CHAPTER 3

MATERIALS AND EXPERIMENTS

3.1 AGAVE ANGUSTIFOLIA MARGINATA (AAM) PLANT

The Agave Angustifolia Marginata (AAM) or Variegated accomplished from the Coimbatore, Tamilnadu, India. The common name of AAM was variegated Caribbean agave. Costa Rica, Mexico was the origin country of the AAM fiber. The AAM is a lovely and beautiful look, very stiff and short, knife-shaped leaves with cream shaded edges. The cultivation of AAM is much necessary, and it could be a little medium growth agave with a thick round rosette of leaf borne from a short trunk, and it shapes bunches.

Table 3.1 Physical Properties of AAM fiber

<table>
<thead>
<tr>
<th>Description</th>
<th>Dimension (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Leaf Height</td>
<td>50-90</td>
</tr>
<tr>
<td>Leaf Spread</td>
<td>50-120</td>
</tr>
<tr>
<td>Length</td>
<td>60</td>
</tr>
<tr>
<td>Width</td>
<td>05-10</td>
</tr>
<tr>
<td>Flower length</td>
<td>05</td>
</tr>
<tr>
<td>Height of the flower stalk</td>
<td>250</td>
</tr>
</tbody>
</table>

The physical properties of the AAM fibers shown in Table 3.1. The growth condition is purely dependent upon the growth rate and climatic conditions of the plant area. The leaves of AAM significantly longer based on the developing nature. The AAM leaves are robust, sword formed and curved
in nature. The color of the leaf is slightly blue or blue with greenish. The flower of the AAM colored with greenish yellow and white. The sprouting season of the AAM plant takes ten years or more (however, not a century) in spring, expire later subsequent blossoming, but instead, new plants may create from pushovers at their base and from bulbils along the bloom stalk.

3.1.1 Culture of AAM Plant

They are fantastic Agaves for partitions due to their little size; they require full sun to little shade or high inside lighting. Plants are not all that dry spell tolerant and require more water than most agave species. Plants ought to be watered and permitted to dry before watering once more. At the point when developed as a houseplant, the temperature ought to never rise and fall under 10° C. Not an ice tolerant, throughout the winter months, just waters enough to shield the leaves from shrinking. The ground developed plant is tolerant to dry spell and salty ocean side conditions. The toothed leaves with sharp spine at the tip are at some point displaced to ensure individuals. The AAM prompted by using suckers, which frequently exposed developing around the base of the plant. Luckily, numerous young or small plants could frame from the inflorescence, offering great open doors for the spread.

3.1.2 Extraction of AAM Leaves

The AAM with a flower as shown in Figure 3.1. The removal of the AAM leaves from the AAM plant as shown in Figure 3.2. The AAM can be developed reasonably, and collected effortlessly, and used for making grounded rope, and the longest enduring apparel, and most exciting medications. Developed on a business scale in the most recent century, AAM gives an economic other option for many oil-based items. It can likewise be utilized to make a variety of items from varnish to auto fuel.
Figure 3.1 AAM plant with flower

Figure 3.2 leaves removal from AAM Plant
AAM is not just a vital yield, it is exceptionally an eco-accommodating as well, not at all like different harvests it can develop in many areas and atmospheres with less water and manure necessities. Long, reliable and robust, AAM fibers are around 60% of cellulose and contain low levels of lignin (around 10-14%).

3.1.3 Usage of AAM Plant Fiber

AAM fibers identified as high rigidity and low density. As a result of this, historically, they were used as a part of assembling twines and ropes for angling and agricultural purposes. Further, these fibers can develop for the generation of nets, rugs, floor coverings, door mats, packs, sacks, fish stringers, furniture webbing, wrap, upholstery cushioning, saddle cushions. It can also use to make pad stuffing, brush bristles, wicker bin, wrist trinkets, headbands, shoes, ornamental things, garments and other woven articles. Papers additionally have been produced using lower grade AAM plant fibers. Research findings proved that AAM fiber could likewise use for other modern purposes. There can be a potential use of AAM fiber in composites and additionally in Nonwovens. Bio-plastics, Geotextiles, floor coverings, fiber sheets, dart sheets and formed furniture can be made utilizing AAM fibers.

3.1.4 Environmental Benefits of AAM Plant and Fibers

AAM plants have four times more cellulose than the quickest developing eucalyptus tree (flowering trees and shrubs), and it is successfully caught CO₂ from the climate. It requires watering system just three to four times each year. AAM fibers have an insignificant ecological effect. The generation does not require farming chemicals. While the handling of AAM fibers, just natural waste is delivered which can regularly reuse. After producing and handling of the agave appears as a bio-degradable natural
substance which can be utilized as "fertilizer" or as a natural material to come back to the area and as fuel for biogas generation. Along these lines, they improve soil fertility. Not at all like engineered (human-made) fibers, AAM fiber is 100% biodegradable in their fullest lifetime, AAM fabricated ropes, and different items can reuse as paper. The plants can likewise utilize as a shield harvest, land from hunters and the deep root structure lessens soil disintegration in dehydrated ranges.

3.2 EXTRATION OF AAM FIBER

3.2.1 Mechanical Method

The hand decortication finished by urban society through which the leaves beat and the pulp smashed away with a knife. Hand decortication is tedious and needs a considerable measure of labor. These days, decortication should be possible effectively by utilizing mechanical decorticator. In the mechanical decoration method, leaves are milled and beaten by a pivoting wheelset with limit blades, so just fibers remain. Some decorticators nourished by hand and the pulp first smashed from half of a leaf, the leaf pulled back, and after that, the inverse half embedded for scratching. In a few machines, the entire leaf decorticated in a single process. In a sectional perspective of the most vital parts of fiber stripper/decorticator. AAM plant leaves stimulated through the mouthpiece, at that point, it goes through the fluted bolster rollers, which clamp the leafs as they are sustained against a stationary bar, while the stripping drum is thumping out the vegetable concern as the leaf goes amongst it and the mixer bar. The stripping drum distance across, width and speed change as indicated by various makes. The drum, scratching against the leaf, held in position by the thumping bar and sustain rollers thumps off the central part of the vegetable matter and leaves the fibers relatively crumpled and with a deposit of vegetable matter staying upon it. In
this method, the AAM fiber extracted by mechanical decoration procedure. The AAM fiber extraction technique is shown in Figure 3.3. After the process of decortication and washing; the fibers are dried in the sunlight up to 48 hours to remove the moisture content entirely.

![Figure 3.3 Extraction of the AAM fiber]

The operations of fiber expulsion, washing and drying must be done quickly after the leaves cut. The gums in the leaves solidify, making the mash follow with the strands and making it hard to clean the strands legitimately. Mechanical extraction techniques are not proficient in the
evacuation of solidifying mixes (generally waxes, hemicelluloses, lignin, and hydrocarbons) between fibers (Bledzki et al. 2010).

3.3 ALKALI TREATMENT

Sodium Hydroxide (NaOH) or Alkali or Mercerisation is a frequently applied chemical treatment method for natural fibers when used to strengthen the thermoplastics and thermosets (Sukmawan et al. 2016). The important change done by soluble (alkali) treatment is the distraction of hydrogen holding in the network structure, consequently expanding surface roughness. The stages required for the basic treatment of fibers. This treatment expels a specific measure of lignin, wax, and oils covering the outer surface of the fiber cell wall, depolymerized cellulose and uncovered the short length crystallites. The exact concentration of sodium hydroxide (NaOH) to natural fiber advances the ionization of the hydroxyl gathering to the alkoxide (Yılmaz & Nazire 2013, Serrano et al. 2013). Thus, soluble handling directly impacts the cellulosic fibril, the level of polymerization and the extraction of lignin and hemicellulosic mixes. The AAM fibers soaked with NaOH solution with different time shown in Figure 3.4. The NaOH solution plays major roles in bleach, clean and make changes in the surface structure of the cellulose in plant fibers. AAM fibers were treated with 2, 5, 10 and 15 % (2, 5, 10 and 15g/L) sodium hydroxide solution at normal room temperature and fibers were immersed in the alkali solution as 1h, 2h, 4h, 6h & 8h time duration. Then, washed the NaOH treated fibers with distilled water and tap water repeatedly to neutralize the chemicals.

Further, The 2.5% Hydrochloric acid (HCl) also used to neutralize the chemicals. Then the fibers were kept in sunlight for up to 48 hrs. The raw and alkali treated AAM fiber shown in Figure 3.5. The more concentration of the NaOH Solution resulted in substantial damage to the fiber surface and less
concentration of NaOH only removed the insufficient amount of surface impurities. The average concentration of sodium hydroxide was recommended to achieve the best results.

Figure 3.4 Fibers soaked with 2%, 5%, 10% and 15% NaOH solution
Figure 3.5 Alkali treated fibers with different NaOH concentrations

3.4 PHYSIO-CHEMICAL AND MECHANICAL TESTS OF AAM FIBER

3.4.1 Breaking Strength and Elongation

The breaking strength of the raw and treated fibers tested as per ASTM D 3822 by using the Instron 5500R machine. In this method, single raw fibers and 2.5, 10 and 15 % of NaOH treated fibers with different holding time were tested separately. The mean value of breaking strength, CV % strength, elongation and CV % of elongation calculated as per above standard.

3.4.2 Fiber Length

The fiber length was measured by using ordinary steel rule. The leaves vary significantly in length according to growing conditions but can reach up to 60 cm (or more).
3.4.3 Diameter

Image analysis was used to calculate the diameter of the raw and Alkali treated fibers. The accurate results achieved with the help of image analyzer and used in 20 different locations of AAM fibers, and finally, average mean values noted.

3.4.4 Fiber Density

The ASTM D 792-13 (water displacement method) standard followed to find the density of the raw and Alkali treated AAM fibers. The one-gram amount of fiber immersed in the water and volumetric displacement was noted. The weight measured by the high precision weighing scale machine. Finally, based on the weight to volume ratio the density value was measured (Mylsamy & Rajendran 2010).

3.4.5 Weight Loss of the Fiber

The AAM fibers exposed to various NaOH base concentration as 2, 5, 10 and 15% with differing holding time as 1, 2, 4, 6 and 8 hours. The weight reduction of the AAM fibers with impact on changing NaOH concentration investigated. The weight loss measured by high exactness measuring scale machine. Dry cleaned fiber before soaking as \( W_1 \) and the measure of fiber was weighed again in the soaking as \( W_2 \). The weight loss percentage ascertained as

\[
\% \text{ weight loss} = \frac{(W_1 - W_2)}{W_1} \times 100
\]  

(3.1)

3.4.6 Water Absorption Properties (Raw & Treated)

Water absorption studies on treated and untreated AAM fibers were carried out by ASTM D570-10. The AAM fiber inclines absorbing water
up to its particular weight. In this case, the raw AAM fibers and NaOH (Alkali) base treated AAM fibers subjected to an absorption test by immersing the individual specimens in a clean water bath at room temperature (29 ºC). The holding period of 24 hours provided. The natural fiber retains water because the cell wall polymers have certain hydroxyl and other oxygenated groups that draw moisture amid hydrogen holding. The hemicellulose is for the most part in charge of water absorption in the common natural fiber (Sgriccia et al. 2008). At regular time intervals, the fiber weights measured and the moisture content calculated as

\[
\text{Moisture content (\%) = \left(\frac{W_2 - W_1}{W_1}\right) x 100}
\]

(3.2)

Where, W1 and W2 denoted the initial weight of dried AAM fiber and weight after time ‘t’ of soaking respectively.

3.4.7 Aspect Ratio (L/d)

The uniformity of the natural fiber is hard to maintain the growth of the fiber depends on the region and climatic conditions. Hence, the measurand of the fibers was taken on an average basis to maintain the regularity. Different samples of fibers taken, and each measurand is noted in different positions on the same samples to arrive at the average values. The average values computed for length and diameter. This procedure followed on both raw fibers and all the samples of alkali treated fibers. From the result, it was shown clearly when the Alkali concentration goes higher, the fiber diameter decreases rapidly, and the lengths also reduced.

3.4.8 Wax Content

Waxes make up the part of the fiber, which can extract with organic solutions. These waxy materials consist of different types of alcohols,
which are insoluble in water and several acids, such as, palmitic acid, oleaginous acid, and stearic acid. The Wax content measured with the aid of sox lot apparatus. Petroleum benzene liquid heated to 70°C, and one gram of fiber dipped in the liquid. Then the fiber sample was dried for one hour, after that, it weighed, and the weight difference provides the amount of wax content.

3.4.9 Cellulose Content

The weighted quantity of AAM fiber immersed in NaOH 1.72% and some drops of sulfuric acid mixed with the water. The soaking time was provided up to 1 hour by using suction process, the excess fluids removed, and ammonia added. The remaining fiber was washed by distilling water maximum 5 times, and it was allowed dry at room temperature. Then the percentage of cellulose weight loss was noted, and the difference recognized.

3.4.10 Lignin Content

A weighed amount of AAM fiber submerged in a mixture of sulfuric acid 12.5 and 300 ml water at room temperature. The two hours soaking time given and after that, the solvents removed by using distilled water. Finally, the residue weight noted as the lignin content.

3.4.11 Hemicellulose Content

The dry AAM fiber of weighed quantity was dipped in a mixture of 5% NaOH solution at room temperature for half an hour, and it deactivated with HCl. The fiber was dried in a hot air oven, weighed, and then weight transformation interprets the presence of hemicellulose content.
3.4.12  Tensile Strength of AAM Fiber

The INSTRAN 5500 R-60211 machine identified the AAM fiber tensile properties with a gauge length of 50 mm and a crosshead speed of 2 mm/min. The thirty samples of raw and treated AAM fibers (2, 5, 10 and 15%) subjected to the tensile test and average values taken.

3.5  EPOXY RESIN

The epoxy resin used as polymers because of their different modern applications requiring absolute quality, fantastic bond, excellent substance resistance, and phenomenal execution at raised temperatures. Because of that, they are used as a part of coatings, electrical/electronic overlaps, adhesives and to make leading composites. The composites in car parts or wind turbine sharp edges are new complications for epoxies. To lower the weightiness of vehicles such as planes, trains, cars, and so forth to lessening fuel utilization. The epoxies are presumably the most flexible group of structure/ necessary adhesives since they are perfect with many substrates, and can be used effectually to accomplish the fluctuating properties. Nowadays, the epoxy resin applied as a part of car assembly, substantial, submarine sheets, flying machine, aeronautics and domestic group adhesive and so on. The worldwide epoxy thermosetting polymers production was evaluated to be 2 million tons in 2010 and is predicted to achieve 3 million tons by the year 2017. Regular epoxy resins are low sub-atomic weight pre-polymers or higher sub-atomic weight polymers which typically contains no less than two epoxides collected. The raw materials for epoxy resin manufacture are to a vast extent petroleum determined; the significant resin derived from the addition of Bisphenol A and epichlorohydrin.
Table 3.2 Properties of Epoxy Resin

<table>
<thead>
<tr>
<th>Properties</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aspect (visual)</td>
<td>clear, pale yellow liquid</td>
</tr>
<tr>
<td>Colour (Gardner, ISO 4630)</td>
<td>≤ 2</td>
</tr>
<tr>
<td>Viscosity at 25 °C (ISO 9371B)</td>
<td>10000 – 12000 [mPa s]</td>
</tr>
<tr>
<td>Density at 25 °C (ISO 1675)</td>
<td>1.15 - 1.20 [g/cm³]</td>
</tr>
<tr>
<td>Flash point (ISO 2719)</td>
<td>&gt; 200 [°C]</td>
</tr>
<tr>
<td>Storage temperature</td>
<td>2 - 40 °C [°C]</td>
</tr>
<tr>
<td>Youngs Modulus</td>
<td>3200 MPa</td>
</tr>
<tr>
<td>Poisson’s Ratio</td>
<td>0.35</td>
</tr>
<tr>
<td>Tensile Ultimate Strength</td>
<td>88 Mpa</td>
</tr>
</tbody>
</table>

A few investigations have discovered various levels of Bisphenol A by using city wastewater. Likewise, it has resolved that Bisphenol A is unkind to marine life. The usual epoxy resin has numerous unfavorable impacts on the circumstance, human wellbeing and they increment convergence of carbon dioxide in the air which is greenhouse gas after warm disintegration/ Burning of epoxy resin (polymers). The inexhaustible assets incorporated with bio-based items (plant, creature, or contagious). There is an increasing interest in bio-based, reflective performance materials, where the emphasis is on performance and persistence. The properties of the epoxy resin shown in Table 3.2.
3.5.1 Bio-Based Epoxy Resin

The bio-based epoxy resins are low sub-atomic weight biodegradable polymers which are a group of regular oils (plant oils). Bio-epoxy resins are increasing considerably more significance given their ecologically well disposed, sustainable, green approach, high biodegradability helping the world to manage expanding issues of litter, bring down expenses in make and much lower carbon footprint. Usually, The Biobased epoxy resins got from plant oil crude materials like the most imperative thermosetting resins, which after curing, show great mechanical behavior, great warm, electrical, and chemical resistance, excellent bond to many substrates. In the huge majority of the cases, the bio epoxy resins additionally give a cost reduction when contrasted with that of the oil-based polymer composite, and they are biodegradable in the constrained period. Plant oil based polymers are biodegradable plants (green growth) which devour carbon dioxide for reducing greenhouse gasses. Among the different plant seed oils, non-palatable oils have utilized for the improvement of chemicals and polymers hence maintains a strategic distance from nourishment versus fuel predicament. The unsaturated fat is a vital component to change over to various useful gatherings. Unsaturated triglycerides can be changed over to the epoxy gathering, which was then changed over to the bio-epoxy resin. Since every one of these items does not contain any fragrant gathering, they are UV safe. The functionalization of double bonds in triglyceride to epoxy gives an active compound for biodegradable epoxy resin inclination by utilizing different bio-based hardeners. The acquired resin was environmentally friendly (Zhu et al. 2004).
### 3.5.2 Applications of Bio-Epoxy Resins

Different modern valuable items arranged from these polymers which comprise of printing ink, glossy paint, cover, Indian rakhi, master Ganesha symbol, a thin adaptable straightforward film for bundling. Moreover, also different cast resin articles, letters, play dolls, encapsulated for electronic circuit board, fiber-based composite, green growth oil-silicone oil based cross type sheet with containing unique properties. The essential part of dynamic asset materials based polymers is their biodegradation. The bio epoxy resin arranged from vegetable/green growth oil was subjected to biodegradation under bio-compost. It took ninety days to completely debase the carbon dioxide and water interval under bio-compost conditions (Zhu et al. 2004).

### 3.6 PREPARATION OF MATRIX

The ARALDITE LY 556 grade epoxy with a density of 1.15 g/cm³ and ARADUR HY 951 grade hardener with a density of 0.98 g/cm³ used to produce the natural fiber reinforced polymer matrix composite. The weight proportion of the blend of epoxy and hardener was 10:1. Epoxy is a thermosetting polymer that cures (polymerizes and cross connections) when blended with a hardener. It can make adaptable or unbending, straightforward or hued, quick setting or amazingly moderate. An exact electronic measuring machine was utilized to measure the resin and hardener. The weighing of the epoxy resin, hardener and electronic weight balancing machine shown in Figure 3.6.
3.6 **Figure 3.6 Weighing of epoxy resin and Mixing with Hardener**

3.7 **PREPARATION OF MOULD**

In compression molding machine, the separate mold die used for making composite sheet such as upper die and lower die with dimensions of 300 mm x 300 mm. The mold plate is shown in Figure 3.7.

**Figure 3.7 Mold Plate for preparing composite**

The 3mm distances maintained between the upper and lower die. The square steel frame 300 mm x 300 mm fabricated with 3mm thickness. The finished composite sheet had obtained with the above dimension. The mold plate manufactured with high heat resisting material (EN8). The mold
plate should withstand the heat applied during the manufacturing of the composite.

3.8 FABRICATION OF COMPOSITES

Fabrication of randomly oriented AAM fiber reinforced epoxy Composites

![Figure 3.8 Fabrication of NFRC by Compression Molding Machine](image)

The AAM fiber and resin were weighed separately according to the proportion. The hardener blended with epoxy resin by using glass pole in a bowl. To avoid air bubbles and pore in the mixture, stirring well it by using
glass pole. The upper and lower mold die surface were cleaned and waxed before initiating the fabrication process. The randomly mixed AAM fibers kept on the lower mold surface area, and the blended resin was poured consistently over all regions of the mold surface. After that, the hydraulic pressure applied in between the molded plates up to 103.42 bar by using the CMM. The temperature was gradually applied up to 70 °C, and it was kept up to 1 hour. The mold plates (bottom & top) opened after the assessed time, and it was permitted to attain the room temperature under atmospheric air condition. Figure 3.8 demonstrates the photographic perspective of fabrication of NFRC by using CMM. The impact of fiber/resin proportion and the effective combination was identified based on the varying concentration of resin/fiber such as 60:40, 65:35, 70:30 and 75:25. The impact of fiber length is one of the critical issues in the composite, and it applied the change in dimension of fibers like 10, 30, 50, 70 and 90mm during the fabrication of the different combination of composites. Similarly, the short fiber composite also manufactured based on the above proportion mentioned. Figure 3.9 demonstrates the photographic perspective fabricated composite plates and the ASTM standard specimen shown in Figure 3.10.

Figure 3.9 Sample Composite Plates
3.9 TESTING OF MECHANICAL PROPERTIES

3.9.1 Tensile Test

The specimen prepared for the tensile test is according to the ASTM standards. The Figure 3.11 shows the sample readily loaded to conduct the tensile test by using UTM on which the tensile tests were carried out on the composites. The ASTM D 638 method adopted for conducting the tensile test and during the test the uniaxial load gradually applied on both ends of the specimen loaded area. The advanced load controller with a maximum capacity of 100kN attached load cell computerized Kalpak UTM utilized for testing of the tensile strength of composites as per the above standard. The work samples measured with 20kN load cell capacity with variant conditions. The speed of the gage is allowed to move at 2mm/min and the maximum length fixed up to 50mm. The test performed in a relative humidity of 50 ± 2% with average atmospheric temperature. The test sample fixed over between the heaps of the gripper in a longitudinal pivot. During the test, the strain values noted by using the UTM controller computer. For every
test, there are three samples involved to achieve the mean and average values. The yield point and breakeven point noted during the specimen test.

3.9.2 Flexural Test

The flexural test was performed as per ASTM D 618 method and three-point bending method involved to accomplish the test accurately. The specimen loaded in the Kalpak UTM to conduct the test as shown in Figure 3.12. The specimen measured by 20kN load cell with advanced controllers of UTM. The elongation, yield point and breakeven point of the specimen can be controlled and noted by the computer. The crosshead moves with a speed of 2mm/ min, and gage length maintained 50mm. The test performed in a relative humidity of 50 ± 2% with standard atmospheric temperature. The test sample fixed over between the heaps of the gripper in a longitudinal pivot. During the test, the strain values noted by the UTM controller computer. For every test, there are three samples involved to obtain the mean, and average
values to get final results. The yield point and breakeven point noted during the test.

![Specimen Mounted in UTM](image)

**Figure 3.12 Specimen Mounted in UTM**

### 3.9.3 Impact Test

The ASTM D 256 method directed to complete the impact test by using Izod impact testing machine. By conducting the impact test, the material resistance against shocks and suddenly applied force measured. The specimen mounted on a vertical column, and room temperature maintained throughout the test. The specimen was broken by a sledge in pendulum with single blow and readings were noted. For every test, there are three samples involved to accomplish mean, and average values to get final results. Figure 3.13 showed the specimen attached to an impact testing machine to find the impact behavior of the specimen.
3.9.4 Compression Test

The ASTM D3410 method applied to conduct the compression test of AAM fiber-reinforced composite specimen. The 25 mm x 3 mm x 150 standard sizes fabricated to conduct the test efficiently. The samples maintained under the room temperature with a relative humidity of 50 ± 2%. The crosshead moved at 2mm/min speed. The test sample fixed over between the heaps of the gripper in a longitudinal pivot. During the test, the strain values noted by the UTM controller computer. For every test, there are three samples involved to find the mean, and average values to acquire final results. The yield point and breakeven point noted during the testing of the specimen. Figure 3.14 shows the ASTM specimen and arrangement of the compression test.
3.10 WATER ABSORPTION PROPERTIES

The inclination of water intake found in NFPC. The effect of an increase in moisture retaining percentage of the fiber-matrix interface was affected and reduced the mechanical properties of composites. Water intake behavior of AAM fiber strengthened epoxy composite investigated by using the ASTM D570 method. The 4-digit perfect balance machine was utilized to measure the water uptake properties of samples.

\[
\text{Water Absorption (\%) } = \frac{(W_2 - W_1)}{W_2} \times 100
\]  

(3.3)

Where \( W_1 \) = weight before soaking in water (g) and \( W_2 \) = weight after soaking into water (g)

3.10.1 Water Absorption Test of AAM Fiber

The AAM fiber has the incline of water intake to its specific weight. For this situation, the crude AAM fiber and NaOH treated AAM fibers subjected to a water retention test by soaking the particular specimen in a clean water shower at room temperature (29 °C). The AAM fiber subjected to various soaking time such as 15, 45, 90,150,225 and 300 minutes. The
moisture content present in the natural fiber due to the cell wall polymer of particular hydroxyl and other oxygenated bunches that draw humidity in the midst of hydrogen holding. The hemicellulose is one of the primary factors, and it is responsible for water consumption in the conventional natural fiber.

3.10.2 Water Absorption Test of AAM Fiber Reinforced Epoxy Composites

The water intake test of AAM fiber reinforced composite determined by using the ASTM 570-98 method. The ASTM size of the testing samples dimensions is 76.2 mm X 25.4 mm X 5 mm. The specimens dried in the open atmospheric air till it reached the constant weight. The water assimilation behavior of untreated and alkali treated AAM fiber reinforced polymer specimens are weighted in numbers and submerged in distilled water shown in Figure 3.15, and boiled with water at 100ºC shown in Figure 3.16. The water intake monitored continuously with respect to the time interval. The water consumption rate of every specimen analyzed and evaluated. The various weight percentages of the AAM fibers such as 25, 30, 35 and 40 % subjected to fabricate the composite and also conduct the test as per the ASTM standards. The specimens made as per the ASTM and keep it in a hot air oven at 50° C for the one-hour duration before the start of the experiment. At this moment, the ready samples soaked in the purified water, and the temperature is maintained up to 30° C around 14 days. The samples were again submerged in the water and to allow the prolongation of the absorption rate till the maximum immersion stage reached. That technique was repetitive until the equilibrium achievement, and the water consumed specimens packed by using the polyethylene cover for to test the mechanical behavior.
3.10.3 **Effect of Moisture on the Mechanical Properties of Composites**

The mechanical behavior of AAM fiber composite mainly influenced with the help of fiber length, the orientation of fiber, the strength of the fiber, modulus and fiber-matrix interfacial adhesive strength. The increase in weight of the composite causes the developing interlocked of the water molecules. Hence the water molecules get probabilities to outbreak the interface, peel off, crack initiation and propagation, causing debonding of the fiber and the matrix within in the composite.
3.10.4 Micro-Hardness of the AAM Fiber Reinforced Composites

The AAM fiber composite samples prepared and tested as per the ASTM D2240 method. The set of samples tested by using the tester of the micro-hardness machine as shown in Figure 3.17, and calibrated scale reading was noted. The set of five readings taken and the average specimen value observed.

![Figure 3.17 Microhardness tester](image)

3.11 FOURIER TRANSFORM INFRARED SPECTROMETRY (FTIR)

The FTIR spectrometry performed by using the thermo scientific NICOLET IS10 spectrometer equipment as shown in Figure 3.18 and the room temperature maintained during the test under at 29 °C. The IR light applied to the AAM sample. The reading noted when the IR frequency and vibrational frequency band was on same. The FTIR spectrometer utilized to
note down the interferogram, and it performed by Fourier Transform method to achieve the spectrum. The chemical behavior of the samples determined based on the analyze and outcome of the absorption spectrum. The FTIR spectrum used to prepare the raw fiber, 2, 5, 10 and 15 % NaOH treated AAM fibers.

![FTIR equipment](image)

**Figure 3.18 Fourier Transform Infrared Spectroscopy (FTIR)**

### 3.12 SCANNING ELECTRON MICROSCOPY (SEM)

The Surface Morphology (SEM) test conducted for the raw, alkali-treated fiber and composite with a high-resolution image captured by using Scanning Electron Microscopy machine model JEOL-JSM-6390 as shown in Figure 3.19. The SEM analysis of the worn surface specimen in wear test was made by the SmartSEM (SIGMA) Scanning Electron Microscope. The above instruments scanned the surface layers in high resolution with a high energy electron beam. The surface morphological study performed due to the
electron beam interrelates with the atoms on the surface. The scanning electron microscopy created a very high-resolution image. The accurate electromagnetic ray in the SEM microphotographs created a three-dimensional amplification that has been a better considerate of the surface structure of the sample.

Figure 3.19 Scanning Electron Microscopy

3.13 THERMOGRAVIMETRIC ANALYSIS

The short AAM fiber reinforced composites made in a different combination of fiber-matrix are as follows 25:75, 30:70, 35:65 and 40:60. The pellets shaped samples prepared from the above proportion of AAM fiber reinforced polymer composite samples, and made under thermogravimetric analysis. The weight took 10 mg for every sample. The equipped samples are retained at 100°C for 1 hour by hot air oven. The Netzsch STA 409 machine as shown in Figure 3.20 utilized to finish the thermogravimetric analysis test. During the test, the heating range of 10 °C/min and Nitrogen atmosphere with a flow rate of 50 ml/min sustained.
3.13.1 **Dry Sliding Wear Test**

The wear tests are planned to conduct by using the pin-on-disc machine. The various weight proportions of Alkali treated AAMF reinforced composites were slide against the stainless-steel plate (EN 32 Día of 165 mm and thickness 8mm) of the test rig. The loading range connected from 10, 20, 30 and 40 N with sliding speeds of 1.57, 2.09, 2.61 and 3.14 m/sec. The wear test conducted on a track of 100mm diameter and the period of 15 min with the relative speeds of 300 RPM - 600 RPM at fixed sliding distance length of 1000m. For the accurate result, at least three times the wear test was repeatedly conducted, and the average value was noted.
3.13.2 Composite Preparation for Wear Test

The 5 wt% alkali treated AAM fibers sliced into several short lengths (50mm). The Epoxy Resin and 5 Wt. % alkali treated short length AAM fibers blended with the weight proportion of 75:25, 70:30, 65:35 and 60:40. The composite plate fabricated by using the compression molding machine. The test specimens prepared in a specified ASTM size of 5 X 5 X 10 mm3. The tested samples placed on the bottom of metal pins by using glue and the contact surface located in a parallel to the disc.

3.13.3 Experimental Procedure

The ASTM G99-95 standard used to conduct the wear test experiment. The composite test specimen prepared as per the above standard, and the waterproof silicon abrasive paper was used to develop the roughness on the test sample surface to make the immediate contact with the counter surface of the disc and the sample. The samples and disc cleaned with acetone before the test and the wear setup shown in Figure 3.21. The following equation calculates the frictional coefficient.

\[ \text{Friction coefficient (μ)} = \frac{f}{F} \]  \hspace{1cm} (3.4)

Where, f is a frictional force (N) and F is Normal load (N).

Before the test process starts, the initial weight of test sample \( W_1 \) measured by using an electronic weighing balance machine with 0.0001g least count; then test specimen is allowed to run on the disc with varying parameters. After the test conducted, the sample weight is taken and noted as \( W_2 \). The difference between the weight of the sample before and after test mentioned as the weight loss \( \Delta W \).
Weight loss ($\Delta W$) = $W_1 - W_2$ \hspace{1cm} (3.5)

The wear rate and specific wear rate of the specimen with different speed and load conditions calculated by using the equation 3.6 & 3.7 (Bajpai et al. 2013).

Wear rate = $\Delta W/ \rho \times L$ \hspace{1cm} (3.6)
Specific wear rate = $W/\rho \times L \times F$ \hspace{1cm} (3.7)

Where the weight loss of the specimen during the test, denoted as $\Delta W$, the density of the sample ($\rho$), sliding distance, $L$ (m) and the average load $F$ (N).

Figure 3.21 Pin on disc friction and wear testing machine

3.13.4 Fatigue Life Prediction

The fatigue test conducted as per the ASTM method by using the Multiaxial Fatigue Testing Machine. The AAM fiber reinforced composite prepared with the help of compression molding machine. The fabricated
AAM composite shaped as per the dimension specified in the ASTM. The dimensions of the fatigue samples are 250mm x 25mm x 3mm. The sharp edges of the specimens were to some extent spherical down with emery paper before the screening, for to avoid any pressure focus effect. The different combination of AAM fiber-reinforced polymer composites like 60:40, 65:35, 70:30 and 75:25 were tested using tension-stress fatigue at monotonic behavior with pressure proportion (R) of around 0.15. The 3Hz loading frequency was used to remove the test sample heating. The fatigue behavior was accompanied depending on ASTM D3479 M, using pc managed servo-hydraulic check machine. The device determined the cycles of the test specimens-failure and the number of cycles-to-failure noted by the computer with the aid of data acquisition system. The fatigue testing machine setup and testing samples are shown in Figure 3.22 and Figure 3.23.

Figure 3.22 Fatigue life testing machine
3.14 EXPERIMENTAL FLOW CHART

The various experiments conducted on AAM fiber and the AAM fiber reinforced polymer matrix composites illustrated by using flowchart as shown in Figure 3.24 to Figure 3.27 as follows.
Figure 3.24 Experimental flow chart- AAM Fiber Extraction process by Mechanical Decorticator
Figure 3.25 Experimental flow chart- Alkali Treatment of AAM fiber and its properties
Figure 3.26  Experimental flow chart- Fabrication of AAM fiber reinforced epoxy composite by CMM
Figure 3.27 Experimental flow chart- Finding the mechanical, wear, thermal and Water absorption properties of AAM fiber reinforced epoxy composite fabricated by CMM
3.15 SUMMARY

The AAM fiber carefully extracted by using mechanical decortications method. The fibers were alkali treated with 2, 5, 10 and 15% NaOH solution with different soaking period. The physical, chemical and mechanical properties of the AAM fiber calculated as per ASTM method. Raw and 5% alkali treated randomly oriented AAM fiber of 10 mm, 30 mm, 50 mm, 70 mm and 90 mm lengths reinforced in the epoxy matrix, and the composites attained. The various test specimens according to the ASTM standards were tested to evaluate physical, mechanical, TGA and water absorption behavior of fiber, water absorption of composites (Boiled and cold) and tribological properties. The surface morphology of the failure specimens also analyzed.