater rods used to heat the setup. The voltage of each rod heater is 750W with single phase. The controllers were used to cut off the supply of heater when temperature reaches required values.



Figure 12- Heating rods

1.1.4. Releasing Agent

To reduce the sticking phenomena between metallic die and matrix material, we are using Teflon sheet as releasing agent so that metallic die can be easy open. Teflon sheet is placed on the upper face of lower die and lower face of upper die to prevent the metallic die and fabricated composite specimen also.



Figure 13- Teflon sheet [26]

1.1.5. Electrical Control Panel

The electrical control panel unit consists following components:

1.1.5.1.Thermocouple

It is a device which consists of two wires made of different metals for the measurement of temperature. J-type thermocouple figure 13 is used in the electrical control panel to measure the temperature of the die. The range of J-type thermocouple is -40 °C to 750 °C. Calibration means whether instrument gives a correct reading or not. It is basically a process that helps to calibrate the instrument. The calibration of the thermocouple is done with the help of a thermometer.



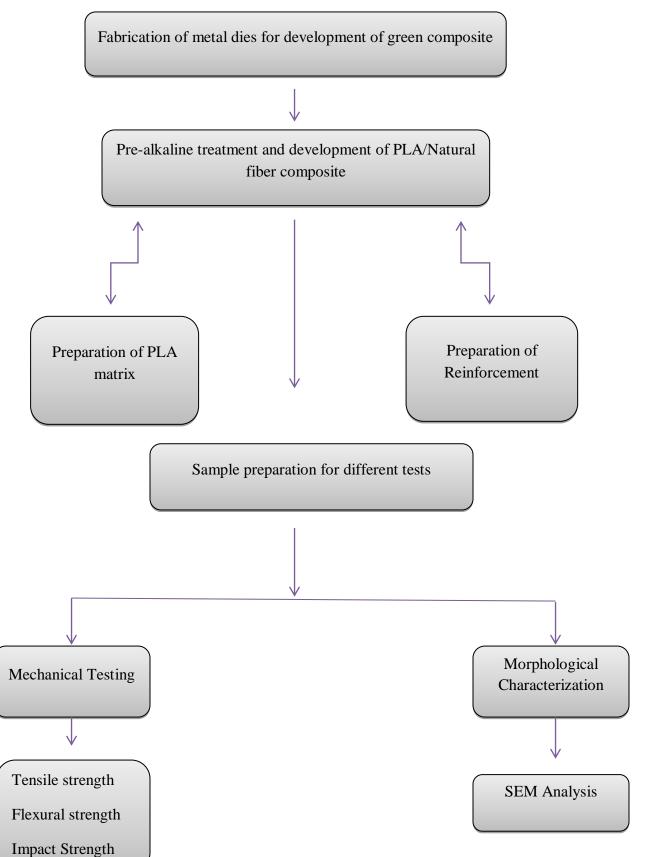
Figure 14- J-type thermocouple

1.1.5.2.Controllers

The main target of this setup is to maintain the die temperature at a required value. So that controllers are used in control panel to control the temperatures of the dies with the help of relays cut off. To begin with, set the temperature value on the display screen figure 14. Once the dies attained the set value controllers cut off its supply by sensing the temperatures of the dies with the help of thermocouples.



Figure 15- Controllers



CHAPTER-6: RESEARCH METHODOLOGY

1. MATERIALS USED

Natural fibers are used as the reinforcement and PLA is used as the matrix material to make the green composite.

1.1.Reinforcement Material

The reinforcement such as jute used is balanced and symmetrical jute fiber collected from the local sources



Figure 16- Jute fiber

1.2.Matrix Material

The matrix used is PLA pallets collected from Nature-Tech India Pvt .Ltd. Chennai, India.



Figure 17- PLA

1.3. Surface Treatment Material

For the current, we are using alkaline treatment method, which requires a material known as sodium hydroxide i.e. NaOH. The material is in the form of small pellets.

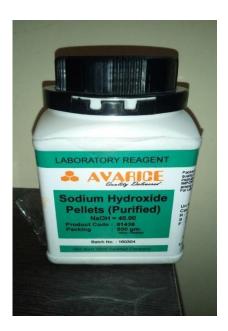


Figure 18- Sodium hydroxide

2. CALCULATION OF VOLUME FRACTION

Fiber volume fraction means the amount of fiber present in the composite material. The Fiber fraction obtained after fabrication of composite is cross verified with the formula.

 $V_{f}=1 - (W-W/\rho * V) \dots [16]$

Where,

 V_f 'is the volume fraction of fiber

'W' is weight of fabricated composite

' ρ_m 'is the density of resin

'V' is volume of fabricated composite

3. MECHANICAL CHARACTERISATION

3.1.Tensile Test

The tensile test will be performed on the universal testing machine. It is load bearing capacity per unit area of cross section. Through this test decision of its application and site of using is, done easily by according to the ASTM standard D 3039. The specification of samples for the tensile test as per the ASTM standard is 250x25x4mm.

3.2.Flexural Test

The flexural testing will be done on the universal testing machine. 3 point bending test will be performed according to the ASTM standard D790-02. The results obtained showing the effects surface treatment on flexure strength. The specification of samples for flexure test as per the ASTM standard is 120x15x4mm.

3.3.Impact Test

It is the amount of energy absorbed by the material during fracture. Izod V notch test is conducted on each specimen of composite material. The ASTM D256-02 is used for Izod testing. The specifications of samples for impact test are as per the ASTM standard is62.7x12.7x4mm.

3.4.Hardness

The Barcol hardness test characterizes the indentation hardness of materials through the depth of penetration of an indenter, loaded on a material sample and compared to the penetration in a reference material. The method is most often used for composite materials such as reinforced thermosetting resins or to determine how much a resin or plastic has cured. The test complements the measurement of glass transition temperature, as an indirect measure of the degree of cure of a composite.



Figure 19- Barcol hardener [26]

3.5.Morphological Characterization

The structural analysis will be done by Scanning electron microscopy (SEM). The images of samples are produced with an electron microscope by keeping it in a focused electron beam. The surface of the test piece is firstly cleaned with acetone and kept in vacuum for approximately 15 minutes to completely dry the surface. After this, the piece is kept on the microscope for the proceedings for SEM. The SEM results are taken at different magnifications and then analyzed.

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