DESIGN AND DEVELOPMENT OF THERMAL AND CHEMICAL PARAMETERS FOR RECYCLING AND RESTORATION OF GLASS FIBERS FROM COMPOSITE WASTE

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CERTIFICATE

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DEDICATION

With profound reverence,

We dedicate this project to our **beloved parents** and **respected teachers** whose guidance and persistent support helped us throughout the process.

ACKNOWLEDGEMENTS

Praise be to Almighty Allah who made us capable to do our best and get through this project.

For the successful completion and execution of our final year project, we sincerely acknowledge all the advice and support we have received from our Supervisor Dr. Muhammad Irfan. Without his diligence, we would not have been able to project our true capabilities for this demanding task.

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ABSTRACT

Previous studies show that Fiber Reinforced Polymers are dumped in landfills after usage or are burned or decomposed by combustion methods. These processes generate environmental waste and landfill problems. The disposal of these composite manufacturing waste and end-of-life composite products in an environmentally friendly manner is one of the most important issues currently faced by the industrial and academic composites community. For the sustainable development of the local industry and economy, these composite wastes need to be recycled. So, to resolve this issue, we are recycling Glass Fibers from Fibers Reinforced Polymers by thermal treatment such as Pyrolysis, but the issue with thermal treatment is strength loss in the recycled fibers.

To recover this lost strength, we have done chemical treatment such as Hot Alkaline treatment, which restores the strength of single fibers to 200% compared to the pyrolyzed fibers, by etching the surface defects and by removing residue. And for the composite samples, the strength was restored to 110% compared to the composite sample made from pyrolyzed fibers.

Tensile testing was performed on both the pyrolyzed glass fibers as well as on the chemically treated fibers for comparison. The results of these tests and Weibull Distribution showed that these chemically treated fibers can be effectively used for making composite materials.

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CHAPTER 1

INTRODUCTION

1.1 Overview

The development and advancement in the field of material Science in the twentieth century have made the Composite the liability, to be utilized in every industry application such as Construction, Aerospace, marine, and sports, due to its ability to modify the Material characteristics to meet the specific requirement in a certain application [1]. GFRP (Glass Fiber Reinforced Polymer) composite is widely utilized in many different industries because of its superior mechanical qualities than more conventional materials like steel or aluminum. Some of the advantages of the GFRPs are:

1.1.1 High Strength-to-Weight Ratio

GFRP composites are renowned for having an outstanding strength-to-weight ratio making them perfect for use in aerospace, automotive, and sports goods sectors, providing a reduction in weight along with high strength and stiffness while being lightweight.

1.1.2 Corrosion Resistance

Contrary to metals, GFRP composites having high levels of corrosion resistance, are capable of withstanding extreme conditions in the marine, chemical, and infrastructural industries that deal with corrosive materials.

1.1.3 Design Flexibility

GFRP composites are capable of being molded into complicated forms, providing for the fabrication of complex structures, and allowing shaping into different shapes and designers to realize imaginative notions, with complex geometries.

1.1.4 Dimensional Stability

GFRP composites exhibit a minor dimensional change, offering extra stability and dependability over time, considering a variety of environmental circumstances, this is critical in industries like aerospace or precision manufacturing where dimensional accuracy and predictability are crucial.

1.1.5 Fatigue Resistance

GFRP demonstrates strong fatigue resistance, enabling it to bear and sustain numerous loading cycles without degrading its mechanical characteristics. Due to this characteristic, GFRP is feasible for applications with dynamic stresses or cyclic loads.

The polymer matrix in GFRP composites is commonly composed of thermosetting resins like epoxy, polyester, or vinyl ester, see Fig 1. These resins give the matrix dimensional stability, durability, and chemical resistance. The composite is given strength, stiffness, and impact resistance by the glass fibers, which are the reinforcement phase [2]. In more than 90% of all these composites, glass fiber (GF) is used as the reinforcement of choice since it is compatible with organic matter, incombustible, and has dimensional stability, and a combination of these properties [1].

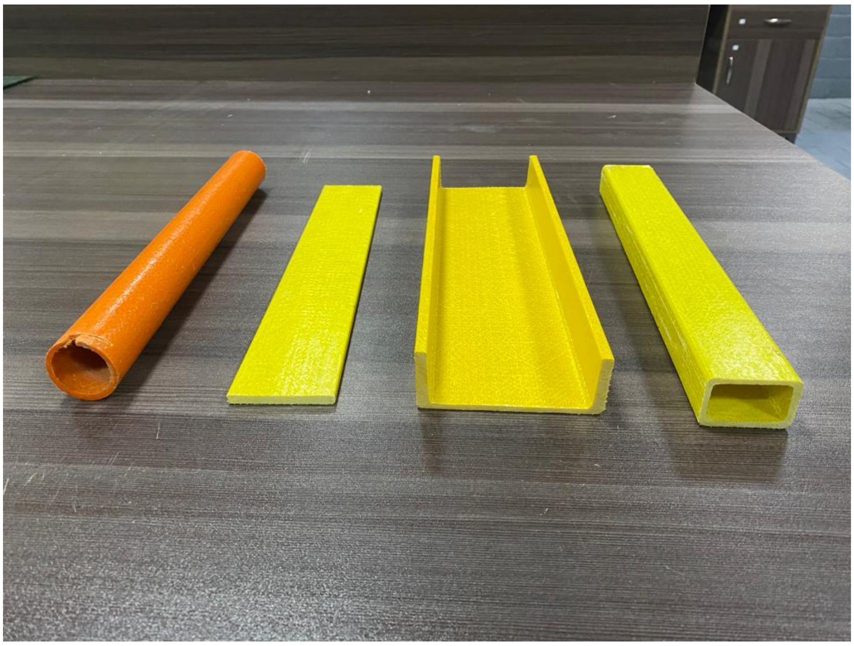


Fig 1: Industrial Composite material

1.1.6 High Tensile Strength

Glass fibers can withstand pulling or stretching forces well because of their high tensile strength. This characteristic aids in the effective distribution of stress within GFRP composites, allowing them to sustain applied loads.

1.1.7 High Stiffness and Rigidity

High stiffness and rigidity are provided by glass fibers for GFRP composites. This rigidity improves the composite's structural integrity by allowing it to hold its shape and withstand deformation under stress.

1.1.8 Thermal Stability

Glass fibers have a high melting point and provide excellent thermal stability making (GFRP) composites well suited for heat resistance or thermal insulation capabilities applications providing high-temperature properties.

1.2 Challenges

Global production of fiber-reinforced composite materials reached over 10 million tons in 2015, with glass fiber (GF) accounting for more than 90% of these composites. Nearly 5 million tons of E-glass fiber were reproduced. The global composites market has reached £80 billion by 2020, leading to a significant volume of waste at the end of the materials' life cycle [3]. Most of the waste generated by GF production between 0.5 and 1 million tons is disposed of in landfills, see Fig 2. Around 70% of GF is used in thermoset-based composites (GRP), which also produce 15% of the production waste. It is challenging to recycle GRP materials, including manufacturing and end-of-life waste, and landfilling is becoming more and more expensive because of regulations [1].

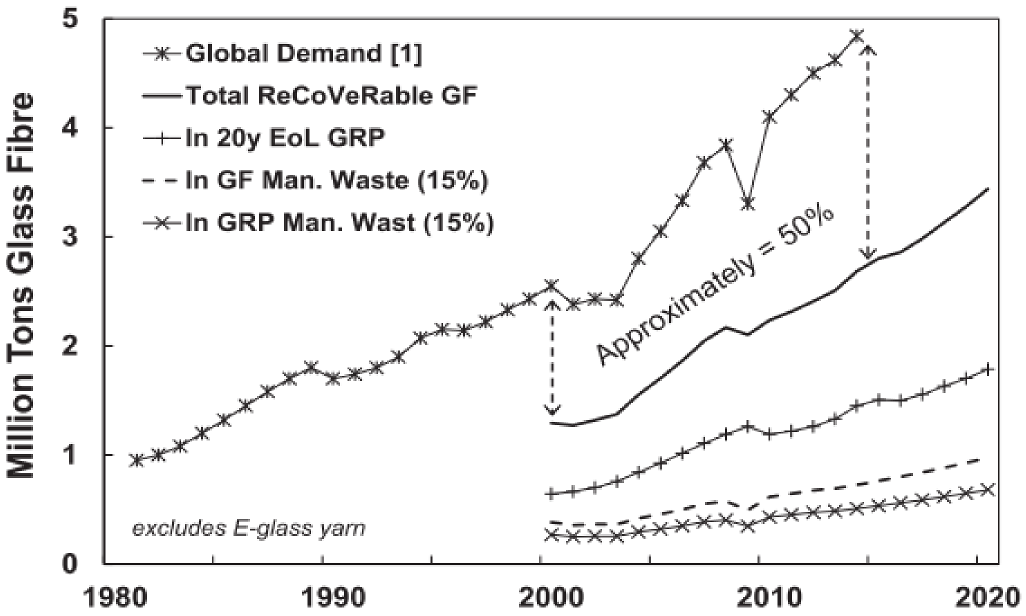


Fig 2: Glass fiber global demand and availability for recycling [1]

The composites sector has a major challenge with the disposal of waste from composite manufacturing and end-of-life composite products. Due to laws and expanding landfill prices, the disposal of these products in landfills is becoming increasingly unsustainable, see Fig 3. Due to the expanding usage of composites in industries including transportation and wind energy, this issue has attracted attention. The advantages of GFRP composites have led to their widespread use. However, direct remelting or remolding is not possible due to its thermoset nature. Additionally, GFRPs are difficult to recycle due to the use of inorganic fillers [1].

Fig 3: Composite waste from Industry [4]

1.3 Problem Statement

Regarding the effects on the environment, disposing of composite production waste and end-of-life items is a substantial difficulty. Traditional ways of disposing of composite trash have detrimental environmental implications, which are quite concerning [3]. Also, the glass fibers recycled through the pyrolysis process have reduced strengths due to the generation of surface defects during recycling and hence are not suitable for high-tech applications.

Several proposals have been made forward to address the recycling of thermoset composites. These methods may be divided into three types in general:

1.3.1 Mechanical Comminution

This includes mechanically decreasing the size of composite waste using techniques including crushing, shredding, or milling. The finished product is often utilized as reinforcement in other composites, even though the method decreases water and air pollution.

1.3.2 Thermal Processing:

An alternate option for disposing of the composite waste is thermal recycling, such as energy recovery. Thermal recycling includes burning waste to produce energy rather than immediately recycling composite material.

Even though thermal recycling is unable to directly recycle composite materials, it nonetheless contributes to sustainable waste management by keeping trash out of landfills. It lessens the environmental effect associated with conventional disposal techniques by using the waste's potential for electricity.

1.3.3 Chemical Recycling (Pyrolysis):

Chemical recycling entails utilizing chemical techniques for separating the composite material. This can involve techniques like pyrolysis, which breaks down composite materials into their components by applying intense heat. The matrix material may be recovered and separated from the reinforcing fibers or particles via chemical decomposition of the matrix. It has the benefit of handling a wider variety of composite materials, such as thermoset composites.

Some Studies reported that 25% of the original glass fiber was recycled and a 35% reduction in the tensile strength of glass fibers recovered through pyrolysis. The 80-90% reduction in TRGF performance (and value) in comparison to its original state is a critical technical issue in the development of GFRP recycling technology [5].

This degradation in performance and value can be dependent upon various factors, such as the degradation of the matrix material during pyrolysis, causing harm to the reinforcing recovered glass fibers reducing their strengths due to the generation of surface defects during recycling and hence making them not suitable for high-tech applications [6].

1.4 Solution

Surface chemical treatments can be designed/developed to remove the surface defects of thermally recycled glass fibers and hence restore their strengths rendering them suitable for the fabrication of new high-strength composites.

This project on recycling glass fiber from end-of-life composites significantly contributes to the United Nations' Sustainable Development Goals (SDGs). It aligns with SDG 12, promoting responsible consumption and production by reusing and recycling materials, and reducing waste. Through pyrolysis and post-oxidation techniques, glass fibers are recovered from composite waste, extending their lifespan and diverting them from landfills. This supports SDG 9, encouraging innovative solutions for sustainable manufacturing and circular economies. The chemical treatment procedures restore fiber strength, fostering resource efficiency and enhancing the performance of recycled materials. Overall, this project supports sustainable material management, reduces environmental impacts, and advances a circular economy, addressing multiple SDGs simultaneously.

1.5 Objectives

The objectives of our project were the:

1. **Recovery of the Glass fiber from GFRPs.**
   Extraction and recovery of glass fiber from glass fiber-reinforced polymers (GFRPs) is the initial goal. To remove the glass fibers from the polymer matrix and pyrolyze the composite material, high temperatures must be applied to the GFRP.
2. **Restoration of the lost strength through Chemical Treatment.**
   The strength of the recovered glass fibers is the second goal, which is its focus. The fibers lose their strength because of the heat stress created by the pyrolysis process. This is remedied by using chemical processes that include ABS (acrylonitrile butadiene styrene), hydroxide solutions like NaOH and KOH, and other substances.
3. **Manufacturing of the Composite with the recovered glass fibers.**
   The final goal is to utilize the recovered glass fibers with their restored properties in the production of composite materials. The primary concern will be ensuring that the composite's tensile strength satisfies the requirements as compared to the composites constructed with pristine fibers.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Our study focused on effectively recovering glass fibers from fiber-reinforced composites using pyrolysis and applying chemical recovery processes to these recovered fibers to restore their strength, basically designing a chemical surface treatment to smoothen the defective surface of the recovered fibers which were generated during the thermal treatment. To lay down the foundation for our study, a thorough literature review was conducted to assess the status of the research in the respective field and identify further investigations.

Composites are favored in many industries because of their superior mechanical properties. Particularly, the aerospace, automotive, construction, and sports equipment sectors employ fiber-reinforced polymers (FRPs) [6]. However, the growing usage of these composites presents an engineering community with a new challenge: Recycling these composites rather than landfilling and decomposing them in incinerators? The disposal of waste from composite manufacturing and end-of-life composite goods is a significant concern for the composites industry [1].

Recycling composites, especially thermoset composites, poses challenges due to their complex composition and the nature of thermosetting resins. Thermoset composites typically consist of multiple components, such as fillers, fibers, and resins. Unlike thermoplastics, which can be melted and reshaped, thermoset composites become permanently rigid once their resin molecules are cross-linked [3].

This cross-linking process makes it difficult to remelt or remold thermoset composites, limiting their recyclability. The most used thermosetting resins, such as polyester and epoxy resin, cannot be depolymerized to their original constituents, further complicating the recycling process. The traditional ways of recycling include Mechanical communication and minimizing the size of the grains to be used as reinforcement in bulk molding compounds [5].

The other technique is thermal processing which can be thermal decomposition and chemical decomposition making it reusable in the form of heat energy or residue. We decided to employ the thermal method of pyrolysis since our goal is to recycle glass fiber so that it may be used in new composites rather than as filler material (the byproduct of mechanical sizing). The fluidized bed thermal process is an additional thermal process. However, in this procedure, the composite feed must be shredded into pieces that are 10 mm or smaller due to the high operating temperature of the bed (which is required for full combustion of the polymer phase) [7].

The most significant advantage of pyrolysis is that it does not require pre-treatment composite waste before the recovery process. In this experimental technique, a batch pilot plant that can process whole composite material pieces is used. There are criteria for thermal decomposition since the pyrolysis is done at different temperatures, namely 500, 550, and 600°C for certain times 10, 20, and 30, minutes. An oxidation process is carried out to further investigate the characteristics and behavior of the carbonaceous residue. In this stage, isothermal conditions in a muffle furnace are applied to the glass fibers with the carbonaceous residue. The fibers are maintained at temperatures of 500 or 600 °C for intervals of 10 to 60 minutes, performing the cleaning action, converting carbonaceous residue as carbon oxides. All the experimentation was carried out in a Protherm tube furnace including pyrolysis and post oxidation using the Argon gas to provide inert atmosphere and air as oxidizing atmosphere, making it the feasible utility for thermal treatment.

Following the successful recovery of glass fibers through the pyrolysis procedure, chemical treatment is the next stage in the procedure. This stage is essential for rebuilding the fibers’ strength, which is lost due to the surface defect that occurred during the thermal treatment. According to some reports, glass fibers can lose up to 70% of their strength after being pyrolyzed. Due to the harsh conditions employed in this procedure, the glass fibers suffer from a severe loss in strength, and therefore cannot be reused in many forms of composite applications [8].

These damaged filaments can however be reused as reinforcements if their strength is restored using chemical treatment. The restored strength glass fiber strength is given after a short immersion in an alkaline solution specifically diluted hydrofluoric acid (HF), heat conditioned at 450–600 C. In addition to being a recognized chemical etchant, HF is also believed to reinforce glass by removing angular, severe surface defects. However, because HF is extremely hazardous by nature, its commercial application for reinforcing the strength of glass fibers is difficult. Different alkaline solutions such as NaOH, LiOH, and KOH were used as chemical etchants and were prepared of different molarities as 2, 3, and 5 M, for certain treatment times and temperatures e.g., 95 ^{0}C [3]. The standard treatment time was 10 minutes, but this was varied in a subsequent experiment (at 2, 5, 20, and 30 minutes) to investigate the effect on fiber properties. After being treated in an alkaline solution, the fibers were rinsed in 5% HCl solution for 5 minutes before being rinsed with deionized water for 1 minute. This is the procedure that we've adopted for our experiment, having the fibers treated in 3M NaOH for 10 minutes, consequently rinsing in 5% HCl and deionized water followed by drying in an oven at 100 ^{0}C for around 6 hours to remove any moisture left within the fibers.

Tensile testing of single fibers under certain conditions is essential for our study to collect data for comparison. It is necessary to demonstrate that the fibers' strengths significantly alter both after chemical treatment and after pyrolysis. Since single fibers are challenging to work with, we sought to discover an appropriate technique to prepare our samples without causing any harm to the fibers.

The samples were prepared according to ASTM C1557-03 standard, the card tabs were prepared by using this standard, and single glass fibers were mounted on card tabs using polyester resin. Fiber diameters were measured with an optical microscope before the card tabs had been attached to the UTM [3]. The low-modulus glass fibers were tested by direct gripping with a load cell of 50 N and around 50 samples were tested to obtain a set of representative set of results. Tensile tests were carried out until failure at a cross-head displacement rate of 1 mm/min. And with a displacement rate of 1.5mm/min. We have conducted our investigation using the Universal Testing Machine UTM with 30 single fibers cards frame prepared according to the standard and having a cross-head displacement rate of 1.2 mm/min. The composite samples were prepared using ISO 527 standards having a gauge length of 12of mm and a load cell of 50 kN was used. Tensile tests were carried out until failure at a cross-head displacement rate of 2 mm/min.

The explanation of these results provides significant luminescence on single fibers' mechanical behavior. The greatest strengths of individual fibers and the likelihood of survival are plotted against this maximum strength to get the Weibull modulus or Weibull form factor, which may be used to visualize the dispersion in the strength data using the Weibull distribution. This Weibull modulus may also be used to determine the probability that a certain sample dataset will fail. The Weibull distribution gives us the failure rate of the fibers that can be used for making composite [9].

To fully understand the mechanisms at work, such as brittle failure and strength regeneration, we also need to be familiar with the many methods we might characterize our samples. A HITACHI SU-6600 field emission scanning electron microscope (FE-SEM) may be used to examine surface morphology.

The same study looked at the observable mid-infrared chemical vibrational modes in alkali-treated fibers using a portable diffuse interface for an Agilent Technologies 4100 FT-IR with an external reflectance instrument. SEM analysis will be used in our study to comprehend the fracture surface of untreated, pyrolyzed, and chemically treated fibers, determining the strength comparison. We have used FTIR and SEM for the previously stated objectives.

To comprehend the structural modifications in the fibers, FT-IR analysis was done. It was shown that the alkali treatments at first increased fiber tensile strength by interacting with surface silicate groups and altering the affected surface layer.

The recovery and restoration of glass fibers from fiber-reinforced composites was the focus of our work. We sought to overcome the difficulties of recycling thermoset composites and improve the strength of the fibers by pyrolysis and chemical treatment. We carried out studies under various treatment circumstances and assessed the fiber characteristics using various techniques. According to our research, alkaline treatments can alter the top layer of fibers to increase fiber strength, but too much treatment can harm the fiber's structural integrity. This study adds to our understanding of glass fiber regeneration and recovery by revealing new possibilities for its repurposing in composite materials. To improve the chemical treatment procedure and investigate further recycling options for thermoset composites, more research is required.

2.2 Effects of thermal treatment on Glass Fiber

The behavior of glass fibers is significantly influenced by thermal treatment. According to a study [8], glass fibers can significantly lose strength at failure at room temperature when thermal treated between 250 and 600 °C, like those that occur during the recycling process of end-of-life composites. According to the study, silane and water-sized E-glass fibers responded differently to thermal conditioning. Water-sized fibers first demonstrated greater strength than silane-sized fibers, which had a protective surface layer. However, their strength at room temperature was significantly reduced by conditioning at temperatures more than 250 °C. Water-sized fibers, on the other hand, showed a linear decline in tensile strength with rising conditioning temperature.

Two distinct thermal conditioning protocols bundle thermal conditioning and single fiber thermal conditioning were created and used in the study [10]. In bundle thermal conditioning, single fibers were retrieved for testing after bundles of fiber were treated at the desired temperature and cooled. In contrast, single fiber heat conditioning required removing them from a bundle and cementing them to separate wireframes. Then, these frames underwent batch thermal conditioning. The fibers or fiber bundles were fixed at both ends during both operations to avoid applying longitudinal tensile stresses. Additionally, to allow for thermal expansion without creating longitudinal tensile stress, the fibers were placed with considerable slack along their length. It should be noted that any bending stresses caused by convective air currents were concentrated at the fiber ends, which were not part of the tested gauge length.

In conclusion, the strength and strain at failure of glass fibers can be significantly affected by the thermal treatment procedures used. Due to their protective coating, silane-sized fibers initially have a higher strength, but as the temperature rises, this strength quickly falls. As the conditioning temperature rises, the strength of water-sized fibers declines linearly. Specific thermal conditioning processes, such as bundle, and single-fiber approaches, have been developed to enable controlled testing while reducing the influence of external stresses.

2.3 Significance of Alkaline Solutions

Numerous studies have been conducted on the significance of alkaline solutions on glass fibers in terms of their corrosion behavior and the impact on fiber failure stress. Study [11] examined the effects of various chemical aging solution compositions, such as NaOH and cement, on the corrosion process of AR-glass and basalt fibers. The investigation discovered that while the failure stress reduction showed clear variations, both treatments produced highly corroded states at high temperatures. The failure stress gradually dropped in a NaOH solution, with brief periods of increased stress. In contrast, the filaments did not exhibit a substantial reduction in failure stresses in cement solution over a broad range of temperatures and time. These findings highlight the importance of understanding specific aging conditions and their impact on the durability and failure susceptibility of glass fibers.

The study also stressed that the susceptibility of the fiber to failure might not be fully captured by focusing merely on mass loss as a measure of chemical resistance. Although mass loss is frequently employed in standards to gauge a fiber's chemical resistance, this method lacks information about the fiber's failure characteristics, which are essential for gauging a fiber's resistance. Strong aging regimes, especially in NaOH solution, might result in the production of peeling shells that eliminate the applied sizing. This makes it difficult to make judgments regarding the efficacy of sizing based purely on filament tensile tests.

Additionally, the study [12] describes the mechanism of water reaction with glass, which is important to comprehend when examining the impact of diluted alkali solutions. Glass is first attacked at the surface, which produces a hydrated silica coating from which water-soluble glass components leach into the solution. The remaining coating serves as a barrier, gradually decreasing the rate of attack. The glass attack is accelerated by adding alkali to water because the thinner, more porous hydrated silica coating is more soluble in alkaline solutions. In terms of the rate of glass attack, NaOH solutions up to 0.5 N strength typically outperform sodium silicate solutions. The presence of silica in solution retards the removal of the barrier or the hydrated high-silica residual glass film, leading to a linear increase in the glass-attack rate over time in weakly alkaline solutions.

In summary, the significance of alkaline solutions on glass fibers lies in their ability to induce corrosion, affect failure stress, and alter the glass-attack rate, see Fig 4. Understanding the specific chemical compositions of the solutions, the behavior of the protective layers, and the interplay between alkali and hydrated silica films is crucial for evaluating the durability and performance of glass fibers in various applications.

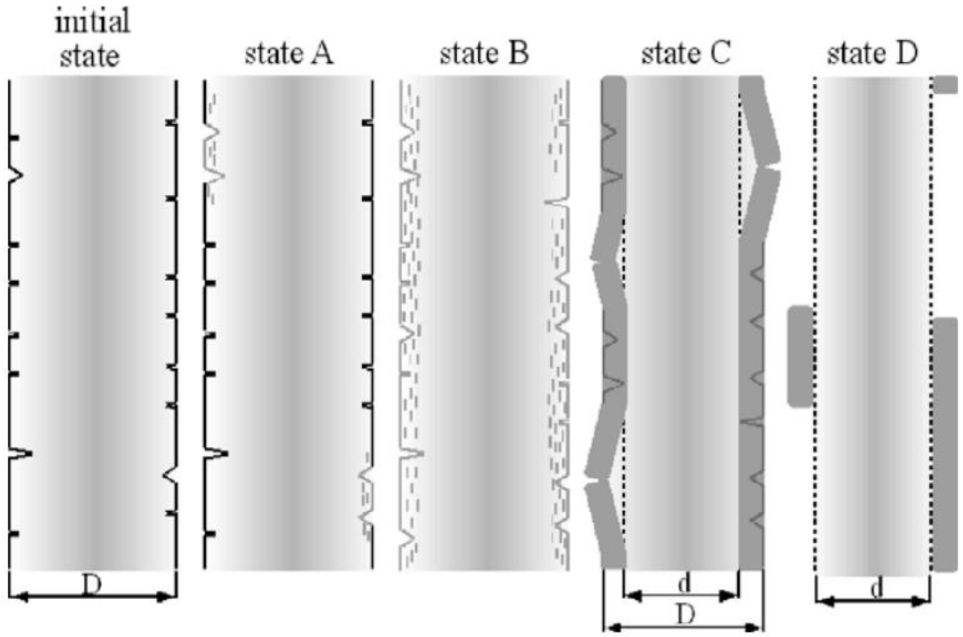


Fig 4: Different stages of etching using hot NaOH [11]

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter focuses on the experimentation that was carried out to successfully recover the glass fibers from composite waste, followed by restoration of lost strength, and finally the manufacturing of composite material from these recovered and restored glass fibers.

3.2 Pyrolysis & Post Oxidation

3.2.1 Pyrolysis

Pyrolysis is a critical process employed in this project to recover glass fibers from glass fiber-reinforced composites. It involves subjecting the composite samples from Fiber Craft Industries to high temperatures in the absence of oxygen, causing the material to undergo thermal decomposition. During pyrolysis, the organic matrix surrounding the glass fibers is decomposed and vaporized, leaving behind the glass fibers. The carbonaceous layer, a residue of the decomposed matrix, adheres to the glass fibers and needs to be eliminated to restore their mechanical properties. Pyrolysis serves as the initial step to break down the composite material and isolate the glass fibers, facilitating their subsequent recovery and further treatment. By understanding the purpose of pyrolysis, we can effectively recover the valuable glass fibers from the composite material, which can then be utilized in various applications, contributing to the circular economy, and reducing waste in the industry.

3.2.2 Post-Oxidation

Post-oxidation, carried out after pyrolysis, plays a crucial role in the removal of the carbonaceous layer that remains on the surface of the glass fibers. The carbonaceous layer forms because of incomplete decomposition of the organic matrix during pyrolysis. To effectively restore the mechanical properties of the glass fibers, it is necessary to eliminate this carbonaceous residue. Post-oxidation involves subjecting the recovered fibers to controlled oxidation in the presence of oxygen at elevated temperatures. The carbonaceous layer reacts with oxygen, leading to its combustion and subsequent removal. The application of post-oxidation ensures the purification of the recovered fibers, eliminating the carbonaceous impurities and enhancing their mechanical strength.

3.2.3 Equipment:

- Protherm tube furnace.
- Quartz boat.
- Glass tube.
- Argon cylinder.
- Compressor.
- Transparent beaker.
- Weighing balance.
- Pipes.
- Rubber stopper.
- Rod.

3.2.4 Procedure

Pyrolysis and post-oxidation were integral stages in this project, aimed at the recovery of glass fibers from glass fiber-reinforced composites and the subsequent removal of the carbonaceous layer left behind after pyrolysis. The pyrolysis process, performed before post-oxidation, involved subjecting the composite samples to a temperature of 400 °C for a holding time of 20 minutes. To eliminate the carbonaceous layer, post-oxidation was carried out at 600 °C, with different holding times (5, 10, and 20 minutes) employed for various experiments. These procedures were based on the work of Loris Giorgini (2016), which outlines the application of post-oxidation after pyrolysis to eliminate the carbonaceous residue.

For the pyrolysis and post-oxidation processes, a Protherm tube furnace was employed, see Fig 6. The samples were placed in a quartz boat, ensuring that they were not covered by the boat from any side, see Fig 5. The boat, with the samples, was then carefully positioned at the center of a glass tube using a rod that was then put inside the furnace. To establish an inert atmosphere during pyrolysis, argon gas was supplied from a cylinder. The glass tube was sealed at both ends using rubber stoppers equipped with pipes for argon and oxygen supply. During the experimental setup, safety measures were followed, including the use of gloves to handle the samples.

To begin the pyrolysis process, the tube furnace was powered on, and a ramp rate of 10 °C per minute was set. For pyrolysis, the temperature was maintained at 400 °C for a ramp duration of 40 minutes, with a holding time of 20 minutes. Subsequently, post-oxidation was conducted at 600 °C, following the completion of pyrolysis, with a ramp duration of 20 minutes and a holding time of 20 minutes (5 and 10 minutes in different experiments). To establish an inert atmosphere during pyrolysis, argon gas was supplied by connecting one end of a pipe to the argon cylinder, while the other end was placed in a water beaker to control the flow rate.

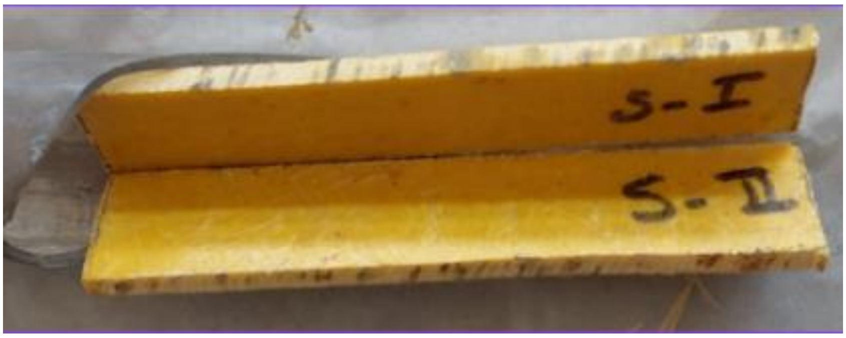


Fig 5: Sample from Industry

Fig 6: Protherm Tube Furnace

Fig 7: Carbonaceous Layer Present on Sample

After the completion of the pyrolysis process, the gas supply was switched from argon to oxygen for the post-oxidation step. The post-Oxidation step is necessary to remove the carbonaceous layer present in samples, see Fig 7. A compressor was used to provide air, and its flow rate was adjusted to ensure controlled oxidation. The flow rate was monitored by observing the bubbles appearing in the water beaker, with adjustments made to the compressor knob to achieve a controlled and steady flow. Following post-oxidation, the tube furnace was turned off, and the samples were carefully removed from the furnace using appropriate precautions. Clean samples were obtained after this step, see Fig 10.

Weighing the composite samples using a weighing balance was conducted before and after the recovery process. The weight change was recorded as a measure of the recovery efficiency. The weighing balance ensured accurate measurement of the sample mass, facilitating the evaluation of the impact of pyrolysis and post-oxidation on the samples' weight.

Fig 8: Sample After Pyrolysis

Fig 9: Protherm Tube Furnace

Fig 10: Sample after Post-Oxidation

3.3 Chemical Treatment

The chemical treatment procedure, aimed at regenerating the strength of the glass fibers recovered after pyrolysis and post-oxidation, involved several steps. The recovered fibers were treated in a solution of NaOH heated to a temperature of 95 °C on a hot plate, see Fig 12. This treatment was conducted for a duration of 10 minutes. Subsequently, the fibers were treated with 5% HCL for a period of 7 minutes. The fibers were then rinsed with water to ensure the effective removal of residual deposits formed on the fiber surface because of the interaction with the alkaline solution. The specifications and parameters for the chemical treatment procedure were derived from the research paper authored by Thomason et al. (2017), which guided optimizing the treatment conditions for the strength regeneration of the fibers.

3.3.1 Equipment

- Weighing balance
- Spatula
- Beakers
- Hot plate
- Thermometer
- Fume Hood

3.3.2 Procedure

To carry out the chemical treatment procedure, several accessories, like a beaker, spatula, thermometer, etc., were utilized. A hot plate was used to heat the NaOH solution to the desired temperature of 95 °C. Beakers were employed to contain the solution during the treatment process. A thermometer was used to monitor and maintain the temperature of the solution accurately. A pipette facilitated the precise measurement and transfer of the required amounts of HCL solution. Tweezers were used for the careful handling of the fibers during the treatment stages. A spatula was used to measure and add the appropriate amount of NaOH pellets to create the desired concentration of the solution. Aluminum foil was utilized to store the treated fiber bundles. Finally, drying was carried out by putting these fibers in a furnace for six hours, see Fig 13. This removed any moisture that could disrupt the network when the composite is made.

To prepare the NaOH solution with the correct concentration, the mass of NaOH pellets needed was calculated using the formula:

Mass = Molarity x Volume of solution in liters x Molar mass.

This calculation ensured the accurate determination of the quantity of NaOH required to achieve the desired concentration for the treatment solution. The required mass of pellets was then weighed before being added to water. For our experiments, we used 12 grams of NaOH with 100 ml of water.

By following this methodology, the glass fibers recovered from the pyrolysis and post-oxidation stages underwent a chemical treatment process utilizing NaOH and HCL solutions and more clean fibers were obtained, see Fig 14. This treatment aimed to regenerate the strength of the fibers, and the procedural details, along with the equipment used, provided a systematic approach to effectively carry out the chemical treatment procedure.

Fig 11: Thermally Treated Fibers

Fig 12: Hot NaOH

Fig 13: Oven for Drying

Fig 14: Chemically Treated Clean Samples

3.4 Tensile Testing of Fibres

3.4.1 Equipment

- Universal Testing Machine
- Micro-tweezers
- Custom-made stamp
- Hammer
- Scissors
- Cutter
- Dropper

3.4.2 Procedure

The tensile testing of single fibers was conducted using a Universal Testing Machine (UTM) following the ASTM C1557-03 standard for sample preparation and testing. The methodology involved the production of card tabs with a window length of 20mm, which matched the gauge length of the fibers, see Fig 15. Initially, rectangular pieces of the card tabs were cut, and a window with a length of 20mm (representing the gauge length) was created using a custom-made stamp.

Single fibers were then carefully selected from the bundles and placed on the card tabs using micro tweezers. Drops of epoxy were applied to secure the fibers onto the card tabs using a dropper, to ensure proper adhesion. The samples were allowed to cure for a day, ensuring the epoxy had been fully set. Following the curing period, the diameter of the fibers was measured at three positions using an optical microscope, and the average diameter was recorded.

For the tensile testing, a load cell with a capacity of 5 kN was used in conjunction with a strain rate of 1.2 mm/min. The prepared samples, with the fibers securely attached to the card tabs, were fixed onto the UTM, see Fig 16. To ensure that the entire load was applied to the single fiber, the thin sections of the card tabs along the window area were carefully cut off. This ensured that the load was concentrated solely on the fiber within the designated gauge length.

A total of 30 samples were created and tested for each specific condition, including pristine fibers, pyrolyzed and post-oxidized fibers, and chemically treated fibers.

The tensile testing procedure involved applying a controlled load to the fibers until failure occurred, measuring the corresponding stress and strain values.

By following this detailed methodology, adhering to the ASTM standard, and employing appropriate sample preparation techniques, the tensile properties of single fibers could be accurately determined using the UTM, see Fig 17. The methodology ensured consistent and reliable testing across different fiber conditions, providing valuable data for evaluating the effects of pyrolysis, post-oxidation, and chemical treatment on the strength characteristics of the fibers.

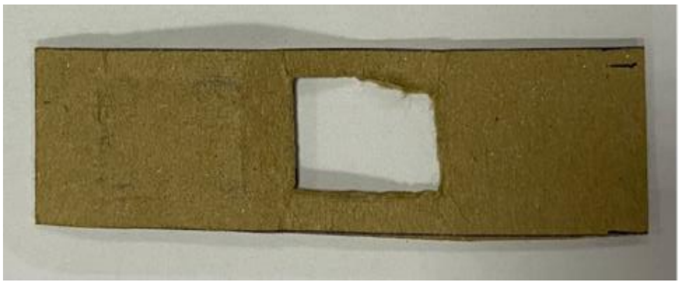


Fig 15: Card Tab with Single Fiber

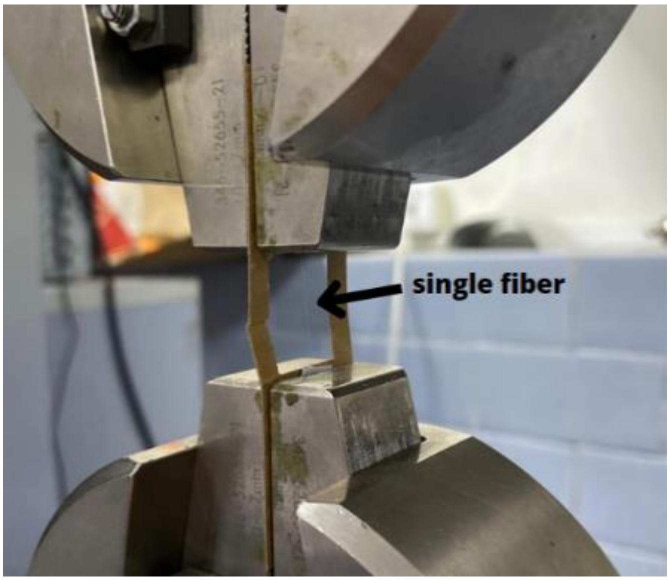


Fig 16: Card Tab Sample Clamped in UTM Jaws

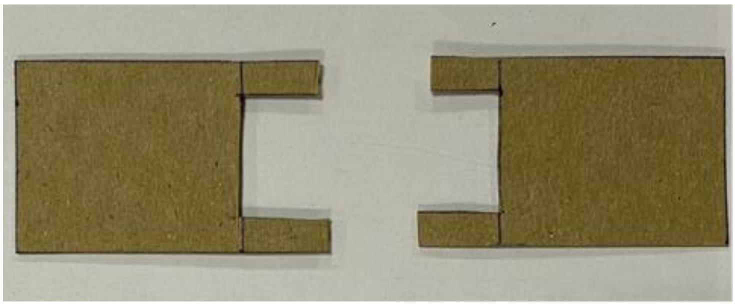


Fig 17: Card Tab after Tensile Testing

3.5 Composite Manufacturing

3.5.1 Equipment

- Mould
- Hammer
- Weigh balance
- 10 Kg weights
- Bottom parts of plastic bottles
- Grinder

3.5.2 Procedure

The composite manufacturing stage involved the fabrication of composites using polyester resin and continuous fibers, see Fig 18 and Fig 19. The initial step was to create composites using pristine fibers. The casting method was employed, where the fibers were carefully placed within a mild steel mold, see Fig 20. Subsequently, the polyester resin was poured over the fibers, ensuring even distribution. To consolidate the composite, the mold was closed, and weights were applied to provide adequate pressure for proper fiber impregnation. This step facilitated the formation of a uniform composite structure. The composites were then allowed to cure for a period of 24 hours, enabling the polyester resin to solidify and bond the fibers effectively.

The purpose of creating these composites was solely for subsequent tensile testing. For each composite, fibers with a length of 82mm were utilized, and the mass of fibers incorporated in a single composite was measured to be 3.5g using a weighing balance. The polyester resin was prepared by combining 100g of polyester with 1% Methyl Ethyl Ketone Peroxide (MEKP) as the hardener and 0.5% cobalt acetate as the initiator. Again, a weighing balance was used for this purpose. The addition of the hardener and initiator served distinct roles in the polyester resin formulation. The hardener, MEKP, initiates the crosslinking reaction within the polyester resin, enabling it to cure and develop its mechanical strength. The initiator, cobalt acetate, aids in promoting the polymerization process, ensuring effective resin curing and overall composite integrity.

Following the curing period, the composite samples were carefully removed from the molds. These samples were then transported to the mechanical workshop, where they were cut precisely to the desired lengths using a grinder for subsequent tensile testing of the composites. The cutting process ensured that the samples had consistent dimensions and met the specific gauge length requirements for accurate tensile testing, see Fig 21. After this stage, the next step was to attach the tabs to the composite samples gripping section, these tabs were used to ensure that all the tensile loads should be applied to the gauge length section. For making these tabs, a glass fiber mat was used, and polyester resin was used to attach these tabs to the composite sample, see Fig 22. After a curing time of 24 hours, final composite samples were obtained, see Fig 24

Furthermore, composites were also manufactured using a similar procedure but utilizing fibers obtained after the pyrolysis and post-oxidation stage, as well as fibers treated with the chemical treatment process. The objective was to investigate and compare the mechanical properties of composites fabricated from different types of fibers, allowing for a comprehensive evaluation of the effectiveness of the recovery and treatment processes on the resultant composite strength.

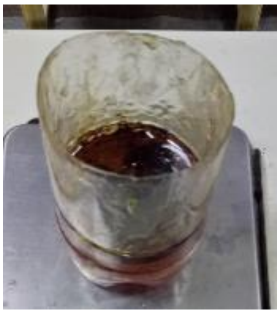


Fig 18: Polyester Resin

+

Fig 19: Chemically Treated Fibers

↓

Fig 20: Mold with Fibers plus Resin

↓

Fig 21: Composite sample

↓

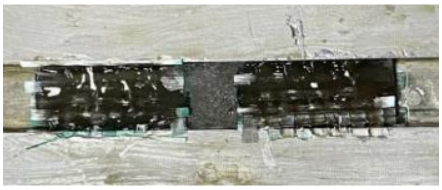


Fig 22: Tabs attached on Grip Section

↓

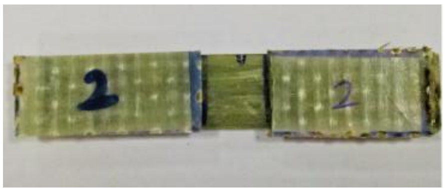


Fig 23: Final Composite Sample

3.6 Tensile Testing of Composite

3.6.1 Equipment

- Universal Testing Machine
- Mould
- 10 Kg weights
- Filer

3.6.2 Procedure

The next stage of the project involved conducting tensile testing on the fabricated composites. To ensure standardized testing procedures, the International Organization for Standardization (ISO) 527 standard was followed for sample preparation and testing. Tensile testing was performed using a universal testing machine (UTM) equipped with a 50 kN load cell, and a constant strain rate of 2mm/min was applied during the testing process.

During the initial testing attempts, a challenge was encountered regarding the gripping of the composite samples on the jaws of the UTM. The composites exhibited slippage as the load was applied, resulting in inaccurate test results. To address this issue, a modification was introduced in the sample preparation stage. Composite tabs were created using the hand lay-up method and were attached to the composite samples to enhance gripping. This modification involved shaping the composite samples into a dog-bone configuration by affixing tabs to the ends of the samples using polyester.

However, during testing, the interface between the composite sample and the attached card tabs proved to be weak, leading to premature fracture at this interface. Recognizing this limitation, a revised approach was implemented. Instead of creating composite tabs separately and then attaching them to the composite samples, the card tabs were directly molded onto the composite samples using the same mold that was employed for fabricating the composite samples. The load was applied consistently during testing, ensuring a stronger interface between the composite sample and the attached composite tabs. The composite samples on which the tabs were attached were also not allowed to fully cure. Moreover, the roughness of the sample over the surface where the tabs would be attached was increased through a mechanical file. This approach eliminated the weak interface issue and provided more reliable and representative test results.

Moreover, the fiber used in the composite tabs was in woven form. The choice of woven fibers for the tabs was made to enhance the interlocking effect between the composite and the tabs, thereby further improving gripping and preventing slippage during tensile testing. The woven fiber structure provided increased mechanical interlocking, enhancing the overall integrity and reliability of the gripping mechanism during the testing process.

By employing these modifications in the sample preparation and testing procedure, the tensile testing stage aimed to obtain accurate and consistent mechanical properties of the composites, ensuring reliable data for further analysis and comparison.

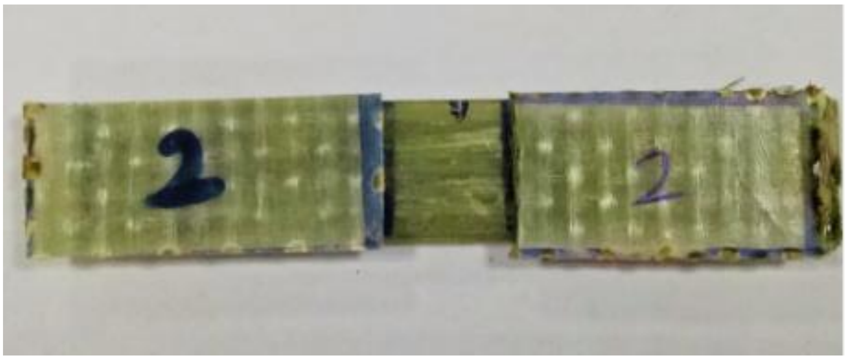


Fig 24: Final Composite Sample

Fig 25: Composite Sample in UTM Jaws

Fig 26: Fractured Tested Samples

CHAPTER 4

CHARACTERIZATION TECHNIQUES

4.1 Introduction

To study the composition, morphology, and strength of these fibers we carried out some well-known characterization techniques. These include Optical Microscopy, Scanning Electron Microscopy (SEM), Fourier Transform Infrared (FTIR) Spectroscopy, and Tensile Testing of single fibers as well as composite.

4.2 Optical Microscopy

Optical microscopy was used to visualize the residue that remains on the glass fiber's surface after post-oxidation, and it is removed after chemical treatment. It was also used to measure the diameter of a single glass fiber which was further used in tensile testing.

Its significance stems from its capacity to deliver high-resolution imaging and thorough characterization of a variety of materials and biological specimens. We can analyze the morphology and structure of samples using optical microscopy, which aids in the understanding of fundamental processes and occurrences.

The optical microscopy working principle involves the use of visible or near-visible light to illuminate the sample under examination. An illuminating source, objective lenses, eyepieces, and detectors are among the major components of a microscope. The sample is positioned on a stage beneath the objective lens. Depending on the nature and qualities of the sample, light undergoes numerous phenomena such as absorption, reflection, scattering, and refraction when it travels through or interacts with it. The transmitted or reflected light is collected by the objective lens, which creates an image of the sample. The image is then enlarged and passed through additional lenses or optical components before arriving at the eyepiece or detector. The eyepiece allows the researcher to observe the image directly, whilst detectors such as cameras capture the image digitally for future study and documentation. [13]

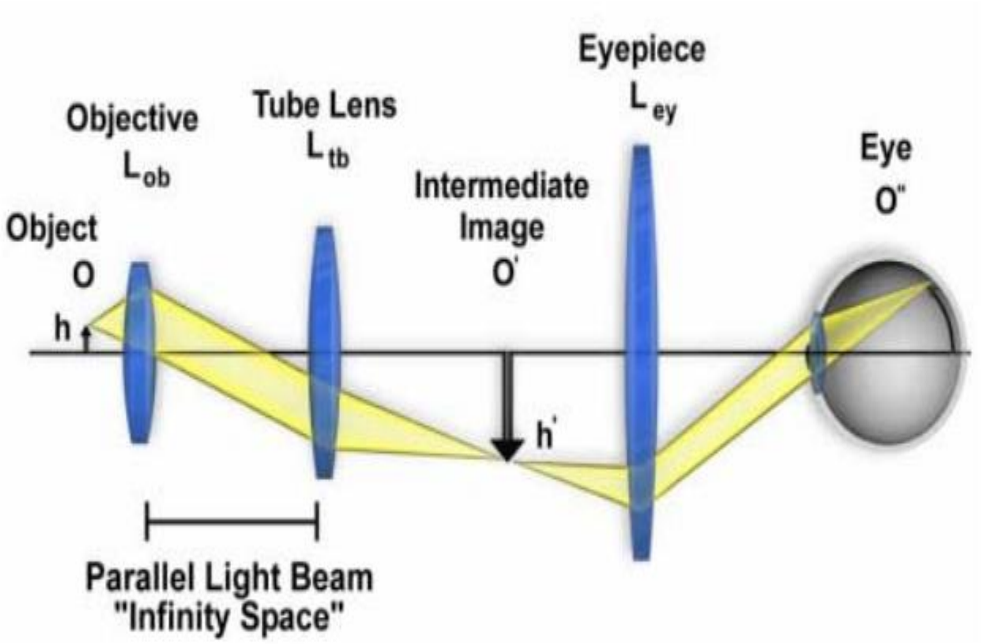


Fig 27: Working Principle of Optical Microscopy [13]

4.3 Scanning Electron Microscopy

SEM was used to analyze the fracture surface of single glass fibers on which tensile testing has been performed. Those samples were selected from the fractured ones which have a very small length remaining part attached to the card tabs.

SEM is a powerful imaging technology that is widely utilized in materials science, metallurgy, and a variety of other disciplines of study. It has high magnification, resolution, and depth of focus, allowing us to investigate sample surface morphology and composition in detail.

Surface morphology, topography, and microstructure of a variety of materials, including metals, alloys, ceramics, polymers, and biological specimens, can be visualized and analyzed using SEM. This data is critical for comprehending material behavior, surface flaws, and the impacts of processing or treatment.

Its working principle is based on the creation of a finely focused electron beam with the help of a high-voltage electron source, often a tungsten filament or a field emission gun (FEG). The electron beam has a far shorter wavelength than visible light, enabling high-resolution imaging. The primary electron beam is concentrated and directed onto the specimen under the investigation's surface. Elastic scattering occurs when electrons in the beam bounce off the atoms in the sample without losing energy. These are secondary electrons, and they are detected using a secondary electron detector. Topographical information is provided by secondary electrons, resulting in high-resolution surface images.

An electron gun, detector, focusing lenses, and sample chamber comprise the SEM configuration. The electron gun discharges high-energy electrons at the surface of the sample in the sample compartment. The electron beam is focused onto the sample by the focusing lenses, and the secondary electrons emitted are detected by the detector. To prevent air molecules from diverting the electron beam, a vacuum pump is utilized to produce a vacuum inside the chamber.

Sample preparation needs to be done before SEM analysis as it is only carried out for conductive samples. If a sample is not conductive, then a sputter coater is used to deposit a conductive layer on the sample, i.e., gold. This causes excessive electrons to flow through the sample surface to obtain a high-resolution image. [14]

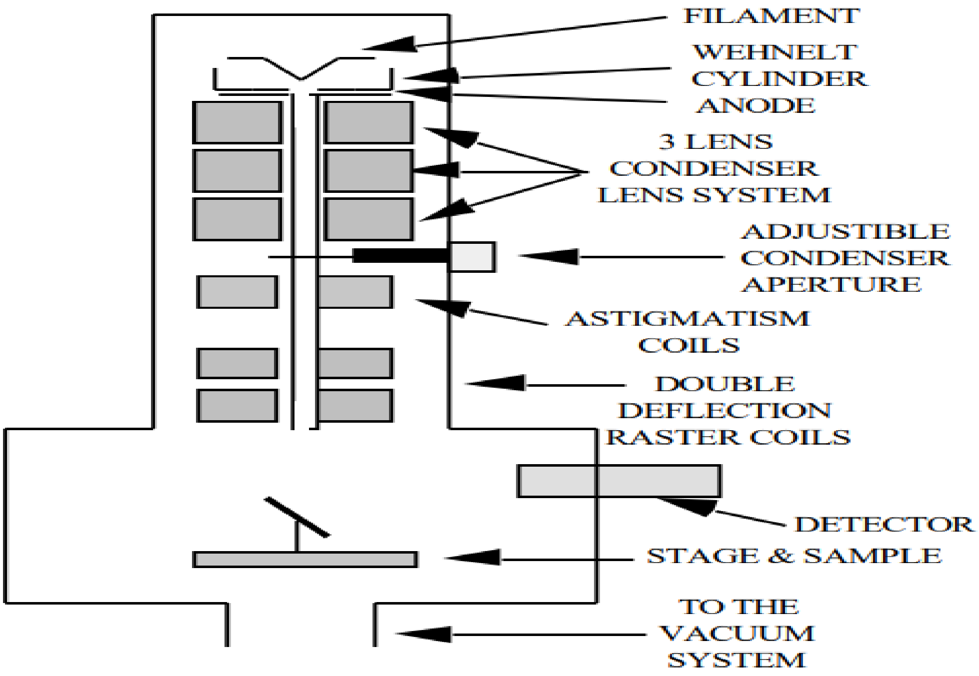


Fig 28: Schematic of SEM Column and Specimen Chamber [14]

4.4 FTIR Spectroscopy

FTIR Spectroscopy was used to analyze the functional groups present in pristine, post-oxidized, and chemically treated fibers.

Organic and inorganic substances are identified and characterized using FTIR spectroscopy. It may assess the existence of functional groups and provide vital information about a sample's chemical makeup by analyzing the distinctive absorption patterns of molecular bonds. We can use this technique to explore the molecular structure and conformation of substances. We can identify the different types of bonds present by investigating the specific frequencies and intensities of infrared absorption bands, such as O-H, C=O, or C-H bonds. This data assists in comprehending the arrangement and connectivity of atoms within a molecule.

FTIR spectroscopy operates by analyzing the interaction of infrared light with a sample to provide valuable insights into its chemical composition and molecular structure. The process starts with preparing the sample, in the form of a small solid pallet. An infrared light source emits a wide range of infrared wavelengths, which an interferometer divides into two beams. One beam is aimed at the sample, while the other is used as a reference. Certain wavelengths of infrared light are absorbed by the sample as it interacts with it, dependent on its chemical composition and molecular interactions. A sensitive infrared detector detects the transmitted or reflected light from the sample and reference beams. The collected electrical signals are then processed using the Fourier transform to translate them from the time domain to the frequency domain, yielding an infrared spectrum. This spectrum is analyzed to find characteristic peaks and absorption bands, allowing the chemical components and molecular structure of the material to be identified and analyzed. FTIR spectroscopy is a versatile and strong analytical technique that is used in a variety of scientific domains to perform qualitative and quantitative analyses on a wide range of substances. [15]

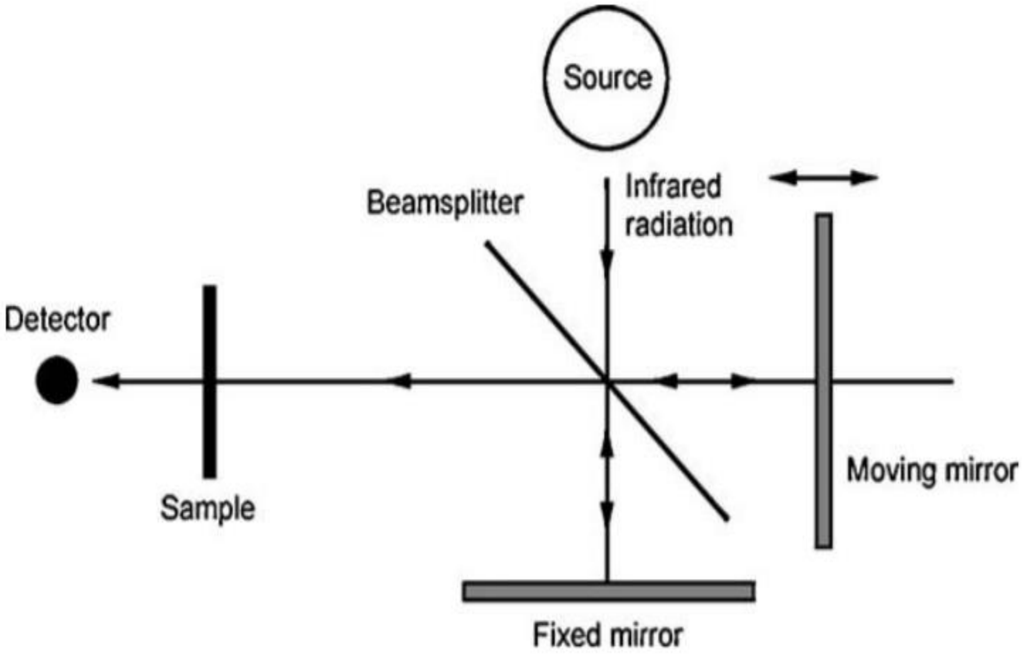


Fig 29: Schematic of Basic Principle of FTIR Spectroscopy [16]

4.5 Tensile Testing

To measure the strength of recovered and restored glass fibers we performed tensile testing on these single glass fibers, according to ASTM C1557-03 standard. Also, tensile testing was performed on the composite samples made from these recovered and restored glass fibers, according to ISO 527 standards [3].

Tensile testing operates on an incredibly simple principle. A test specimen is constructed with precise dimensions, sometimes in the form of a standardized shape such as a dog bone or cylinder. The specimen is next firmly fastened into a testing machine, which exerts a tensile force along its longitudinal axis that gradually increases. The specimen elongates as the force is applied, and the machine records the accompanying load or stress and deformation or strain.

The load and displacement are continually measured during the test, allowing for the generation of a stress-strain curve. This curve depicts the material's response to applied force, demonstrating how it deforms and eventually breaks. The elastic region, where the material responds linearly and can revert to its original shape if the load is removed, and the plastic region, where permanent deformation occurs, can be isolated from this curve.

CHAPTER 5

RESULTS AND DISCUSSIONS

5.1 Introduction

After performing the characterization techniques, desired results were obtained. This chapter focuses on these results obtained after performing several characterization techniques on the samples. The discussion of these results is also present in this chapter.

5.2 Morphology

First, the samples were analyzed by the naked eye to get an idea about the morphology of glass fibers obtained after pyrolysis, post-oxidation, and chemical treatment. Their morphology was compared with the pristine glass fibers on which no thermal or chemical treatment has been performed.

We can see in Fig 30, that these pristine fibers are completely clean. However, in Fig 31, the pyrolyzed glass fibers have a carbonaceous layer present on them which remains on the fibers after pyrolysis. This carbonaceous layer is not required because if this layer remains on the fibers, then we cannot separate the fibers for single fibers tensile testing. So, this carbonaceous layer was removed by using a post-oxidation treatment which was carried out at 600 ^{0}C, and clean fibers were obtained, as we can see in Fig 32. But still, some residue at the micro level remained on these post-oxidized fibers which we had observed in optical microscopy, see. To remove this residue, we performed a chemical treatment, and more clean fibers were obtained, as we can see in Fig 33. These chemically treated fibers are almost as clean as pristine fibers.

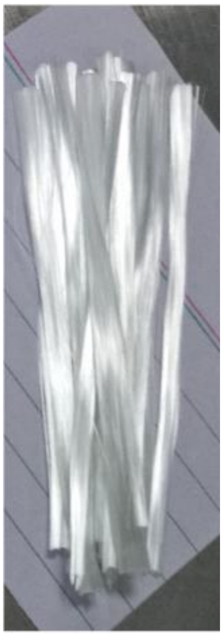


Fig 30: Pristine Fibers

Fig 31: Pyrolyzed Fibers

Fig 32: Post-Oxidized Fibers

Fig 33: Chemically Treated Fibers

5.3 Optical Microscopy

Optical microscopy was used to analyze the morphology of single fibers before and after thermal and chemical treatments. Here, we can see in Fig 34, that the single fiber surface is completely clean. However, in Fig 35, some residue is present on the surface of a post-oxidized single fiber, and in Fig 36, the chemically treated single fiber's surface is as clean as the pristine single fiber's surface.

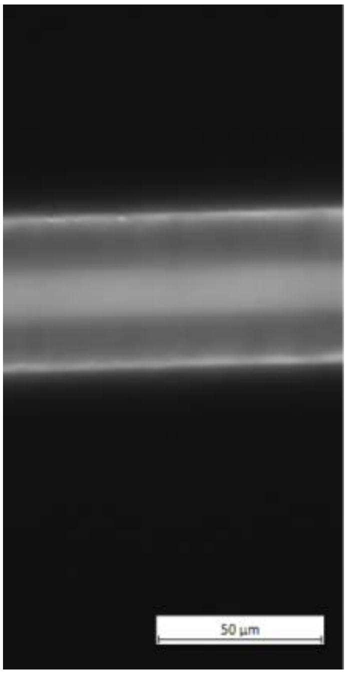


Fig 34: Single Pristine Fiber

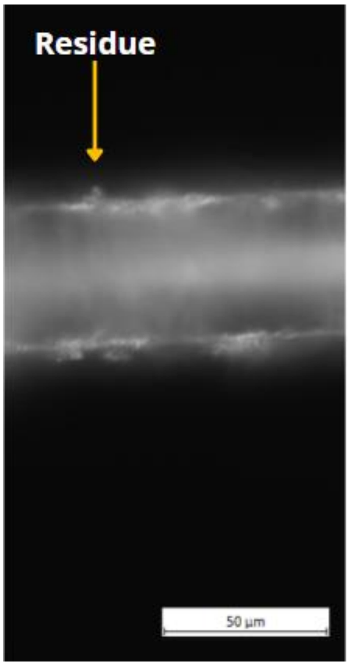


Fig 35: Single Post-Oxidized Fiber

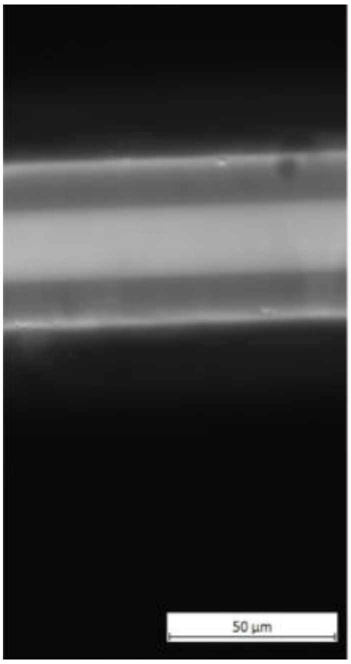


Fig 36: Single Chemically Treated Fiber

Optical microscopy was also used to calculate the diameters of single fibers to use in tensile testing experiments. From these diameter calculations, we also obtained some useful results related to the morphology of these thermally and chemically treated fibers. We can see in Fig 37 and Fig 38, that the diameter is almost comparable, for pristine and post-oxidized fibers. But the diameter of chemically treated fibers, see Fig 39, is significantly reduced. This indicates that the residue as well as surface defects are removed from these fibers' surfaces after chemical treatment. As these surface defects, which play a major role in the strength reduction of these fibers after thermal treatments, are removed now so, the strength of these fibers is recovered. These defects are removed due to the etching effect of the hot alkaline NaOH solution that we are using in the chemical treatment procedures.

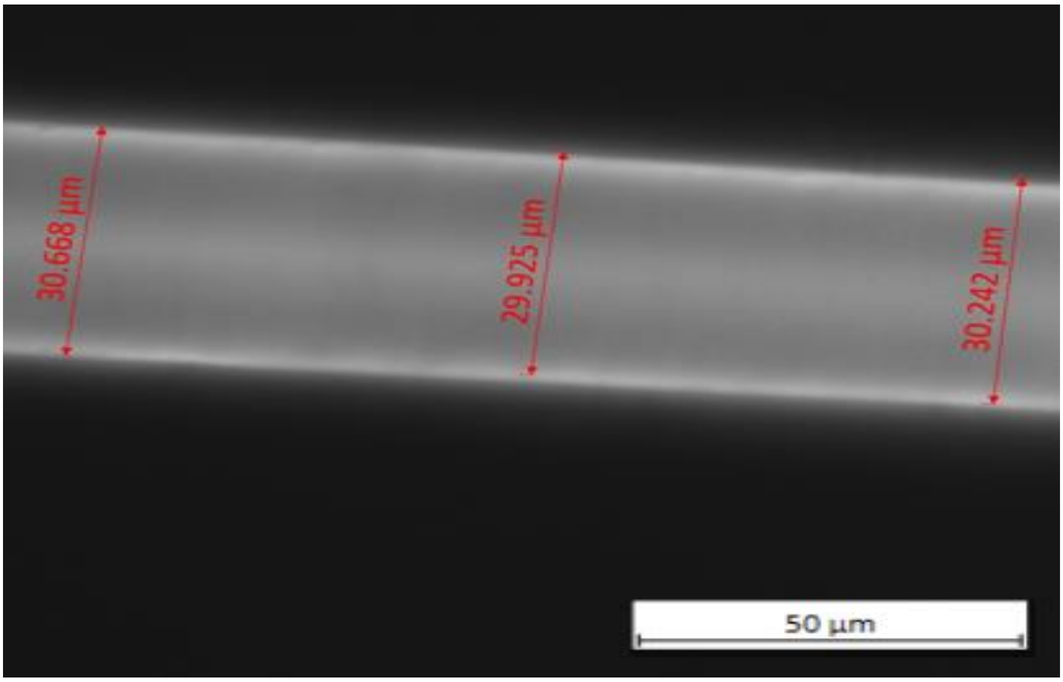


Fig 37: Pristine Fibers Dia 100X

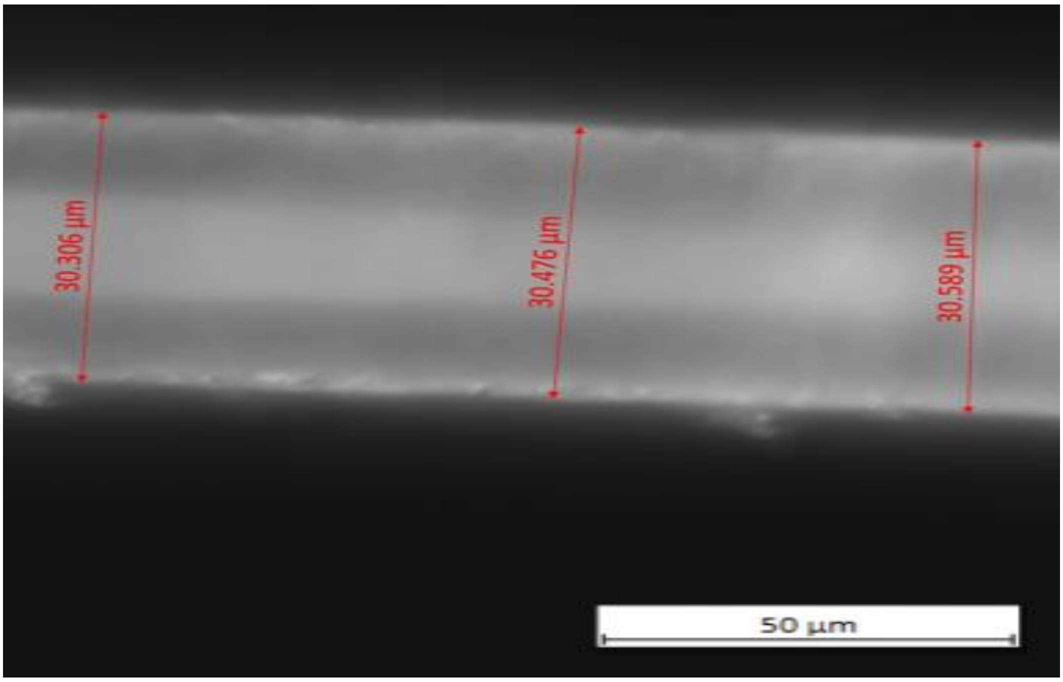


Fig 38: Post-Oxidized Fiber Dia 100X

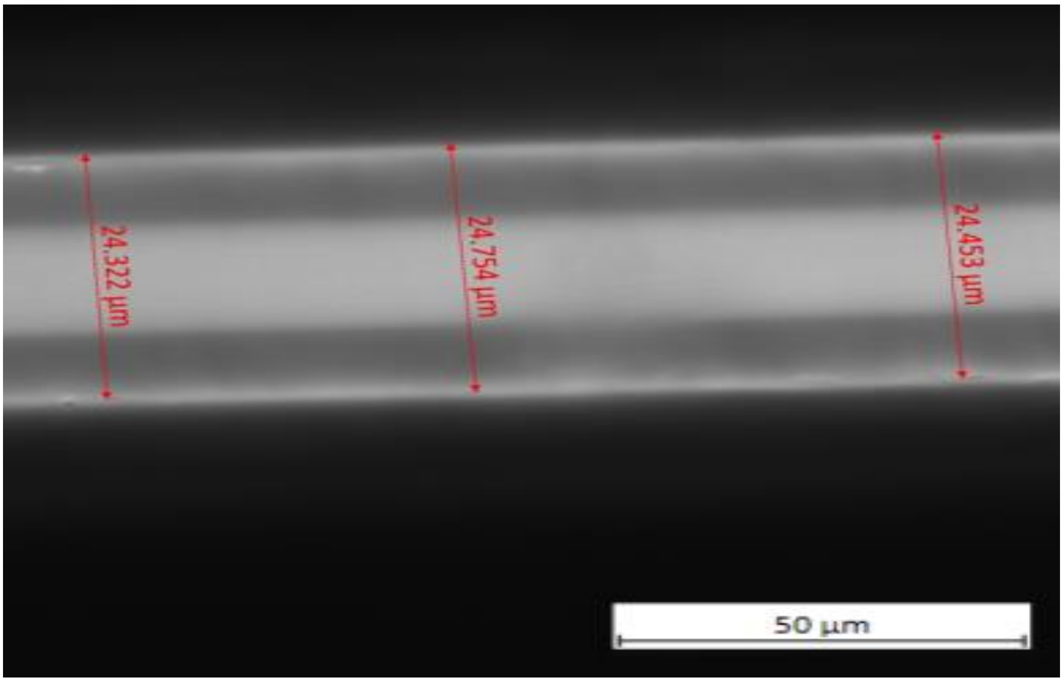


Fig 39: Chemically Treated Fiber Dia 100X

5.4 Weibull Analysis

The Weibull Distribution is a continuous probability distribution that is used to assess life statistics, estimate failure durations, and access product dependability. It is an extreme value of probability distribution that is widely used to describe reliability, survivability, wind speeds, and other data.

Weibull analysis is one of the useful tools to analyze scattering in the values of obtained data (in our case it was tensile strength). It can also give failure probability between samples prepared by two different conditions, i.e., which is more prone to failure.

The following results were obtained after performing a Weibull distribution analysis on tensile strength values of pristine, thermally treated, and chemically treated fibers.

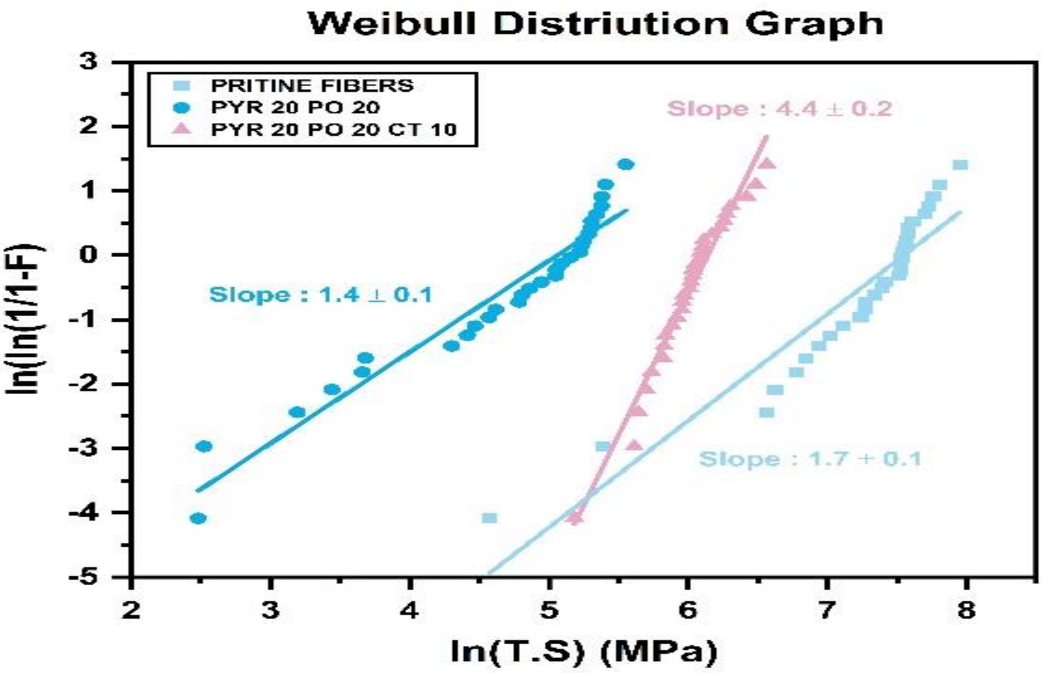


Fig 40: Pristine Fibers VS. 20 Min Conditions

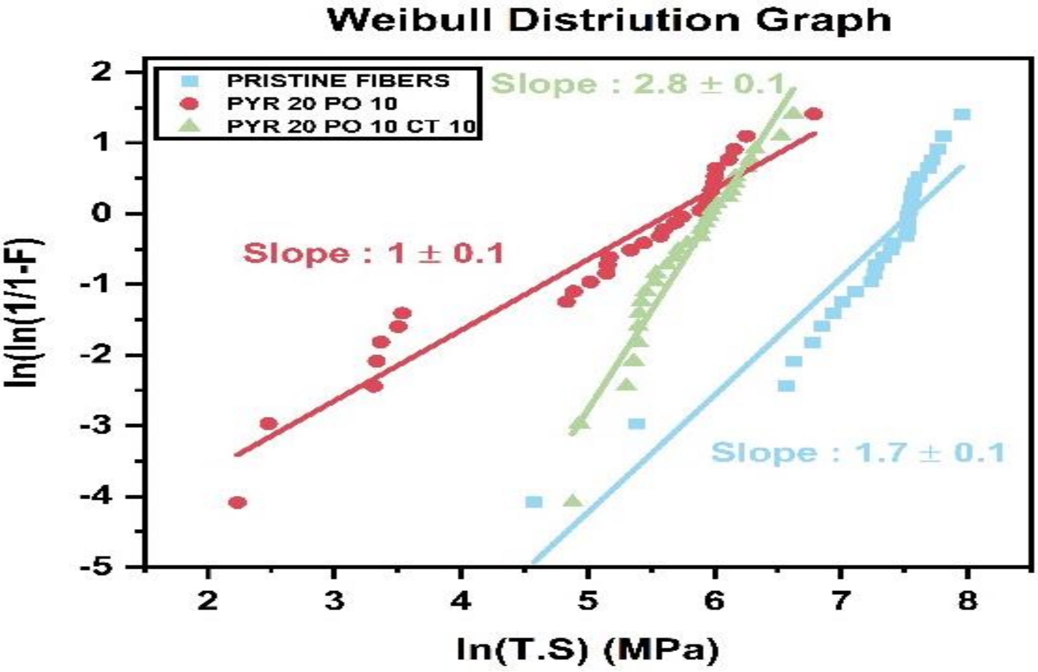


Fig 41: Pristine Fibers VS. 10 Min Conditions

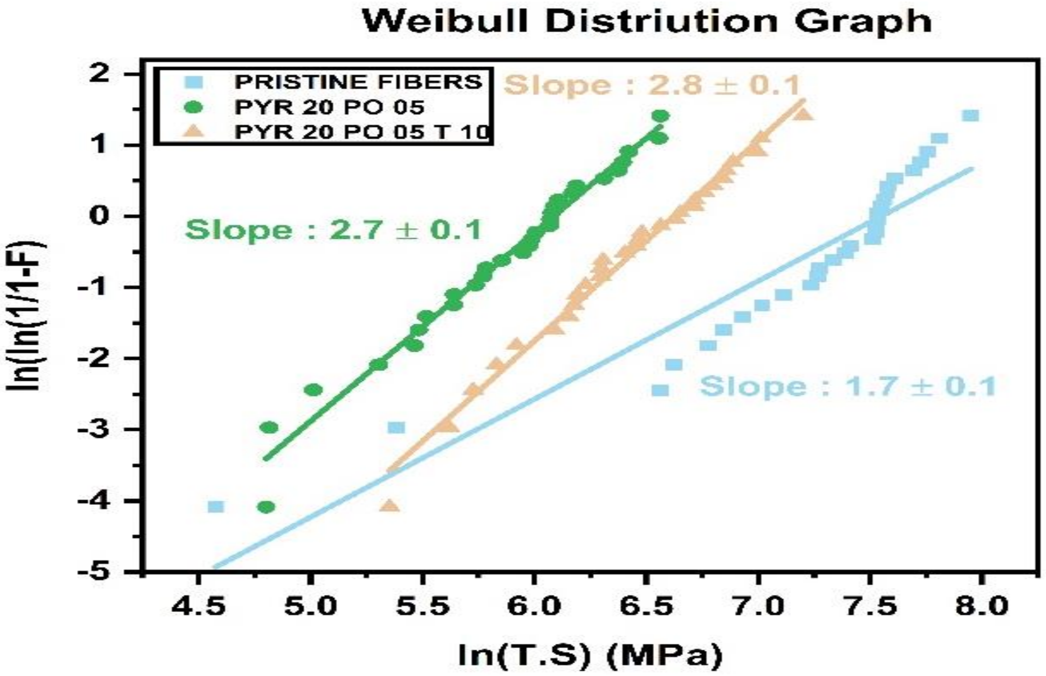


Fig 42: Pristine Fibers VS. 05 Min Conditions

In these figures, we can see the slopes for each condition. Such as, in Fig 40, the slope for pristine fibers is 1.7 ± 0.1, and for post-oxidized fibers, it is 1.4 ± 0.1, and for chemically treated fibers, the slope is 4.4 ± 0.2, and so on in Fig 41 and Fig 42. These slopes are equal to Weibull Modulus (β). The higher the value of Weibull Modulus (β), the narrower will be the distribution of tensile strength values, and as a result, the lesser will be the probability of failure for those samples. In Fig 40, Fig 41, and Fig 42, the Weibull Modulus (β) of chemically treated fibers is more compared to the pyrolyzed fibers. Therefore, it indicates that these chemically treated fibers are more suitable for making composite materials. This statement will be understood from the results in the coming sections of this chapter, see Fig 43. Also, if we compare these chemically treated fibers in Fig 40, Fig 41, and Fig 42, we can see that the Weibull Modulus (β) of chemically treated fibers of, Fig 40, is highest compared to other fibers so, these fibers are more suitable in making composite materials.

Now, in cases where the Weibull Modulus (β) of two slopes becomes equal, i.e., in Fig 42, then we can go for the location of the curve or line in the graph. If it is more toward the right, it means more tensile strength, and if it is more toward the left, it means less tensile strength. Also, if we compare different holding time conditions then we can see in Fig 42 that the highest value of tensile strength is obtained in fibers treated at 5 min post-oxidation plus 10 min chemical treatment with hot alkaline NaOH solution.

5.5 Tensile Strength Comparison of Single Fibers

In Fig 43, a comparison between the tensile strengths of pristine glass fibers, thermally treated glass fibers, and chemically treated glass fibers is present. We can see that the tensile strength of pristine fibers is much higher compared to other fibers. However, if we compare the tensile strengths of thermally treated fibers at different holding times for post-oxidation, then we can see in Fig 43, that fibers that are treated for a 5 min holding time of post-oxidation have a higher strength compared to fibers treated for 20 min and 10 min holding time of post-oxidation. Also, in the case of chemical treatment, these 5 min holding times post-oxidized fibers have a higher strength compared to other treatment times of 20 min and 10 min holding time for post-oxidation followed by chemical treatment.

After thermal treatment, the strength is reduced by 91%, 84%, and 75% in 20 min, 10 min, and 5 min post-oxidized fibers, respectively, compared to pristine fibers. And after chemical treatment, the strength is increased to 200%, 38%, and 72% in 20 min, 10 min, and 5 min post-oxidized fibers, respectively, compared to the thermally treated fibers.

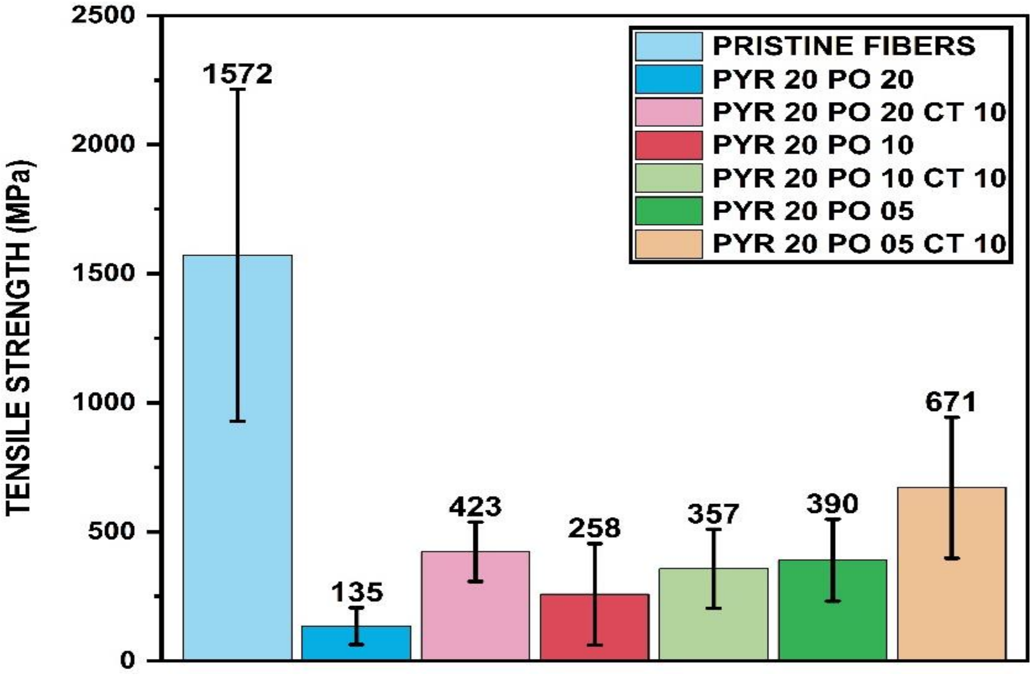


Fig 43: Tensile Strength Comparison of Pristine, thermally treated, and Chemically Treated Fibers at different holding times

These results show that the post-oxidation condition of 20 min holding time followed by chemical treatment with hot alkaline NaOH is the most suitable condition to recover fibers from composite waste.

5.6 Tensile Strength Comparison of Composite

In this characterization, we used the fibers of 20 min holding time conditions, i.e., PYR 20 PO 20, as thermally treated fibers reinforcement, and PYR 20 PO 20 CT 10, as chemically treated fibers reinforcement for composite manufacturing.

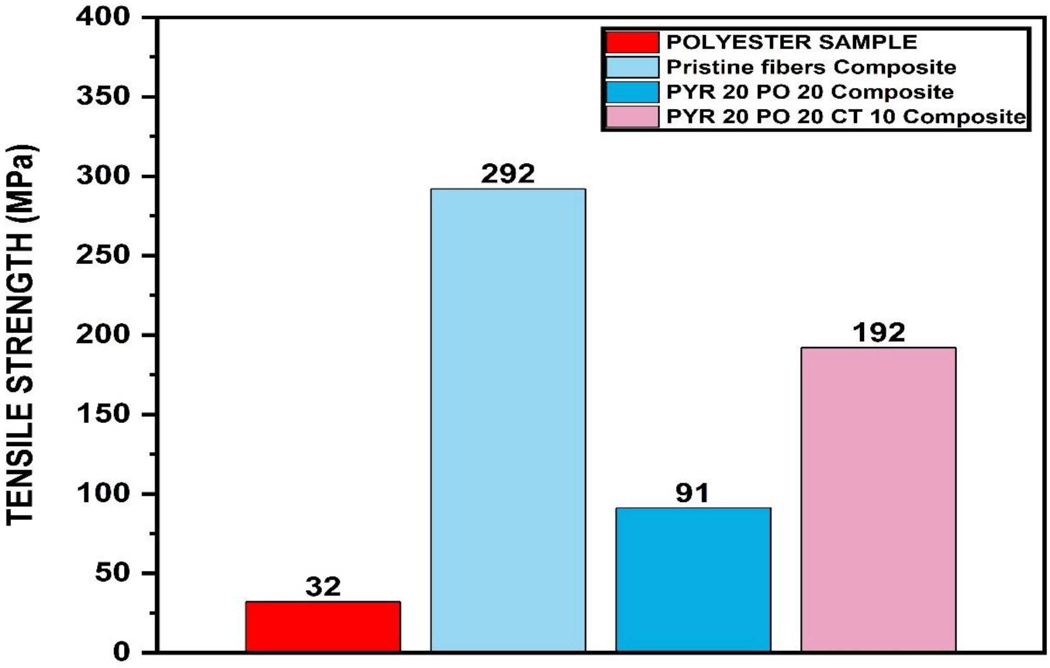


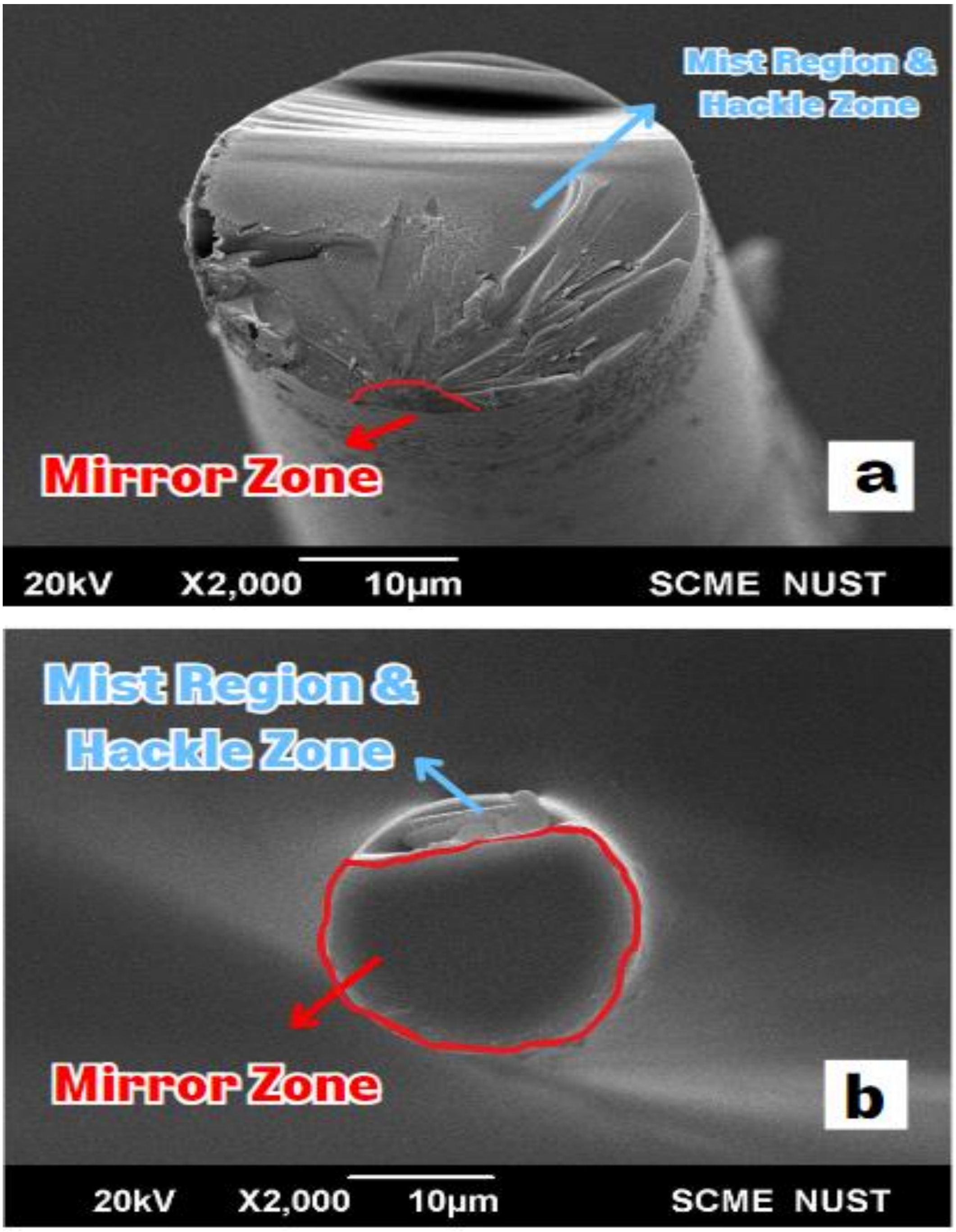
Fig 44: Tensile Strength Comparison of Polyester sample, Pristine fibers composite, thermally treated fibers Composite, Chemically Treated Fibers Composite

We can see in Fig 44, that the strength of the composite is way more than compared to the normal polyester sample because there is no reinforcement present in the normal polyester sample. Now, if we compare the composites samples, we can see that the strength of pristine fibers composite is significantly higher compared to thermally treated and chemically treated fiber composite, which is justified due to the higher strengths of pristine fibers, see Fig 43. However, if we compare the composite samples made from thermally treated fibers and chemically treated fibers, then we can see in Fig 44, that the strength of the composite made from chemically treated fibers is much more compared to the thermally treated fibers.

The strength of thermally treated fibers composite is reduced by 69% compared to pristine fiber composite. After using chemically treated fibers as reinforcement, a 110% increase, compared to thermally treated fibers composite, was observed in the composite material.

5.7 SEM Analysis of Fracture Surface of Single Fibers

SEM Analysis was performed to determine the fracture surface of those single glass fibers on which tensile testing was performed. For this purpose, pristine single fiber, thermally treated single fiber, and chemically treated single fiber were used. The following images were obtained after performing SEM on these samples:



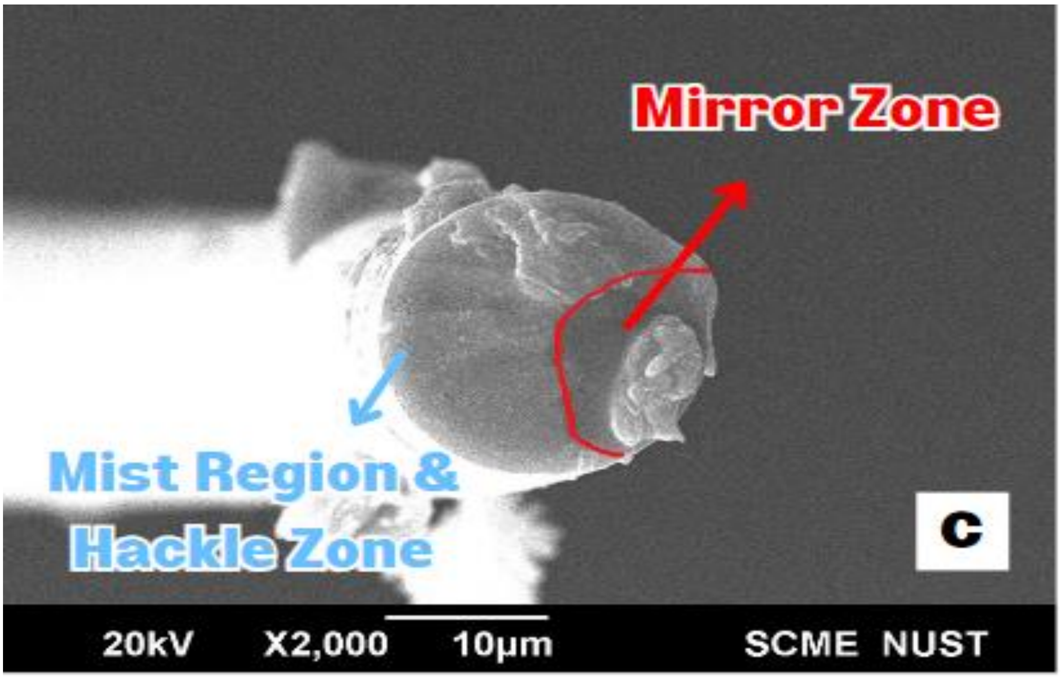


Fig 45: SEM image of Fracture Surface of Single Fibers after Tensile Testing, (a) Pristine Fiber, (b) Pyrolyzed Fiber, (c) Chemically Treated Fiber

The images obtained in SEM revealed three regions on the fracture surface of pristine glass fibers. These regions are the Mirror Zone, Mist Region, and Hackle Zone. The mirror zone is the smooth and featureless surface; the region followed by the mirror zone is the Mist region. In the mist region pitted surface is present and the branches present in this region are referred to as hackle. The fact that these regions exist in the fracture surface of pristine fibers is that the crack initiates from the surface followed by the mirror zone and then the mist region and hackle zone. This also shows that surface features play a major role in the tensile strength of glass fibers. The smaller the mirror zone, the more will be the more strength, and vice versa. We can see in Fig 45, that pristine fibers have the smallest mirror zone, so it has the highest strength, then comes the size of the mirror zone of chemically treated fibers, and in the end the mirror zone of thermally treated fiber, hence their strength is lowest. The size of the mirror zone also indicates the size of the crack. The size of the mirror zone is reduced in chemically treated fibers because, after chemical treatment, the surface of thermally treated fibers was etched which results in the removal of these surface cracks. As these cracks reduced in size so as the mirror zone, see Fig 39.

5.8 FTIR Spectroscopy Analysis

FTIR spectroscopy was performed to analyze the functional groups present in the glass fibers before and after thermal and chemical treatments. For this purpose, pristine fibers, thermally treated fibers, and chemically treated fibers were used.

The following results were obtained from this characterization technique:

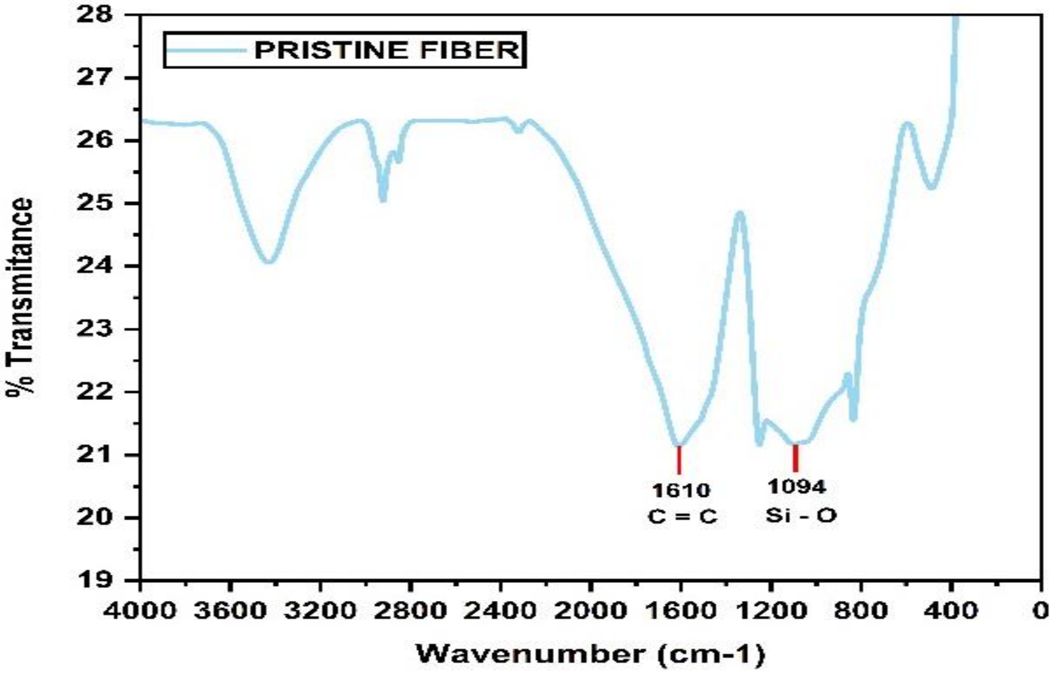


Fig 46: FTIR Spectroscopy graph of Pristine fibers

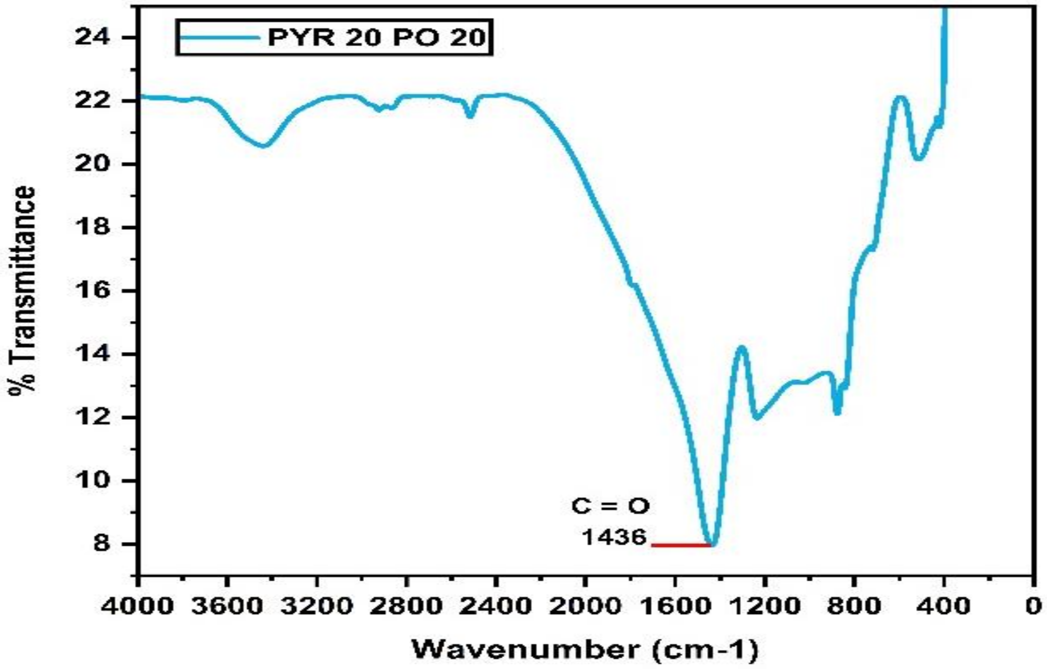


Fig 47: FTIR Spectroscopy graph of Thermally Treated fibers

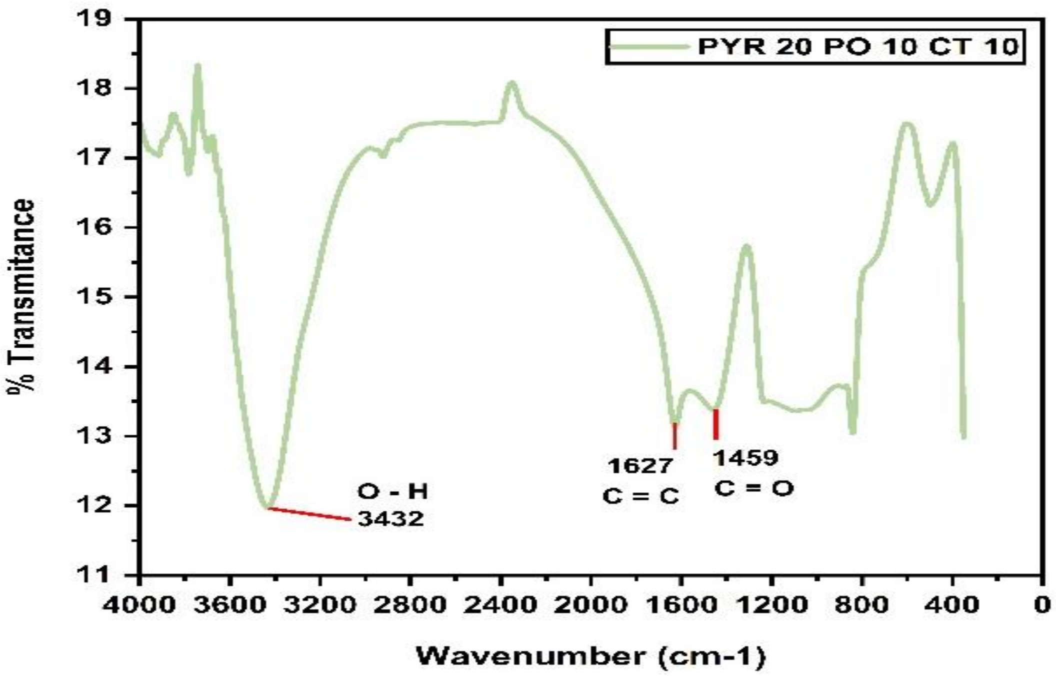


Fig 48: FTIR Spectroscopy graph of Chemically Treated fibers

The main functional groups that we obtained after FTIR spectroscopy were C = C, C = O, Si – O, O – H, etc.

We can see that in Fig 46, C = C and Si – O bonds are more prominent. Here, Si – O bond is because glass is made up of silica, and the C = C group is because some coupling agents are attached and used on these pristine fibers. After all, these fibers must be used as reinforcement with some other material, so these coupling agents are added to assist in bonding. In Fig 47, C = O is the most prominent functional group because these fibers are oxidized in air and this C = O indicates the presence of residue on the thermally treated fibers. In Fig 48, the presence of the O – H group indicates the treatment with a hot NaOH solutions. This O – H group helps in hydrogen bonding between resin and reinforcement during the curing of the composite material which contributes to the strengthening of the new composite.

This O – H group contributes to hydrogen bonding by utilizing O with Si of glass fiber and hence H remains free which can make hydrogen bonding with the elements of resin.

CONCLUSIONS

In conclusion, the strength of glass fibers, which was lost due to thermal treatment, was successfully recovered after treating these fibers with a hot alkaline NaOH solution. We can see in Fig 43 and Fig 44, that after pyrolysis (and post oxidation) strength of single fibers is reduced by 91% and that of composite by 69%. However, after chemical treatment, a 200% increase in the tensile strength of single fibers was observed and a 110% increase was observed in the case of composite, in comparison to thermally treated single fiber and composite. The Weibull distribution gives us the failure rate or failure probability of the fibers that can be used for making composite. The narrower distribution and a higher Weibull Modulus (β) indicate less failure rate, and vice versa [9]. Fig 40, Fig 41, and Fig 42 are showing that the strength distribution for chemically treated fibers is narrow, hence, their failure rate is low as compared to pyrolyzed and pristine fibers. Therefore, these chemically treated fibers can be effectively used for making new composite materials.

From these results, we also concluded that post-oxidation conditions of 20 min holding time are the most suitable conditions for recycling and strength recovery of glass fibers from GFRPs wastes.

Applications like water storage tanks, ladders, boat hulls, etc. are most suitable for incorporating these chemically treated recovered fibers as reinforcement in the composite material used for these applications.

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