EFFECT OF CRYOGENIC TREATMENT ON THERMAL AND MECHANICAL PROPERTIES OF HEMP FIBER COMPOSITE PANELS

A PROJECT REPORT

Submitted by

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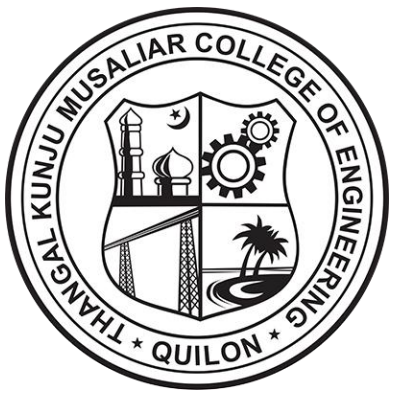
REG NO: TKM21MEIR09

to

The APJ Abdul Kalam Technological University

in partial fulfilment of the requirements for the award of

M. Tech degree in Industrial Refrigeration and Cryogenics Engineering.



Department of Mechanical Engineering

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May 2023

DEPARTMENT OF MECHANICAL ENGINEERING

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CERTIFICATE

This is to certify that this report entitled '**EFFECT OF CRYOGENIC TREATMENT ON THERMAL AND MECHANICAL PROPERTIES OF HEMP FIBER COMPOSITE PANELS**' is the report of project presented by **RAHUL RAJ**, Reg. No: **TKM21MEIR09** during **2022-2023** in partial fulfilment of the requirements for the award of the Degree of Master of Technology in Industrial Refrigeration and Cryogenics Engineering of the *APJ Abdul Kalam Technological University*.

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DECLARATION

I, Rahul Raj, hereby declare that this project report entitled "Effect of cryogenic treatment on thermal and mechanical properties of hemp fiber composite panel", is the bonafide work of mine carried out under the supervision of Dr. Mubarak Ali M., Assistant Professor in the Department of Mechanical Engineering, TKM College of Engineering, Kollam. I declare that, to the best of my knowledge, the work reported herein does not form part of any other project or dissertation on the basis of which a degree or award was conferred on an earlier occasion to any other candidate. The content of this report is not being presented by any other student or any other university for the award of a degree.

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ABSTRACT

Natural fibres have been outstanding materials which are feasible and amble substitute for expensive, non-bio degradable and non-renewable synthetic materials so these organic/natural fibres can be used as replacement for synthetic fibers for various applications. Cryogenic treatment is a type of heat treatment process applied to materials at low temperatures in which fiber is treated at cryogenic temperatures, at this low temperature conditions, materials showcase some physiochemical changes The main objective of these works is to conduct cryogenic treatment on hemp fiber and to study the effect of cryogenic treatment on thermal, mechanical and water absorption properties of hemp fiber reinforced epoxy composite panels. Physical properties such as functional groups of fabrics were studied and compared using Fourier Transform Infrared Spectroscopy (FTIR). It shows treatment on fiber have substantially increased the number of hydrogen bonding in cellulose, which lead to increase in stiffness of the fabric. The thermal behaviour of untreated and treated hemp fiber was studied using the techniques of thermo gravimetric analysis (TGA) and differential thermo gravimetric analysis (DTG). It is observed that after cryogenic treatment, hemp fiber's thermal stability has been significantly enhanced. Mechanical properties such as tensile and flexural strength of Hemp/epoxy panels are studied under untreated and cryogenic treated condition and the result show improvement in mechanical properties under cryogenic treatment. Improvement in mechanical properties was observed in cryogenic treated hemp composite due to increased frictional bond in the composite interface. Thermal conductivity of panels was measured using guarded hot plate method and the results shows decrease in thermal conductivity after cryogenic treatment. Water absorption rate of the fiber increases with time due to hygroscopic nature of fiber.Cryogenic treated hemp fiber shows 12% reduction in percentage of water absorption compared to untreated hemp fiber. These findings indicate hemp fabric as an alternative bio-degradable material with greater thermal stability and physical qualities than synthetic materials for engineering applications.

Keywords: Cryogenic treatment, hemp fiber, thermal properties, mechanical properties, acoustic properties, bio-composite material.

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CHAPTER 1

INTRODUCTION

1.1 REQUIREMENT OF THERMAL INSULATION

Thermal insulation plays a crucial role in maintaining a comfortable and hygienic indoor climate in buildings and homes. It achieves this by impeding the flow of heat, which helps to keep the indoor temperature cool during high ambient temperature conditions and warm during low ambient temperature conditions. In addition to its applications in building and household construction, insulation is also widely used in various industries. For example, it helps prevent damage to articles caused by freezing or high temperatures and reduces heating and cooling costs.

Heat naturally flows from a high temperature body to a low temperature body in a unidirectional manner. Insulation helps to slow down this heat flow and maintain a more stable indoor temperature. However, while there is a significant amount of research being conducted to improve the thermal conductivity of polymer reinforced composites, less attention is given to improving their insulation capacity.

Thermal insulation has a wide range of applications in industries such as refrigeration and air conditioning, building insulation, thermo-flasks, food preservation, aerospace, and automotive industries, among others. Given its importance in various sectors, the development of new and innovative insulation materials and techniques remains an ongoing research focus.

1.2 OVERVIEW OF COMPOSITES

A composite is a combination of two or more materials consisting of two phases. One is called reinforcing phase which consists of fibres, sheets or particles that is embedded on another material called matrix phase. The materials used in matrix and reinforcing phases can be metal, ceramic or polymer. Composites generally have a reinforcing phase that is stronger than the continuous matrix phase and serve as the principal load carrying members. The matrix acts as a load transfer medium between fibers, and in less ideal cases where the loads are complex, the matrix may even have to bear loads transverse to the fiber axis. The matrix also serves to protect the fibers from environmental damage before, during and after composite processing. When designed properly,

the new combined material exhibits better strength than would each individual material.

Composites are used not only for their structural properties, but also for electrical, thermal, tribological, and environmental applications. The history of composites dates back to ancient times for construction applications where straw was mixed with mud to form a building material known as adobe. The straw provided the structure and strength, while the mud acted as a binder, holding the straw together in place.

1.3 MERITS OF COMPOSITES

1. A high performance for a given weight leading to fuel saving.
2. Excellent stiffness and strength to weight ratio can be achieved.
3. High creep resistance, high toughness.
4. It is easier to achieve smooth aerodynamic profiles for drag reduction. Complex curvatures parts can be made in one manufacturing process.
5. Part count is reduced.
6. Production cost is reduced.
7. Composites offer excellent resistance to corrosion and chemical attack.
8. Composites have good dimensional stability.

1.4COMPONENTS OF COMPOSITES

The composite materials in basic is the one which consists of two elements working together to obtain material properties that are different from the properties of the two elements on their own. In general, the composite consists of the bulk material ‘matrix’ and ‘reinforcement’ which is added primarily to increase the strength and stiffness of the matrix.

1.4.1 Matrix

Now a day’s polymer, metal, ceramic, thermosets and thermoplastics are widely used matrix materials which have been used for making composites. The matrix material should be tough and ductile in nature for serving the proper function of composite material. Accordingly different type of reinforcement is being used such as dispersions, particulates, bristle, discontinuous or continuous fibers. The matrix phase provide strength to the composite to resist compressive,

tensile, shear or fatigue loads. Thermosetting or thermoplastic resins are used as polymers in matrix material of composites. Thermoset resins are the polymers which once gets into shape, can’t be reshaped or remoulded. Some of them are mostly use in making composites such as epoxy, polyester, phenolic, vinyl ester, polyurethane, silicone, polyamide and polyamide-imide. Thermoplastic resins are the polymer compound which soften on heating and returns to its original solid state on cooling. Thermoplastic resins are the polymer compound which soften on heating and returns to its original solid state on cooling. Commonly used thermoplastic resins are PET, PVC, polyethylene, poly carbonate, vinyl, polypropylene, nylon.The primary task of a matrix is to hold the reinforcements together. The matrix isolates the reinforcements from one another so as to prevent abrasion and formation of new defects. The matrix should also transfer the stresses between the fibres, provide a barrier against the adverse environments. The selection of matrix material plays an important role in compressive and shear strength, processing of the composite material.

In this case, epoxy resin, a thermosetting polymer, is employed to create the composite. To perform efficiently at greater temperatures, epoxy resin is cured by adding a curing ingredient called hardener in a 2:1 ratio, 2 parts epoxy resin and 1 part hardener. It has excellent mechanical qualities, is hydrophobic in nature, and has a high glass transition temperature. It is critical to use the right ratio of epoxy and hardener to reap the most benefits from the characteristics. Fibre reinforced composites are becoming increasingly used in the manufacturing sector as a means of improving the material's qualities. It is utilised in the development of high-performance items that demand high strength while remaining lightweight, with important applications in automobiles, aeroplanes, sports, medical equipment, household appliances, and industries.

1.4.2 Reinforcement

The purpose of reinforcement is to improve the system's overall mechanical characteristics, including strength, stiffness, and failure resistance. They take the shape of fibres, lamellae, whiskers, or a mesh. Heat and electricity can be conducted or insulated using reinforcement. For the qualities required for a particular application, the choice of reinforcement is crucial. The orientation of the reinforcement is crucial for getting the desired characteristics. Metallic whiskers, carbon, ceramic, natural, glass, metallic, oxides, carbides, and nitrides are often utilised reinforcing materials.

1.5 CLASSIFICATION OF COMPOSITES

The composites are classified mainly based on the type of matrix and also on the type of reinforcement used.

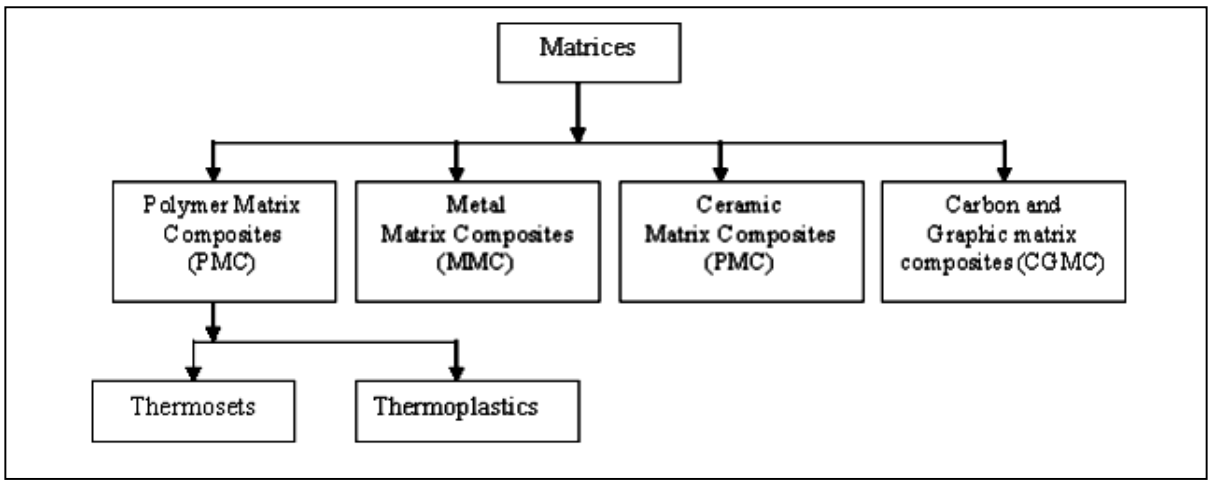

Figure-1.1 Classification of composite materials based on matrix

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

1.5.1 Metal matrix composites:

Metal matrix composites have made great progress in exploiting the required qualities of metals and their alloys as compared to traditional alloying and heat treatment processes. These composites are capable of achieving qualities that are impossible to achieve in single alloys. These reinforcement matrices can be continuous or discontinuous and made up of particles, fibres, or whiskers. Powder metallurgy and melting metal are two processes that embed ceramic particles in metal matrix to improve strength, wear resistance, thermal resistance, and coefficient of thermal expansion.

1.5.2 Ceramic matrix composites:

Due to the numerous advantages of ceramics matrix, such as its light weight, hardness, resistance to corrosion, and high hot hardness, these composites can withstand higher temperatures than any other composites. The most widely used ceramic matrices include oxides, carbides, nitrides, borides, glasses, and silicates. The reinforcements employed are SiC, Si3 N4, Al2 O3, BN, ZrO2, AIN, and C in the form of fibres, whiskers, or particles. Sintering, hot squeezing, hot isostatic squeezing, penetration, compound holding, and ignition are some of the unique processes that are used to create these composites. At the moment, adding cutting devices, wearable composites, space transport tiles, and aircraft components is the real use of creative framework composites. Bio-Earthenware and high temperature ceramic super conduit composite wires for force transmission connections, engines and super leading attractive energy storage systems are further potential applications.

1.5.3 Polymer matrix composites

Polymer matrices are the most preferred matrix materials over all others. When polymers are reinforced with fillers, their strength, thermal strength, and stiffness are greatly increased. The conductivity or insulating qualities of the composite can vary depending on the filler material utilised in the polymer matrix. The popularity of polymer composite stems from its simple construction technique, which is less expensive than any other matrix and does not necessitate highly skilled technicians. Polymer composites are commonly utilised because they outperform any individual polymer in terms of overall qualities. Because of their high ductility or modulus of elasticity, these composites can be easily manufactured without the need of high pressure, temperature, or energy.

1.6 TYPES OF POLYMER MATRIX COMPOSITES

Polymer composites are broadly categorized into two classes based on the reinforcing material.
They are as follows:

- Fibre reinforced polymer
- Particle reinforced polymer

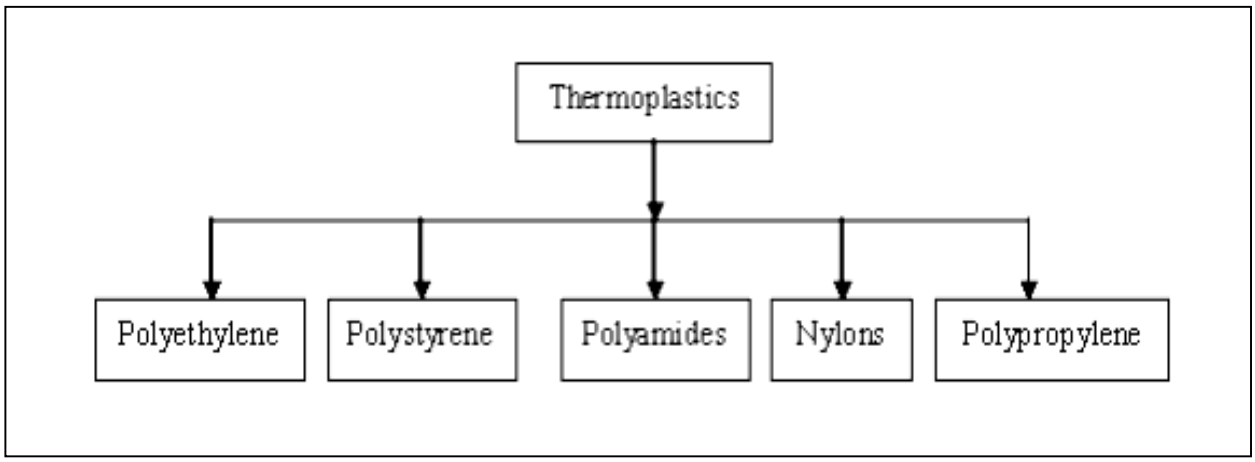

Figure-1.2 Classification of polymer composite materials based on matrix

Polymer is defined as a long chain molecule containing one or more repeating units of atoms, joined together by strong covalent bonds. A polymeric material is a collection of large number of polymer molecules of similar chemical structure but not of equal length.

Polymers are divided into two broad categories: 1.thermoplastics and 2.thermosets.

In a thermoplastic polymer, the individual molecules are not chemically joined together. They are held in place by weak secondary bonds or intermolecular forces, such as Van der Waals bonds and hydrogen bonds. With the heat applied these weak forces can be broken temporarily and the molecules can be moved relative to each other to desired shapes. On cooling the molecules restore their bonds, resulting in new shape. Thus, a thermoplastic polymer can be heat softened, melted and reshaped as many times as desired. In a thermoset polymer, the molecules are chemically joined together by cross links, forming a rigid, and three dimensional network structures. Once these cross links are formed during the polymerization reaction, the thermoset polymer cannot be melted by the application of heat. PMCs are very popular due to their low cost and simple fabrication methods. Reinforcement of polymers by strong fibrous network permits fabrication of PMCs, which is characterized by the following:

a) High specific strength
b) High specific stiffness

c) High fracture resistance

d) Good abrasion and corrosion resistance

e) Good impact resistance

f) Good fatigue resistance

h) Low cost

1.7 CLASSIFICATION BASED ON REINFORCEMENT

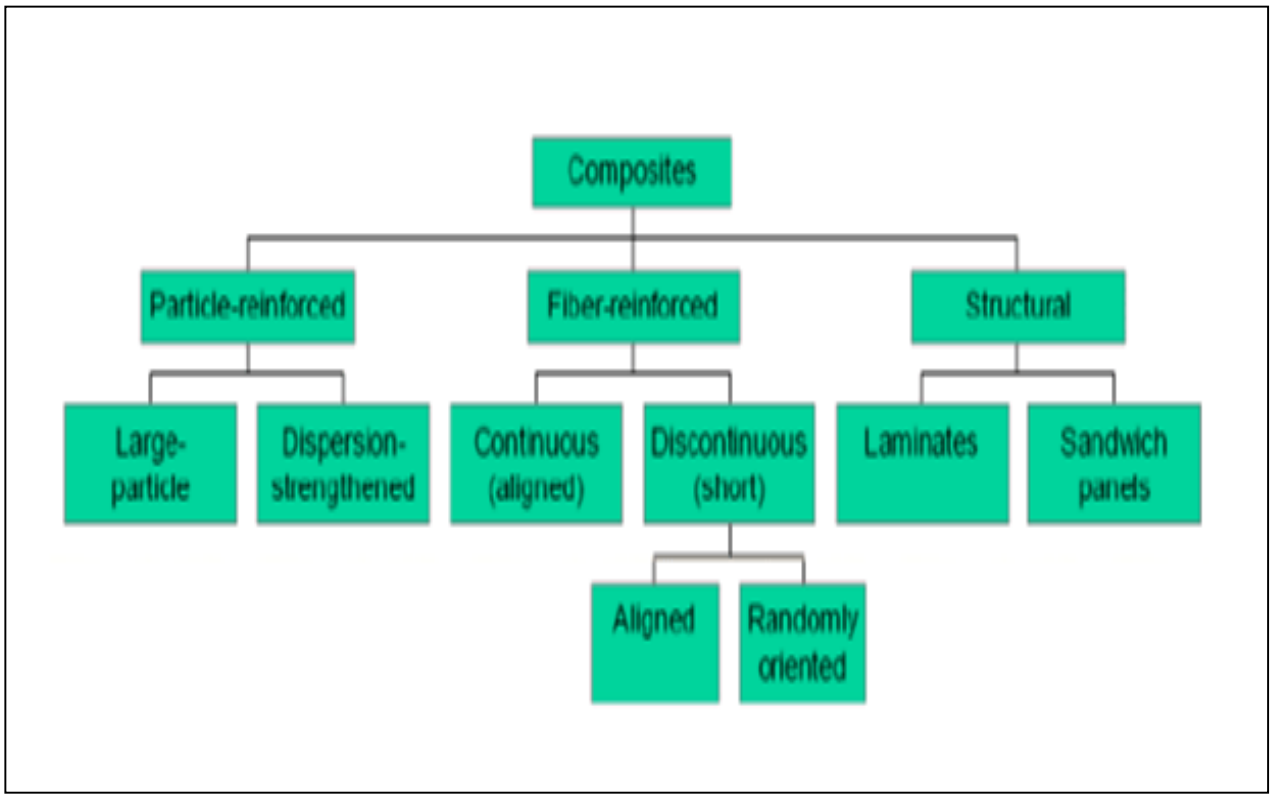

Figure-1.3 Classification of composite materials based on reinforcement

1.7.1 Fibrous composites

A fibre is characterised by its length much greater when compared to its cross sectional dimensions. The dimensions of the reinforcements determine the properties of the composites. Fibre reinforced composites have high fracture resistance compared to matrix material since fibres discourages the growth of cracks normal to the surfaces. The fibres can be arranged randomly or in

a preferred orientation depending upon its needs. Depending upon the length of the fibre used it is classified into short fibre reinforced and long fibre reinforced composites. When fibres are reinforced or implanted in a polymer matrix, they form a composite that is far stronger than a single component. Fibre reinforcement materials include glass, carbon, molybdenum, beryllium, beryllium carbide, beryllium oxide, and natural fibres such as bagasse, jute, banana, paper, wood, or asbestos. Fibre reinforced polymer composites have a wide range of applications, including aerospace, automotive, marine, and construction. The primary source of strength is fibre reinforcement, whereas the matrix holds all of the fibres together in perfect shape and distributes stresses among the reinforcing fibres. The loads are transmitted lengthwise by the fibres. In some circumstances, fillers may be added to ease the fabrication process and reduce manufacturing costs. Common matrix materials used now a days are epoxy, phenolic resin, polyester, polyurethane, vinyl ester etc. Among all these matrix materials, epoxy is generally used because of its higher adherence and less contraction than polyesters.

1.7.2 Particulate composites

In particulate composites the reinforcement is of particle nature. It may be spherical, cubic, tetragonal, a platelet, or of other regular or irregular shape. In general, particles are not very effective in improving fracture resistance but they enhance the stiffness of the composite to a limited extent. Particulate composites have an additional substance element that is only a handful of dimensions and naturally visible/minuscule. However, in a few composites, the added substance constituent is visibly non-dimensional, i.e., theoretically a point rather than a line or zone. Only on the microscopic sizes does it become dimensional, i.e., a molecule, and hence the concept of composite must descend down to the tiny level if it is to embrace all the composite of architects' excitement. Particulate composites differ from fibre chip types in that the conveyance of the added material element is often erratic rather than controlled. As a result, particle composites are often isotropic. This composite category includes scattered solidified compound and cermet. Only on the smallest scales does it become dimensional, i.e., a molecule, and hence the concept of composite must be reduced to the smallest level in order to incorporate all of the designers' passion. Particulate composites differ from fibre chip types in that the distribution of the additional substance element is often erratic rather than regulated. Particulate composites are thus typically isotropic. This composite category includes scattered solidified amalgam and cermet.

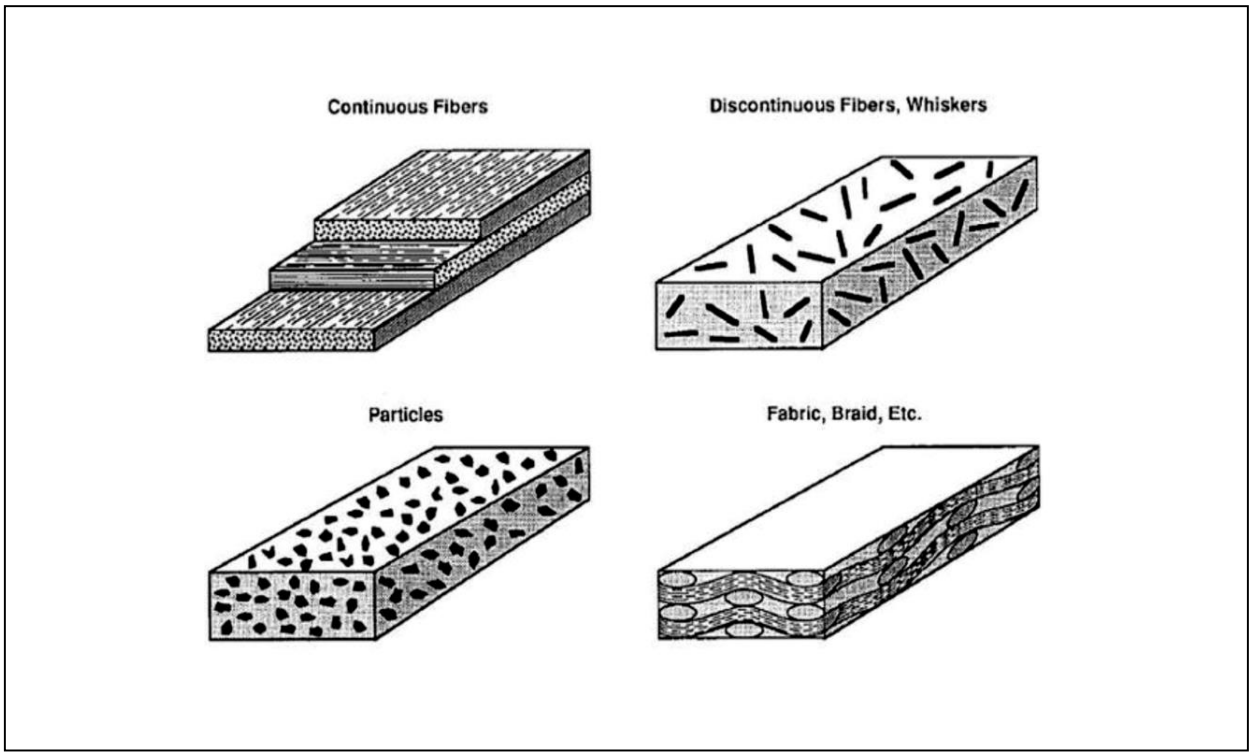

Fig.1.4. Classification of composite materials based on reinforcement

1.8 CRYOGENIC TREATMENT

Cryogenics is defined as the fields of physics and engineering that investigate extremely low temperatures, how they are produced, and how materials behave at such temperatures. Cryogenicists employ the Kelvin and Rankine scales rather than the common Fahrenheit and Celsius temperature scales. The term cryogenics technically means "the production of icy cold"; nonetheless, it is now used as a synonym for the low-temperature state. It is unclear where on the temperature scale refrigeration ends and cryogenics begins. Workers at the National Institute of Standards and Technology in Boulder, Colorado, have chosen to define cryogenics as temperatures below -180 °C (93.15 K). This is a reasonable dividing line because the usual boiling temperatures of so-called permanent gases (such as helium, hydrogen, neon, nitrogen, oxygen, and normal air) are less than -180 °C, whereas the boiling points of Freon refrigerants, hydrogen sulphide, and other common refrigerants are greater than -180 °C. Cryogenic treatment is a one-time permanent treatment technique that affects the entire cross-section of the material. It is often performed after

the end of the standard heat treatment process but prior to tempering. It is thus not a replacement technique, but rather an addition to the normal heat treatment method. It is thought to improve wear resistance, surface hardness, and thermal stability in a variety of materials. Many cryogenic applications make use of liquefied gases such as liquid nitrogen and liquid helium. The most often used ingredient in cryogenics is liquid nitrogen, which is legally available all throughout the world. Liquid helium is also often used and allows for the lowest temperatures possible. These gases are stored in either Dewar flasks, which are about six feet tall (1.8 m) and three feet (91.5 cm) in diameter, or enormous tanks in bigger commercial operations. Cryogenic transfer pumps are the pumps used on LNG piers to transfer LNG from LNG carriers to LNG storage tanks. The liquid nitrogen produced by the nitogen plant is kept in storage tanks.It is directed through a nozzle to a closed vacuum evacuated chamber known as a cryogenic freezer using transfer lines.The delivery of liquid nitrogen into the cryo-freezer is controlled by soleniod valves.Inside the chamber, the temperature gradually drops from room temperature to -196o C at a rate of 2o C/min.When the temperature drops below zero, the specimens are transferred to the nitrogen chamber or soaking chamber, where they are stored for 24 hours with a constant supply of liquid nitrogen.

1.9 OBJECTIVES

- To conduct cryogenic treatment on hemp fiber.
- To fabricate treated hemp fiber reinforced composite panels using epoxy.
- To study the thermal degradation of the fabricated composite.
- To characterize the thermal insulation property of the fabricated composites.
- To study the effect of cryogenic treatment on mechanical properties of fabricated composites.
- To study effect of thermal treatment on moisture absorption in hemp fiber composite panels.

CHAPTER 2

LITERATURE SURVEY

In this chapter, an overview of the survey made on past research which has already available has been presented. The main objective of this literature survey is to present the past research on fiber reinforced polymer composites. It includes the work on mechanical and thermal behavior as well as cryogenic treatment of the fiber reinforced polymer composites and thereby calculating the performance of the polymer composite. It includes the following available research domain:

Elisa Morett et al. [1] focused on development of Mineral fiber (Basalt fiber) insulating panels and investigated thermal properties of basalt fiber panel and compared them with conventional solutions. The panel shows excellent thermal properties: thermal conductivity values are 0.031-0.032 W/(m K) range, depending on the density. Gowtham et al. [2] thermal properties of an innovative insulation system composed of the natural fibers (sisal, jute, flax) were investigated .The eco-friendly panel has a thermal conductivity values between 0.078-0.085W/(m.K) range, lower than other panels. Yiqin Shao et al. [3] investigated the interfacial shearing strength and it was found to be 31% higher than the pristine CNT fiber composite .Mechanical results showed that the strength of CNT fiber was not affected significantly but the tensile strength of the epoxy was increased by 27% after cryogenic treatment. Richard Butler et al. [4] shows Cryogenic temperature has a positive effect on composite and also results in improvement in strength ,modulus, fatigue and thermal properties Contrarily it cause a reduction in ductility, fracture toughness and impact resistance .Wei Chen et al.[5]studied about two types of CNT composite yarns by twisting CNT films with thermoset epoxy (EP) and thermoplastic poly vinyl alcohol (PVA) resins and after cryogenic treatment, the mechanical and electrical properties of CNT/EP and CNT/PVA composite yarns were both enhanced due to improvement of interfacial bonding. Yinnan Zhang et al. [6] investigate the impact of cryogenic treatments on the characteristics and micro-structures of carbon fibres, polyacrylonitrile-based carbon fibers are cryogenically conditioned at both a low cooling rate and a quench rate. The inter-planar distance increases in the fiber axial direction and decreases in the fiber radial direction as a result of slow cooling cryogenic treatment, resulting in a 3% reduction in fiber diameter. Microscopy analysis

reveals slightly larger and deeper rill-like pleats and a 41% increase in fiber surface roughness, resulting in a 30.2% increase in the interfacial shear strength between the fibre and epoxy. After sharp cooling cryogenic treatment, epoxy inter-planar distance, fibre diameter, surface morphology, and interfacial shear strength do not change. Fujun Xu [7] the effects of cryogenic treatment on Kevlar fiber at various cooling rates were studied. Cryogenic treatment of Kevlar fiber improved its tensile, wear, and interfacial shear strength (IFSS) capabilities. Sharp cooling rate conditioning enhanced the tensile strength of the aramid fibre by 24.9%, while low cooling rate conditioning increased it by only a small amount. Furthermore, sharp and low cooling treatments respectively resulted in a 51.6% and 51.1% increase in the fiber's abrasion duration. . Punyapriya Mishra [8] mechanical behavior of epoxy composites reinforced with bagasse fibers at cryogenic temperatures is the subject of this study. Composites were prepared by reinforcing epoxy matrix with bagasse fibres at 10, 15, and 20 wt%. After being cryogenically conditioned, samples have greater flexural strength than those that have been left at room temperature. This is due to contraction of epoxy matrix at low temperatures, creating compressive stresses that improve the function at the interface and account for the greater flexural strength value seen for cryogenically conditioned samples. Sair S. et. al [9] prepared partially biodegradable green composites by including natural hemp fiber as reinforcement. At various loading rates in (H.F), composites of rigid polyurethane (PU) and hemp fiber (H.F) (5%, 10%, 15%, 20%, 25%, and 30%) were created. The effect of fiber content on the composite's thermal conductivity, was studied. A linear relationship between density and heat conductivity is observed for composites. Thakare, Pravin A. et al. [10] thermal characteristics of hybrid FRPs incorporating jute, flax, sisal, and hemp were analyzed. The thermal conductivity of all the panels was less than 1 W/mK, with the flax and hemp panels exhibiting the lowest conductivity at roughly 0.45 W/mK. All panels are observed maintained their thermal properties up to a temperature of 200 degrees C. When compared to H-FRP, which degrades thermally in two stages at around 260 C and 410 C with the wt. loss of 53.72% and 26.79%, respectively. T. Behzad et al.[11] The steady-state temperature decline of samples exposed to a known heat flux was utilized to study hemp fiber reinforced polymer composites. The orientation of fibers was found to have a significant effect on the thermal conductivity of composites. Waikamboo and Ansell [12] evaluated the serviceability of normal fiber filler composites by assessing their physio-mechanical properties. Treated filaments of the highest quality were used as reinforcement for a cashew nutshell fluid grid, and the ductile

characteristics, porosity, and break surface morphology of the composites were determined. The purpose was to broaden the composite's assessment of easy regular assets. They hypothesised that the presence of lignin in untreated hemp fibre provides more cross-connecting destinations, and that the untreated fibre surface is more compatible with CNSL (Cashew Nut Shell Liquid pitch) than the antacid surface. Yongli [13] investigated the mechanical properties of unidirectional flax and glass fiber reinforced cross breed composites, with a focus on the mixed impacts of composites generated from natural and synthetic filaments. Huang et al. [14] investigated how water retention affects the mechanical characteristics of glass/polyester composites. It was discovered that the breaking quality and malleability of the composites decreased gradually with increased water inundation time because of the weakening of holding in the midst of fiber and framework. Li, Xue et al [15] studied natural fibers in fiber-reinforced composites and observed poor compatibility between the fiber and matrix and high moisture sorption limit their effectiveness. To improve the fiber surface properties, chemical treatments such as alkali, silane, acetylation, benzoylation, and others have been studied. These treatments can enhance adhesion between the fiber and matrix, increase fiber strength, and reduce water absorption, leading to improved mechanical properties of the composites. J. Liu et al [16] this paper proposes a new degumming method for hemp fibers, which involves cryogenic and mechanical treatments followed by alkaline cleaning. The cryogenic treatment caused some micropore creation and microcracking, while the mechanical treatment separated the fibers from the bundles. Scanning electron microscopy, Fourier transform infrared spectroscopy, and thermogravimetric analysis were used to investigate the effects of the treatments on the fibers. The cellulose content increased, while hemicellulose and lignin decreased. The treated fibers had a higher thermal stability than the untreated fibers, as evidenced by differential thermogravimetric analysis. The study concludes that cryogenic and mechanical treatment is a feasible method for degumming hemp fibers. Bledzki AK et al [17] this study investigated the effects of various mercerization parameters, such as alkali concentration, temperature, duration time, and tensile stress, on the structure and properties of hemp fibers. The study found that mechanical properties of the fibers could be adjusted by using appropriate mercerization parameters. The researchers manufactured unidirectional EP composites using filament winding and film-stacking techniques with a PP matrix material, and studied the influence of mercerization parameters on the properties of the composites using hemp yarn as an example. The study showed different macromechanical effects in hemp- and flax-PP model

composites with mercerized, MAH-PP-treated, or MAH-PP-treated mercerized yarns.

Bettahally et al [18] this study investigates the mechanical properties of hybrid hemp and jute fiber reinforced epoxy composites under both room temperature and cryogenic temperature. The effects of cryogenic treatment duration (15, 30, 45, and 60 minutes) on tensile, flexural, and impact properties were studied. Results showed that cryogenic treatment significantly affected the properties of the composites, with the maximum tensile strength of 21.13 MPa, flexural strength of 51.95 MPa, and impact strength of 8.935 kJ/m² obtained for untreated specimens. The material becomes harder and more brittle at cryogenic temperature, losing its ductile property due to the difference in thermal expansion coefficient between the matrix and fiber material. Ouajai et al [19] In this study, the thermal degradation behavior of hemp fibers was investigated using thermogravimetry. The activation energy of treated fibers was calculated from the thermogravimetry data and found to be greater than that of untreated fibers, indicating an increase in purity and improvement in structural order. Non-cellulosic components were removed from the fibers by mercerization and enzyme scouring, but structural disruption was observed at higher alkaline concentration and longer scouring time. The FTIR results showed the elimination of non-cellulosic components by mercerization and a specific removal of low methoxy pectin by pectate lyase enzyme. The study also found that an increase in temperature at the maximum rate of degradation and the rate of weight loss was characteristic of the purity and structure of treated hemp fibers.

CHAPTER 3

MATERIALS AND METHODS

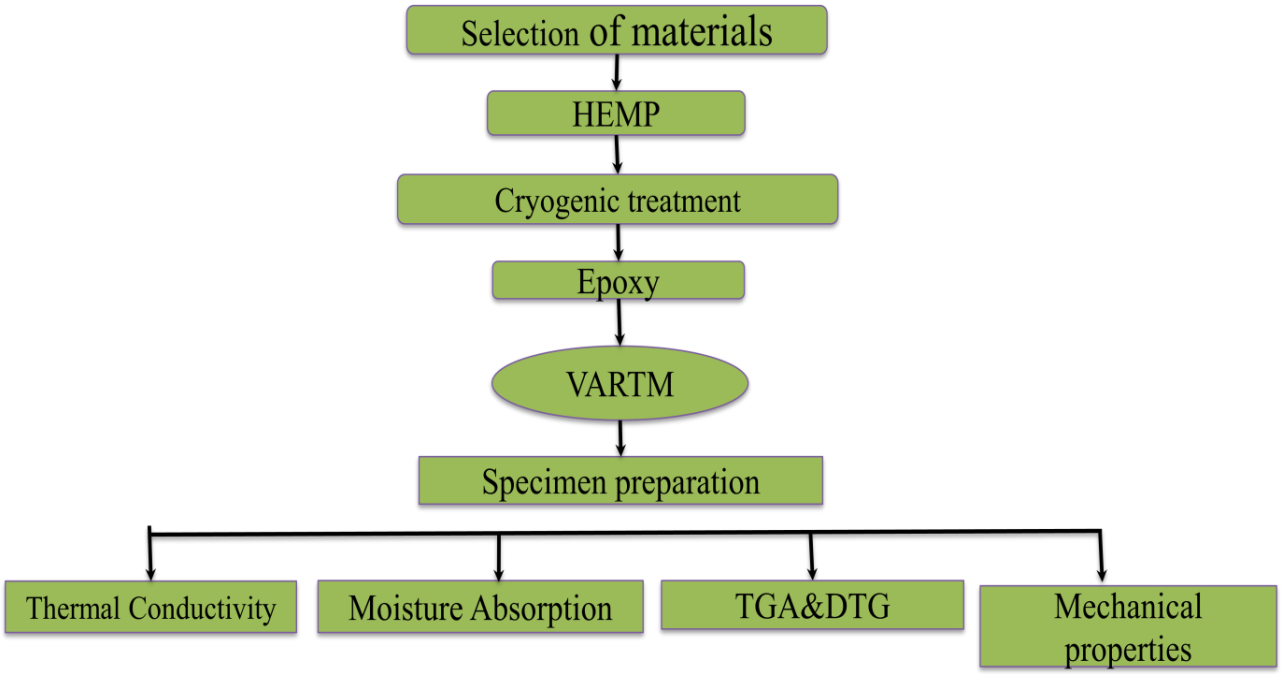

Figure- 3.1 Flow chart of methodology

3.1 MATERIALS AND METHODOLOGY

The thermoset, DGEBA (Diglycidyl ether of bisphenol A) based polymer matrix is used for the present study. The curing agent used is Modified cycloaliphatic amine. The process involves the use of a vacuum to facilitate resin flow into a fiber layup contained within a mold tool covered by a vacuum bag. After the impregnation occurs the composite part is allowed to cure at room temperature. HEMP fiber used for fabrication of panels is of dimension 30cm x 20cm and it consisting of 8 layers. Resin for the composite is prepared by mixing epoxy (300g) and hardener (150 g) in ratio 2:1.

Table-3.1 Materials Used

Hemp fiber (170 GSM)	Cellulose-75-77%, Hemicellulose-10%, Lignin-6.8%, Pectin -2.9%, Waxes-0.90%
Epoxy (DGEBA)	Density (gm/cc)-1.1, Compressive strength (MPa)-90, Tensile strength (MPa)-58, Glass transition temperature (°C)-104
Hardener (Modified cycloaliphatic amine)	Viscosity:250-500 centipoise (cP) at 25°C. Pot life-30-120 minutes Curing time-75h

3.1.1 EPOXY (DGEBA)

Epoxy (DGEBA) is a type of thermosetting polymer that is widely used in various industries for its excellent mechanical and adhesive properties. The full name of this polymer is Diglycidyl Ether of Bisphenol A (DGEBA), and it is made by reacting bisphenol A with epichlorohydrin. The resulting polymer is a clear, colorless, and viscous liquid that can be cured into a hard and durable solid by the addition of a curing agent such as amines or anhydrides. Epoxy (DGEBA) resins are commonly used as adhesives, coatings, and matrix materials for composites due to their excellent adhesion, chemical resistance, and mechanical properties. They can also be used as electrical insulators and are widely used in the electronics industry. Epoxy (DGEBA) resins are also used as a binder in the manufacture of high-performance laminates, such as printed circuit boards and aerospace components. One of the major advantages of epoxy (DGEBA) resins is their ability to form strong bonds with a wide range of materials, including metals, plastics, and composites. They also have excellent chemical resistance and can withstand exposure to harsh chemicals, solvents,

and fuels. Additionally, epoxy (DGEBA) resins have good electrical insulation properties and can be formulated to have a range of mechanical

properties, from rigid and high-strength to flexible and tough. Epoxy is a popular matrix material for Fiber Reinforced Composites (FRC) due to several advantages it offers:

- High strength and stiffness: Epoxy is a rigid and high-strength material that can significantly enhance the strength and stiffness of fiber composites. This is particularly useful for FRC applications that require high structural performance, such as aerospace, automotive, and marine industries.
- Good adhesion to fibers: Epoxy has excellent adhesion to fibers, such as carbon, glass, or aramid, which helps to transfer the loads from the fibers to the matrix, resulting in a strong and durable composite material.
- Chemical and environmental resistance: Epoxy is highly resistant to chemical and environmental degradation, which makes it a suitable material for FRC applications in harsh and corrosive environments.
- Versatile: Epoxy can be easily modified to achieve a wide range of mechanical, thermal, and electrical properties, making it a versatile matrix material for FRC.
- Low shrinkage: Epoxy has low shrinkage during curing, which helps to maintain the integrity and dimensional stability of the FRC component.

Figure - 3.2 Epoxy resin and hardener

3.1.2 HEMP FIBER

Hemp fiber (170 GSM) refers to a type of hemp fabric that weighs 170 grams per square meter. Hemp is a natural fiber derived from the stem of the Cannabis sativa plant, and it has been used for in various applications, including clothing, textiles, and paper. Hemp fiber is known for its excellent strength, durability, and breathability. It is also naturally resistant to mildew, bacteria, and UV light, making it a suitable material for outdoor and high-moisture environments. Additionally, hemp is an eco-friendly and sustainable fiber, as it requires minimal water and pesticides to grow, and it is biodegradable and compostable. The 170 GSM weight of hemp fiber fabric refers to the density of the fabric, with higher GSM indicating a thicker and more substantial fabric. A fabric with 170 GSM would be considered a mid-weight fabric, suitable for a variety of applications, including clothing, upholstery, and bags. Hemp fiber has several advantages over other natural fibers, including:

- Strength and durability: Hemp fiber is one of the strongest natural fibers, with a high tensile strength and excellent durability. It is stronger than cotton, linen, and most other natural fibers, which makes it ideal for use in products that require high strength and durability.
- Environmental sustainability: Hemp is a highly sustainable crop that requires less water and pesticides to grow than other natural fibers like cotton. It also has a lower carbon footprint and can be grown in a wide range of climates.
- Versatility: Hemp fiber can be used to make a variety of products, including clothing, textiles, paper, building materials, and more. It can be blended with other fibers to create fabrics with different properties.
- Breathability: Hemp fiber is naturally breathable, making it comfortable to wear in hot and humid conditions. It also wicks moisture away from the skin, which helps to regulate body temperature.
- UV resistance: Hemp fiber is naturally resistant to UV light, which makes it ideal for use in outdoor products.
- Antimicrobial properties: Hemp fiber has natural antimicrobial properties, which makes it resistant to mildew and bacteria growth. This makes it ideal for use in products that are exposed to moisture and humidity.

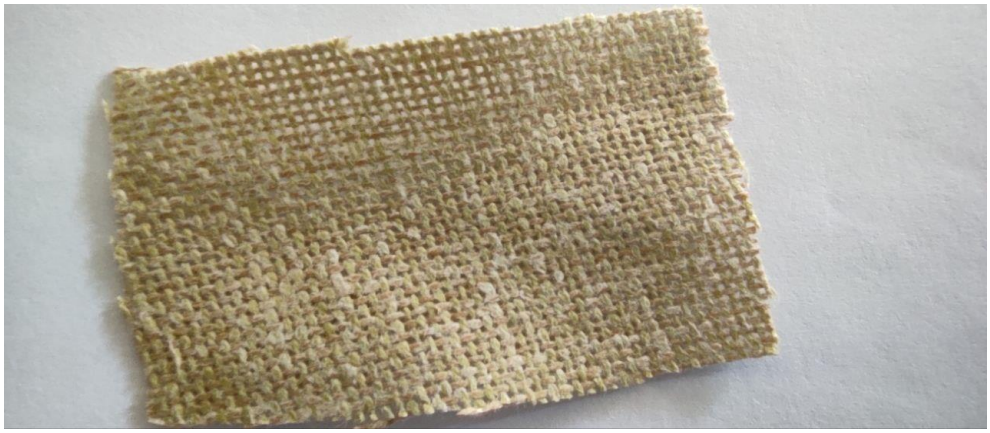

Figure-3.3 Hemp fiber

3.1.3 HARDENER (MODIFIED CYCLOALIPHATIC AMINE)

Modified cycloaliphatic amine is a type of hardener used in epoxy resin systems to catalyze the curing process. Cycloaliphatic amines are cyclic organic compounds that contain an amine group, and when they are modified, their properties are optimized for use as an epoxy hardener.

Modified cycloaliphatic amine hardeners are preferred over other types of hardeners, such as aliphatic or aromatic amines, for several reasons. Firstly, they have a relatively low viscosity and are easy to mix with epoxy resins, which allows for efficient wetting of the reinforcing fibers in composite materials. Secondly, they provide a high degree of toughness and impact resistance to the cured epoxy matrix, making it ideal for applications that require high mechanical strength and durability. Some of the other advantages of modified cycloaliphatic amine hardeners include, excellent chemical resistance: Modified cycloaliphatic amine hardeners provide excellent chemical resistance to the cured epoxy matrix, making it suitable for use in harsh chemical environments. Good adhesion: Modified cycloaliphatic amine hardeners exhibit good adhesion to a wide range of substrates, including metals, plastics, and composites. Low toxicity: Modified cycloaliphatic amine hardeners have low toxicity and are not harmful to human health or the environment, which makes them an ideal choice for applications where safety is a concern.

Low shrinkage: Modified cycloaliphatic amine hardeners have low shrinkage during the curing process, which minimizes the risk of delamination or cracking in composite materials. Modified cycloaliphatic amine hardeners are a p opular choice for epoxy resin systems due to their excellent

mechanical properties, chemical resistance, adhesion, low toxicity, and low shrinkage.

3.2 FABRICATION METHOD

3.2.1 VACUUM ASSISTED RESIGN TRANSFER MOLDING (VARTM)

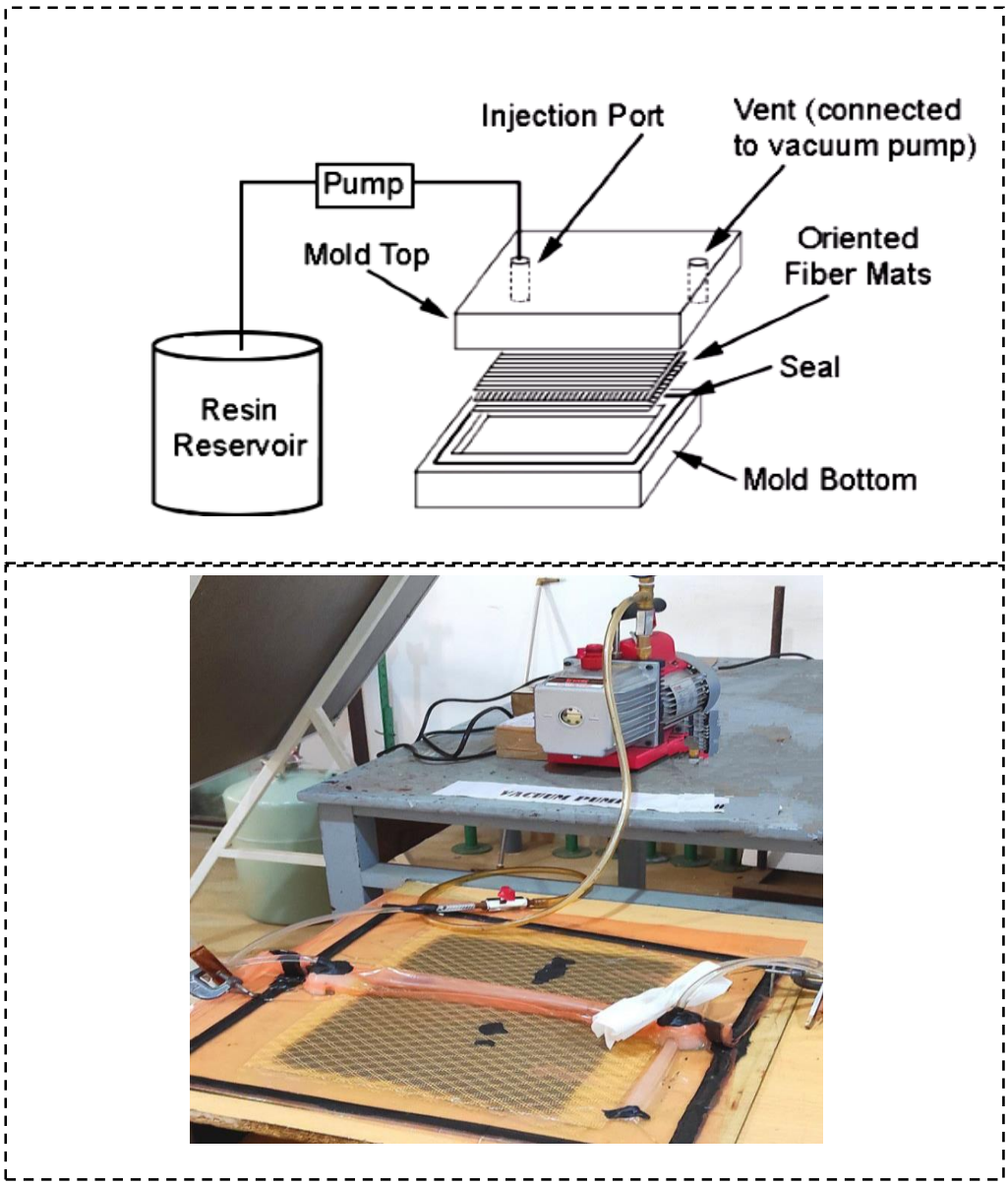

Figure- 3.4 Vacuum assisted resign transfer molding

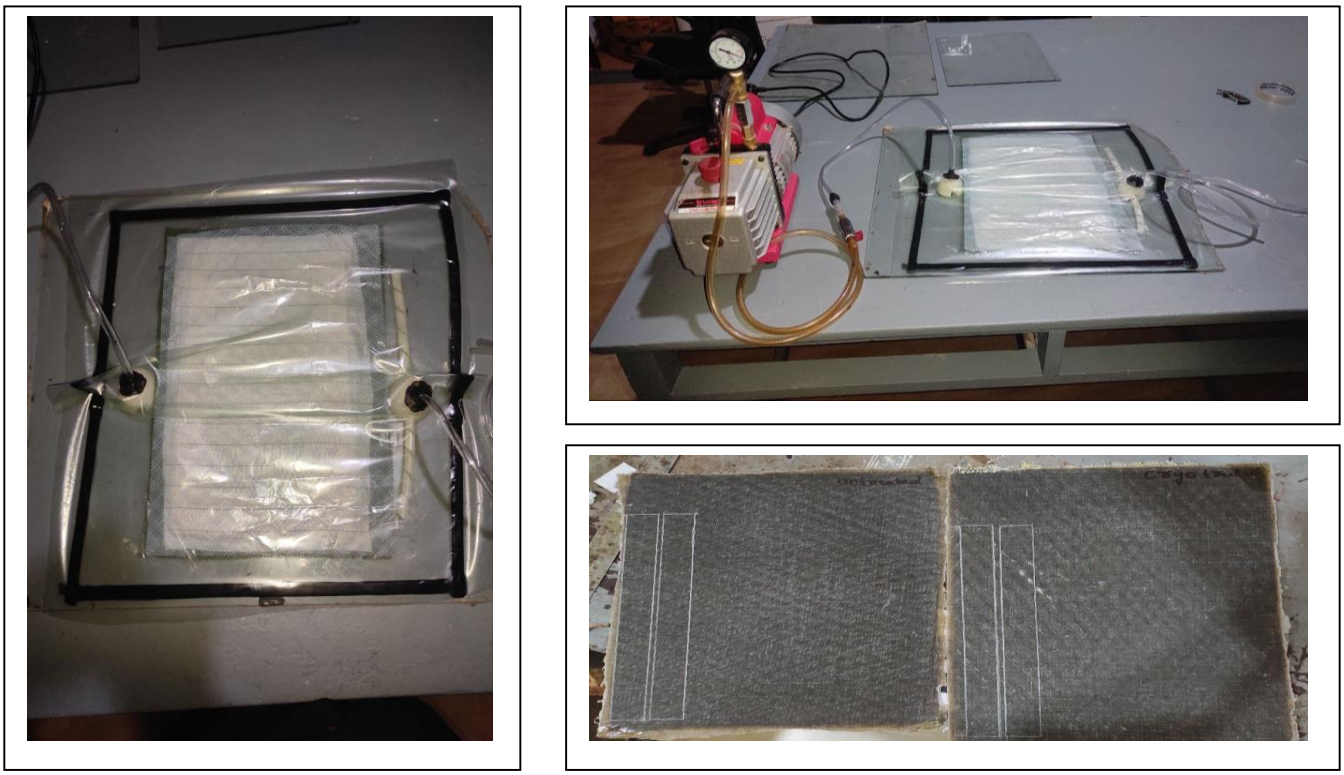

Figure-3.4 VARTM set up

Vacuum Assisted Resin Transfer Molding, which is a composite manufacturing process used to fabricate high-quality composite parts. It is a closed-mold process that involves the injection of resin into a perform of reinforcing fibersunder vacuum pressure.

The advantages of VARTM process include:

- Reduced material waste: The VARTM process allows for precise control of resin flow, which minimizes material waste and reduces overall production costs.
- Improved part quality: The VARTM process can produce high-quality composite parts with consistent fiber volume fraction and low void content.
- Flexibility: The VARTM process can be used to fabricate complex shapes and geometries, making it suitable for a wide range of applications.
- Low cost: The VARTM process is relatively low cost compared to other closed-mold processes like RTM or autoclave molding.

The VARTM process involves several steps:

- Preform preparation: The reinforcing fibers (Hemp fibers) are placed into a mold in a predetermined pattern to create the preform. The preform is typically dry and compacted to minimize voids and ensure consistent resin flow.
- Resin injection: The mold is sealed, and a vacuum is applied to the mold cavity to evacuate any trapped air. The epoxy resin is then injected into the mold cavity through a small hole or network of channels, under vacuum pressure, to ensure even distribution of the resin.
- Cure: The epoxy resin is then allowed to cure in the mold cavity, which is typically done at room temperature. The curing process can be accelerated by heating the mold or using a heated tool to apply pressure.
- Demolding: Once the resin has cured, the mold is opened, and the part is removed. Any excess material is trimmed, and the part is inspected for quality.

3.2.2 CRYOGENIC TREATMENT

Cryogenic treatment is a type of heat treatment applied to materials at low temperatures. Here deep cryogenic treatment is conducted on the hemp fiber with an immersion time duration of 60 minutes. Liquid nitrogen (N2) is the liquid coolant used for the treatment. The coolant is stored at 77K inside a partially thermal insulated box and the fiber is immersed in it.

Cryogenic treatment of hemp fiber involves subjecting the fibers to extremely low temperatures, typically below -150°C (-238°F), in order to improve their mechanical properties and durability. The process is often used in conjunction with other treatments, such as chemical and mechanical processing, to enhance the overall performance of the fiber.

The primary benefit of cryogenic treatment is that it helps to reduce the moisture content of the hemp fiber, which can improve its strength and durability. This is because water molecules can weaken the bonds between the individual fibers, making them more prone to breaking under stress. In addition to reducing moisture content, cryogenic treatment can also help to reduce the amount of impurities in the hemp fiber, such as waxes and lignin. This can further enhance its mechanical properties and make it more suitable for use in a variety of applications, such as textiles, composites, and building materials. Cryogenic treatment is a promising technology for improving the properties of hemp fiber and increasing its potential as a sustainable and versatile material.

However, more research is needed to fully understand the effects of this treatment on the fiber and to optimize the process for specific applications.

Figure-3.5 Cryogenic treatment of Hemp fiber

3.3 CHARACTERIZATION

3.3.1 TENSILE STRENGTH TESTING

Tensile testing involves measuring the mechanical properties of the composite material, such as its strength, stiffness, and elongation, under tensile stress. The procedure for conducting a tensile test on hemp fiber reinforced composites is similar to that of other composite materials, but there are some specific considerations that must be taken into account due to the unique properties of natural fibers, the steps involved in conducting a tensile test on hemp fiber reinforced composite:

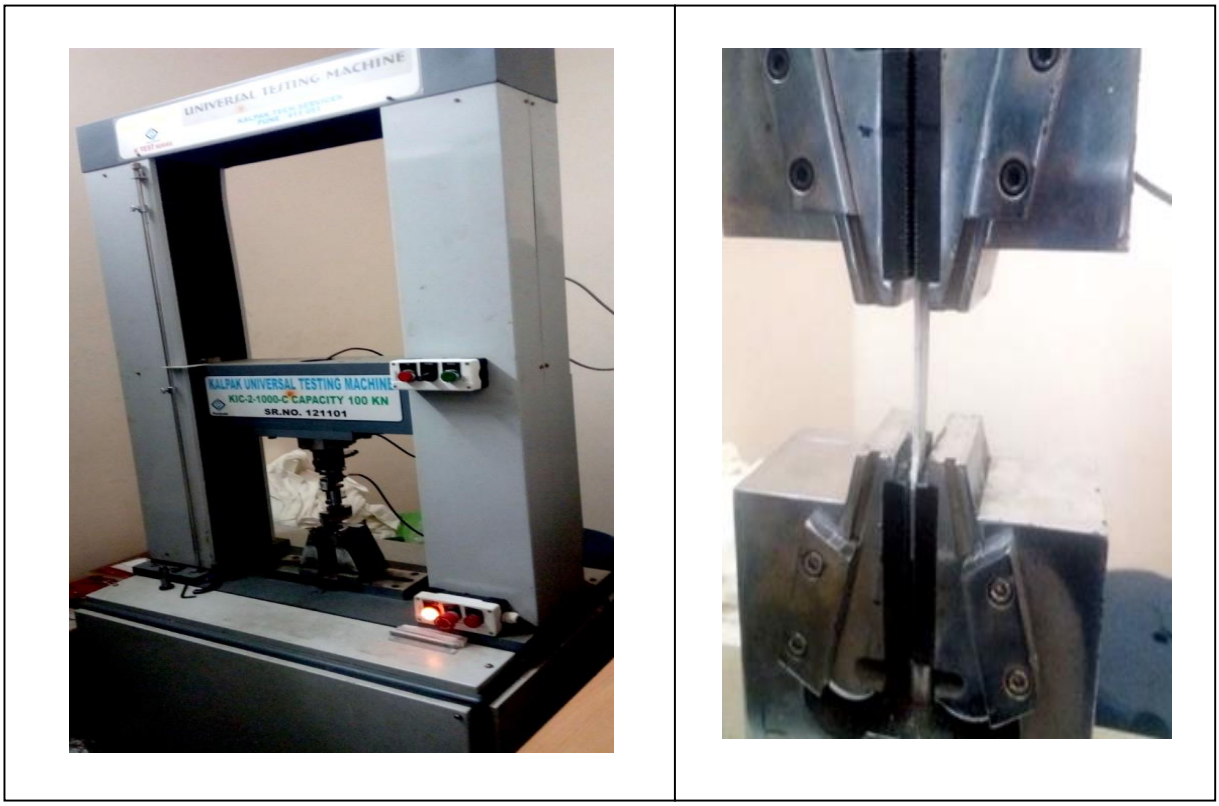

Figure-3.6 Tensile testing of sample

- Sample preparation: Cut a rectangular sample of the composite material using a precision saw or cutter. The dimensions of the sample should be in accordance with the testing standard ASTM 3039 (250 x 25 x 2.5 mm).

- Mounting the sample: Mount the sample on the tensile testing machine with the help of grips. The grips should be carefully aligned with the long axis of the sample to ensure an even distribution of force during testing.
- Preload: Apply a small amount of force to the sample to ensure it is properly seated in the grips and to remove any slack.
- Testing: Apply a continuous tensile load to the sample until it breaks. The load and displacement are recorded throughout the test to generate a stress-strain curve.
- Analysis: Use the stress-strain curve to calculate the mechanical properties of the material, such as its tensile strength, modulus of elasticity, and elongation at break.

To calculate stress and strain from load and displacement data obtained during a tensile test, you can use the following equations:

Stress (σ) = Load (F) / Cross-sectional area (A)

Strain (ε) = Change in length (ΔL) / Original length (L)

Where:

- Cross-sectional area (A) is the initial cross-sectional area of the sample perpendicular to the direction of the applied load.
- Change in length (ΔL) is the change in the length of the sample due to the applied load.
- Original length (L) is the original length of the sample before the load is applied.

Using these equations, you can plot the stress-strain curve for the material, which can provide valuable information about its mechanical properties, such as its elastic modulus, yield strength, and ultimate strength.

3.3.2 FLEXURAL STRENGTH TESTING

Flexural strength testing using the 3-point method is a common test method used to determine the strength and stiffness of a material under bending stress. This test involves placing a beam- shaped sample on two supports and applying a force at the center of the beam. The force causes the beam to bend, and the amount of deflection is measured. From these measurements, the flexural strength and stiffness of the material can be calculated.

The steps involved in conducting a flexural strength test using the 3-point method:

- Sample preparation: Cut a rectangular beam-shaped sample of the material to the required dimensions 125 mm×12 mm x 2.5 mm according to ASTM DD790 standard.
- Mounting the sample: Place the sample on two supports, positioned at a distance of 30 mm apart. The distance between the supports should be in accordance with the testing standards being used.
- Loading: An extension rate of 9 mm/min and load of 5 kN was used .The load is typically applied at a constant rate until the sample fractures or until a specified deflection is reached.
- Deflection measurement: Measure the deflection of the sample at the center of the span during loading. This can be done using a displacement transducer or a dial indicator.
- Calculation: Use the load and deflection data to calculate the flexural strength and stiffness of the material. The flexural strength is typically calculated using the following equation:

Flexural strength = (3FL)/ (2bd^2)

Where:

- F is the maximum load applied to the sample.
- L is the distance between the supports.
- b is the width of the sample.
- d is the depth of the sample.

The flexural stiffness is typically calculated using the slope of the linear portion of the load-deflection curve.

The three point bend test were carried out in the samples immediately after exposure to cryogenic temperature in an UTM 201 machine in accordance with ASTM DD7901 . The former samples after exposure to room temperature and the untreated as-cured samples were tested in short beam shear test at room temperature. All the specimens (composites) were of rectangular shape having dimension with length 125 mm, breadth of 12 mm and thickness of 2.5mm. A span of 30 mm was employed maintaining a cross head speed of 9mm/min.

3.3.3 FTIR ANALYSIS

FTIR (Fourier-transform infrared) spectroscopy is a powerful analytical technique used to identify and analyze the molecular composition of a wide range of materials. The technique is based on the absorption of infrared radiation by molecular vibrations, which can provide information about the functional groups and chemical bonds present in a sample. In FTIR analysis amount of the sample is typically ground into a fine powder and mixed with a spectroscopic-grade KBr (potassium bromide) powder. The mixture is then pressed into a thin, transparent pellet using a hydraulic press. The FTIR spectrometer is set up by first calibrating the instrument with a blank KBr pellet to establish a baseline signal. The sample pellet is then placed into the sample holder and positioned in the beam path of the infrared light. The instrument is then activated and the infrared light is directed through the sample. The instrument measures the amount of infrared radiation that is absorbed by the sample at various wavelengths. The resulting spectrum is a plot of the absorption of infrared radiation by the sample as a function of wavelength. The resulting spectrum can be analyzed to identify the functional groups and chemical bonds present in the sample. The positions and intensities of the absorption peaks in the spectrum can be compared to reference spectra in a database to determine the identity of the sample components. FTIR spectroscopy can be used to identify unknown compounds by comparing their spectra to a database of reference spectra and is commonly used to characterize the chemical composition and structure of polymers and plastics

3.3.4 THERMO GRAVIMETRIC ANALYSIS

TGA (Thermo gravimetric Analysis) is a technique used to determine the thermal stability and composition of a material. It involves the measurement of the weight of a sample as it is heated or cooled under controlled conditions. In TGA a small amount of the sample is weighed and placed into a sample pan. The pan is then placed on the balance of the TGA instrument. The TGA instrument is set up by first calibrating the instrument with an empty sample pan to establish a baseline weight. The sample pan with the sample is then placed into the instrument and positioned in the furnace. The instrument is then activated, and the temperature of the furnace is increased or decreased at a constant rate. As the sample is heated or cooled, the weight of the sample is

measured continuously by the balance. The resulting TGA curve is a plot of the weight of the sample as a function of temperature or time. This curve can be analyzed to determine various thermal properties of the sample, including the thermal stability, decomposition temperature, and the amount and nature of the volatile and non-volatile components.TGA analysis is used to determine the following

- Thermal stability analysis: TGA can be used to determine the thermal stability of a material by measuring its weight loss as a function of temperature. This information can be used to select materials that can withstand specific thermal conditions and to optimize the performance of materials in high-temperature applications.
- Composition analysis: TGA can be used to determine the composition of a material by measuring the weight loss of different components as the material is heated. This information can be used to identify the presence of specific components, to quantify the amount of different components, and to monitor the purity of materials.
- Quality control: TGA can be used as a quality control tool to monitor the composition and purity of materials during production processes. It can also be used to detect the presence of impurities or contaminants in materials.
- Failure analysis: TGA can be used in failure analysis to determine the cause of failures in materials. For example, TGA can be used to identify the presence of contaminants or impurities that may have contributed to a material failure.

3.3.5 DIFFERENTIAL THERMO GRAVIMETRIC ANALYSIS (DTG)

DTG (Derivative Thermogravimetry) is a variation of thermogravimetric analysis (TGA) that provides additional information about the thermal properties of a material. DTG involves taking the derivative of the TGA curve, which results in a curve that shows the rate of change in weight loss as a function of temperature.

Here are some of the key benefits of DTG analysis:

- Improved resolution: DTG provides improved resolution of the TGA curve, which can help to identify and distinguish different thermal events or decomposition processes that may be overlapping in the TGA curve.
- Identification of reaction kinetics: DTG can provide information about the kinetics of

thermal reactions that occur in a material. The shape of the DTG curve can indicate the activation energy of a reaction, which can be used to determine the rate of reaction and the reaction mechanism.

- Prediction of thermal behavior: DTG can be used to predict the thermal behavior of a material under different conditions. By analyzing the DTG curve, it is possible to estimate the temperature at which a material will decompose and the amount of weight loss that will occur.
- Quantitative analysis: DTG can be used for quantitative analysis of materials. By comparing the area under the DTG curve for different samples, it is possible to determine the relative amounts of different components in a material.

3.3.6 WATER ABSORPTION TESTING

Water absorption testing is a process used to measure the ability of a material to absorb water. This testing is important in determining the suitability of a material for applications where it may be exposed to water. Immersion testing is a method used to measure the ability of a material to absorb water or other liquids. This test involves immersing a sample of the material in the liquid for a specified amount of time and then measuring the amount of liquid absorbed by the material. The purpose of immersion testing is to determine the degree of water absorption of the material and its performance when exposed to water.

To perform an immersion test, a sample of the material is cut to a specific size and weighed before immersion. The sample is then completely immersed in a container of water or other liquid for a specified time, such as 24 hours. After the immersion period, the sample is removed from the liquid, excess liquid is removed by blotting or shaking, and the sample is weighed again. The difference in weight before and after immersion is used to determine the amount of liquid absorbed by the material.

The results of an immersion test can be reported as the percentage of weight gain, which represents the amount of liquid absorbed relative to the weight of the sample before immersion. This information can be used to evaluate the performance of the material in applications where it may be exposed to liquids.

The composite sample underwent the water absorption test in accordance with ASTM D 2842-

01.The specimen was 40 x 40 x 20 mm. The composite specimen was subjected to a water absorption test by being submerged in distilled water in a beaker at room temperature for seven days (168 hours). The specimen was periodically removed from the water, and any surface water was wiped away with a tissue paper. After reweighing, the specimen was submerged once more. The percentage of water absorption is determined from the following equation:

$$W(\%) = \frac{m_{2-}m_2}{m_2} \times 100$$

Where m_2 and m_1 are weight of wet sample and dry samples espectively.

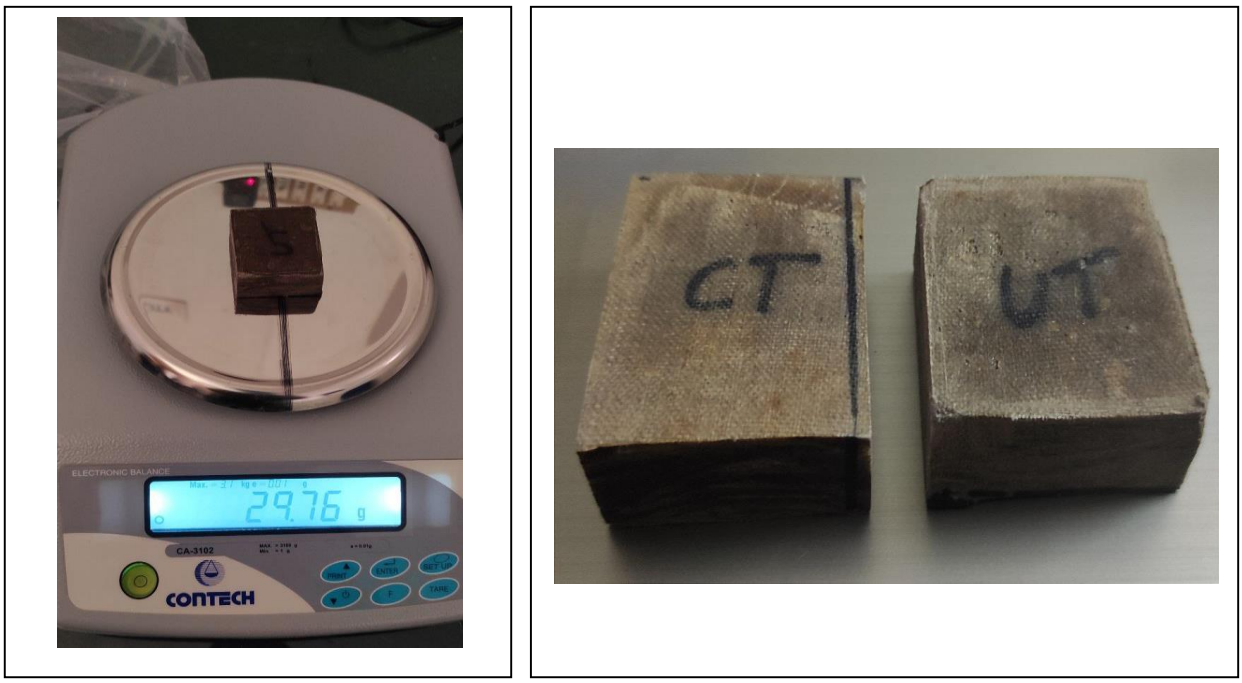

Figure-3.7 Untreated and cryogenic treated specimen for water absorption test

3.3.7 THERMAL CONDUCTIVITY MEASUREMENT

Thermal conductivity is a property of materials that describes their ability to conduct heat. It is defined as the amount of heat that passes through a unit area of a material in a unit time when a temperature difference is maintained between two sides of the material. The SI unit of thermal conductivity is watts per meter-kelvin (W/mK). The thermal conductivity of fiber reinforced composites depends on various factors such as the type of fiber, the matrix material, the fiber volume fraction, and the orientation of the fibers. Generally, natural fibers have lower thermal

conductivity compared to synthetic fibers, and therefore the thermal conductivity of natural fiber reinforced composites is typically lower than that of composites reinforced with synthetic fibers.

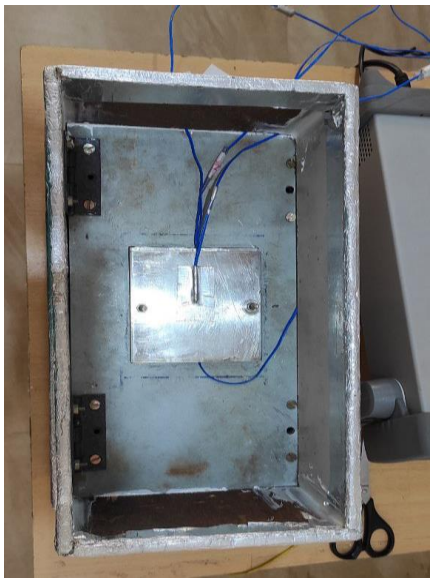

Figure-3.8 Set up for thermal conductivity measurement

The guarded hot plate method is a commonly used technique for measuring the thermal conductivity of materials. This method involves sandwiching a material sample between two parallel metal plates, one of which is heated and the other cooled, and measuring the heat flow through the sample. The apparatus used in the guarded hot plate method consists of a central heater plate, a pair of thermometers, and two guard plates that surround the sample and minimize heat loss from the edges of the sample. The temperature of the hot plate and the cold plate are measured using the thermometers, and the heat flow through the sample is calculated based on the temperature difference between the plates and the thermal conductivity of the sample. To perform the guarded hot plate measurement, the sample is first cut to the appropriate size and shape and placed between the two guard plates. The heater plate is heated to a specific temperature, while the cold plate is cooled to a different temperature. The temperatures of the plates are measured using the thermometers, and the heat flow through the

sample is calculated using Fourier's Law of Heat Conduction. The thermal conductivity of the sample is then calculated by dividing the heat flow by the product of the sample thickness and the temperature difference between the plates. The guarded hot plate method is a highly accurate and reliable technique for measuring thermal conductivity, particularly for materials with low thermal conductivity values.

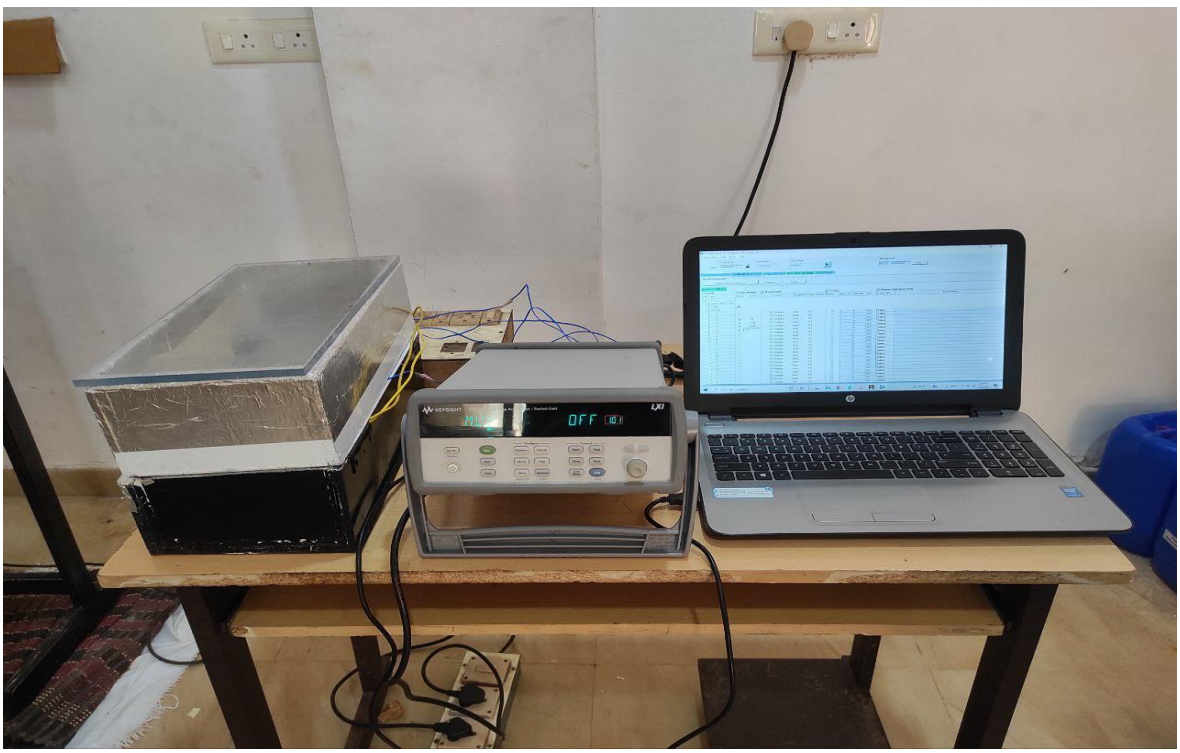

Figure -3.9 Guarded hot plate method for thermal conductivity measurement

The material is kept under homogeneous compressive load between two gleaming surfaces, each of which is tested at a different temperature. In the lower surface, the heat flow transducer has been calibrated. Heat passes through the sample from the upper plate at high temperature, causing an axial temperature differential in the pile. Following thermal equilibrium, the temperature gradient across the sample is measured using the heat flow transducer output. The thermal conductivity of the composite sample is calculated using the measured values of temperature difference T and sample thickness x. Temperature sensors in the extremely conductive metal surface layers on

either side of the sample measure the temperature trickle across the specimen. For one-dimension heat flow, the equation is given as:

$$Q = KA\frac{T_1 - T_2}{X}$$

Where, Q is the heat flux (Watt), K is the thermal conductivity (W/m K), A is the cross-sectional area (m2), (T1 – T2) is the difference in temperature (K), x is the thickness of the sample (m).

CHAPTER 4
RESULTS AND DISCUSSION

Thermo gravimetric analysis (TGA), Differential Thermo gravimetric analysis (DTG), and Fourier Transform Infrared Spectroscopy (FTIR) analysis were used to characterize the physiochemical properties of cryogenically treated hemp fiber. Then Comparing the experimental outcomes of treated and untreated fabrics

4.1 FTIR SPECTROSCOPIC ANALYSIS

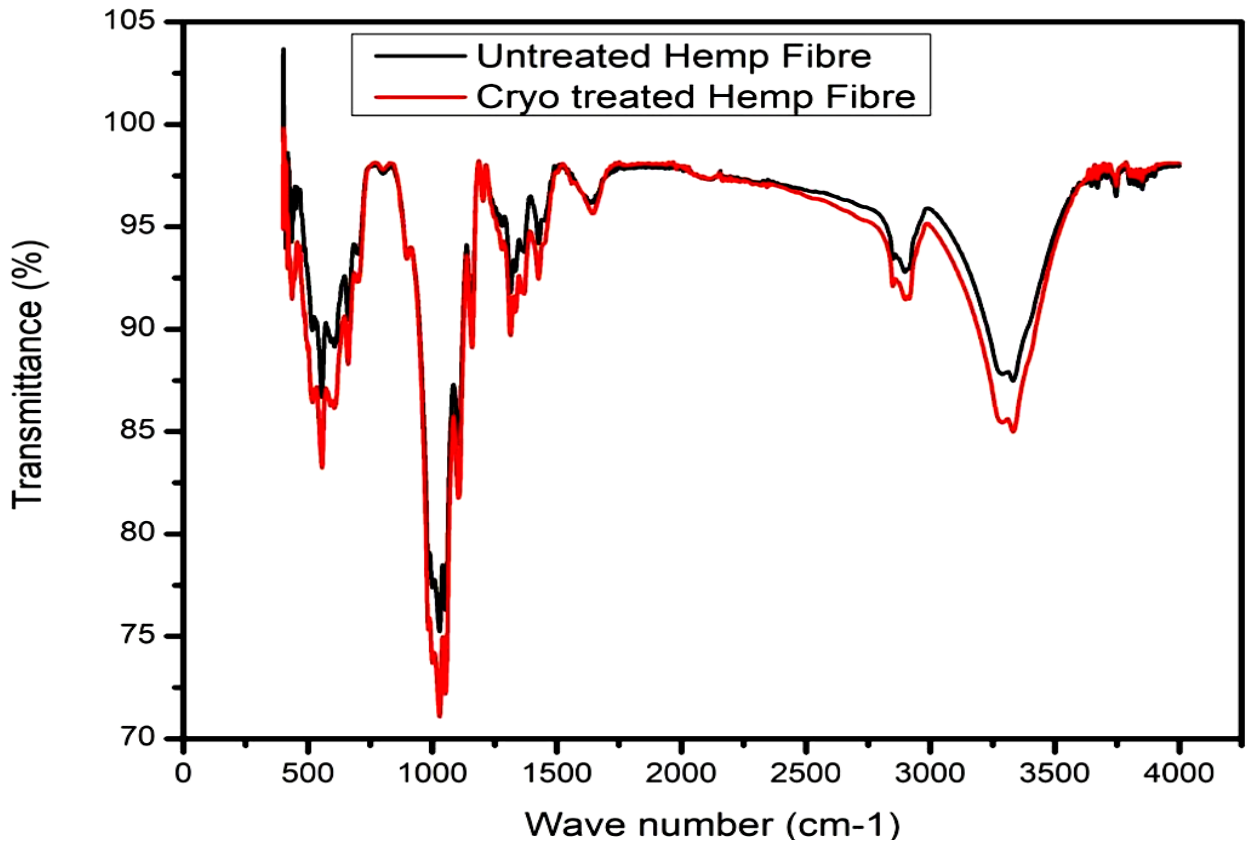

Figure-4.1 FTIR spectrum of untreated and cryogenic treated hemp fiber

Figure 4.1 shows an FTIR spectroscopic investigation on a hemp fiber subjected to cryogenic treatment. The difference between the absorbance bands of treated and untreated materials shows that there are more hydrogen bonds in the treated material. According to Beer Lamberts law there is a increase in OH bonds in the range of 3000-3500 cm^-1. The asymmetric and symmetric

methyl and methylene stretching groups were associated to the vibration absorption peak at 2900 cm-1, which also indicates the existence of lignin in the fabric. The FTIR spectrum's absorbance peaks about 1600-1800cm-1, which correspond to the wave numbers of carboxylic ester (C=O) in pectin and waxes, show that these constituents have been reduced as a result of cryogenic treatment. Removal of aromatic functional groups is thus observed in the range of 1600-1800 cm^1.The C=O stretching vibration in hemicellulose was the cause of the absorption peaks from 1020 to 1040 cm^-1. Figure 4.1 demonstrates how treated hemp fiber has a wider band and more vibration absorbency than untreated fabric. Two curves in figure trace the same path, which indicates both the specimens possess similar functional groups even after cryogenic treatment of fiber.

4.2 MECHANICAL POPERTIES

4.2.1 TENSILE TEST

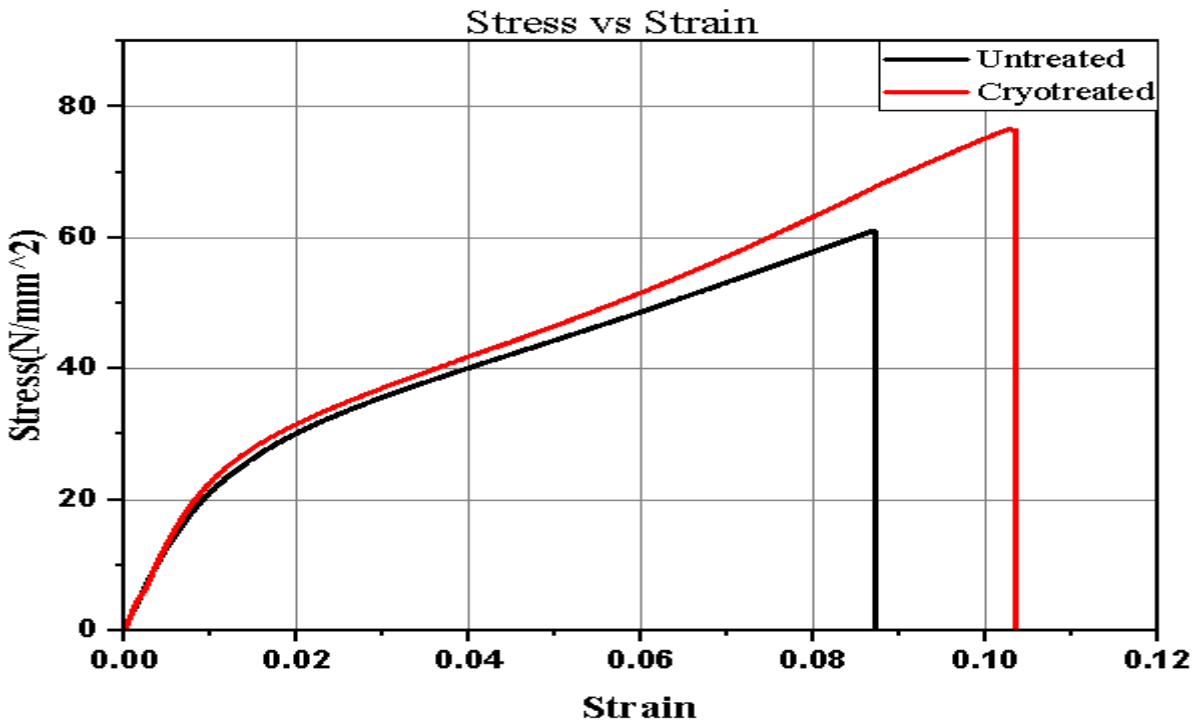

Figure-4.2 Tensile stress-strain curve of untreated and cryogenic treated composite

The experiment was conducted at room temperature using ASTM D 3039 universal testing equipment, which is a standard test method for tensile properties of polymer matrix composite

materials. From the experiment, five samples with dimensions 150mm x 15mm x 3.2mm were prepared and evaluated for each variation. The crosshead speed during the test was set at 2 mm/min, and a data acquisition system (DAQ) was used to record the load-displacement data. The tensile strength and modulus were then computed from this data An important factor in the stiffness and strength of the composite is reinforcement. When employed as filler in epoxy matrixes, hemp fibers outperform other natural fiber fillers in terms of specific strength. The strength of natural fiber is mostly derived from its cellulose and hemicellulose components. Cryogenic treatment caused the fibers to exhibit brittle behavior, resulting in a composite that is susceptible to brittle fracture. The tensile strength and modulus of the cryogenic treated specimen increased by 25% compared to untreated specimen.

4.2.2 FLEXURAL TEST

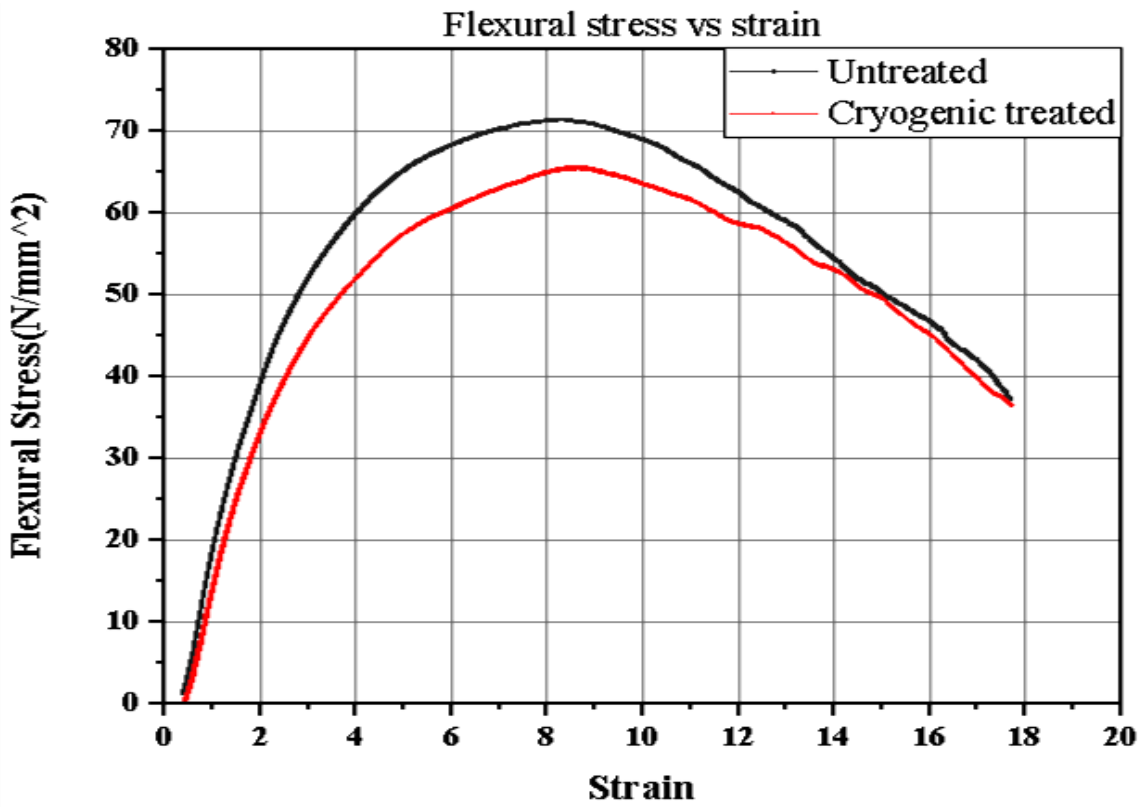

Figure-4.3 Flexural stress-strain curve of untreated and cryogenic treated composite

A three-point bending test was used to conduct a flexural test. The test is run with a 0.1mm/min strain rate. For each variation eight number of samples with the dimensions 125mm 3 12.7mm 3

3.2mm were evaluated. Understanding the composite's bending strength and the interfacial connection between the reinforcement and the matrix depends on its flexural properties. This study shows how epoxy resin adheres to both untreated and cryogenic treated hemp fiber composite panel. Figure 4.3 provides evidence of improved epoxy-reinforcement interfacial bonding following cryogenic treatment. Increased flexural strength of the modified composite is also a result of reduced hemicellulose and lignin content, which causes pores to form in the composite. Induction of brittle behavior due to cryogenic treatment is evident with the flexural behavior of the composite. The flexural strength of the cryogenic treated specimens exhibited a 13% increase compared to the untreated specimens. This suggests that cryogenic treatment can enhance the flexural properties of composite materials made from hemp fibers and epoxy matrix, despite inducing brittle behavior.

Table-4.1 Values of mechanical properties of hemp fiber composite panel

Specimen	Tensile strength (N/mm^2)	Tensile Modulus (N/mm^2)	Flexural Strength (N/mm^2)
Untreated	61.032	701.51	62.52
Cryogenic treated	76.5	743.48	71.05

4.3. THERMAL PROPERTIES

4.3.1 DTG CURVES OF UNTREATED AND CRYOGENIC TREATED HEMP FABRIC

The derivative of thermo gravimetric (DTG) plot for time displays the substance's rate of percentage weight deterioration. The plot compares the untreated with cryogenic treated hemp/epoxy showing a significant increase in peak degradation temperature at all stages of decomposition. At the tertiary stage of degradation, which occurs between temperatures of 255°C and 460°C, hemicelluloses, lignin, and glycosidic linkages in cellulose undergo thermal depolymerization. In the fourth stage, which takes place between 475°C and 585°C, a-cellulose and lignin decompose, leaving behind remnants of epoxides. The modified composite material exhibits higher thermal stability than the untreated composite, as evidenced by a significant increase in the peak inflexion point.

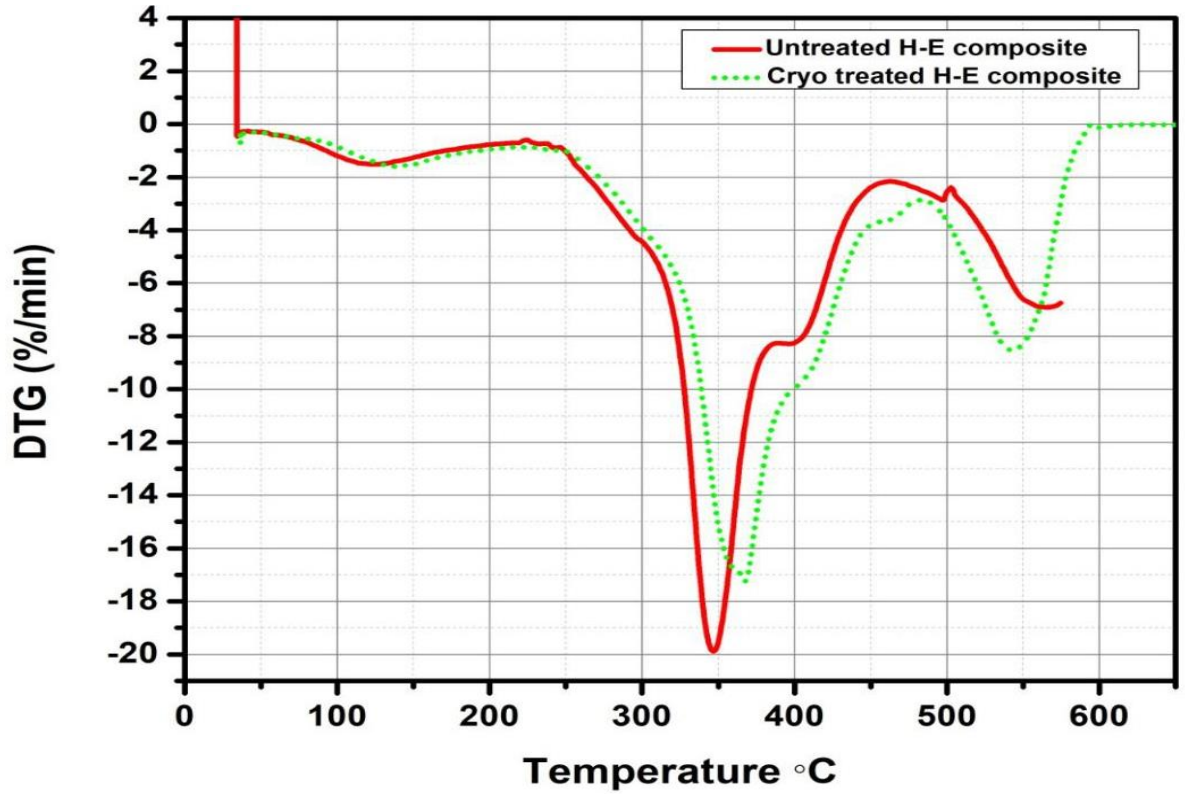

Figure-4.4 DTG curves of untreated and cryogenic treated hemp fiber composite panel

4.3.2 TGA CURVES OF UNTREATED AND CRYOGENIC TREATED HEMP FABRIC

A controlled temperature program was employed to conduct TGA, which measured the mass constituents of the substance by recording the change in mass with increasing temperature and time intervals. The first stage, occurring between 75°C and 125°C, represents moisture evaporation from the substance. Cryogenic treatment of the hemp/epoxy composite led to a lower mass loss during this stage, indicating a significant reduction in moisture absorption by the hydrophilic cellulosic fibers. In the secondary stage, mass loss represents degradation of the epoxy polymer. The modified composites exhibited greater mass loss during this stage than the unmodified ones, indicating an increase in the polymer-fiber ratio and improved wettability of the modified hemp fibers. The tertiary stage involved the degradation of residual polymer and fiber constituents, the main constituents of the composite. Finally, the fourth stage indicated the overall residual substance content of the composite.

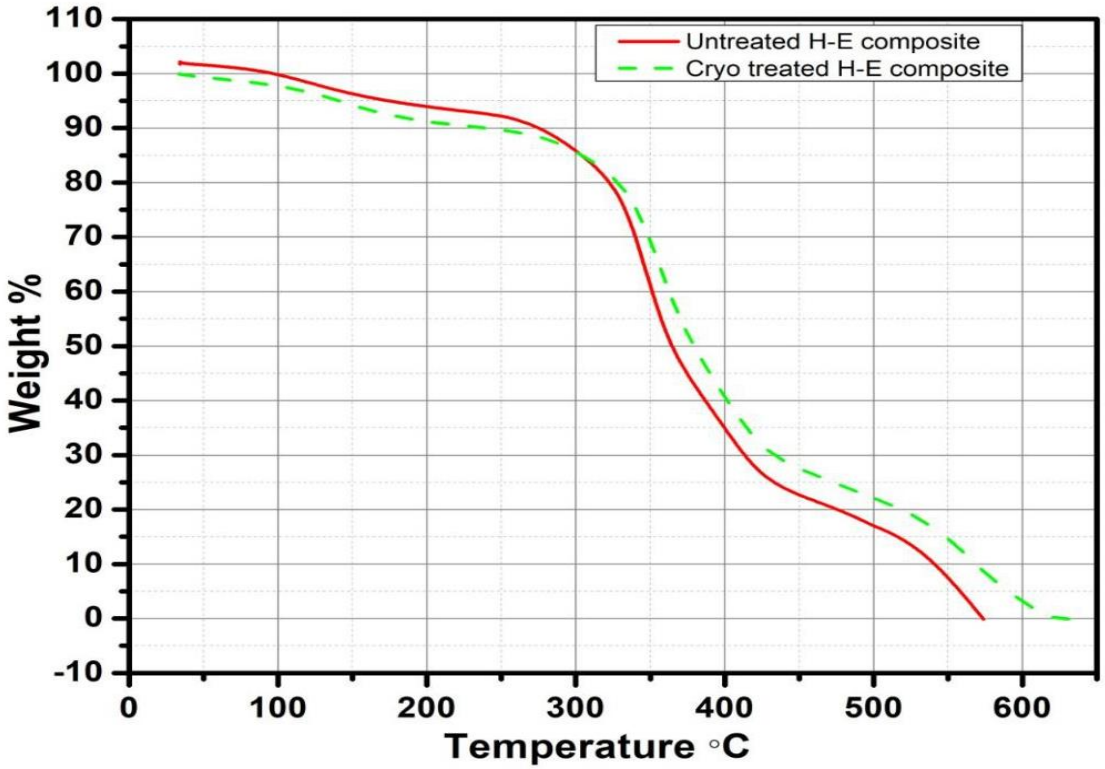

Figure-4.5 TGA curves of untreated and cryogenic treated hemp fiber composite panel

4.3.3 THERMAL CONDUCTIVITY STUDY

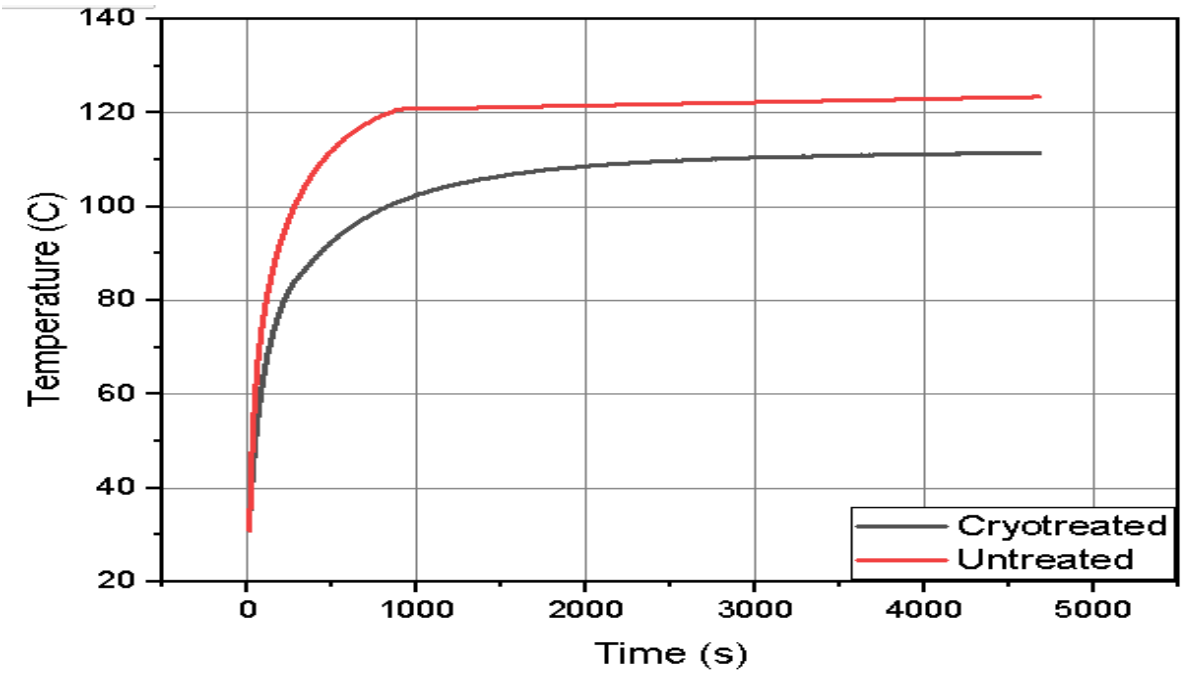

Figure-4.6 Temperature vs time for hemp fiber panel

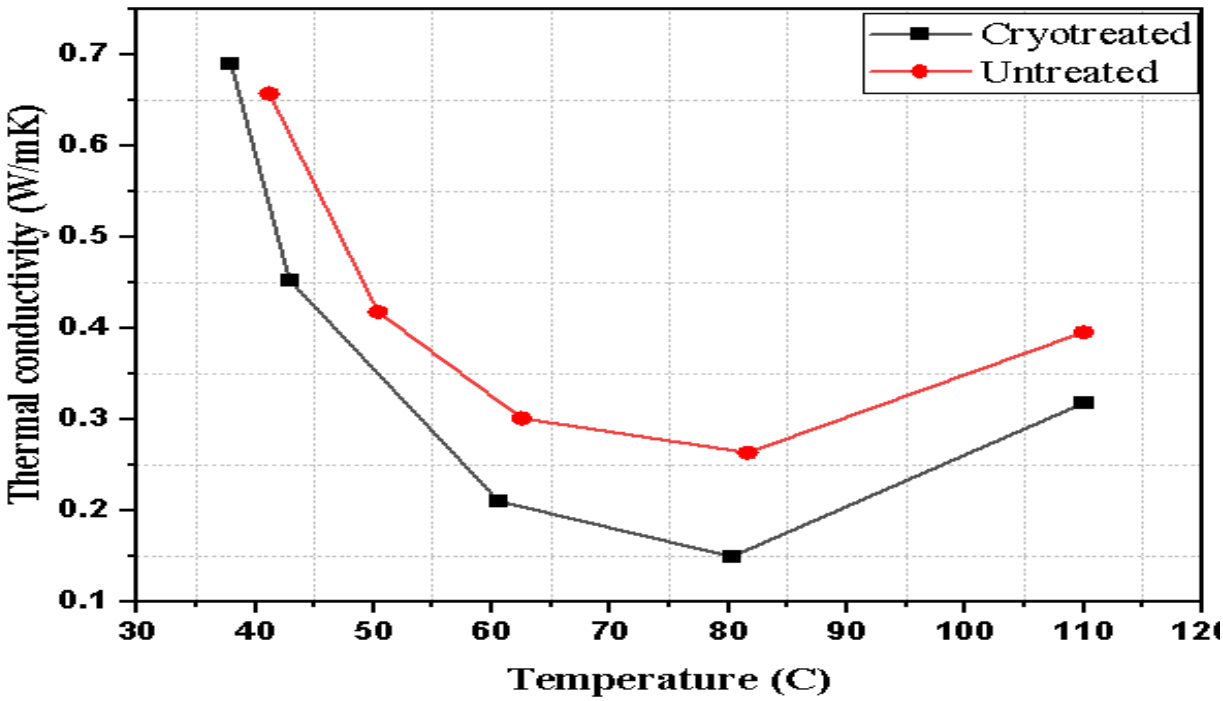

Figure 4.7 Thermal conductivity vs temperature graph for untreated and cryogenic treated hemp fiber panel

Thermal conductivity is defined as the amount of steady state heat flux transferred through unit thickness materials that has unit area induced by unit temperature difference across the cross section. Thermal conductivity is the effectiveness of the material to conduct heat. If the thermal conductivity values become low, it indicates that the materials have very high insulating property. In these work thermal conductivity of untreated and deep cryogenic treated hemp /epoxy composite is analyzed .The fig 4.7 shows the comparison of thermal conductivity vs temperature of untreated and cryotreated specimen from these plot it shows a decrease in thermal conductivity of 15 % when compared with untreated specimen. The treated samples developed in the work shows a higher thermal resistance .The lowest value of thermal conductivity is observed at 80°C and its value is equal to 0.15 W/mK and 0.3 W/mK for cryotreated and untreated specimen respectively

4.4 PERCENTAGE OF WATER ABSORPTION

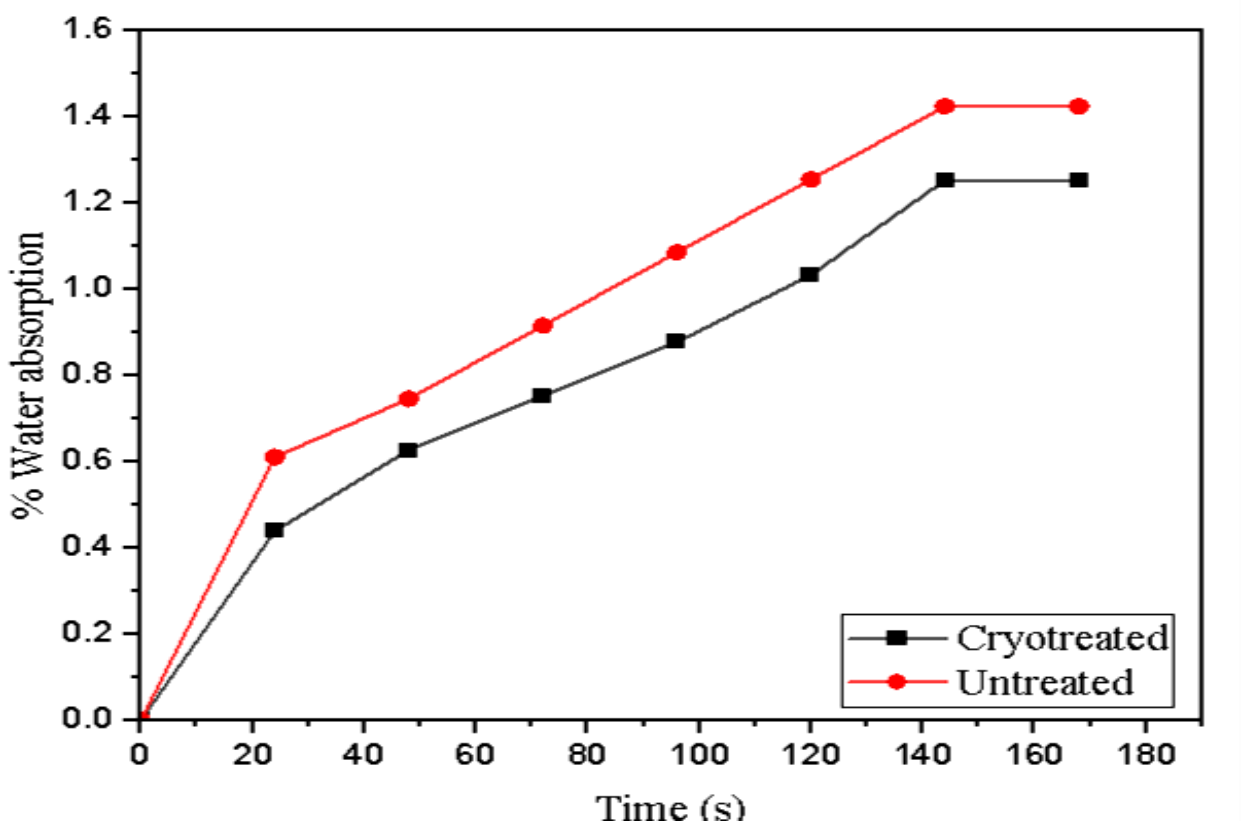

Figure-4.8 percentage of water absorption for untreated and cryogenic treated hemp fiber composite

The water absorption rate of the natural fiber composite materials depends on the fiber size, surface area, cellulose and hemicelluloses content. The sample developed in this work shows a percentage of water absorption rates of 0.43% and 0.61% for cryogenic treated and untreated specimen respectively. Water absorption rate of the fiber increases with time due to hygroscopic nature of fiber. Thus cryogenic treated hemp fiber shows 12% reduction in percentage of water absorption compared to untreated hemp fiber. Cryogenic treatment can be an effective method to reduce the moisture absorption of hemp fiber. It can be presumed that the freezing process can induce some chemical changes in the hemp fiber. The low temperatures may cause some of the water molecules to be removed from the fiber structure, which can lead to a reduction in the number of active sites available for water absorption. Additionally, the low temperatures can also cause some of the lignin and hemicellulose compounds in the fiber to break down, which can make the fiber more resistant to water absorption.

CHAPTER 5
CONCLUSION

The purpose of this research was to investigate how modifying hemp fibers through cryogenic treatment would impact their thermal and mechanical properties of polymer composite. Through the study, it was observed that subjecting hemp fabric to cryogenic treatment resulted in an improvement in its physical characteristics due to an increase in the number of hydrogen bonds within the cellulose. The treatment removed waxes and pectin from the lignocellulosic fiber, which created more room for covalent bonding between cellulose OH groups. This ultimately led to an enhancement in the stiffness of the fabric. The hydrophobic behavior of hemp fabric was improved through cryogenic treatment, as indicated by a lower derivative weight during the initial stage of degradation. Improved physical properties are observed with an increase in the amount of extensive constituents such as cellulose, and a decrease in hemicellulose and pectin. The stiffness of the fiber was improved by an increase in its cellulose composition. The removal of hemicellulose, lignin, waxes, and pectin from the fiber partially increased its thermal stability since these elements degrade earlier than raw cellulose. When cryogenic treated fibers were combined with epoxy, the resulting composite exhibited high thermal stability compared to the unmodified composite. The TGA curve indicated an increase in polymer content, suggesting that the fibers became more wettable after cryogenic treatment. It is believed that the increase in mechanical bonding contributed to the improved tensile strength. The improved flexural properties also suggest better interfacial bonding between the matrix and reinforcement due to cryogenic treatment. The thermal conductivity of the hemp fiber reinforced composite panel decreases about 15% after cryogenic treatment of hemp fiber. Thus cryogenic treatment can be used as effective method to improve thermal resistance of hemp fiber composite panel. Cryogenic treatment of hemp fiber decreases the water absorption rate by 12.4% .It is presumed that low temperatures can also cause some of the lignin and hemicellulose compounds in the fiber to break down, which can make the fiber more resistant to water absorption. The findings suggest that cryogenic treatment of hemp fibers can enhance their thermal stability, thermal conductivity, and physical properties, making them suitable for various applications. Consequently, the treated fibers may offer superior performance compared to untreated hemp fibers.

Reference

[1] Elisa Morettia, Elisa Bellonia, Fabrizio Agosti, Innovative mineral fiber insulation panels for buildings: Thermal and acoustic characterization, Applied Energy 169 (2016) 421–432.

[2] Gowtham, Rajakumar, Investigation of Natural Fiber Composite Material for Building Cooling System, International Journal of Science and Research (2018): 7.426

[3] S. Sair, A. Oushabi, A. Kammouni, O. Tanane, Y. Abboud, A. El Bouari ,Mechanical and thermal conductivity properties of hemp fiber reinforced polyurethane composites, Case Studies in Construction Materials 8 (2018) 203–212.

[4] Zsombor Sapi, Richard Butler, Properties of cryogenic and low temperature composite materials, cryogenics 111(2020) 103190.

[5] Chen W, Wang Y, Zhang K, Xu F. Characterization and comparison of properties of cryogenic conditioned CNT reinforced thermoset (epoxy) and thermoplastic (poly vinyl alcohol) composite yarns. Journal of Composite Materials. 2021;55(30):4503-4511.

[6] Yinnan Zhang, Fujun Xu, Chuyang Zhang, Junjie Wang, Zhemin Jia, David Hui, Yiping Qiu, Tensile and interfacial properties of polyacrylonitrile-based carbon fiber after different cryogenic treated condition, Composites Part B: Engineering, Volume 99, 2016, Pages 358-365,ISSN 1359-8368

[7] Xu, F., Fan, W., Zhang, Y., Gao, Y., Jia, Z., Qiu, Y., & Hui, D. (2017). Modification of tensile, wear and interfacial properties of Kevlar fibers under cryogenic treatment. Composites Part B: Engineering, 116, 398–405.

[8] Kanthavel, Karuppusamy & Ramanaicker, Palanisamy & S, Vivek. (2015). Investigation of the Mechanical Properties of Bagasse Fiber-Reinforced Epoxy Composite using Taguchi and Response Surface Methodology. BioResources. 10. 3749-3756. 10.15376/biores.10.2.3749-3756.

[9] Sair, Said & Oushabi, Abdessamad & Kammouni, A. & Tanane, Omar & Abboud, Younes & Abdeslam, El Bouari. (2018). Mechanical and thermal conductivity properties of hemp fiber reinforced polyurethane composites. Case Studies in Construction Materials. 8. 10.1016/j.cscm.2018.02.001.

[10] P.A. Thakare, N. Kumar, V.B. Ugale, Thermal Behaviour of Hybrid Composites Reinforced with Natural Fibers and Kevlar, International Journal of Recent Technology and Engineering (IJRTE) ISSN: 2277-3878 (Online), Volume-8 Issue-2, July 2019.

[11] Behzad, Tayebeh & Sain, Mohini. (2007). Measurement and prediction of thermal conductivity for hemp fiber reinforced composites. Polymer Engineering & Science. 47. 977 - 983. 10.1002/pen.20632.

[12] T. Zhidong and L. Nielsen, “Thermal Conductivity of Carbon Nanotubes and Their Polymer Nanocomposites: A Review”, Vol.14, pp. 1449-1471, 2011.

[13] Yongli Zhang, Yan Li, Hao Ma and Tao Yu, “Tensile and Interfacial Properties of Unidirectional Flax/Glass Fiber Reinforced Hybrid Composites”, Composites Science and Technology, Vol. 88, pp. 172–177, 2013.

[14] Huang, G. & Sun, H. “Effect of Water Absorption on the Mechanical Properties of Glass/Polyester Composites”, Materials & design, Vol.28, pp.1647-1650. 2007.

[15] Li, Xue & Tabil, Lope & Panigrahi, Satyanarayan. (2007). Chemical Treatments of Natural Fiber for Use in Natural Fiber-Reinforced Composites: A Review. Journal of Polymers and the Environment. 15. 25-33. 10.1007/s10924-006-0042-3.

[16] J. Liu, Z. Guan, and Z. Li, Oct. 2018, “Application of cryogenic and mechanical treatment in degumming of hemp stems,” *Biosyst. Eng.*, vol. **174**, pp. 144–152.

[17] Bledzki AK, Fink HP and Specht K. Unidirectional hemp and flax EP- and PP-composites: influence of defined fiber treatments. J Appl Polym Sci 2004; 93(5):2150–2156.

[18] Bettahally, Vinod & L J, Sudev. (2019). Investigation on Effect of Cryogenic Temperature on Mechanical Behavior of Jute and Hemp Fibers Reinforced Polymer Composites. Applied Mechanics and Materials. 895. 76-82. 10.4028/www.scientific.net/AMM.895.76.

[19] Ouajai, Sirisart & Shanks, Robert. (2005). Composition, structure and thermal degradation of hemp cellulose after chemical treatments. Polymer Degradation and Stability. 89. 327-335. 10.1016/j.polymdegradstab.2005.01.016.

[20] Karvanis K, Rusna’kova’ S, Krejc, et al. Thermal analysis of postcured aramid fiber/epoxy composites. Rev Adv Mater Sci 2021; 60(1): 479–489.