CHAPTER 3
MATERIALS AND
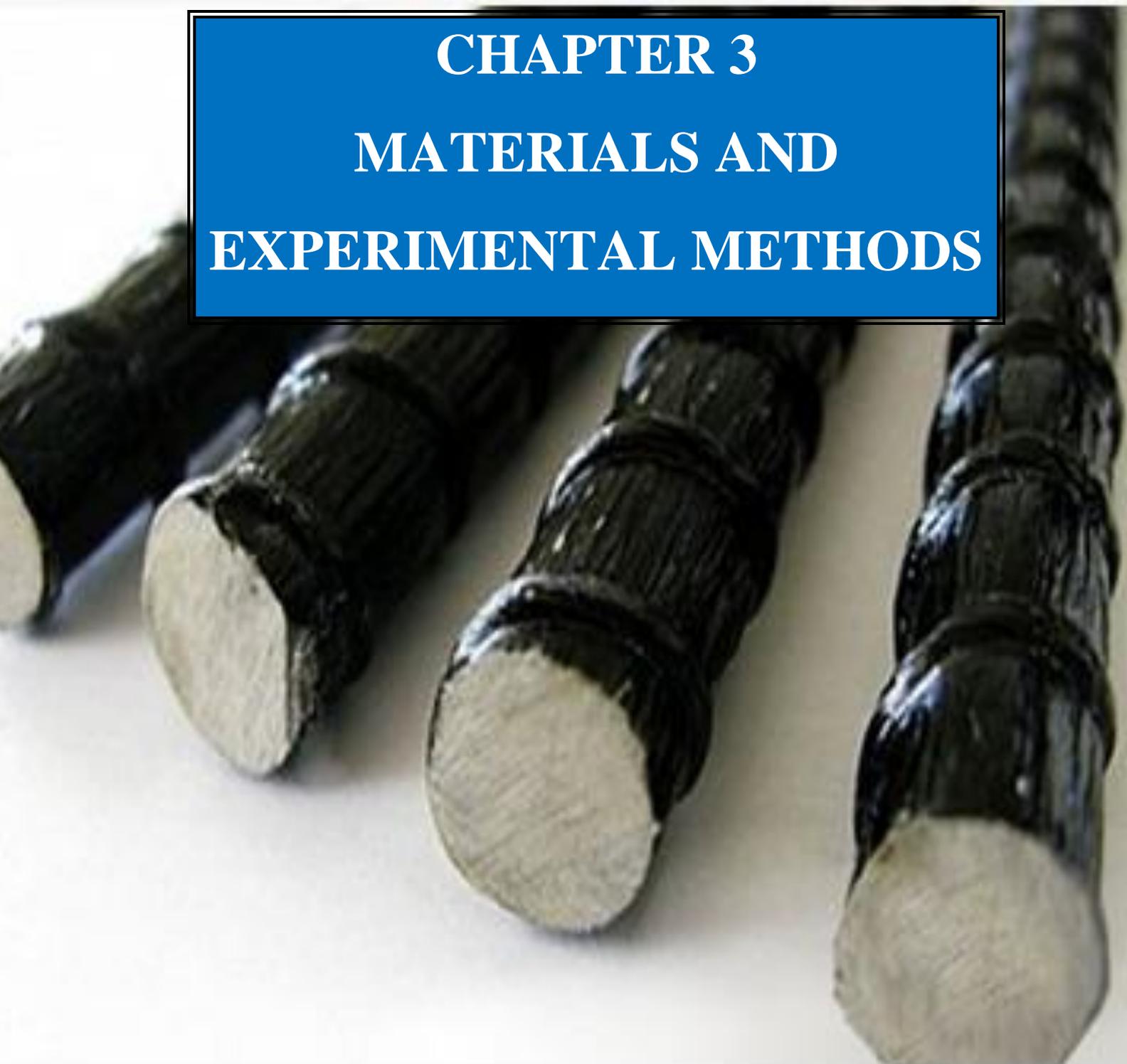# EXPERIMENTAL METHODS

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CHAPTER 3

MATERIALS AND EXPERIMENTAL METHODS

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3.0   Introduction

This chapter provides a comprehensive overview of the materials and methodologies employed in this research. The efficacy and reliability of any research study largely depend on the appropriateness of the materials used and the robustness of the methodologies applied. This section serves as a comprehensive guide to the procedures and techniques employed to achieve the research objectives and validate the hypotheses. The selection of appropriate materials and the implementation of a rigorous methodological framework are pivotal in ensuring the reliability and validity of the study's findings. This chapter is divided into three sections, Section 1 - Machine Modification, Section 2 - Rope Preparation, and Section 3 - Rod Preparation, each detailing a specific aspect of the research design, including the selection of materials, data collection methods, experimental setup, and analytical techniques. Through this structured approach, we aim to provide a transparent and replicable account of the research process, facilitating a thorough understanding and critical evaluation of the study.

After assessing the performance of yarn, rope, and rod prepared from a given material of available size and dimension, predicting the beyond size is challenging. To predict the values of ropes & rods beyond the tested sizes, mathematical modeling becomes essential. For this study, the tensile behaviour of synthetic fiber ropes made from materials like polypropylene, nylon basalt, and their blends have been mathematically analysed. Here goal is to generate well-documented experimental statistics and develop simplified stress-strain constitutive laws that describe the ropes' tensile response. Here to predict this behaviour of rope and rod, various mathematical formulation based on python programs is used. Methods like Artificial Neural Networks (ANN), and Curve Optimization are used. All these methods and their outputs are discussed in a separate chapter.

To have a comprehensive understanding of the work, Chapter 3 has been further divided into different sections. Section I deals with machine modification, Section II deals with rope preparation of different diameters with the help of varied materials and Section III deals with the preparation of composite rods from the prepared ropes.

Section I

3.1   Machine Modification

The existing biaxial braiding machine was modified to facilitate the addition of third axial yarn which will be introduced at zero degrees, during braiding. Such a braiding machine is known as Tri-axial braiding. Apart from this, the take-up mechanism was also modified to achieve different take-up rates. Thus, modification was carried out in the existing machine and was made versatile for the manufacturing of biaxial as well as triaxial braided products [1–5].

Initial modification was made at the base of the machine i.e. at the creel section from where the core yarns are supplied. An additional creel was designed to accommodate an additional 8 supply packages. This creel was kept at the bottom of the machine, exactly below the horn gear (Figure 3.1). This will facilitate the addition of yarn exactly at zero degrees between the intertwining yarns. Additional spring and washer type of tensioning devices were also incorporated on the creel for individual yarn to maintain constant yarn tension and it will also prevent extra and loose yarn from being braided.

The second modification was conducted at the centre of the machine. i.e. between creel and take up section. A half-threaded solid bolt was provided to hold the horn gear and track plate assembly (Figure 3.2), which was replaced by a specially designed hollow bolt of the same dimension. The replacement of the hollow bolts at all positions (Figure 3.3, a &b) will not only hold the horn gear and track plate firmly but also will allow the central thread from the bottom creel to pass through it and clockwise and anticlockwise yarn will braid towards the central take-up unit [5–8].

Figure 3.1 Tri-axial braiding machine

Figure 3.2 Nut &bolt at horn gear assembly for biaxial machine

(a)

(b)

Figure 3.3 Modification at horn gear assembly for triaxial machine

<table>
<tr><td>(a)</td><td>(b)</td></tr>
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(a)   *(b)*

Figure 3.4 Modification at take up zone for braiding machine

Further, in addition to the introduction of the third axial yarn, modification was also made to the take-up mechanism of the machine (Figure 3.4 & 3.5). The take-up mechanism controls the tension and speed of the braided material as it winds onto a spool. It is an important way to adjust the tightness factor of the braided structure, especially the angle of the braid. The modification of this mechanism enables the machine to achieve different take-up rates, providing greater flexibility in the manufacturing process.

Figure 3.5 Take up zone for braiding machine

Overall, the modifications made to the existing biaxial braiding machine make it versatile, and capable of producing both biaxial and triaxial braided products. This versatility is essential for meeting the specific requirements of different applications and expanding the range of products that can be manufactured using the modified machine. The adaptation of the machine to accommodate the third axial yarn and adjust the take-up mechanism demonstrates an innovative approach to enhance its functionality and broaden its applications in the field of braided materials manufacturing [9], [10].

Section II

3.2   Rope Preparation

We aim to make a braided composite rod to be used for civil engineering applications and the strength and diameter requirement of these rods should be comparable with the existing steel rods. So, high tenacity and high denier yarns of three different variants like Polypropylene, Nylon & Basalt were procured from Arrow Tech Textiles, Mumbai (Figure 3.6). These basic yarns and their combination blends i.e. Basalt - Nylon and Basalt - Polypropylene were used for rope preparation.

Figure 3.6 Yarns procured for braiding

Researchers consider yarn strength to be a very important factor. Stronger yarns make better final products. Therefore, higher denier synthetic yarns, are used because they offer greater tensile strength & other mechanical characteristics compared to lower

denier. For these trials, higher denier yarns with high tenacity material of three different variants, namely basalt, nylon, and polypropylene are tried to get variety in the product range. To get more into to product category, blends like basalt - nylon and basalt - polypropylene also have been tried. The basic raw material characteristics are as follows shown in Table 3.1.

Table 3.1 Yarn characteristics

Yarn type	Yarn count	Maximum load [gf]	Maximum extension [mm]	Stress [gpd]	Strain (%)
3 ply PP	1500 Den	9269.64	24.29	6.17	24.29
4 ply Nylon	3550 Den	25532	20.15	7.19	20.15
Basalt	7200 Den	33082	2.86	4.59	2.87

3.2.1 Tri-axial Rope

The high-tenacity yarns procured were used as raw material for the manufacturing of triaxial braided ropes. Circular braided ropes are manufactured on a B&B braiding machine. This machine is running on the Maypole principle, which has 16 carriers. The diameter of the manufactured ropes was increased by the braid over braid technique. These desired diameter ropes are used as a reinforcement material and were further converted into rods using the method described in section 3.3.

1st lot of tri-axial braided rope production started with polypropylene yarn of given properties. Here in tri-axial braided rope regular braiding positions rotate in clockwise and anti-clockwise direction. i.e. 8 clockwise and 8 anti-clockwise, total 16 bobbins. Apart from these 8 bobbins were placed at zero-degree position along with a single yarn core for the first layer. i.e. total of 25 yarns in the first layer. In this position about 100 meters of braided rope were prepared. From this about 20 meters of rope were cut and placed apart for 1st layer rope testing and rod preparation. The remaining 80 meter of rope is now ready for second layer build-up.

Now in the core, 1st layer braided rope was fed and second layer was braided above the first layer keeping all other parameters same. Similar kind of arrangements like 1st layer

i.e. 8 bobbins will rotate in clock wise direction, 8 bobbin will rotate in anti-clockwise, total 16 bobbins. Along with it 8 bobbins were placed at zero-degree position. i.e. Total 24 yarns and 1 core. This type of arrangement is known as braid over braid arrangement. In this condition we will start the machine for another 80 meter braided rope. This is known as second layer braiding. From this about 20 meter of rope were cut and placed apart for 2nd layer rope testing and rod preparation. Remained about 60 meter of rope is now ready for third layer build up.

In this similar manner, the third, fourth, and fifth layers are prepared each of 20 meters. The basic aim of adopting this braid over braid technology is to gain the higher diameter of braided rope and further with the rod. This higher diameter may contribute towards high tensile strength and other favourable mechanical properties.

The below given parameters describes the development of a variety of triaxial braid-over-braid ropes using different materials and techniques to achieve a range of strengths and other characteristics for a cost-effective product line.

Material Exploration:
- Experimented with various raw materials having different properties to create a diverse product range in terms of strength and other characteristics.
- Similar to the previously discussed polypropylene (PP) ropes, designed and produced triaxial braid-over-braid ropes using nylon and basalt fibers.

Diameter Variation:
- Using the same braiding technique, manufactured five different diameters for each type of rope (PP, nylon, and basalt).

Blended Rope Development:
- To further expand product offerings, created blended ropes using the triaxial braid-over-braid technique.
- This involved modifying the bobbin placement on the braiding machine.

Basalt-PP Blend:
- For the basalt-PP blend, position 100% basalt fibers in the axial direction (0 degrees) for maximum strength.
- The remaining braiding positions were filled with 100% polypropylene fibers at an angle.
- Similar to the pure PP ropes, produced five different diameters of this blended rope,

each 20 meters long.

Nylon-Basalt Blend:

- Followed the same methodology to create a nylon-basalt blended rope with similar properties and production details.

In essence, created a variety of triaxial braid-over-braid ropes using different materials (PP, nylon, basalt) and blends (basalt-PP, nylon-basalt) with varying diameters to cater to a wider range of techno-commercial needs in terms of strength and other functionalities.

3.2.2 Testing for Rope

The yarn procured was tested for its linear density and tensile properties. The rope prepared from the material was also evaluated for its linear density, braid geometry and tensile properties.

3.2.3.1 *Grams per linear meter test*

The grams per linear meter test for rope, also known as the linear density test, is a measure of the weight of a rope per meter of length. It is typically expressed in grams per meter (g/m) or kilograms per kilometre (kg/km). The linear density of a rope is an important factor in its performance, as it affects its strength, weight, and handling characteristics.

Figure 3.7 GLM testing

For measuring the linear density of rope, a simple weighing machine was used (Figure 3.7). The weight of a known length of rope was obtained which was then divide by the length. For example, if you weigh a 10-meter length of rope and find that it weighs 500 grams, then the linear density of the rope is 50 g/m.

The linear density of rope is an important consideration when choosing a rope for a particular application. For example, a rope with a high linear density will be stronger and heavier than a rope with a low linear density. This means that a rope with a high linear density will be more suitable for applications where strength is important, such as rock climbing or rappelling. However, a rope with a high linear density will also be heavier and more difficult to handle, so it may not be suitable for applications where weight and ease of handling are important, such as camping or backpacking

3.2.3.2   *Tensile testing for rope*

Tensile testing is a crucial method for evaluating the strength and performance of ropes. It involves applying a pulling force (tension) to a rope specimen until it breaks or reaches a predetermined strain (elongation). Tensile Testing machine equipped with grips to hold the rope specimen and a mechanism to apply a controlled pulling force. The machine measures and records the force applied and the resulting elongation of the rope. 40 cm length of rope is cut according to the testing standard ISO 2307 with a 10 mm/min speed (Figure 3.8) Textiles, The ends of the rope knotted to prevent unravelling & slippage while load application [9], [10].
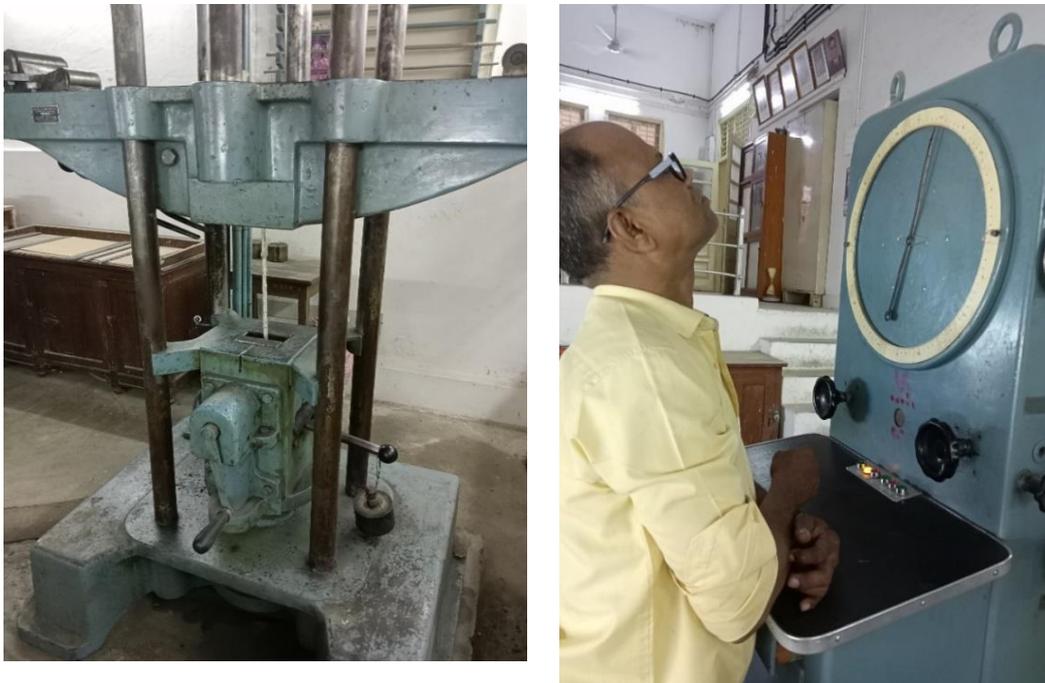

Figure 3.8 Braided rope during tensile testing

Section III

3.3   Rod Preparation

For the preparation of Braided composite rods, the prepared braided ropes are to be used as reinforcement and the matrix material needs to be applied. Selection of the right matrix material is very important, as it will finally decide the proper bonding with reinforcement for the desired application. Various types of resin and hardener combinations are available in the market. After a few initial trials with a couple of epoxy resins as well as a polyester base resin, we chose to go with the epoxy resin CTE556 and the hardener material CTAH951. The basic details of the resin and hardener used are as given in Table 3.2, 3.3, 3.4. [Procured from Composite tomorrow, Vadodara].

Table 3.2 Characteristics of resin& hardner

Characteristic	Test Method	Unit	CT/E-556 [Resin]	CT/AH-951 [Hardener]
Viscosity at 25°C	ASTM-D 2196	mPa·s	9,000 - 12,000	110 - 120
Flash Point	ASTM-D 93	°C	> 200	> 200
Density at 25°C	ASTM-D 4052	g/cc	1.15 - 1.20	0.97 - 0.99
Epoxy Content	ASTM-D 1652	Eq/Kg	5.3 - 5.45	-

Table 3.3 Mechanical and Physical Properties of resin& hardner

Property	Test Method	Unit	Value
Tensile strength	ASTM:D638	N/mm²	70-80
Elongation at break	ASTM:D638	%	2.0-2.2
Flexural strength	ASTM:D790	N/mm²	90 - 100
Glass transition temperature (DSC)	ASTM: D3418	°°C	70-80

Table 3.4 Electrical Properties of resin& hardner

Property	Test Method	Unit	Value
Dielectric strength	IEC 60243-1	KV/mm	23-25
Dielectric dissipation factor	IEC 60250	%	1.2 -1.3
Dielectric Constant	IEC 60250	-	4-4.2
Volume Resistivity	IEC 60093	Ohm cm	> 10^15

CTE556 is a modified epoxy laminating resin designed for high-performance applications. CTAH951 is a modified polyamine-based hardener suitable for high mechanical performance applications in static and dynamic load conditions.

Figure 3.9 Epoxy resin with hardener

The hardener CTAH951 of this system can provide a pot life of >5 hours at 25ºC with low exothermic reactions, even when it is used for higher thickness components of large size. The low initial viscosity of this system ensures fast and complete impregnation of reinforcement in fibers such as glass, carbon, polyamide, and polyester. It allows components to be produced by various process techniques with high consistency in performance properties. The components cured at room temperature provide excellent handling strength (Figure 3.9).

3.3.1   Hand Lay Process

The application of a matrix on the surface of a rope is essential, especially when dealing with braid over braid ropes. The binding of individual layers can be challenging, and this is where the resin comes into play. To ensure uniform resin penetration inside the rope matrix, a small calendaring machine was developed. This machine, as depicted in Figure 3.10, consists of two heavy rolls housed in a bearing and mounted in a steel frame. One of the rollers is equipped with a handle to facilitate rotation. The resin-impregnated rope is passed through these rollers, allowing for proper penetration of the resin into the layers and removal of excess matrix material. This process is repeated

two to three times to ensure proper impregnation of resin into the reinforcement material.

It was noted that the shape of the prepared Braided Composite Rod (BCR) became deformed due to squeezing, and a proper circular cross-section was not achieved. To rectify this, the braided ropes were passed through desired circular shape pre-forms. This process allowed the deformed braided rope to regain its original shape and size.

Figure 3.10 Rope calendaring equipment

The resin solution used in the braided ropes was a mixture of CTE556 (Epoxy resin) and hardener CTAH951, maintained at a ratio of 10:1. The curing of this resin within the braided rope is a crucial factor, as it needs to result in a straight rod-like structure. The curing process takes place at room temperature. Arrangements were made to hang the resin-applied ropes vertically, allowing the ropes' self-weight to straighten them. The ropes were left in this position for about 24 hours to cure, hardening the rope and converting it into a rod-like structure.

Figure 3.11 illustrates the preparation of the resin mixture. The rope is converted into a composite form using a hand-laying method. This involves manually mixing the resin and hardener and applying them to the braided ropes. The material is then left on the desired base for the next 24 hours at room temperature.

Figure 3.11 Resin mixture preparation

Due to the viscosity of the resin, it would have been challenging for it to penetrate the core of the rope. To improve its penetration, the braided rope was unwound from the bobbin, passed through the resin bath, squeezed manually, and then dipped again into the resin bath. This process allowed the air from the sample to escape and facilitated resin impregnation to the core. The rope was squeezed again before drying to ensure proper impregnation of the resin into the reinforcement material (Figure 3.12). The repetitive squeezing disturbed the circular shape of the braided rope. To restore its shape, the resin-impregnated rope was immediately passed through a circular-shaped funnel, giving the rod a proper diameter. Different layers of ropes required different funnel diameters. All the samples were then hung on a wire for the next 24 hours at room temperature (Figure 3.13). After 4 to 5 hours, the samples began to solidify, and complete solidification required 24 hours.

A total of 25 samples, each with 5 layers and 5 raw material combinations, were prepared using this hand-lay method. All braid rope samples have now been converted

into composite rod form, referred to as a Braided Composite Rod (BCR). The braid angle and diameter of the BCR are mentioned in the table below. All 5 materials were braided with a uniform take-up speed to maintain almost uniform braiding parameters, as shown in Table 3.5. The entire sample set is now ready for testing. All the samples were precisely cut into the required shape and size to carry out tests as per the standards.

Figure 3.12 Manual hand lay preparation

Figure 3.13 Curing treatment for braided rod preparation

Table 3.5 Parameters of braided composite rods

Composite sample Code	Layer (mm)	Braid angle (Degree)
BCR	1	28
BCR	2	34
BCR	3	41
BCR	4	48
BCR	5	54

Figure 3.14 Specimen diameter check through digital Vernier

3.3.2 Testing for Rod

Detailed analysis was carried out during different stages of FRP rod manufacturing. The yarn procured was tested for its linear density and tensile properties. The rope prepared from the material was also evaluated for its linear density, braid geometry and tensile properties. The prepared rods were further analyzed for their diameter, fiber volume fraction, and tensile properties (Figure 3.14). The prepared rods were also subjected to the preparation of cemented blocks and beams, for checking the performance of prepared rods inside the cement structure in terms of bonding and flexural strength respectively

3.3.2.1 Grams per linear meter test

The linear density of FRP bar is an important consideration when choosing an FRP bar for a particular application. For example, an FRP bar with a high linear density will be stronger and heavier than an FRP bar with a low linear density. In order to evaluate the GLM, the total available length of 20 meters was weighed, and then the weight per meter was calculated. FRP bar with a high linear density will be more suitable for

applications where strength is important, such as structural reinforcement or rebar replacement. However, an FRP bar with a high linear density will also be heavier and more difficult to handle, so it may not be suitable for applications where weight and ease of handling are important, such as aerospace or automotive applications [9].

3.3.2.2 Fiber volume fraction (FVF) analysis

The Fiber Volume Fraction (FVF) is a critical parameter in composite materials, indicating the volume of fiber present in the composite. It is typically evaluated using the ASTM D2584 standard. Here by dividing the volume of the resin by the total volume, we get the volume fraction of the fiber.

 The formula used to calculate the FVF. Here's how it works:

FVF= volume of the resin/ (volume of the resin + volume of the composite)

The volume of composite is nothing but the volume of resin + volume of fiber.

Given:

Resin Density (RD) = 1.25 g/cm³ (for Epoxy Resin)

Rope Weight (RW)

Fiber Density (FD)

Resin Weight (RWe)

The formula for Fiber Volume Fraction (FVF) is

$$FVF = \frac{RD \times RW}{(RD \times RW) + (FD \times RWe)}$$

In this method, all the parameters regarding weight is taken from g/lm to get accurate results. Also, this method assumes that there are no voids or defects within the composite material, which might not always be the case in practical scenarios. Therefore, while this formula provides a good approximation, the actual FVF might vary based on the manufacturing process and the quality of the composite material [13].

3.3.2.3 ATR-FTIR spectroscopy

 Attenuated Total Reflection (ATR) is a widely used technique for measuring Fourier Transform Infrared (FT-IR) spectra. It allows the study of chemical bonding in various materials, including solids and liquids, without causing significant damage. ATR-FTIR

is particularly convenient due to its non-destructive nature and ease of application. In an ATR-FTIR experiment, a sample is placed in contact with a single-bounce diamond crystal attached to the ATR accessory (Figure 3.15). The crystal interacts with the sample, allowing infrared light to penetrate and generate a spectrum. For this study, a polymer liquid sample was analyzed using a Shimadzu (Bruker Globals) spectrometer equipped with a deuterated triglycine sulfate (DTGS) detector. The measurements were conducted at ambient temperature, and the resulting ATR-FTIR spectra covered the range from 4000 to 400 cm⁻¹ with a resolution of 4 cm⁻¹.

Figure 3.15 FTIR Spectrometer

3.3.2.4 Tensile testing for rod

ASTM D638 is a standard test method for determining the tensile properties of plastics. It is widely used to evaluate the strength and ductility of plastic materials. The test is performed by applying a tensile force to a standard dumbbell-shaped specimen until it breaks [14–17]. The following is a summary of the test procedure:

Specimen Preparation:

1. Obtain a representative sample of the plastic material to be tested.
2. Prepare standard dumbbell-shaped specimens from the sample with a sample size of 70 cm. The dimensions of the specimens are specified in ASTM D638.
3. Condition the specimens to the desired test temperature and humidity.

Test Procedure:

1. Mount the specimen in the grips of a universal testing machine maintaining gauge length of 500 mm.

2. Set the test standard value of the testing load rate with 200KN/Min. The test speed is typically specified in the ASTM D638.

3. Apply a tensile force to the specimen until it breaks.

4. Record the data during the test.

ASTM D638 is a versatile test method that can be used to evaluate a variety of plastic materials. It is a relatively simple test to perform, and the results are easy to interpret. The test is widely used in the plastics industry to ensure that materials meet the required specifications. In between the upper and lower jaw of UTM Specimen fixed & continuously load applied on to it as per the 5.0 mm/ min speed (Figure 3.16).

(a) *(b)*

Figure 3.16 Specimen tensile testing (a) Specimen marking, (b) Specimen before Test

3.3.2.5 Bond strength of FRP

The bond strength of FRP bars to concrete is typically determined using a direct pull-out test, which is described in ISO 10406-1:2015. This test involves embedding an FRP bar in a concrete block of 15 cm x 15 cm and then applying a tensile load to the bar until it pulls out of the concrete. The bond strength is calculated as the maximum load divided by the cross-sectional area of the bar.

The direct pull-out test is a relatively simple test to perform, but it can be affected by a number of factors, such as the size and shape of the concrete block, the embedment length of the bar, and the loading rate. As a result, it is important to carefully control these factors when performing the test in order to obtain accurate results [18–22].

The testing procedure for ISO 10406-1:2015, "Fibre-reinforced polymer (FRP) reinforcement of concrete - Test methods - Part 1: FRP bars and grids", is as follows:

(a) Material mixing

(b) Preparation of cement blocks

(c) Insertion of BCR inside blocks

(d) Curing of prepared blocks

Figure 3.17 Bond strength specimen preparation

Preparation of specimens:

1. Prepare concrete specimens according to Clause 5 of ISO 10406-1:2015 i.e. M20 grade. The dimensions of the concrete specimens should be such that the embedment length of the FRP bar is at least 10 times the nominal diameter of the bar.

2. Clean and dry the FRP bars and concrete specimens.

3. Apply a release agent to the ends of the FRP bars to prevent them from bonding to the testing apparatus.

4. Put rebar in a concrete mold of 15cm x 15 cm filled with concrete material, and insert de-bonding pipe for rest of rod dia.

Let it cure for approx. 28 days before initiating the test (Figure 3.17).

Figure 3.18 Bond strength specimen testing

Test setup:

1. Mount the concrete specimen in the testing apparatus. The specimen should be aligned such that the FRP bar is perpendicular to the loading direction.

2. Position the loading grips on the FRP bar. The grips should be positioned such that the load is applied uniformly to the bar (Figure 3.18).

Test procedure:

1. Apply a tensile load to the FRP bar at a constant rate of displacement. The rate of displacement specifies a loading rate of 1.2 mm/min and should be such that the test is completed within approximately 5 minutes.

2. Record the load of the test.

3. Continue the test until the FRP bar pulls out of the concrete specimen.

3.3.2.6 Beam flexural strength

Flexural strength testing of a small concrete beam involves following the ASTM D790 standard, utilizing the simple beam with third-point loading method. Here is a simplified description of the process:

Sample Preparation:

- Prepare a concrete mix of M20 grade according to design specifications.
- Place a braided bar at approximately 20 mm height in molds.
- Fill the beam molds of 100 mm x 100mm x 500 mm, with fresh concrete, compacting it thoroughly using a vibrator machine to eliminate air voids
- Allow the concrete to set and cure in a water tank for 28 days (Figure 3.19).

Test Setup:

- After the beams have cured, remove them from the molds.
- Measure and record the dimensions of the beams, including width, height, and length.
- Set up the testing machine with appropriate support rollers or blocks, ensuring the beam is centered under the loading head.
- Apply a three-point load at the specified rate of 1.5 N/mm²/min at the center of the beam using the loading head of the testing machine.[IS 516-1959]
- Load the beam at a constant rate until failure occurs.
- Record the maximum load applied and the corresponding deflection (Figure 3.20).

(a) Prepared BCR

(b) Prepaartion of BCR cages

(c) Addition of concrete

(d) Final cured beam

Figure 3.19 Flexural strength specimen preparation

Figure 3.20 Flexural strength specimen testing