

## Methodology

The methodology followed for using different techniques and tools in the investigation are described in detail under the following headings. It was conducted in five phases and are presented in Figures (1a – 1e)

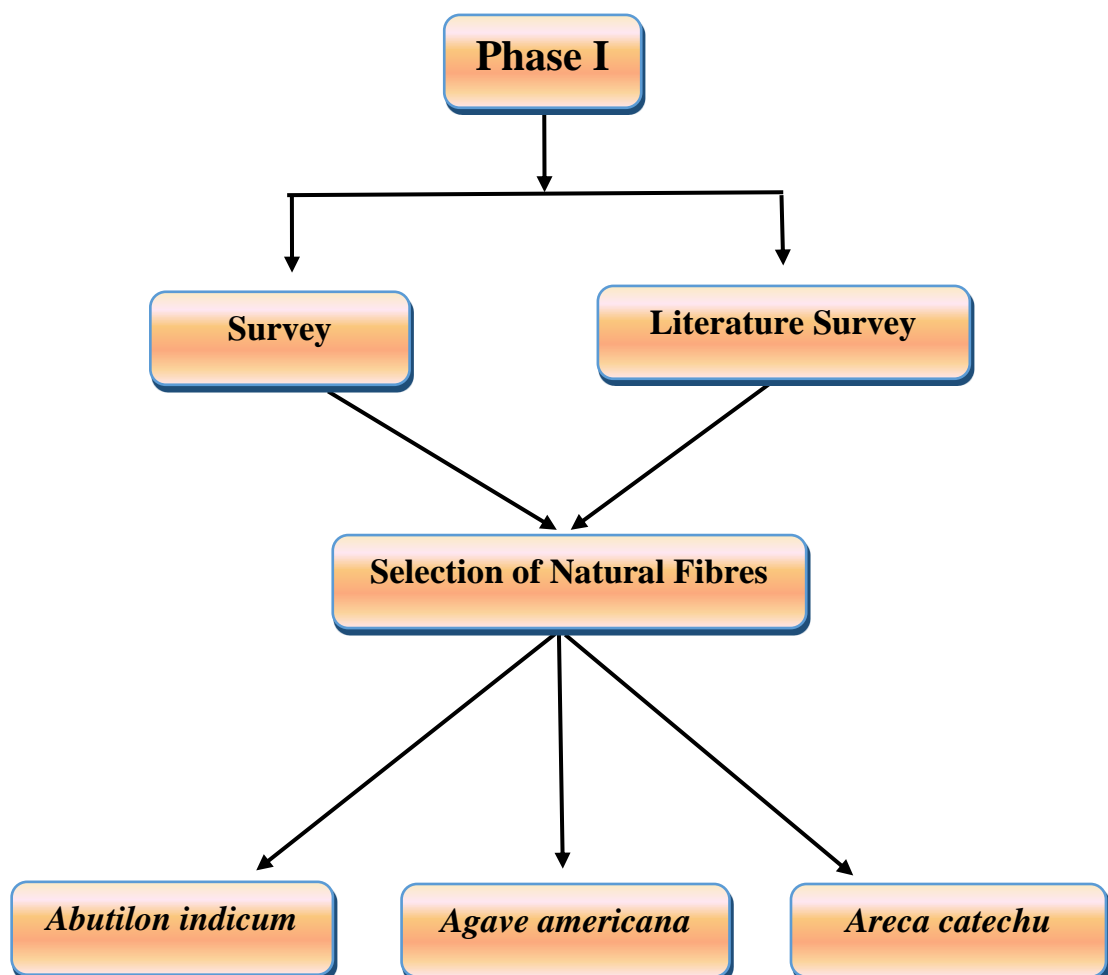


Figure 1a Phase I Survey and Fibre Selection

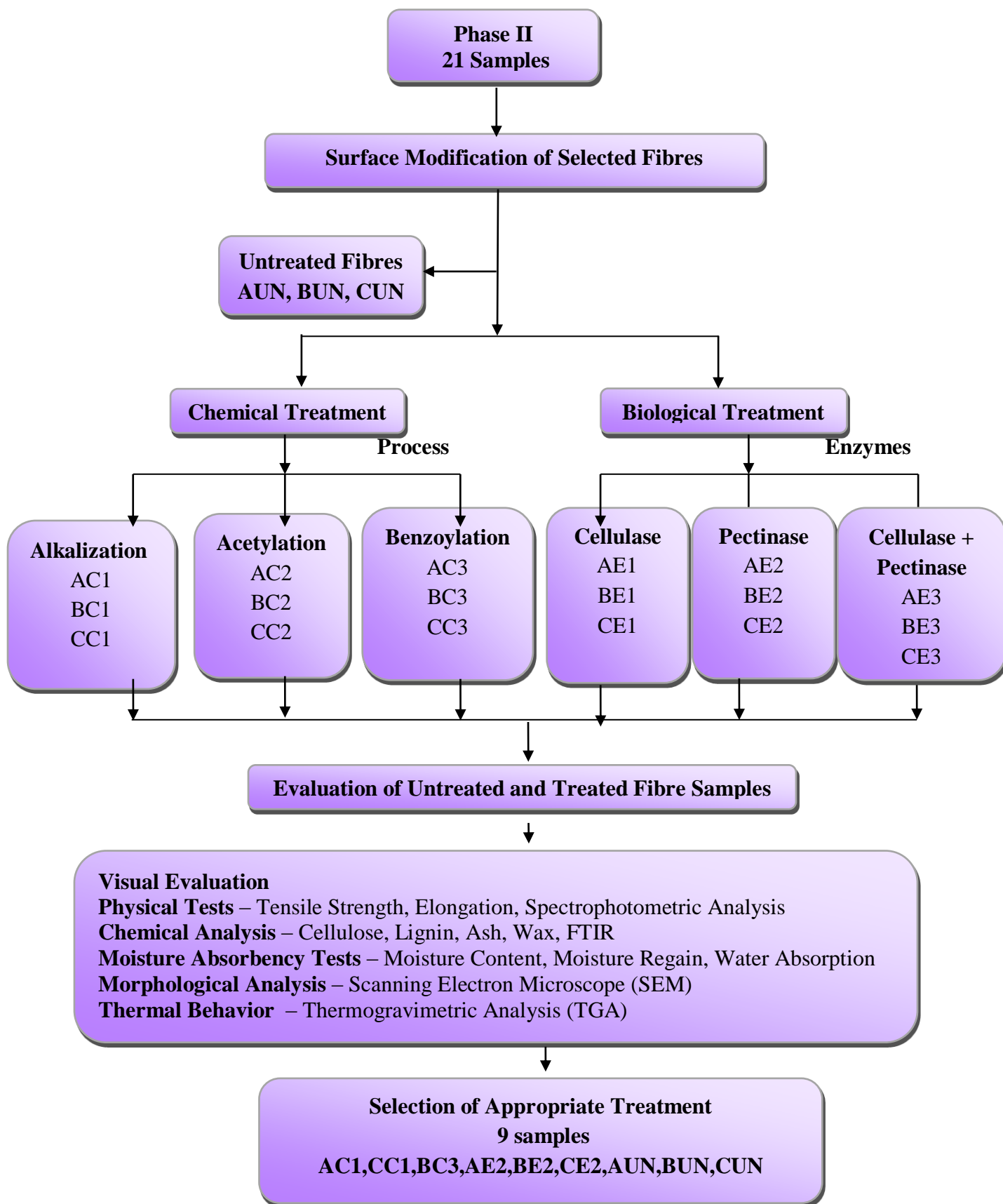


Figure 1b Phase II Selection of Treated Fibres

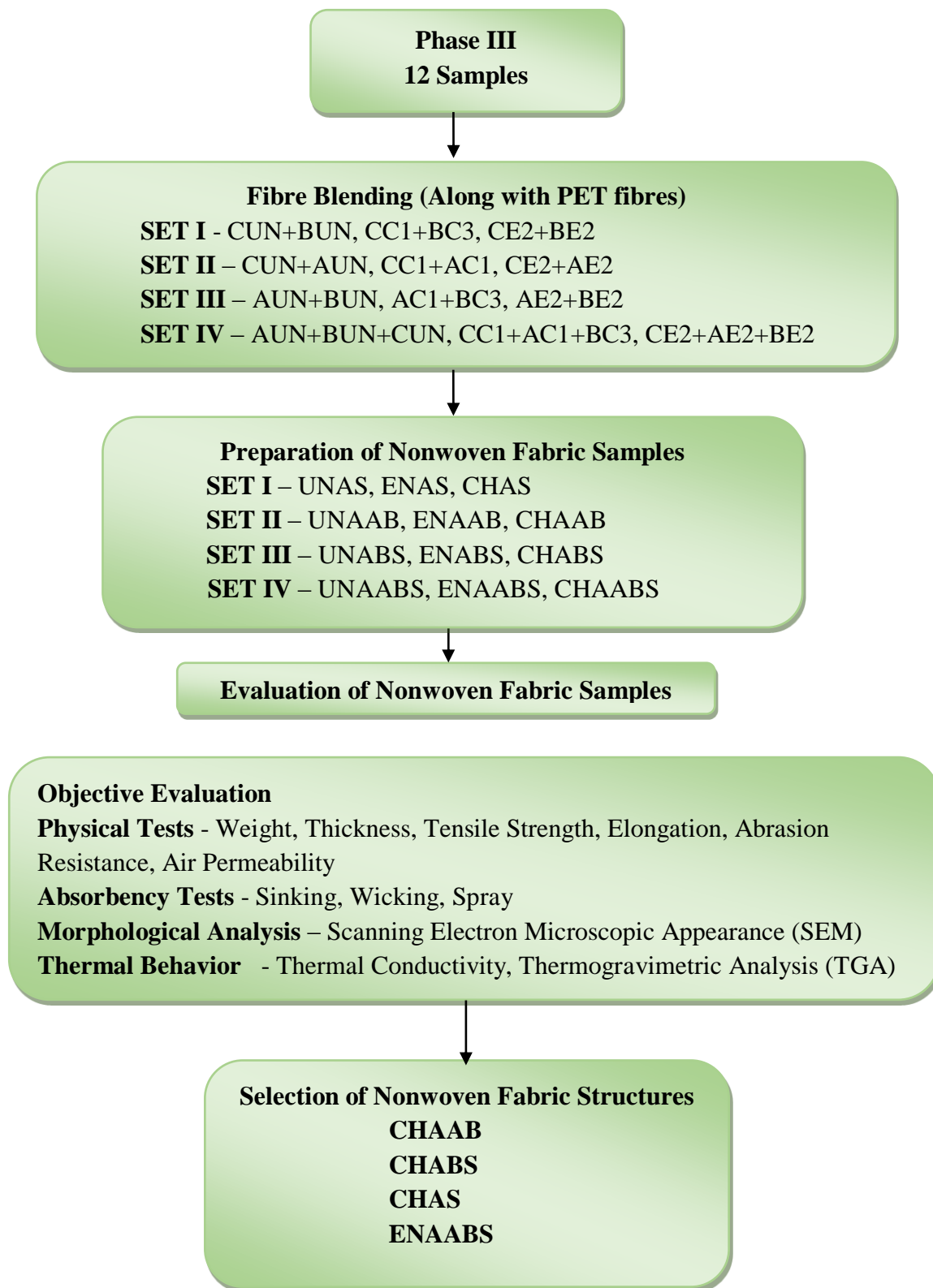


Figure 1c Phase III Nonwoven Fabrication, Evaluation and Selection

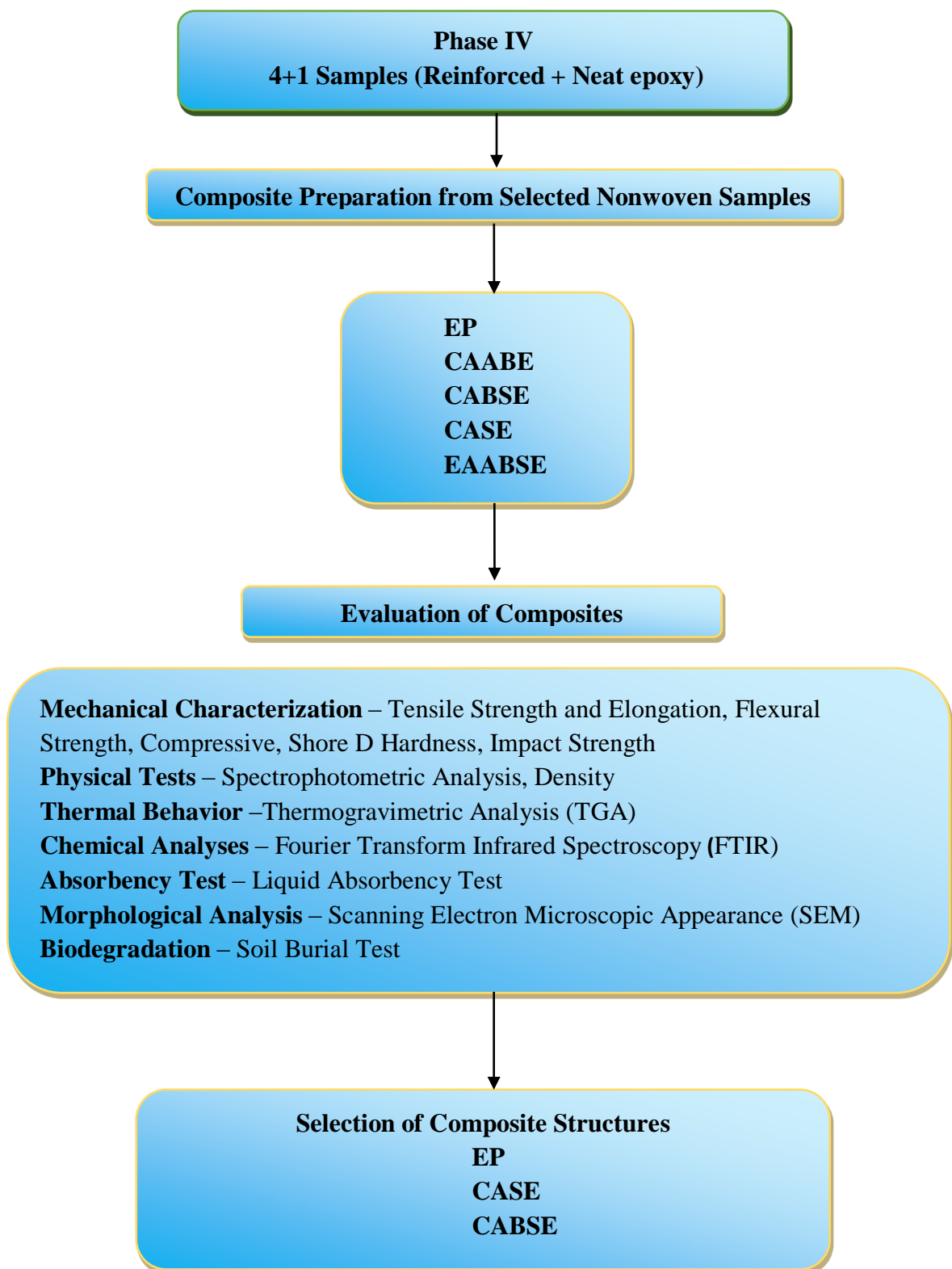


Figure 1d Phase IV Composite Preparation, Evaluation and Selection

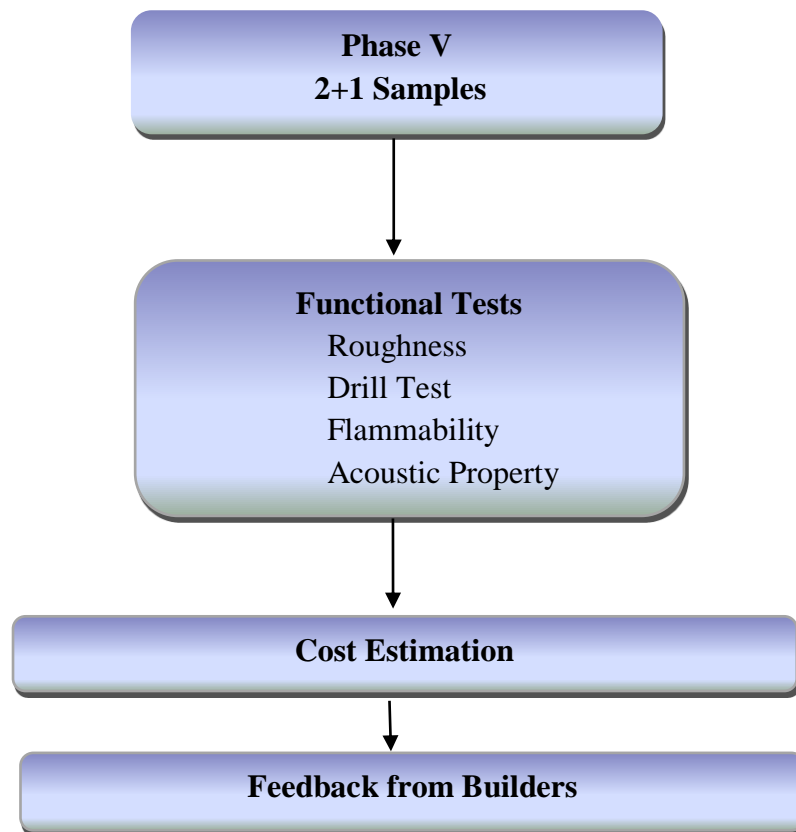


Figure 1e Phase V Functional Tests for Selected Composites

### **3.1 Phase 1 – Survey and Fibre Selection**

The method for survey conducted and selection of plant source are explained under the following heads.

#### **3.1.1 Conduct of survey**

A survey was carried out to gather information regarding the existing use and potentiality of natural fibres in civil applications. The purposeful sampling technique was used for the study because it is frequently employed in qualitative research to find and choose information to make the most of the resources (Patton 2002). It also involves selecting people or groups of people who have particular expertise in or experience with an area of interests (Creswell and Clark 2011). This survey was conducted among civil engineers, architects, builders and building contractors and the information related to the use of materials and problems in civil field was also obtained with a sample size of fifty. For gathering quantitative primary data, questionnaires are typically the primary approach employed. Utilizing a questionnaire enables the systematic collection of quantitative data, guaranteeing its internal coherence and suitability for analysis (Roopa and Rani 2012). So, the tool used for survey was questionnaire which comprised of twenty-five questions. The questions were framed with suitable options following the five-point Likert Scale. Pretesting was done and required modifications were carried out while preparing the questionnaire. The certificate obtained for ethical clearance from Institutional Human Ethical committee and the Proforma prepared for survey are presented in Appendix Ia and Appendix Ib.

The questionnaire comprised of questions based on demographic aspect, employment details, materials preferred for construction, type of materials used, factors considered for material selection, usage of natural fibres in construction field, awareness about use of natural fibres, existing application of natural fibres, fabricated structures used and suitability, local availability of construction materials, other fibres used in construction field, ecofriendly products, reusable materials, satisfaction about existing materials challenges faced in civil field and to understand the potentiality of natural fibres in various civil application. The prepared questionnaire was sent online to members in the civil field and were also approached directly. During survey the investigator explained to the respondents about the purpose of the survey and collected first-hand information. The data collected from the survey were systematically recorded and the results are presented in Chapter 4.

### **3.1.2 Selection of Fibres**

The bast fibres are the stiffest of all natural fibres with higher tensile strength and flexural strength and these constitute major utilization in composite manufacturing (Tanushree and Chanana 2016). *Abutilon indicum* is a plant belonging to Malvaceae family that yield fibres from the bast part of the plant (Plate 1a). Leaf fibres are obtained from the plant *Agave Americana* which is a xerophytic, monocarp, semi-perennial fibre producing plant that belongs to Asparagaceae family (Plate 1b). The fibres obtained from this plant are coarse and hard with appreciable strength (Kumar *et al.*, 2017b). The *Areca catechu* that belongs to Arecaceae family (Plate 1c) yields fibre from the fruit portion which is stiffer with high strength and can be an alternative for synthetic fibres in both structural and nonstructural applications (Venkateshappa *et al.*, 2012). Considering the facts from literature survey and for the reasons of easy local availability, these three plant sources were selected to combine the positive attributes of the stalk, leaf and fruit fibres.

### **3.2 Phase II - Procurement, Treatment and Evaluation of Fibres**

The treatment and evaluation methods carried out in the Phase II are presented under the following heads.

#### **3.2.1 Procurement of Fibres**

The fibres selected from different sources were confirmed and authenticated for the botanical names before procurement by providing a leaf and blossom specimen of all the three selected plants to 'Botanical Survey of India, Tamil Nadu Agricultural University, Coimbatore'. The confirmation certificate is presented in Appendix II. The fibres were extracted from the *Abutilon indicum* and *Agave americana* by using stagnant water retting method. Beating and scrapping was done for the retted stalk of *Abutilon indicum* and leaf of *Agave americana* after twenty-one days and twenty-three days of soaking respectively. The separated fibres were then washed thoroughly in soft water and dried in shade. The *Areca nut* fibres were extracted using machine. These fibres extracted by different means namely retting, decortication and mechanical processing were procured from Sudha Industries, Salem District, Tamil Nadu.



**Plate 1a** *Abutilon indicum*  
plant



**Plate 1b** *Agave americana*  
plant



**Plate 1c** *Areca catechu* tree



**Plate 1d** *Abutilon indicum*  
fibres



**Plate 1e** *Agave americana*  
fibres



**Plate 1f** *Areca catechu*  
fibres

**Plate 1 Selected Plant Sources and their fibres**

### 3.2.2 Surface Modification of Fibres

The natural fibres are modified using chemicals that alter the plant fibres permanently by grafting the fibre surface. It also provides better dimensional stability and giving resistance to fungal attack (Xie *et al.*, 2010). The biological treatment improves the flexural and tensile strength of fibre reinforced composites, leading to increased interfacial bonding (Bledzki *et al.*, 2010). Considering these facts, the selected fibres, namely *Abutilon indicum*, *Agave americana*, and *Areca catechu*, were cleaned, washed thoroughly for surface modification.

#### 3.2.2.1 Chemical Treatment

These fibres were subjected to treatment using chemicals through the processes namely alkalization, acetylation and benzoylation.

In the process of **alkalization** the raw fibres were treated with 5% NaOH solution for two hours to remove the unwanted soluble constituents from the fibres. The fibre weight to solution ratio was maintained at 1:25 and after two hours the fibre samples were washed thoroughly in distilled water to remove excess NaOH and dried at 60°C for 24 hours

(Bledzki and Gassan, 1999). Accordingly, all the fibres namely *Abutilon indicum*, *Agave americana* and *Areca catechu* fibres were treated with alkali for surface modification (Plate 2a).

For performing the "**acetylation**" process, the selected fibres were first soaked in distilled water for one hour. The fibres were then submerged for an hour at 30°C in a 5% NaOH solution. Following the alkali treatment, the fibres were soaked in glacial acetic acid for an hour at 30 °C, then drained and given an acetic anhydride treatment by adding one drop of sulfuric acid for five minutes. After treatment, to get rid of the traces, the fibres were thoroughly rinsed with distilled water (Mishra *et al.*, 2003). This method was carefully carried out for all three selected fibres (Plate 2b).

For the **benzoylation** process, the alkali pretreatment was used to activate the hydroxyl groups of the cellulose and lignin in the fibres. So the fibres were suspended in 10% NaOH and benzoyl Chloride solution for 15 minutes. These were then soaked in ethanol for one hour to remove the benzoyl chloride, then washed with water and dried in the oven at 80°C for 24 hours. (Li. *et al.*, 2007). This was followed for all three selected fibre samples (Plate 2c)

### **3.2.2.2 Biological Treatment**

Biological treatment was done using the enzymes namely cellulase and pectinase. The pectinase and cellulase treatments produced by *Aspergillus niger* fermentation were purchased from Resil Company, Tirupur. The fibres were treated as per the instructions given by the Resil company with enzymes at 50°C about 30 minutes and pH was maintained at 7 with continuous agitation. Each of the selected fibres was subjected to biological treatments using cellulase, pectinase, and their combinations using the same procedure (Plates 2d-f).



**Plate 2a Alkalization**



**Plate 2b Acetylation**



**Plate 2c Benzoylation**



**Plate 2d Cellulase**



**Plate 2e Pectinase**



**Plate 2f Cellulase and Pectinase**

**Plate 2a-2f Surface Modification of Fibres**

### **3.2.3 Nomenclature of Fibre Samples**

The nomenclature of the untreated and treated fibres used in the explanation and discussions of the study are presented in the Table I

**Table I**  
**Nomenclature of Untreated and Treated Fibre Samples**

S.No	Description	Nomenclature
	<b><i>Abutilon indicum</i> Fibres</b>	<b>A</b>
1	Untreated <i>Abutilon indicum</i>	AUN
2	<i>Abutilon indicum</i> after alkalization	AC1
3	<i>Abutilon indicum</i> after acetylation	AC2
4	<i>Abutilon indicum</i> after benzooylation	AC3
5	<i>Abutilon indicum</i> after cellulase treatment	AE1
6	<i>Abutilon indicum</i> after pectinase treatment	AE2
7	<i>Abutilon indicum</i> after cellulase and pectinase treatment	AE3
	<b><i>Agave americana</i> Fibres</b>	<b>B</b>
8	Untreated <i>Agave americana</i>	BUN
9	<i>Agave americana</i> after alkalization	BC1
10	<i>Agave americana</i> after acetylation	BC2
11	<i>Agave americana</i> after benzooylation	BC3
12	<i>Agave americana</i> after cellulase treatment	BE1
13	<i>Agave americana</i> after pectinase treatment	BE2
14	<i>Agave americana</i> after cellulase and pectinase treatment	BE3
	<b><i>Areca catechu</i> Fibres</b>	<b>C</b>
15	Untreated <i>Areca catechu</i>	CUN
16	<i>Areca catechu</i> after alkalization	CC1
17	<i>Areca catechu</i> after acetylation	CC2
18	<i>Areca catechu</i> after benzooylation	CC3
19	<i>Areca catechu</i> after cellulase treatment	CE1
20	<i>Areca catechu</i> after pectinase treatment	CE2
21	<i>Areca catechu</i> after cellulase and pectinase treatment	CE3

### 3.2.4 Evaluation of Fibres

The following heads provide the evaluation procedures that were used to compare treated and untreated fibres. Chapter 4 presents the findings on the tested fibres.

#### 3.2.4.1 Visual Evaluation

The visual evaluation was done to obtain feedback about the untreated and treated fibre samples. The judges, numbering twenty-five were requested to assess the fibre samples for colour, luster, texture and general appearance by displaying the samples. The obtained results were recorded and tabulated. The proforma used for obtaining feedback is presented in Appendix III.

#### 3.2.4.2 Spectrophotometric Analysis

The objective evaluation for analyzing the whiteness, yellowness and brightness indices of fibres was carried out with the spectrophotometer as per standards. It was

challenging to measure loose fibres because they have a tendency to protrude into the sphere, which could cause inaccuracies in the reflectance measurement. Falling into the instrument is also risky. Consequently, a superior technique was employed in which a compression cell containing the precise quantity of fibres was filled while maintaining a constant pressure (Plate 3a). All the fibre samples were thus tested and recorded.

### **3.2.4.3 Chemical Constituents**

Depending on the type of fibre and its sources, different natural fibres have different chemical compositions. The main components of fibre are cellulose, hemicellulose, pectin, and lignin. The overall characteristics of the fibres are influenced by the characteristics of each element. The percentage composition of each of these components varies for different fibres. The chemical compositions namely cellulose, lignin, wax and ash of untreated and treated fibres were tested in chemistry laboratory at SITRA, using standard in-house procedures.

### **3.2.4.4 Moisture Content, Regain, Water Absorption and Density**

**Moisture Content** of fibres was obtained by knowing the initial weight of the fibres which was recorded as ( $M_1$ ) after which the samples were oven, dried. The fibres were then weighed again after drying in the oven at 105°C for 24 hours and recorded as ( $M_2$ ). The moisture content was determined using the standard formula which is presented in the Appendix IVa.

**Moisture Regain** is defined as the amount of water or moisture expressed as the percentage of the oven-dry weight of the material. The moisture regain test was carried out for fibres according to ASTM D 5229. The formula used to calculate moisture regain is given in the Appendix IVb.

**Water Absorption** was assessed after immersing the samples in water at room temperature for twenty four hours. Five specimens from each sample were weighed before and after immersion in water. The formula used to calculate the water absorption is given in the Appendix IVc.

The **Density** of the fibres was assessed using the standard Method: SITRA/TC/FCC/03 in the SITRA laboratory, Coimbatore. The results obtained for all the fibre samples were recorded in g/cc.

#### **3.2.4.5 Tensile Strength and Elongation**

The untreated and treated fibres were analyzed for tensile strength, elongation, and time taken for break in 'South Indian Textile Research Association (SITRA)' laboratory. The equipment used was Zwick Roel for assessing the properties as per standard methods.

#### **3.2.4.6 Thermogravimetric Analysis (TGA)**

The thermogravimetric analysis of the fibres was conducted using the thermogravimetric analyzer EXSTAR/6300 in the CNR RAO laboratory, "Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore". About twenty milligrams from the sample of each set of fibre were taken and it was heated up at a range between 50°C to 400°C in nitrogen atmosphere. To get perfection, the analysis has been done two times for each of the samples (Plate 3b). The weight loss of the untreated and treated fibres at higher temperatures was obtained and recorded.

#### **3.2.4.7 Scanning Electron Microscope (SEM)**

Surface morphology of untreated and treated fibre samples was examined using Zeiss Scanning Electron Microscope in the SITRA laboratory, Coimbatore (Plate 3c).

#### **3.2.4.8 Fourier Transform Infrared Spectroscopy (FTIR)**

The presence of functional groups was analyzed using FTIR in the range of 16 cm<sup>-1</sup> to 6000 cm<sup>-1</sup> according to standard ASTM E 168-06 by using FTIR Spectroscopy Shimadzu FTIR 8400S. This was carried out for both untreated and treated fibre samples (Plate 3d).

From Phase II based on the properties exhibited by the fibre samples, the treatments which gave favourable results for the study were selected.



**Plate 3a Spectrophotometer**



**Plate 3b Thermogravimetric analyser**



**Plate 3c SEM analyser**



**Plate 3d FTIR Analyser**

**Plate 3 Evaluation of Fibre samples**

**3.3 Phase III – Nonwoven Fabrication**

The nonwoven fabrication of selected fibres includes mechanical and thermal bonding of fibres. The method followed for the fabrication of selected fibres into nonwoven fabrics is presented under the following headings:

**3.3.1 Nomenclature of Nonwoven Samples**

The samples were designated, and the nomenclature used are expressed in Table II.

**Table II**  
**Nomenclature of the Nonwoven Fabric Samples and others**

S.No	Nomenclature	Details	
		Nomenclature of the Nonwoven Samples	
1	UNAS	Set I	Untreated <i>Areca catechu</i> and <i>Agave americana</i>
2	ENAS		Enzyme treated <i>Areca catechu</i> (Pectinase) and <i>Agave americana</i> (Pectinase)
3	CHAS		Chemical treated <i>Areca catechu</i> (Alkalization) and <i>Agave americana</i> (Benzoylation)
4	UNAAB	Set II	Untreated <i>Areca catechu</i> and <i>Abutilon indicum</i>
5	ENAAB		Enzyme treated (Pectinase) <i>Areca catechu</i> and <i>Abutilon indicum</i> (Pectinase)
6	CHAAB		Chemical treated <i>Areca catechu</i> (Alkalization) and <i>Abutilon indicum</i> (Alkalization)
7	UNABS	Set III	Untreated <i>Abutilon indicum</i> and <i>Agave americana</i>
8	ENABS		Enzyme treated <i>Abutilon indicum</i> (Pectinase) and <i>Agave americana</i> (Pectinase)
9	CHABS		Chemical <i>Abutilon indicum</i> (Alkalization) and <i>Agave americana</i> (Benzoylation)
10	UNAABS	SET IV	Untreated <i>Areca catechu</i> , <i>Abutilon indicum</i> and <i>Agave americana</i>
11	ENAABS		Enzyme treated <i>Areca catechu</i> (Pectinase), <i>Abutilon indicum</i> (Pectinase) and <i>Agave americana</i> (Pectinase)
12	CHAABS		Chemical treated <i>Areca catechu</i> (Alkalization), <i>Abutilon indicum</i> (Alkalization) and <i>Agave americana</i> (Benzoylation)

### 3.3.2 Mechanical Bonding

Needle punching technique was used for converting the selected fibres directly into nonwoven fabric. The treated fibres that were selected were cut into small pieces of 3 centimetre length. The fibres were blended thoroughly for carding. The proportion of fibres used for blending is presented in Table III.

**Table III**  
**Blend Proportion of Fibres for Nonwovens**

S.No	Nonwoven Fabric	<i>Abutilon indicum</i> (gms)	<i>Agave americana</i> (gms)	<i>Areca catechu</i> (gms)	PET (gms)
1	UNAS	-	40	40	20
2	CHAS	-	40	40	20
3	ENAS	-	40	40	20
4	UNAAB	40	-	40	20
5	CHAAB	40	-	40	20
6	ENAAB	40	-	40	20
7	UNABS	40	40	-	20
8	CHABS	40	40	-	20
9	ENABS	40	40	-	20
10	UNAABS	26.6	26.6	26.6	20
11	CHAABS	26.6	26.6	26.6	20
12	ENAABS	26.6	26.6	26.6	20

**Carding** is a crucial mechanical procedure that begins with fibre bundles. The fibres are spread open and sent to the card, where a carding machine, which consists of a rotating sequence of card wire-covered drums, combs them into a web. The type of fibre used and the desired base weight determine the exact card layout. The majority of the fibres can be laid in the machine direction when the web is parallel-laid. In the machine direction, well-parallel laid carded webs have strong strength, low elongation, and low tear strength; in the cross direction. The carded laps were taken and laid on the needle punching machine. These nonwovens are created by repeatedly penetrating the fibrous web with a set of barbed needles to create coherent and self-locking materials (Tejyan *et al.*, 2011).

The punching of the web was done with barbed needles. The number of punches done was 1000 per minute and the speed rate was 10 mm per stroke. About 40 grams of each selected fibre along with 20 grams of PET were blended together, and the web was formed. The webs were lapped one over the other using three layers, and these were needle punched using the Trytex miniature nonwoven system lab model needle punching machine was used. It comprised of 60 mm downward stroke and 1310 needles with a capacity to produce 500 mm width fabric. The fabric was given needle punching on both sides to get compactness. Each time 600 mm x 300 mm fabric was produced (Plates 4 a–4d).



Plate 4a  
Weighing Fibres



Plate 4b  
Blending of Fibres



Plate 4c  
Carding of Fibres



Plate 4d  
Needle Punching



Plate 4e  
Thermal Bonding



Plate 4f  
Prepared Samples

#### Plate 4 Nonwoven Fabrication

##### 3.3.3 Thermal Bonding

The needle punched fabrics for twelve fabric samples were placed in thermal bonding machine individually. The fabrics were placed on the Teflon sheets and passed through the heat roller. Heat 260°C was applied for melting PET and to obtain better bonding of the fibres. The blended, carded and needle punched structures were thus hot

calendared and cooled for bonding. The low-melt PET binder component and the Teflon sheets assisted in perfect bonding. For thermal bonding Trytex Koriyan thermal bonding lab model machine was used. The speed of the machine was two rpm. The fabric dimension of 600 mm x 300mm was produced individually for each specimen (Plates 4e and 4f). Thus, twelve thermal bonded fabric samples, namely UNAS, ENAS, CHAS, UNAAB, ENAAB, CHAAB, UNABS, ENABS, CHABS, UNAABS, ENAABS and CHAABS were produced for the study.

### **3.3.4 Evaluation of Nonwoven Structures**

All the nonwoven structures were evaluated for their essential properties by standard methods after preconditioning as per ASTM D 1776. The methodology followed for carrying out the tests is vividly explained under the following headings:

#### **3.3.4.1 Thickness**

Thickness plays an important role in different potential applications. As per ASTM D 5729, the thickness of prepared samples was carried out using Analog thickness tester. The samples were placed between pressure foot of 25 mm and 10 mm. The weight of 4.14 kPa was placed on the top pan of the tester and the reading was noted. The dial gauge with a minimum count of 0.01 mm and a maximum displacement of 1.5 mm was attached to the thickness tester. Thus, the thickness was assessed for all the samples (Plate 5a).

#### **3.3.4.2 Weight**

The Fabric weight is expressed in grams per meter square (GSM). The fabric weight was determined according to the standard ASTM D 3776. The GSM cutter and electronic weighing balance were used to measure the weight of the prepared samples (Plate 5b).

#### **3.3.4.3 Bulk Density**

The fabric bulk density was determined from the fabric weight per unit area and thickness. The bulk density of fabric increases with an increase in punch density because bonding caused by needling leads to higher compactness and lower thickness. (Rakshit *et al.*, 1990) The bulk density of the nonwoven samples was calculated using the following formula.

$$\text{Bulk density g/cc} = \frac{\text{Area density (g/cm}^2\text{)}}{\text{Thickness (cm)}}$$



Plate 5a Thickness Tester



Plate 5b GSM Cutter



Plate 5c Tensile Strength Tester



Plate 5d Abrasion Resistance Tester



Plate 5e Air Permeability Tester



Plate 5f Spray Tester

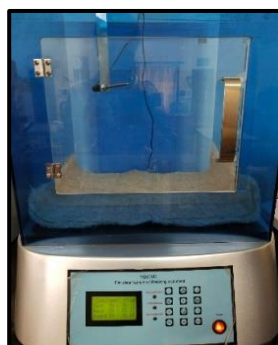


Plate 5g Thermal Conductivity Tester



Plate 5h Acoustic Tester

Plate 5 Evaluation of Nonwoven Fabric Samples

#### **3.3.4.4 Tensile Strength and Elongation**

The conditioned fabric samples were subjected to tensile strength test using the cut strip method, which assures the breaking force and elongation of nonwoven fabrics as per ISO 9073-3. The MAG unistretch equipment was used with the test speed of 100mm/min and a gauge length of 200mm. The specimen was cut with dimensions of  $25 \pm 1$  mm wide and  $150 \text{ mm} \pm 5$  mm long with the length parallel to the direction of testing and force application (Plate 5c). The data obtained for five measurements in each specimen was recorded. Thus, breaking force and elongation at maximum stress and time taken were obtained for all the prepared nonwoven structures.

#### **3.3.4.5 Abrasion Resistance**

Abrasion was carried out using geotextile abrasion tester to determine the abrasion resistance of nonwoven materials as per ISO 13427. The machine is comprised of a lower plate and an upper plate with dimensions of 50 mm x 200 mm, the stroke length of  $25 \pm 1$  mm and speed of ninety cycles per minute at test cycles of 750. It is power driven and the sample size taken was  $50 \times 300 \pm 1$  mm and the pressure imparted was  $6 \pm 0.01$  kg (Plate 5d). The abrasion was calculated using the formula  $100 (A-B)/A$ , (Where A is the tensile strength of the reference specimen, B is the tensile strength of the abraded specimen). This was carried out with five samples for all the prepared nonwoven samples and the resistance to abrasion was expressed as the percentage loss in tensile strength of the specimen.

#### **3.3.4.6 Moisture Regain**

Moisture regain of all the nonwoven samples was carried out as per standard ASTM D 5229. The same method used for fibre in 3.2.4.4 was followed (Appendix IVb)

#### **3.3.4.7 Air Permeability**

Air permeability indicates the breathability of fabrics. The specimens were preconditioned as per ASTM D 1776 and the test was carried out using the ASTM D 737-80 method in the testing laboratory at Bharathiar University, Coimbatore. The test was carried out by placing the sample on the test head with adequate tension in the fabric. The air was forced through the fabric by a suction fan, and the pressure was adjusted until there was an air pressure drop of one centimetre or 10 mm WG of water head across the fabric. This was achieved by opening the airflow of the rotometers and adjusting the flow rates. It is the volume (cm) of air passing through  $1 \text{ cm}^2$  of fabric per second at a pressure difference of 1 cm head of water. The sum of all the rotometer values indicated the rate of air flow in litres

per hour (Plate 5e). The test was carried out for ten replications, and the average value was calculated. The air permeability was calculated using the formula.

$$\text{Air permeability } R = r \times 1000/60 \times 60 \times a$$

Where r, - Rotameter reading in liter per hour, a -Treat Area (4or10cm<sup>2</sup>)

### **3.3.4.8 Absorption Tests**

#### **Water Absorption Test**

The water absorption nonwoven fabric samples were weighed before and after immersion in deionised water. The calculation for water absorption was done using the standard formula (Yusriah *et al.*, 2014) which is presented in Appendix IVc

#### **Sinking Test**

This test was carried out as per standard procedure (Savile, 1999). About five samples were cut into 2 cm x 2 cm square from the nonwoven fabric samples. A beaker (1000 ml) was filled with distilled water, and a few drops of wetting agent were added to the distilled water. The fabric sample was dropped on the surface of the water from a fixed height. Following the standard procedure, five readings were taken to calculate the mean value. The same method was repeated for all the nonwoven specimens.

#### **Wicking Test**

The vertical wicking test was conducted as per AATCC 197. A vertically hanging piece of fabric measuring 30 cm by 2 cm had the lower edge submerged in a reservoir of distilled water. The capillary rise of the dyed water contained in beaker was noted for a stipulated time for all the samples.

#### **Spray Test**

As per ISO 4920, the conditioned fabric was spread over an embroidery hoop at an angle of 45° without wrinkles, with 200 mm below a burette positioned to deliver 250 ml of water sprayed at a rate of 25 seconds. A drop of distilled water was allowed to fall on the cloth. The stopwatch was started immediately, and the time required for the drop of water to lose its spectacular reflectance and appear as a dull wet spot was noted in seconds. The average of five readings was taken (Plate 5f).

### 3.3.4.9 Thermal Conductivity

Thermal conductivity test was carried out as per standard ASTM D 1518 using the tester YG6061 (Plate 5g). It is an automatic instrument for measuring, calculating, and directly displaying the insulation rate, heat transfer coefficient, and CLO value on an LCD screen. It has microcomputer-controlled data processors and quality temperature sensors. The temperature range was adjusted between 20-50°C, the preconditioned sample of 250 mm x 250 mm was placed and the test was conducted. This was repeated for all the nonwoven specimens and the results obtained for thermal conductivity were recorded.

### 3.3.4.10 Thermogravimetric Analysis (TGA)

Understanding the considerable thermal behavior of natural fibres at high temperatures of 220°C for at least a few minutes is crucial because thermogravimetric analysis (TGA), which bases its findings on mass change, defines the thermal stability of natural fibres throughout the heating process. (Kiziltas *et al.*, 2016). Using the tester 'EXSTAR/6300 TGA' the thermal stability of non-woven fabrics was analyzed. Fabric sample weighing 6 to 7 mg was heated between 50 and 450°C in a nitrogen gas atmosphere. Thermogravimetric analysis was used to investigate the fabric thermal resistance and weight loss when exposed to higher temperatures. This was repeated for all nonwoven Structures.

### 3.3.4.11 Scanning Electron Microscope (SEM)

The morphological analysis was done using the "TESCAN-MIRA3 XMU scanning electron microscope" by obtaining images of the fabric surface at various magnification to assess the fibre interlocking in the nonwoven fabric structure.

### 3.3.4.12 Acoustic Test - Sound Absorption Coefficient (SAC)

The acoustic properties of the prepared nonwoven samples were measured using an impedance tube as per ASTM E 1050 (ASTM standard E1050-12 - 2008) for the frequency ranges from 220 Hz to 4000 Hz. All the nonwoven samples were cut into circular sections with a diameter of 33 mm (Plate 5h)

$$(K_0) = \frac{2\pi f}{C_0}$$

where  $K_0$  the wave number,  $f$  is the frequency and  $C_0$  is the speed of sound

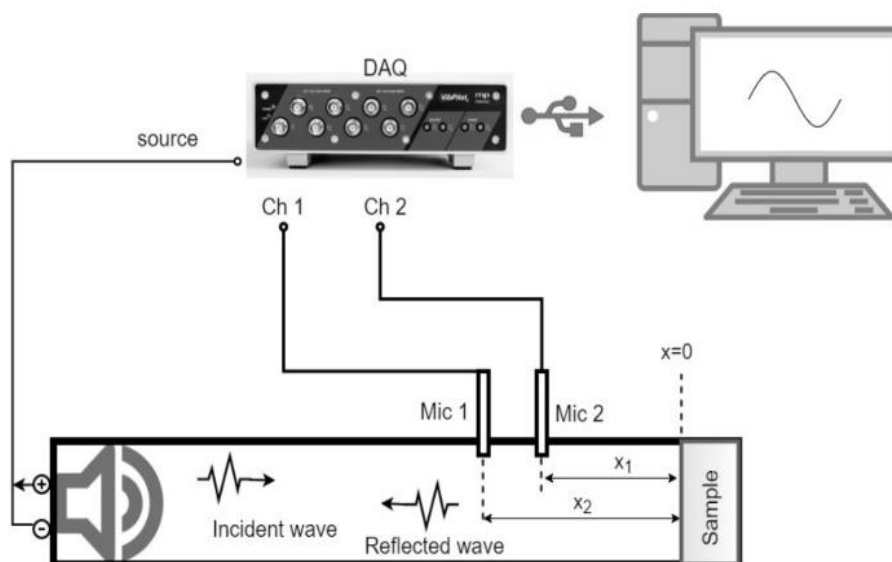
$$(H_{12}) = \frac{p_2}{p_1} = \frac{e^{jk \frac{x_2}{2}} + r e^{-jk \frac{x_2}{2}}}{e^{jk \frac{x_1}{2}} + r e^{-jk \frac{x_1}{2}}}$$

where  $H_{12}$  - acoustic transfer function,  $p_1$  and  $p_2$  - measured acoustic pressure of the two microphones,  $x_1$  and  $x_2$  - the distances between the reference plane (the sample position

of  $x = 0$ ) and the two microphones (Figure 2). Thus, the sound absorption coefficient ( $\alpha$ ) was calculated using the formula:

$$R = \frac{e^{-jks} - H_{12} x e^{2jk(l+s)}}{H_{12} - e^{jks}}$$

$$(\alpha) = 1 - |R|^2$$



(Source: Yuvaraj L *et al.*, 2021)

**Figure 2 Schematic Diagram of Impedance Tube**

### 3.3.4.13 Correlation Between Thickness and Acoustic Property of Nonwovens

A correlation in statistics is a relationship between two variables that is predictive. One may determine the type, strength, and direction of the relationship between two variables using this dependence measure (Kumar and Gautham 2020). This was used to find and express the association between thickness and acoustic properties between the prepared nonwoven structures. The results obtained are presented in Chapter 4. From Phase III, based on the properties exhibited by nonwoven samples and those which gave favourable results were selected for further study.

### 3.4 Phase IV – Fabrication of Composite Structures

In phase IV the fabrication of composite structures was done using the selected nonwoven fabrics, for which the materials, methods used, and steps involved are presented under the following headings.

### 3.4.1 Nomenclature of the Composite Structures

The designation used for the composite samples and the details are presented in Table IV.

**Table IV**  
**The Nomenclature of Composite Samples**

S.No	Samples	Details	Treatment
1	EP	Neat Epoxy	-
2	CAABE	Chemical treated <i>Areca catechu</i> + <i>Abutilon indicum</i> fibres	<i>Areca catechu</i> and <i>Abutilon indicum</i> – Alkalization
3	CABSE	Chemical treated <i>Abutilon indicum</i> + <i>Agave americana</i> fibres	<i>Abutilon indicum</i> – Alkalization <i>Agave americana</i> – Benzoylation
4	CASE	Chemical treated <i>Areca catechu</i> + <i>Agave americana</i> fibres	<i>Areca catechu</i> – Alkalization, <i>Agave americana</i> – Benzoylation
5	EAABSE	Enzyme treated <i>Areca catechu</i> + <i>Abutilon indicum</i> + <i>Agave americana</i> fibres	Pectinase enzyme

### 3.4.2. Selection of Reinforcement

Natural fibre has gained increased attention as a glass substitute over the past ten years due to the potential benefits of natural fibre reinforced composites (Todor *et al.*, 2018a). The treated and selected *Abutilon indicum*, *Agave americana* and *Areca catechu* fibres which were converted into nonwoven fabrics and those which gave favourable results with respect to strength, were utilized for conversion into composite structures. These nonwoven thermal bonded fabrics namely CHABS, CHAS, CHAAB and ENAABS were used as reinforcements. These nonwoven fabrics were cut to dimensions of 150 mm X 150 mm for composite making and the composite samples were designated as CAABE, CABSE, CASE, and EAABSE.

### 3.4.3. Selection of Polymer Matrix

The thermoset polymers are highly cross-linked polymers with great strength and modulus (Mohammed, *et al.*, 2015). In the marine sector, epoxy resin is renowned for its

extraordinary durability and strength for bonding. It can flex and strain more with the fibres without microfracturing, and it has good water resistance. This resin bonds well to all sorts of fibres and gives fibres excellent results in repair ability when it is used to bond two different materials together (Guduru, *et. al.*, 2016). Epoxy resin is the traditional type, produced through polymerization and utilized as a thermoset polymer for composites and adhesives. It is smooth, watertight, and two times stronger than concrete. (Ray, *et.al.*, 2016). Considering the predominant properties of the epoxy resin, it was selected and purchased for the study. "Epoxy LY556" is a clear pale-yellow transparent liquid with a density between 1.15 and 1.20 gm/cm<sup>3</sup> at 25° C. "Epoxy resin LY556" and suitable "hardener HY951" which were purchased from Covai Seenu Company, Coimbatore, were utilized for the study (Plate 6a).

#### **3.4.4. Fabrication of Composite Slabs**

The fabrication of composite slabs consists of the following steps.

##### **3.4.4.1 Preparation of Mould**

To prepare the composite structure, first the mould of dimension 150×150 mm was taken and sprayed with silicone free spray, which is mould releasing agent. Then a thin layer of aluminium foil sheet of the same dimension was marked, cut, and spread as the base and top layers of the composite, hence forming the 3 mm depth. The silicone-free spray was sprayed in order to avoid the sticking of the composite material to the surface of the mould and to get the perfect structure of the composite (Plate 6b).

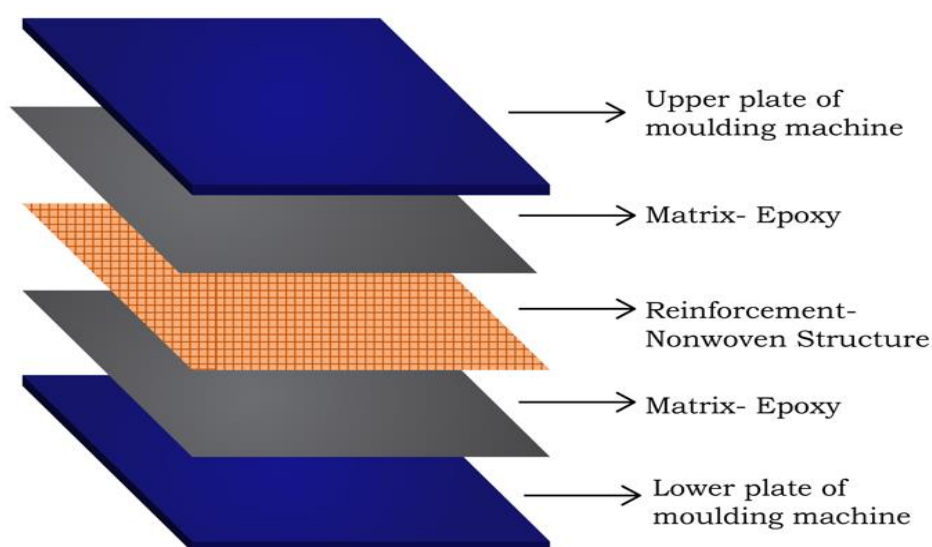
##### **3.4.4.2 Neat Epoxy Slab**

The Epoxy Resin LY556 and the hardener HY951 were mixed in a ratio of 10:1. (Gujjala *et al.*,2014). For easy removal of the composite from the iron mould, the aluminium foil sheet of 150×150 mm was used as the releasing sheet on which the silicone spray was sprayed. Using the stirrer, the resin was thoroughly mixed with the hardener and poured on the sheet evenly without any formation of voids for a depth of 3 mm. Then the aluminium foil sheet of 150×150 mm was placed over the poured resin. This process was carried out at room temperature and this setup was left for about twenty-four hours for complete curing. Thus, a neat epoxy slab was prepared. (Plate 6c)

##### **3.4.4.3 Development of Fabric Reinforced Composite Slabs**

To prepare the composite, the Unipolymer Compression moulding machine with Hydraulic pressure was used. The binding material called epoxy resin and the reinforcement

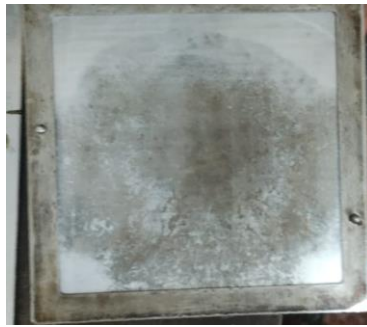
material, which is the thermal bonded material were the two materials used to form the nonwoven fabric reinforced polymer composite slab. The "Epoxy Resin LY556" and the "hardener" were mixed in a ratio of 10:1. For easy removal of the composite from the iron mould, an aluminium foil sheet of 15×15 mm was used as the releasing agent silicone spray was sprayed. Using the stirrer, the resin was thoroughly mixed with hardener, poured and spread evenly on the sheet without any formation of voids into the mould of depth 3 mm the dimension being 150×150 mm. The thermal bonded material of 150×150 mm was weighed and placed over the resin. Another layer of epoxy resin was poured evenly on the surface of the thermal bonded material using the stirrer tool. The aluminium foil sheet was placed followed by the layer of poured resin. The top plate of the mould was placed over the aluminium sheet and closed tightly. This process was carried out at the temperature of 130°C and pressure of 30kg/cm<sup>2</sup>. This was kept without disturbance for about one hour for complete curing until the temperature reduced from 130 °C to room temperature. Then this setup was left for 24 hours at room temperature for complete curing. This procedure was carried out for all the fabric reinforced composite slabs in the ratio 70:30 of resin and fabric. Thus, the composite samples, namely CABSE, CASE, CAABE and EAABSE were prepared. (Plate 6d-6l). The schematic diagram for the preparation of composite samples is presented in Figure 3.



**Figure 3 Schematic Diagram for Composite Preparation**



**Plate 6a**  
Resin and Hardener



**Plate 6b**  
Mould



**Plate 6c**  
Neat Epoxy



**Plate 6d**  
Weighing reinforcement



**Plate 6e**  
Mixing of Resin and Hardener



**Plate 6f**  
Pouring resin on  
reinforcement fabric



**Plate 6g**  
Compression Moulding  
Machine



**Plate 6h**  
Weighing Composite Sample



**Plate 6i**  
Sample CAABE



**Plate 6j**  
Sample CABSE



**Plate 6k**  
Sample CASE



**Plate 6l**  
Sample EAABSE

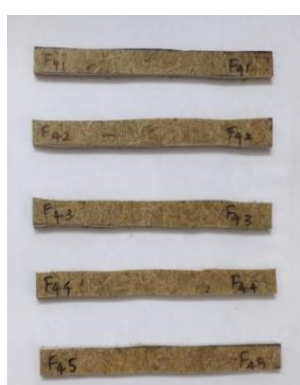
**Plate 6a-l Preparation of Composite Samples**

### 3.4.5 Machining of the Composite Structures

The machine used in this process was a semi-electric machine, in which the sample to be cut was placed beneath the machine's zigzag-shaped blade, and using the operator's assistance, the samples were cut into the desired shape by tilting the sample in accordance with the line that was marked on the surface of the composite structure. The sample was labeled in accordance with the required standard before the slabs were machined. For the tensile strength test, the dimensions of the ASTM D 3039 standard were followed. By using oil as a lubricant, cutting samples were done with minimal friction. For the flexural strength, a dimension of 125×13×3mm was cut in a rectangular shape according to ASTM D 790. In accordance with the ASTM D 3410 standard, the sample was marked with dimensions of 150×25×3mm, yielding a rectangular shape for the compression test. Similar markings were made for the impact test, a rectangular-shaped sample 65×13×13 mm to meet the standard ASTM D 256, for hardness ASTM D 2240 with dimensions 30 ×125 ×3 mm. The dimensions of 20×20×3 mm in accordance with the ASTM D 5229 standard were cut for the water absorption test. All the prepared composite slab specimens were marked as per standards clearly using marker pen and cutting was carried out neatly. Thus, fine specimens were cut from each specimen, namely EP (ASTM D 638 Tensile strength) CAABE, CABSE, CASE, and EAABSE for evaluation. (Plates 7a-d)



**Plate 7a**  
Tensile Strength



**Plate 7b**  
Flexural Strength



**Plate 7c**  
Impact Strength



**Plate 7d**  
Compression

**Plate 7 Machined Specimen of Composites**

### **3.5 Evaluation of Prepared Composite Structures**

The process of analyzing the mechanical properties of natural fibre polymer composites is of paramount importance. The mechanical properties of fibre reinforced composites greatly depend on the polymer matrix, fibre distribution, fibre orientation, and fibre-matrix interfaces (Athijayamani *et al.*, 2009). Hence, the prepared composite structures were characterized for mechanical properties, thermal behavior, liquid absorbency, presence of chemical components, and morphology by subjecting the samples to various pertinent tests. The Computerized "Universal Testing Machine (UTM)" was used for testing all the samples. The features of the machine are as explained: Loading Frame - Powder coated with compact footprint Floor Mounted; Testing area; interchangeable load cells; Software - Kalpak's Windows based software; Load Capacity-100KN; Maximum speed-200 mm/min; Drive Mechanism- Ball Screw driven by variable speed at servo drive (Ramakrishnan and Sampath, 2017). All the prepared composite samples were transmitted to evaluate the essential mechanical properties namely Tensile strength, Flexural strength, and Compression using this machine and changing the settings as per the standards required.

#### **3.5.1 Tensile Strength and Elongation**

The capacity of the material to resist breaking under stress is an important property to analyze for structural applications. The force per unit area required to break a material is the ultimate tensile strength at break (Johnson *et al.*, 2018). Tensile strength and elongation of composite slabs were investigated according to ASTM D 3039 standards. The test was done using Universal Testing Machine. The machine worked at the speed rate of two millimeters per minute with a gauge length of 50 millimeters and an average peak load of 3526.075 N. Five specimens were tested for each composite slab, namely CAABE, CABSE, CASE, and EAABSE and the average results were obtained for tensile strength and elongation (Plate 8a).

#### **3.5.2 Flexural Strength**

Flexural strength, is also referred to as rupture modulus, bending strength, or fracture strength. It is a mechanical characteristic of brittle materials that is characterized as a substance's capacity to withstand deformation under force. The Flexural Strength Test for all the composite samples was conducted on the same machine UTM. According to the standard ASTM D790-03, with dimensions of 125×13×3 mm was cut. The peak load,

flexural strength (MPa) and flexural Modulus (GPa) were noted for five specimens for each composite at the rate speed of 2 millimeter per minute with a gauge length of 50 mm with 3410. About 130 N peak load and the average values were obtained. The average flexural characteristics were determined for five successful tests in each specimen for the composite slabs and the average was calculated and recorded. (Plate 8b)

### **3.5.3 Compression**

According to the test method ASTM D 3410, the compression test was carried out on a universal testing machine at a cross sectional area of  $112.5 \text{ mm}^2$  at a speed of 2 mm/min with a gauge length of 50 mm. The machined specimen of dimension  $150 \times 25 \times 3$  mm was taken. The strength of a material under applied crushing loads was assessed using compression test machines (CTM), which are typically used to impart compressive pressure to a test specimen. The cross sectional area ( $\text{mm}^2$ ), peak load (N), and compressive strength ( $\text{N}/\text{mm}^2$ ) were noted, and the average values were calculated (Naveen *et al.*, 2015). It was repeated for all the composite samples, with five specimens in each sample (Plate 8c).

### **3.5.4 Shore D Hardness**

As per ASTM D 2240, a Rockwell hardness tester was used to determine the hardness of the composite materials. The Shore D durometer is a device for measuring the hardness of materials, including polymers, elastomers, and rubbers. Higher numbers on the scale denote tougher materials with better indentation resistance and lower values indicate less resistance and softer materials. The hardness or indentation hardness after a certain amount of time was measured with a durometer. The basic test requires consistently applying the force, without shock, and measuring the hardness (depth of the indentation). A plastic cutter was used to prepare the composite specimens, which had dimensions of 30 mm x 12.5 mm x 3mm (Plate 8d) Five such specimens in each composite slab sample were tested and the average value was calculated.

### **3.5.5 Impact Strength**

The impact strength test was conducted as per ASTM D 256-05 with a specimen size of  $65 \times 13 \times 3$  mm. Impact strength is the material's capacity to withstand a sudden application of load. In this test, the amount of kinetic energy required to start the fracture and maintain it until the specimen broke was measured. With the aid of grippers, the test object was held vertically while the pendulum was blasted from one side, striking it

vigorously. (Plate 8e). This procedure was repeated for all the composite slabs and an average of five samples were taken and recorded.



Plate 8a Tensile Strength Tester



Plate 8b Flexural Strength Tester



Plate 8c Compression Tester



Plate 8d Hardness Tester



Plate 8e Impact Tester



Plate 8f Liquid Absorption Test



Plate 8g Soil Burial Test

**Plate 8 Evaluation of Composites**

### **3.5.6 Spectrophotometric Analysis**

Brightness is the measurement of the light reflectance of a specific wavelength of blue light (Choudhury 2006). The description of the spectrophotometric analysis followed for composites was the same method used for the fibre analysis explained in 3.2.4.2. All three aspects namely whiteness, yellowness and brightness indices were observed for the prepared composite samples, namely CAABE, CABSE, CASE and EAABSE.

### **3.5.7 Thermogravimetric Analysis (TGA)**

Thermal characterization of composite samples was carried out using TG/ DTA EXSTAR 6300 in Alumina pan in a "Nitrogen gas" environment with a heating rate of 20° C/min. The TGA measures weight changes in a material as a function of temperature (or time) under a controlled atmosphere. Each of the six samples, weighing 6 grams was placed in the chamber. The analysis was done with a temperature range of 50 to 600 °C, and a heating rate of 10 ° C. For situations involving temperatures above ambient, such as fire damage, curing, or processes involving heating, the thermal behavior is also of practical interest. Different thermal reactions are evaluated for a number of works, especially in terms of the thermogravimetric properties of natural fibre polymer composites (Monteiro *et al.* 2012). The analysis measures the quantity of rapid mass degradation in a composite. The weight loss due to degradation of the samples namely CABSE, CASE, CAABE and EAABSE was observed and recorded.

### **3.5.8 Density and Volume Fraction of Voids**

The results obtained for density and volume fraction of voids are explained under the following headings

#### **3.5.8.1 Density of Composites**

The density of all the prepared composite samples was determined as per the standard ASTM D 792 with dimensions of 20 x 20 x 3 mm using the water displacement method applying Archimedes Principle. The specimen was immersed in the deionized water for weighing after knowing the weight of composites in air, the weight was observed using an electronic weighing balance before and after immersing in water. The mass (m) and volume (v) were assessed, and the density was obtained by using the formula, Density =  $r = \text{mass/volume}$ , which was expressed in  $\text{g/cm}^3$  (Pandita *et al.* , 2013).

### 3.5.8.2 Volume Fraction of Voids

The volume fraction of voids was obtained by calculating various parameters using the following formulae.

$$\text{Fibre weight fraction } wf = \frac{wf}{wc} \frac{\text{Mass of fibres}}{\text{Mass of composite}}$$

$$\text{Fibre volume fraction} = vf = Vm = (1-wf) \frac{\rho_c}{\rho_m}$$

(Volume fraction of matrix in composite)  $vf = 1 - Vm$

$$\frac{\text{Density of composite} \times \text{Fibre weight fraction}}{\text{Density of fibres}}$$

Density of composite was calculated using the formula

#### Density of Composite

$$= \frac{\text{(weight of fibres} \times \text{Density of fibre} + \text{weight of resin} \times \text{Density of resin)}}{\text{Weight of composite}}$$

Volume fraction of voids

$$V_v = \frac{\rho_c - \rho}{\rho_c}$$

where  $V_v$  = volume fraction of voids,  $\rho_c$  = theoretical density,  $\rho$  = experimental density

The density of the respective treated fibres considered were taken from the fibre density Table in Phase I

### 3.5.9 Liquid Absorbency Test

Samples of nonwoven fabric reinforced composite specimens, namely CABSE, CASE, CAABE and EAABSE of dimensions 20x20x3 mm were used for the liquid absorption test according to ASTM D 5229. The absorbency was assessed after the first, second, third, fourth, fifth, and sixth days of immersion in three different liquids, namely ionized water, deionized water, and kerosene. After drying in an oven at 105<sup>o</sup>C for twenty-four hours, the samples were weighed periodically before and after immersion in liquid (Korla., 2010). The specimen was immediately put back into the respective liquids

(Chakrabarty *et al.*, 2012). (Plate 8f). Thus, after the specified periods of immersion of the samples, the mass was calculated as per the formula:

$$\text{Water absorption (\%)} = \frac{(\text{Final weight} - \text{Initial weight}) \times 100}{\text{Initial weight}}$$

where final weight is the weight of the sample measured after liquid immersion and initial weight is the weight of the dry sample.

### **3.5.10 Scanning Electron Microscopic Appearance (SEM)**

The morphology of the composite was analyzed using a scanning electron microscope (FESEM - TESCAN -MIRA3 XMU. This was carried out at different magnifications to determine the bonding and compatibility between the reinforcement and the matrix. This was analyzed for all the prepared composite slabs, namely CABSE, CASE, CAABE and EAABSE.

### **3.5.11 Fourier Transform Infrared Spectroscopic Analysis (FTIR)**

The "Shimadzu FTIR 8400s machine equipped with MIRACLE 10 (ZnSe)" was used to obtain an infrared spectrum of absorbed emissions. In order to produce light across a large range of infrared wavelengths, a continuum source of light was used. After going through an interferometer, the infrared light is then focused on the sample. Each sample was scanned 45 times with a resolution of 16 cm<sup>-1</sup>. FTIR assisted in determining the change in functional groups due to the incorporation of the various fibres in the matrix. This was carried out for all the prepared composite slabs, namely CABSE, CASE, CAABE and EAABSE.

### **3.5.12 Soil Burial Test**

Biodegradation is the decomposition of organic materials by the action of bacteria and fungi or by other biological means. This process leads to the decomposition of material in the environment. The completed biodegradation process is called mineralization (Tahri *et al.*, 2013). The standard procedure followed for soil burial test is presented in Appendix V. The biodegradation test was performed following the method adopted by earlier study (Jumaidin *et al.*, 2020). This was carried out for all the composite samples namely CAABE, CABSE, CASE and EAABSE (Plate 8g)

### **3.5.12.1 Weight Loss**

The quantitative method for testing the biodegradation of a polymer composite is to determine the mass loss of the material (Hermann *et al.*, 1998). The soil buried samples namely EP, CASE, CAABE, EAABSE and CABSE were analysed for weight loss. Three replicates of each sample were buried in soil. At the end of the testing period, namely 20, 40, and 60 days, each of the buried composite samples were removed, cleaned, washed, and dried for 48 hours before being weighed. The change in weight was calculated using the equation "Weight loss (%) =  $(w_i - w_t) / w_i \times 100$ ." where  $w_i$  is the initial weight of the sample and  $w_t$  is the weight of the sample after the established time.

### **3.5.12.2 Degradability**

The degradability of the materials was calculated using the mass loss and time taken for degradation for each sample. The degradability of a material is determined from the rate of degraded mass at a certain time duration ( $\Delta t$ ) using the following equation (Mahyudin *et al.*, 2020).

$$\text{Degradability} = \text{Mass loss} / \text{Time duration}(\Delta t)$$

### **3.5.12.3 Visual Evaluation**

The soil buried samples, namely EP, CASE, CAABE, EAABSE and CABSE were visually analyzed by the judges, numbering twenty-five, who were requested to evaluate the soil buried samples with the unburied samples for colour, texture, and general appearance. The proforma prepared without disclosing the days of burial was distributed to the judges for obtaining the response. (Appendix VI)

### **3.5.13 Comparison Between the Mechanical Properties of Composite Structures**

All the prepared composite structures that were subjected to various tests were analyzed using a radar chart as a confirmation for deciding about the selection of composite structures to be utilized for further study. For this purpose, normalization of the readings obtained for different mechanical properties was done considering the standard deviation values of the six mechanical properties namely impact, compression, flexural, tensile strength, elongation, and hardness.

From the test results and analysis, the samples that gave satisfactory results were selected for further evaluation in the phase IV.

### **3.6 Phase V Evaluation of Selected Composites**

Based on the test results and confirmation with the radar chart, the two specimens that gave favorable results were selected for further analysis, along with neat epoxy sample for comparison.

#### **3.6.1 Acoustic Test**

The acoustic test is the process by which the acoustic characteristics of materials are determined in terms of absorption and transmission loss, and the procedure followed is explained under the following headings:

##### **3.6.1.1 Sound Transmission Loss**

The sound transmission loss of the prepared composites was measured using the impedance tube setup as per ASTM E2611-17 as shown in Figure 3. The test was carried out for the selected composite samples CASE, CABSE and neat epoxy at frequencies ranging from 100 to 2000 Hz, which were compared.

##### **3.6.1.2 Sound Absorption Coefficient**

Sound Absorption Coefficient (SAC) is defined as the fraction of randomly incident sound energy that is absorbed by the surface (Shiny and Premlet 2014). The absorption coefficients of the selected samples, namely EP, CASE and CABSE for frequencies ranging from 200 to 4000 Hz were analyzed and compared.

##### **3.6.1.3 Comparison between Nonwoven Fabric and Composites**

The alteration in the acoustic properties caused by the polymer coating in the nonwoven fabric reinforced rigid structures was compared with the nonwoven flexible material. The results obtained in terms of acoustic properties for both materials are repeated in the Table LXIII in Chapter 4 for comparison.

##### **3.6.1.4 Noise Reduction Coefficient (NRC)**

Noise Reduction Coefficient (NRC) is the ability of a surface to absorb sound, allowing incoming sound energy to be absorbed or not reflected by the surface. The formula for calculating the noise reduction coefficient is expressed in the following equation (Kaamin *et al.*, 2020).

$$\text{Noise Reduction Coefficient} = \frac{\alpha_{500} + \alpha_{1000} + \alpha_{2000} + \alpha_{4000}}{4}$$

4

This was followed for calculating NRC for the samples EP, CHAS, CHABS, CASE and CABSE.

### **3.6.2 Roughness**

The roughness test was carried out only for the selected composite slabs, namely CASE and CABSE and was compared with a neat epoxy slab. A laser profilometer was used to analyze the surface roughness of composite structures (Plate 9). Surface profiles and pores were studied using a Zeta-20 3D Optical Profiler as per standard procedure. This was carried out for all the samples, namely EP, CASE and CABSE (Plate 9).

### **3.6.3 Drill Test**

The quality of a hole is very important in the fabrication process. During the machining process, there are chances of cracks and damage. In industries like automotive or aerospace, for the fastening process or the riveting process of structural components and assemblies, drilling is done using a twist drill (Vinay *et al.*, 2020). Among different machining processes, drilling is the most useful process for assembling various pieces. Composites will experience a delamination process throughout the drilling operation, which will impair their strength and effectiveness (Patel *et al.*, 2017). The driller used was a Saw Classic Master SWC-FF-BSH-13RE. It works at 220V- 50HZ/780 W and 2800 rotations per minute.

**The drill bit** High-speed Steel (HSS) with twists was used for the study (Plates 10a–c). This drill bit is a steel tool used for drilling hard materials with varying drill bits, namely 5 mm, 4 mm and 3 mm which were used for drilling all the composite samples. Three holes were drilled using each drill bit for each specimen. The aspects noted during the drilling process were damage occurring at the entrance and exit points of the composite samples, time taken for penetration, fibre pullout, hole damage, and the perfection of the hole drilled (Plate 10a – 10c).

### **3.6.4 Flammability Test**

Flammability of natural fibre reinforced composites is to be considered an important factor because the flaming properties of polymers and natural fibres are quite high when compared to other materials (Vishnu *et al.*, 2016). The specimen size of 300 mm × 76 mm was cut and tested as per standard procedure ASTM 6413. The flammability test was done for all the prepared composite samples EP, CASE and CABSE for measuring ignition and

flame spread. The sample was vertically placed and subjected to a small igniting flame, which was applied at the bottom edges, with a flaming time of 12 seconds and a flame height of 40 mm. The parameters recorded were burning time, afterglow time, and concurrency of melting and dripping (Plate 11).



**Plate 9 3D Profilometer**



**Plate 10a 2 mm**



**Plate 10b 3 mm**



**Plate 10c 4 mm**

**Plate 10 Drill Bit**



**Plate 11 Flammability Tester**

### **3.6.5 Cost Estimation of Composite Slabs**

The cost of the composites was calculated taking into account a number of aspects, including raw materials – Fibre, chemicals used for treatment, resin used for fabrication and labour involved. The estimated cost is presented in Chapter 4.

### **3.6.6 Feedback of the Selected Composite Samples**

The prepared composite samples, which were assessed for mechanical properties and acoustic tests, were analyzed for their future performance by sending the material to fifty members comprising of builders and civil engineers. The various aspects assessed about the prepared composite samples were general appearance, idea about utilization of underutilized fibres, suitability for building material, future application, cost effectiveness, market potentiality in the future, environment friendliness, reason for preference of eco-friendly materials, and willingness to recommend the prepared slabs to builders (Appendix VII)

### **3.6.7 Statistical Analysis and Diagrammatical Representations**

The results obtained in the study in each phase have been analyzed statistically. In the study, various analysis methods were used, including the t-test, ANOVA, and correlation. For better expression of the data, various charts also have been used, including a bar chart, Pareto chart, and Radar chart.

The t-test, a statistical test, is used to evaluate assumptions about the population's mean from a small sample when the population's standard deviation is unknown. The t-test is performed to evaluate whether there is a significant difference between the means of two groups. To ascertain whether a process has an impact on both samples or if the groups differ from one another, it is utilized for hypothesis testing (Sapkota 2022). A statistical technique called analysis of variance (ANOVA) is used to identify variations in experimental group means. In experimental designs with numerous experimental groups within one or more independent variables and one continuous parametric numeric result measure as the dependent variable, ANOVA is appropriate (Sawyer 2009). The visual aid used in Pareto analysis is a Pareto chart, which presents the relative importance of issues in a way that is simple to understand (Leavengood and Reeb 2002). Multivariate data can be viewed using radar charts. Plotting one or more groupings of values over a number of related variables is done using them. By displaying numerous polygons, overlaying them, and decreasing the opacity of each polygon, multiple observations can be included in a single chart (Hanna and Carter 2016).