EXPERIMENTAL PROCEDURE

CHAPTER III

EXPERIMENTAL PROCEDURE

The research study is designed based on the experimental and exploratory format of woven fabric structure using cellulosic minor fibers. The main aim of the study was to develop woven fabrics with different weaves for sound resistant fabrics as front/topmost layer, for which the fibers were enzyme treated and a commercial set-up was an additional motto. The study was also intended to analyze the absorption of sound with and without the application of resin onto the woven fabrics and a sound absorption instrument was installed in the Department of Clothing and Textiles, Faculty of Family and Community Sciences, The Maharaja Sayajirao University of Baroda, Vadodara for the testing of the fabrics. Hence, this chapter deals with the materials and methods followed for fulfilling the objectives of the study.

The experimental procedure of the study has been further subdivided and discussed under various three phases:

3.1. Phase 1: Selection and analysis of raw materials

3.1.1 Procurement of the raw materials

a. Cellulosic minor fibers
b. Enzymes for softening of the fibers

3.1.2 Preliminary data of sisal and ramie fibers

a. Determination of microscopic properties
b. Determination of fiber length
c. Determination of fiber diameter

3.1.3 Fabrication of beating machine and combing tool for the fibers

3.1.4 Softening treatment on the fibers

3.1.5 Assessment of the sisal and ramie fibers

a. Bundle Strength
b. Fiber fineness
c. Whiteness Index
d. Scanning Electronic Microscopy (SEM)
e. Fourier Transform Infrared Spectroscopy (FTIR)
f. X-Ray Diffraction (EDS)
g. Energy-dispersive X-ray spectroscopy (XRD)

3.1.6 Yarn preparation

a. Hand Spinning
b. Machine Spinning

3.1.7 Assessment of the yarns

a. Determination of Yarn Count
b. Determination of Yarn strength
c. Determination of Yarn twist (TPI - Twist per Inch)

3.2. Phase II: Development of different types of fabrics

3.2.1. Preparation of woven fabrics using various weaves

a. Plain weave fabrics
b. Broken twill weave fabrics using treated yarns
c. Double cloth fabrics using treated yarns

3.2.2. Preparation of nonwoven fabrics using scoured fibers

3.2.3. Assessment of the prepared fabrics

a. Determination of fabric weight per unit area
b. Determination of fabric count (EPI & PPI)
c. Determination of fabric thickness
d. Determination of the air-permeability of sound absorbing materials.
e. Cover Factor
f. Anti-microbial and anti-fungal activity
g. Flammability Test
h. Sound absorbing coefficient (dB)
i. Noise Reduction Coefficient (NRC)

3.3. Phase III: Sound absorbing instrument, finishes and analysis

3.3.1 Fabrication of sound absorbing instrument for testing

3.3.2 Selection and application of natural resin on the woven fabrics

3.3.3 Evaluation of the resin finished fabrics

a. Determination of surface characteristics of the fabric samples using scanning electron microscope.
b. Sound Resistant (dB)
c. Noise Reduction Coefficient (NRC)

RESEARCH DESIGN:

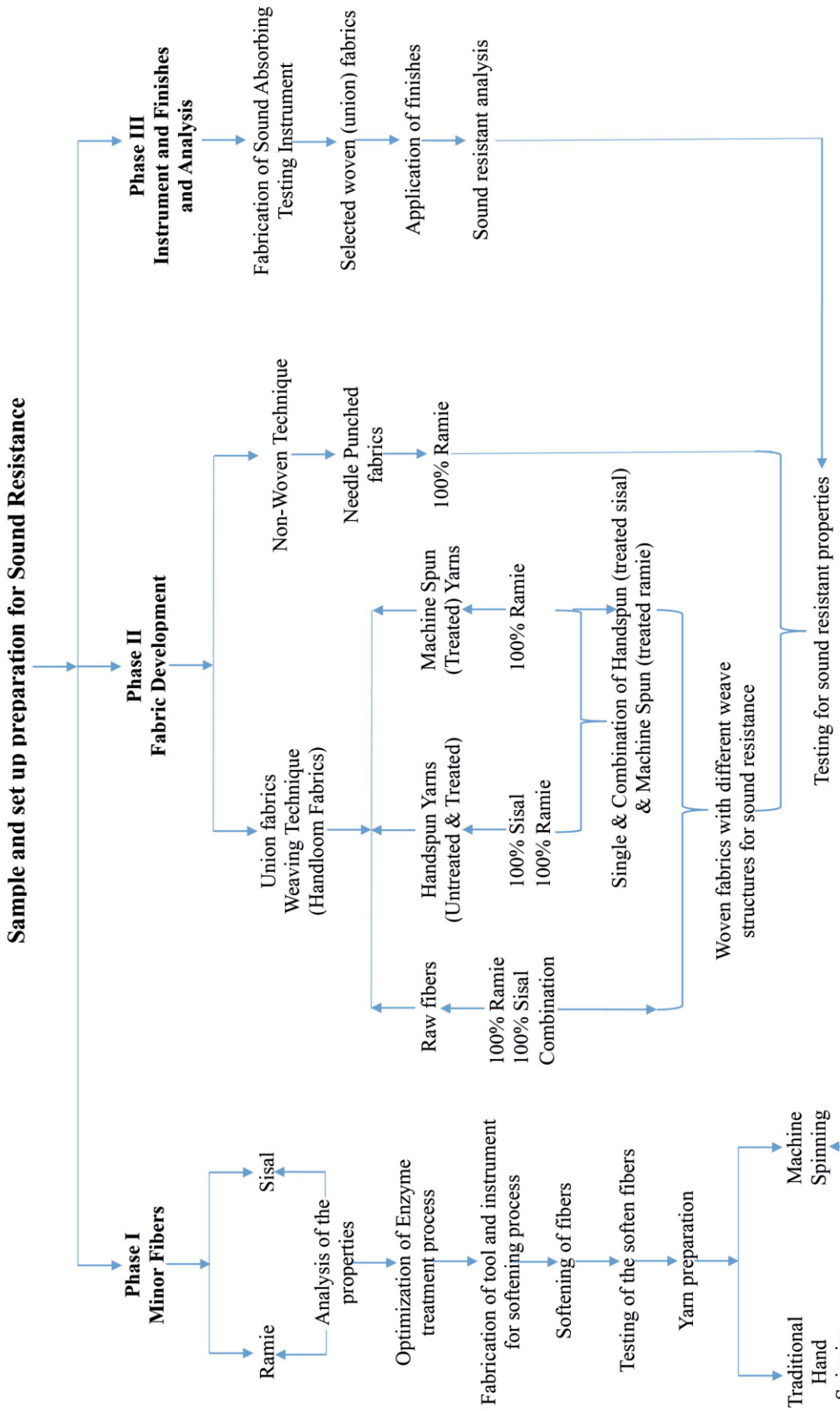


Figure 3.1: Flow chart of Research design

3.1. Phase 1: Selection and analysis of raw materials

The selection of materials was done with the aim of sustainability and acoustic application. Sisal and ramie fibers were procured from different places, further eco-friendly treatment was applied on the fibers to enhance their properties.

3.1.1 Procurement of the raw materials

Natural minor fibers utilized for the experimental and exploratory research study were based on its availability and to introduce the less explored fibers for woven sound resistant materials. Enzymes were also procured to soften the fibers thereby to increase its pliability as well as texture of the fabric as a finished product.

a. Cellulosic minor fibers

Selection of two different minor fibers were done purposively based on its availability and fiber structure for sound absorption purpose. The two selected fibers were Sisal and Ramie. Both the fibers procured were in the raw form i.e. un-degummed fibers and Table 3.1 contains all the details of the fibers.

Table 3.1: Procurement details of the cellulosic minor fibers

Sr. No.	Name of the fiber	Scientific Name	Name of the Species	Supplied by
1	Sisal	Agave Sisalana Perrine	A. Sisalana	Sisal Research Station, (CRIJAF), Barrakpore, Odissa.
2	Ramie	Boehmeria Nivea	B. Nivea	Ramie Research Station, (CRIJAF – ICAR), Sorbhog, Barpeta, Assam.

b. Enzymes for softening of the fibers

Four different enzymes for softening of the fibers were procured from different places based on its availability. Table 3.2 indicates about the procurement details.

The procured fibers were treated with different enzymes using different treatment process to enhance its properties which involves mainly combing, beating and padding mangle. The table 3.3 shows the different stages of treatment and process sequence.

Table 3.2: Procurement details of the enzymes

Sr. No.	Name of the Enzyme	Components affected	Supplied by
1	Denilite® || S	Laccase	Novazymes A/S Krogshoejvej 36, 2880 Bagsvaerd, Denmark
2	Greenboost liquid	Pectinase	Rossari biotech limited 201 a & b, Ackruti corporate park, lbs marg, kanjurmarg west, Mumbai - 400 078
3	Biocool z-20 powder	Hemicellulase	
4	G-zyme axe liquid	Cellulase	

Table 3.3: Coding of the raw and treated sisal and ramie fibers

Sr. No.	Codes	Description
1.	Sr	Sisal Raw
2.	Shc	Sisal – High per cent concentrated - combing
3.	Slc	Sisal - Low per cent concentrated - combing
4.	Shcp	Sisal - High per cent concentrated - combing - Padding
5.	Slcp	Sisal - Low per cent concentrated - combing - Padding
6.	Shcbc	Sisal - High per cent concentrated – combing - beating - combing
7.	Slcbc	Sisal - Low per cent concentrated – combing - beating - combing
8.	Shcbc4	Sisal - High per cent concentrated (4hrs treatment without changing water) – combing - beating – combing
9.	Shcbc4t3	Sisal - High per cent concentrated (4hrs treatment time) – combing - beating – combing – Launder O Meter trial
10.	Rr	Ramie Raw
11.	Rhc	Ramie - High per cent concentrated - combing
12.	Rlc	Ramie - Low per cent concentrated - combing
13.	Rhcbc	Ramie - High per cent concentrated – combing - beating – combing
14.	Rlcbc	Ramie - Low per cent concentrated – combing - beating – combing
15.	Rhcbc4	Ramie - High per cent concentrated (4hrs treatment without changing water) – combing - beating – combing
16.	Rhcbc4t3	Ramie - High per cent concentrated (4hrs treatment time) – combing - beating – combing – Launder O Meter trial

3.1.2 Preliminary data of sisal and ramie fibers

To identify the basic properties of both the fibers certain analysis was conducted, which further assisted in the study in terms of optimizing the appropriate treatment process for softening of the fibers.

a. Determination of microscopic properties

Longitudinal view of the fibers was observed under the microscope with the magnification of 15X power to identify and relate the structure of fibers with sound absorption. Further both the untreated and treated fibers were also analyzed under SEM for both longitudinal and cross section view.

b. Determination of fiber length

Length of both the fibers was identified individually with the help of steel ruler. The edge of the fiber was clipped with the ruler at one end and length analyzed on the other end. An average of 50 readings was calculated to identify the length of both the fibers.

c. Determination of fiber diameter

To determine the fiber diameter the single fiber was observed under the compound microscope with micrometer lens and an average of 20 readings was considered in µm.

3.1.3 Fabrication of beating machine and combing tool for the fibers

Beating machine and combining tool were fabricated under the study for the easy and bulk processing of the fibers. It has been installed in the Department of Clothing and Textiles, Faculty of Family and Community Sciences, The Maharaja Sayajirao University of Baroda, Vadodara. Both the fabricated machine and tool were the part of commercial set up. They are portable and less expensive, hence installation in rural areas could be done for income generation.

Fiber Beating machine

The un-degummed fibers are inclusive of impurities like pithy material, waxy material and dust. In earlier times such impurities were removed by continuous beating of the un-degummed fibers in wet condition onto the rock or hard wooden substances. Later flax and linen cleaning process were carried out by beating process, where the

beating of the fibers was constantly done between the wooden base and wooden hammer like structure to remove the impurities. Thus, the inception of basic concept came from the above mentioned processes. A handy and portable beating machine was fabricated for the easy cleaning process of the sisal and ramie fibers (Plate 3.1). The machine was such that the fibers are to be placed on the wooden base and the wooden hammer having the metal plate will continuously beat the fibers on constant speed, with the help of electric motor attached and which will maintain rotation per minute. Thus, replacing and passing of bundle of the fibers was only done manually as well as single-handedly. Another advantage was to have soften fibers due to the changes in the fiber bonds by continuous beating.

Plate 3.1: Fabricated Beating Machine

Combing tool for the fibers

Combing also known as heckling or hackling involves cleaning, splitting, separating and straightening of the filaments. Dirt, small fibers and entanglements were removed using combs (Plate 3.2). Two metal combs (No.32) were fixed adjacent to each another on the wooden block as a base having approximately 2.5 cm gap in between two metal combs. Nature of the fibers are such that if the metal combs were kept diagonally, the fiber would break and thereby wastage would be more. For further easy working, the tool can be fixed using clamps on the table. It also assisted in having aligned bundle fibers for further process.

Plate 3.2: Fabricated Combing Tool

3.1.4 Softening treatment on the fibers

Amongst the fiber's sisal being stiffer and less cohesive makes it difficult to spun into 100% sisal yarn, while bark fiber - ramie having a rough texture can be spun but, the fabric will have a rough textured. Sound resisting materials using these fibers, will be used for various applications like partition panel, wall mounting, etc. which might have little contact with the human skin. Thus, an eco-friendly treatment using enzyme will impart smooth feel, lustre and handle of the fiber. The enzymes used for the study will also modify the fiber properties that would be suitable for absorbing the sound.

The procured fiber are un-degummed fibers containing many kinds of impurities, which are essential to be removed for the equal and appropriate penetration of enzymes. Thus, scouring a pre-treatment is given to both the fibers individually that would change the natural colour of the fiber as well as it will remove the impurities.

Scouring of the fibers:

The fibers were scoured to remove various impurities like wax, pithy substances and any foreign matter. It helps in proper application and penetration of the enzymes for softening treatment (Plate 3.3). Scouring was carried out mixing 2g per liter of commercial detergent and 2g per liter of soda ash in the material liquor ratio of 1:30 at 60℃ temperature and the treatment was carried out for 45 mins. The fibers were then washed thoroughly under running water and air dried. The scouring process also helps in making the fibers lustrous with little change in the colour.

Plate 3.3: Scouring treatment

After scouring, enzyme treatment was given to increase softness and cohesiveness of the fibers and thereby to create an appropriate yarn for the end application.

Experimental set up for softening process:

Enzyme treatment is comparatively less used for the reason of its application method. Thus, study aims at standardizing enzyme treatment recipe and identifying the possible commercial set up for continuous and bulk treatment process.

Four different enzymes were used for softening treatment of the fibers, where each enzyme plays a vital role in enhancing the fiber properties. This modification makes the fibers more smooth and softer, which might be helpful in absorbing or scattering of sound passing through it.

Enzyme treatment with sisal and ramie fibers needs adequate amount of water as well as proper rotation with accurate temperature for penetration and activation of the enzymes. Thus, initially the process was carried out using the lab utensils. Initially, as a pilot work scoured sisal fibers were treated using previous recipes to understand the modification in the properties and feel of the treated fibers.

Further based on the fiber testing analysis, the recipes where been applied onto ramie fibers. Finally, the selected recipes were processed using two different laboratory machines for commercializing the entire process.

Softening treatment using utensils:

Sisal fibers were treated with four enzymes - cellulase, hemicellulase, pectinase and lacase individually and in combination, to have a smooth and soft fiber for spinning. The enzymes act and reacts with each layer of the fiber amongst which lignin is the main component that keeps the fiber stiffer. These enzymes interrelate with bonds and thereby the modification in the properties takes place by breaking or weakening the bonds.

The treatment was carried out in huge aluminum bowl (Plate 3.4), to have a wider space for fibers to be soaked completely into the solution and also for the easy rotation of fibers. Initially, previous recipe and application procedure was applied onto the fibers with variation in per cent concentration of the enzymes and treatment methods keeping M:L ratio i.e. 1:30, temperature 55℃ and 5 pH as constant. Further, the application of

modified recipe was also conducted with the above mentioned variables. The detailed recipe and treatment steps are mentioned in Table 3.4.

Plate 3.4: Softening process

Table 3.4: Stepwise enzyme treatment process of sisal fibers

Sr. No.	**Steps**	**Sr**	**Shc**	**Slc**	**Shcp**	**Slcp**	**Shcbc**	**Slcbc**	**Shcbc4**
1.	Beating	-	-	-	-	-	10mins	10mins	10mins
2.	Padding	-	-	-	3 pounds	2.5 pounds	-	-	-
3.	Combing	-	-	-	Combing of the fibers in small bundles				
4.	Pectinase	-	2%	1%	2%	1%	2%	1%	2%
5.	Laccase	-	10%	5%	10%	5%	10%	5%	10%
6.	Cellulase	-	7%	5%	7%	5%	7%	5%	7%
7.	Hemicellulase	-	5%	3%	5%	3%	5%	3%	5%
9.	Oil	-	25%	25%	25%	25%	25%	25%	25%
10.	Batching	-	24 Hours				Overnight		
11.	Combing	-	Combing of the fibers in small bundles						

Sr: Sisal Raw, Shc: Sisal-High % concentrated-Combing, Slc: Sisal-Low % concentrated-Combing, Shcp: Sisal-High % concentrated-Combing-padding mangle, Slcp: Sisal-Low % concentrated-Combing-padding mangle, Shcbc: Sisal-High % concentrated-Combing-Beating-Combing, Slcbc: Sisal-Low % concentrated-Combing-Beating-Combing, Shcbc4: Sisal-High % concentrated-Combing-Beating-Combing (4hrs treatment without changing water)

After the enzyme treatment fibers are washed thoroughly under the running water and immersed in oil emulsion for 10mins. Then, the treated fibers were directly wrapped

into the aluminum foil and kept for the overnight batching treatment. Batching treatment helps in increase the pliability of the fibers by smoothing the texture of the fiber. Finally, the completely treated samples were air dried for 24 hours at room temperature.

All the samples of different processing steps were analyzed through feel and touch test, SEM, bundle fiber strength and by hand twisting the bundle of fibers to check its spinnability. Based on which Shcp and Slcp treatment were eliminated, due to reduction strength and flattening of fibers.

Further, similar enzyme treatment including all the variations in the procedure was applied onto ramie fibers to identify its effectiveness and the details are mentioned in Table 3.5.

Table 3.5: Stepwise enzyme treatment process of ramie fibers

Sr.No.	Steps	Rr	Rhc	Rlc	Rhcbc	Rlcbc	Rhcbc4
1.	Beating	-	-	-	10mins	10mins	10mins
2.	Combing	-	-	-	Combing of the fibers in small bundles		
3.	Pectinase	-	2%	1%	2%	1%	2 %
4.	Laccase	-	10%	5%	10%	5%	10%
5.	Cellulase	-	7%	5%	7%	5%	7%
6.	Hemicellulase	-	5%	3%	5%	3%	5%
7.	Oil	-	25%	25%	25%	25%	25%
8.	Batching	-	24 Hours	Overnight			
9.	Combing	-	Combing of the fibers in small bundles				

Rr: Ramie Raw, Rhc: Ramie-High % concentrated-Combing, Rlc: Ramie-Low % concentrated-Combing, Rhcbc: Ramie-High % concentrated-Combing-Beating-Combing, Rlcbc: Ramie-Low % concentrated-Combing-Beating-Combing, Rhcbc4: Ramie-High % concentrated-Combing-Beating-Combing (4hrs treatment without changing water)

Amongst all the treated sisal and ramie samples, Shcbc, Shcbc4, Rhcbc and Rhcbc4 were selected on the basis of water consumption concept during the process, fiber strength and softness needed for the spinnability. Further, these recipes were experimented using two different lab instruments - Infracolour and Launder-O-Meter machine for bulk and accurate treatment as well as for commercializing the process.

Softening treatment using lab instruments:

Infracolor Machine:

Infracolor machine (Plate 3.5) is a sample dyeing machine installed in R & D section of Colourtex, Surat. The machine was utilized for softening treatment using scoured fibers in form of hanks. Twelve small beakers of onelitre capacity and a beaker which works as a sensor was attached in the center. The sensor monitors the temperature during the process. For the pilot analysis process only the two beakers and a sensor beaker were used. The machine is fully automatic hence, certain data like temperature (55℃), rate (2), speed (100) and time (as per the recipe) was to be inserted before starting the process.

For the enzyme treatment the pH of distilled water was reduced to 5pH by using acetic acid. 50gms of fibers after scouring, beating and combing process were individually wrapped in polypropylene net to prevent fiber entanglements and kept for wetting. As per 1:10 M:L, luke warm water was added into the beaker. Then the application process for softening of the fibers with (Shcbc and Rhcbc) and without (Shcbc4 and Rhcbc4) changing the water was carried out. The detailed softening process of sisal and ramie fibers are mentioned in Table 3.6.

Table 3.6: Softening process of the fibers using Infracolor machine

Sr. No.	Enzyme	% Concentration	Time
1.	Pectinase	2%	15 mins
2.	Laccase	10%	30 mins
3.	Cellulase	7%	45 mins
4.	Hemicellulase	5%	60 mins
5.	Oil emulsion (water + Rice brain oil + Non-ionic detergent)	Oil – 5% Detergent – 25%	10 mins
6.	Batching	-	Over night

During the process with the samples Shcbc4 and Rhcbc4 water was not changed, the enzymes were added into solution and stirred by removing the hank. Washing for the four different samples was carried out before the oil treatment under the running water for deactivating and removal of all the enzymes. Constant rotation with required

temperature was the main advantage in this process, but due to the capacity of the machine bulk treatment was time consuming.

(a)Infracolor machine **(b) Control panel**

Plate 3.5: Infracolor sample dyeing machine and its control panel

Launder-O-Meter:

Launder-O-meter (Figure 3.7) is basically used for the evaluation of wash fastness properties. A mechanical washing device includes a water-bath and a rotor attachment for fixing the beakers. The rotor rotates at the speed of 40+2 revolution per minute and a thermostat to control the temperature of water bath as per the need. Controlled temperature and rotation of the instrument were the main advantage for the experimentation of bulk fiber softening treatment.

Plate 3.6: Polypropylene Net **Plate 3.7:** Launder-O-meter

The pre-treated fiber sample of 500gms each were divided into two and wrapped in a polypropylene net (Figure 3.6) to avoid the entanglements. These bundles of fibers were then fixed either sides of the beaker holders using a plastic string (Figure 3.8), which holds the bundle tightly and withstand the temperature during the process. As the entire

process was executed within the water bath the M:L was constant i.e. 1:40. While, the recipe was the same as Infracolor process (Table 3.6).

Plate 3.8: Bundle of fibers arranged on the rotor panel of launder-O-meter

Both the Infracolor and launder-O-meter treated samples were evaluated by feel and touch test and hand twisting the bundle of fibers to check its spinnability. The bulk and final commercial treatment process were also taken into consideration during the evaluation of treated fiber samples.

Standardized enzyme recipe Shcbc4 and Rhcbc4 using Launder-O-Meter was finalized as commercial process for softening treatment based on fiber analysis. This was also a convenient process for bulk treatment with less of entanglements, proper penetration and activation of enzymes, adequate space for immersion and rotation of the fibers, temperature was maintained and water consumption was also less. Thus, the treatment was carried out for the entire study using this process and maintaining the M:L i.e.1:40, to have proper and equal treatment onto the fibers and the commercial set up for minor fibers was identified.

3.1.5 Assessment of the sisal and ramie fibers

Soften fibers were analyzed and the results further directed to finalize the fiber treatment process for the study. Various tests conducted are as follows:

a. Bundle Strength test

The Sisal and ramie fibers are naturally in bundled form, so it is difficult to separate each fiber. Secondly, with the Beasley strength test the fibers were slipping. So, with the final application for sound absorbing materials, wherein the fibers were used in

bundle form. So, the conventional method of bundle strength test was carried out on Llyod Instron Tensile Test (Plate 3.9) having variable capacity of load application. In this method fibers were hold by the jaws with which the fibers could sustain the force applied on it.

For the testing 10 cm length of fiber were cut and weight to calculate the Denier. The instrument works on the principle of constant rate of extension (CRE). Average of five readings was considered to identify the strength of untreated and treated fibers. Test was conducted using ASTM D 3822, in the Department of Textile Engineering, Faculty of Technology and Engineering, The Maharaja Sayajirao University of Baroda, Vadodara.

Plate 3.9: Llyod Instron Tensile Testing Instrument

b. Fiber fineness

ASTM D 7025 standard was followed to determine the fiber fineness of both the fibers. Tex was calculated by the average weight of 20 readings of 100 cm length of the fiber and using the formula:

$$\text{Tex} = \frac{W \times l}{L}$$

Where, W = Weight of the fiber
l = Unit length of the sample
L = Length of the sample

Fiber fineness was also determined by indirect system of yarn numbering using Beesley's yarn balance. The instrument consists of a hook and a pointer at two ends. The alignment of the balance was checked and adjusted. A standard balance was hung on the notch of the beam. Template was used to cut the length of the fibers based on cotton

count system; the cut fibers were then added on to the hook until the pointer is opposite to the datum line. The count is the number of filaments used to balance the beam.

c. Whiteness Index

Whiteness Index test was determined using CIE and ASTM D 1925 standard on Spectrophotometer instrument. It was used to measure the yellowness of the untreated and treated fibers. Analyses were also done with the naked eye and touch and feel method for determination of colour and texture of the treated fibers were compared with the untreated i.e. raw fibers.

d. Scanning Electronic Microscopy (SEM)

Longitudinal and cross section of both the fibers were analyzed using SEM. Fiber sample was mounted on SEM Stub of 12mm X 12mm diameter using carbon tabs. The samples other than metal are polymeric materials which are very sensitive to electron beam and get charged in electron beam. The charging of the specimen causes artifacts' (Astigmatism) and also focusing problems in the SEM. To avoid charging, the specimen was then coated with thin layer of conducting material such as gold or gold palladium using Sputter Coater for one min. After coating with Au/Pd in sputter coater, the specimen was than scan in Philips XL 30 SEM (Plate 3.10). Using suitable magnification and accelerating voltage, micrographs were recorded.

Cross sectional view of the fibers was also, where the fibers were the bundle of fibers were passed though the tube and fixed it compactly. Then the tube filled with fibers were sharply cut into small pieces and placed on the plate after coating. Carbon tapes were used to mount rest of the area to avoid charging, the specimen was scan using suitable magnification and micrographs were recorded. Both the longitudinal and cross section of the fibers were analyzed at CIRCOT, Mumbai.

Plate 3.10: Scanning Electronic Microscopy instrument (SEM)

Plate 3.11: Fourier Transform Infrared instrument (FTIR)

e. Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR analysis of the untreated and optimized enzyme treated samples was conducted on Shimadzu IR Prestige 21 (Plate 3.11) analyser at Chemistry Lab, Applied Chemistry Department, Faculty of Technology and Engineering, The Maharaja Sayajirao University of Baroda, Vadodara. It was performed to identify the presence of certain functional groups in a molecule. It works on the relationship between time and frequency. The wavelength of 400 to 4000 cm^{-1} which is mid-IR range was kept standard for all the samples.

Sample was prepared by freezing the chopped untreated and optimized enzyme treated fibers of sisal and ramie in liquid nitrogen and pulverizing it to yield a fine powder capable to being cast into traditional KBr pellets for IR analysis. For the analysis the scanning range was from 400 to 4000 cm-1 and also observed at two different Wavenumber i.e. 500 and 1000 for clarity.

f. Energy-Dispersive X-ray Spectroscopy (EDS)

For the identification of elements present in the untreated and optimized enzyme treated samples EDS (Plate 3.12) analysis was conducted at EDS Department, Electrical Research and Development Association (ERDA), Vadodara. The X-rays are emitted from the specimen when bombarded by the electron beam to identify the elemental composition of the specimen. The analysis is done to know if any elements are removed or added and whether the elements are within permissible limits.

Plate 3.12: Energy-dispersive X-ray spectroscopy instrument (EDS)

Plate 3.13: X-ray diffraction instrument (XRD)

g. X-ray Diffraction (XRD)

X-ray diffraction (Plate 3.13) was determined of both the untreated and optimized enzyme treated sisal and ramie fibers. The bundle of fibers was mounted and scanned in Transmission mode 10-40̊. Test was conducted at The Bombay Textile Research Association (BTRA), Mumbai.

3.1.6 Yarn preparation

The untreated and treated fibers were converted into yarns using different spinning techniques depending on the fiber characteristics, twist holding capacity and end use. Sisal being stiffer and less cohesive hand spinning was the only possible technique for yarn preparation. Similarly, ramie fibers were difficult for hand spinning thus machine spinning technique was finalized. Thus, table 3.7 shows yarns spun using different techniques.

Table 3.7: Coding of the different sisal and ramie yarns

Sr.No.	Coding	Description
1	SUT	Sisal, Untreated, Traditional (Hand) spinning technique
2	STH	Sisal, Treated, Hand spinning technique
3	RUT	Ramie, Untreated, Hand spinning technique
4	RTR	Ramie, Treated, Rove spinning technique
5	MC	Mercerized Cotton yarn

a. Hand Spinning

The scoured sisal and ramie fibers were spun using two different hand spinning techniques. Various thickness of yarns was spun by the Kachchh spinner and women of self-help group, Aadhar, a non-governmental organization in Ahmedabad.

100% sisal and 100% ramie yarns were hand spun using drop technique as shown in Plate 3.14. The fibers were separated and aligned for easy plucking of the strands. Then 4-5 strands were bundled and spun using the thumb and first finger. It was a continuous process of spinning and winding of the spun yarn in ball form. Twist was so tight that the even yarn structure was formed.

Plate 3.14: Traditional Drop spinning technique

Another bundle of untreated sisal and ramie fibers were spun using thumb and first finger and then twisting further between the palm (Plate 3.15) by the spinners. As each fiber is different in thickness, approximately 7-8 strands of fibers were spun together. For the continuous yarn, loop was made at the end from which the next strands of fiber were passed and twisted and in this technique of spinning was carried out for the preparation of yarns. Once the spinning was done the protruding fibers were cut using sharp scissors in the dry state and cones were used for winding the yarns as shown in Plate 3.16. While, waste fibers were used for the stuffing of double cloth sample.

Sisal, being a straight and good length fibers were easy to spun compare to ramie. Thus, this spinning technique was finalized and applied further with modification in the process for soften sisal fibers only. Bundle of soften fibers were aligned using metal comb and soaked into water tub to keep the fibers in wet condition. Wet fibers were easy

to pluck, reduced entanglements and increased the strength of the fiber which was essential for pliability.

Plate 3.15: Hand Spinning Technique

Plate 3.16: Handspun Sisal yarns

b. Machine Spinning

Machine spinning of ramie yarn using rove technique was conducted at NIRJAFT, Kolkata. Enzyme treated fibers were kept for overnight drying in the fiber storage room consisting Dehumidifier instrument, which controls the humidity of the storage area. Next day an emulsifier was prepared (by mixing oil, water and plasticizer) which was sprayed on both the sides of the fibers to have a smooth carding process and kept for 36 hours conditioning in a wooden drum under the weight.

After conditioning the fibers were divided into bunches of equal proportion. Bunch of fibers were passed though the flax carding machine to convert it into silver. Silver were laid on the flat bed and bundle of 12 layered silver of required length was again passed under the carding machine to have thin and equally layered silver.

Then the silver was laid and bundle of five layered silver of particular length was inserted into the 1st Jute drawing machine having two rollers and output of fine silver was collected into four canes (division of canes was decided by the expert based on the number of spindle and expected count) and tex of the silver was 19.02kg/tex. Again, five layered silver was prepared. Further again five layered silver was prepared and passed under 2nd Jute drawing machine having two rollers and an output of silvers was collected into two canes. Finally, the silvers were inserted in the rove yarn machine to convert ramie silvers into ramie rove yarn of 1200 tex. The Plate 3.17 shows stepwise fiber to ramie rove yarn preparation.

Enzyme treated ramie fibers

Fibers in Dehumidifier storage area for drying

Spraying of emulsifier

Bundled fibers converted into carded sheet on flax carding machine

Layering and bundle preparation of carded sheet

Re-carding process

Bundles of carded fibers for converting into sliver

Sliver preparation on Jute drawing machine

Layering of sliver for yarn preparation

Sliver inserted in Rove yarn spinning machine

Winding of Rove yarn

Rove yarn

Plate 3.17: Stepwise carding of the fiber, sliver and ramie rove yarn preparation

3.1.7 Assessment of the yarns

Yarns were manufactured using different techniques and thus to determine the quality of fabric in terms of thickness and strength for the sound resistant materials, three different analysis was conducted.

a. Determination of Yarn Count

The ASTM D 7025 standard was followed to determine the fiber fineness of both the fibers. Tex was calculated by the average weight of 20 readings of 100 cm length of the fiber and using the formula:

$$\textbf{Tex} = \frac{\textbf{W X l}}{\textbf{L}}$$

Where, W = Weight of the sample
l = Unit length of the system
L = Length of the sample

The Fiber fineness was also determined by indirect system of yarn numbering using Beesley's yarn balance. The instrument consists of a hook and a pointer at two ends. The alignment of the balance was checked and adjusted. A standard balance was hung on the notch of the beam. Template was used to cut the length of the fibers based on cotton count system; the cut fibers were then added on to the hook until the pointer is opposite to the datum line. The count is the number of yarns used to balance the beam.

Further, Linen count another indirect system of yarn count was also calculated for both all the different yarns to identify the change in yarn count. The formula used:

$$\textbf{N} = \frac{\textbf{L X w}}{\textbf{l X W}}$$

Where, L = Length of the sample
w = unit weight of system
W = Weight of the sample
l = Unit length of the sample

b. Determination of Yarn strength

The tensile strength of the yarns was tested on Llyod Instron Tensile Tester having variable capacity of load application. For the testing the of 10cm length were cut and weight was taken to calculate the Denier. The instrument works on the principle of constant rate of extension (CRE). Average of five readings was considered to identify the strength of untreated and treated fibers. Test was conducted using ASTM D 3822, in

the Department of Textile Engineering, Faculty of Technology and Engineering, The Maharaja Sayajirao University of Baroda, Vadodara.

c. Determination of Yarn twist (TPI - Twist per Inch)

The type and amount of twist was calculated on Alfred Suter twist tester (Plate 3.18). The 10inch length of sample was fixed in the jaws under tension. An average of 10 readings was considered for the analysis. Testing was carried out at the testing lab of Century Rayon Limited, Shahad (Mumbai) under the guidance of Mr. Nishith K. Sheth, Assistant Manager, Tyre cord and continuous spun rayon filament yarns.

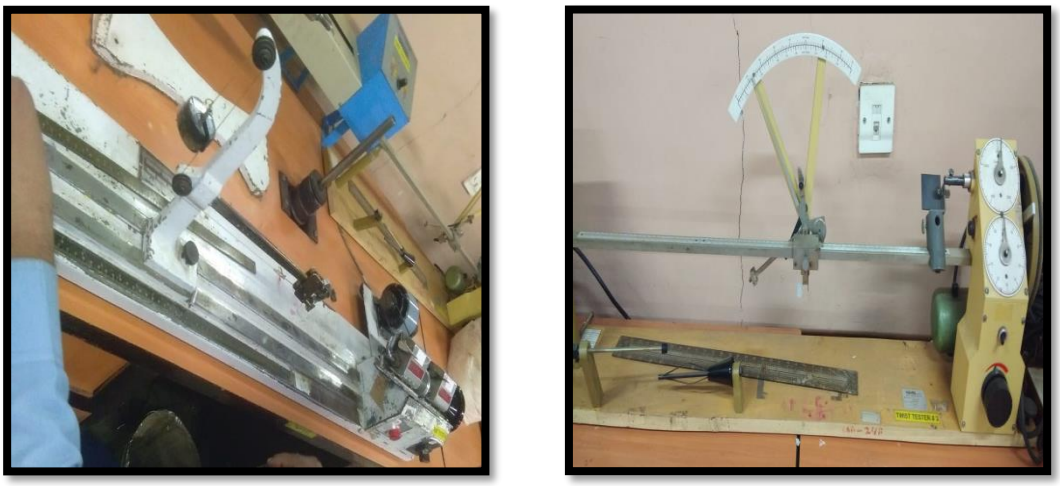


Plate 3.18: Alfred Suter Twist tester

3.2. Phase II: Development of different types of fabrics

Woven and nonwoven fabrics using 100% sisal and 100% ramie yarns were created. The combination of both fabrics will be analyzed for absorption or scattering of the sound passing through it. Both functional and aesthetical purpose were taken care while creating these fabrics. The details of the various fabrics are mentioned in Table 3.8.

Table 3.8: Coding of the fabrics constructed using sisal and ramie

Sr. No.	Sample Code	Description
Plain weave using fibres		
1	S FUP	Sisal Fiber Untreated Plain Weave
2	R FUP	Ramie Fiber Untreated Plain Weave
3	R/S FUP	Ramie and Sisal Fiber Untreated Plain Weave

Untreated Plain Weave		
4	SUP	Sisal Untreated handspun yarn Plain Weave
5	RUP	Ramie Untreated handspun yarn Plain Weave
Treated Plain weave		
6	STP	Sisal Treated handspun yarn Plain Weave
7	RTP	Ramie Treated rove yarn Plain Weave
Broken Twill weave		
8	STBT	Sisal Treated handspun yarn Broken Twill Weave
9	RTBT	Ramie Treated rove yarn Broken Twill Weave
Double Cloth variations		
10	R/S TDC	Ramie and Sisal Treated yarn Double Cloth Weave without stuffing
11	R/S TDC_S	Ramie and Sisal Treated yarn Double Cloth Weave with stuffing
12	R/S TTCs	Ramie and Sisal Treated yarn Tubular Cloth Weave with stuffing
Nonwovens		
13	R_{620}	Ramie needle punch of 620 GSM
14	R_{814}	Ramie needle punch of 814 GSM
15	R_{919}	Ramie needle punch of 919 GSM

3.2.1. Preparation of woven fabrics using various weaves

Basic weaves were explored on table loom (Plate 3.20) of weaving lab, Institute of Fashion Technology, Faculty of Family and Community Sciences, The Maharaja Sayajirao University of Baroda, Vadodara. The samples were analyzed based on the parameters of sound absorbing materials.

Tillberg (2012), stated that acoustic properties of textiles differ depending on how the textile structure is created - through knitting or weaving or other techniques. In the structure of textiles, if small air pockets are formed it will affect the acoustic properties. Thus, three different weaves – Plain, Twill and Double Cloth were explored for the study.

Plate 3.19: Weaving using Fiber Strands

Plate 3.20: Exploration of weaves on table loom

Source (3.19): http://www.dsource.in/resource/palm-fiber-craft-ernakulam-kerala/making-process

a. Plain weave fabrics

Plain weave the most fundamental weave being strong and durable and also used for furnishing fabrics was initially developed using raw and scoured fiber strands (Plate 3.19) and untreated and enzyme treated yarns. The fabric created was less thick with a balanced look and was analyzed for sound absorption. Based on the analysis and needed fabric parameter two more weaves were explored.

b. Broken twill weave fabrics using treated yarns

The uneven structure, design of the fabric, intersection points and compactness of the fabric affects the sound resistance. Hence, the broken twill weave structure was explored with an assumption that the uneven twill will create less open structure. Also, the sound which will strike and pass will dissipate or scatter thereby the resistance will be the resultant.

Both the plain and twill weave fabric samples were developed on handloom by Prakashbhai Siju, carpet weaver (Plate 3.21), Bhujodi, Kachchh. Details of the loom for constructing the plain and broken twill woven fabrics are as follows:

Table 3.9: Details of the Handloom

Type of Loom	**Treadle loom**
Number of Shafts	04
Loom width	32 inches
Reed number	14 (7picks in 1”)

Plate 3.21: Yarn winding and fabric preparation on handloom

c. Double cloth fabrics using treated yarns

Dense and thicker the fabric with an uneven surface, greater the resistance will be. The double cloth weave structure was explored with and without stuffing using combination of both the treated yarns. Also, tubular cloth with the stuffing were also explored under the study to analyze the sound resistance of the materials. The samplers were developed on the table loom having eight shafts by the researcher at the Mumbai Weavers Service Center (Plate 3.22), under the guidance of R.S. Gokhale, Assistant Director (Weaving) and the team.

Plate 3.22: Construction of Double cloth fabric

3.2.2. Preparation of nonwoven fabrics using scoured fibers

Nonwoven was prepared only from the scoured ramie fibers and of different GSM. Carding process of 4kgs scoured ramie fibers were conducted on flax carding machine at NIRJAFT, Kolkata. Carding was done twice to remove all the impurities and

carding waste (87gms) was collected, which will be further used as stuffing material in the preparation of double cloth fabric.

Hand opening of the carded sheet

Sprinkling of the water

Laying of hand opened carded fibers

Web formation

Nonwoven cloth beam

Layering of the nonwoven sheet

Insertion of layered sheet in needle punch machine

Winding of Nonwoven cloth beam

Edge finishing of Nonwoven sample

Plate 3.23: Fiber to nonwoven fabric preparation of various GSM

The machine and the fabric parameters to prepare the nonwovens of various GSM were decided by the NIRJAFT depending upon the softness, properties of the fabrics and based on the end application of the fabric under the study. The pilot needle punch machine having two sets of needle boxes each containing approx. 1200 needles were used for preparing the nonwoven fabrics.

The fibers were passed through the carding machine and further through the doffer the combing process is carried out to prepare a web of 100 GSM. Further, the web was layered up as per the required GSM nonwoven fabrics. After various trials finally three fabrics of different GSM – 620 GSM, 814 GSM and 919 GSM were prepared. The Plate 3.22 shows stepwise process of carding to product finishing of nonwoven.

Specification of nonwoven machine are as follows.

i. Needle – 40/cm width
ii. Stitch density – 90/cm (approx.)
iii. Advance stroke (up and down) – 4.5/mm
iv. Feed take up – 0.40mt/min (i.e. conveyor belt where the fibers were feeded)
v. Doffer speed – 19.9mt/min (provides the carded fibers)
vi. Cross lapper speed – 20.2mt/min
vii. Feeding card – 550 gm/mt^2 (approx.)

3.2.3. Assessment of the prepared fabrics

All the woven and nonwoven fabric samples were analyzed for the application of the same as sound resisting materials for indoor application. The various tests are as follows:

a. *Determination of fabric weight per unit area*

Sample of 5*5 cm were cut, weighted and calculated using formula.

$$\textbf{GSM} = \frac{\text{Weight in grams of sample X 100 X 100}}{\text{5 X 5}}$$

b. Determination of fabric count (EPI & PPI)

Fabric count (the number of yarns per inch), determines the closeness of the weave structure. ASTM D 3775-98 fabric count was analyzed by counting number of threads in one inch, weft and warp direction using pick glass. Average of 10 readings was taken.

c. Determination of fabric thickness

Using ASTM test method D1777-96, fabric thickness was measured by compressometer thickness tester. The fabric sample was placed between the anvil and

pressure foot, thickness was indicated by the needle on the dial of the gauge was recorded. Average of 10readings was recorded as the final reading.

d. Determination of the air-permeability of sound absorbing materials

Air permeability or air flow resistance is one of the important parameters of sound absorbing materials. Friction will be created within the material which will help the sound to dissipate and thus absorption takes place. It is the measure of the resistance of fabrics for passage of air through them, more the passage of air means less sound absorption. The Air-Permeability test was conducted in the Department of Textile Engineering, Faculty of Technology and Engineering, The Maharaja Sayajirao University of Baroda, Vadodara. Average of three readings was taken and reported in m3/mt2/m to determine air permeability of sound absorbing materials.

e. Cover Factor

Cloth cover, a measure of the fraction of area covered by both the warp and weft threads in a given fabric was calculated using thread count and yarn number (cotton count number) for respective fabric. Equation for calculating cloth cover

Cloth cover = Cover factor warp + Cover factor weft – [(Cf warp X Cf weft)/28]

Where, Cover factor (Cf) = threads per inch/√Yarn number

Although this formula for estimating cloth cover was develop for plain weave cotton fabrics, it was applied to all cellulosic fabrics of various weave structure under the study.

f. Anti-microbial and anti-fungal activity

The natural resin finished fabrics samples were analyzed by Choksi Lab, Makarpura, Vadodara for identifying the quantitative growth of microbes and fungi using the test method - USP 42,61 “Microbiological examination of nonsterile products: Microbial enumeration tests”. The test was conducted using agar method and the procedure was:

1) Preparation of culture medium - Positive control used E. coli culture (100 CFU)
2) Sterilization of the samples - 121C, 15LBS for 15 Min.

3) Inoculation: - 100 ml soyabean case in digest medium
4) Incubation: - Bacterial count - 72 Hours
   Yeast and mold count - 5 Days

After incubation, a clear area of interrupted growth underneath and along of the test material indicates antimicrobial activity of the specimen (Plate 3.24).

Plate 3.24: Antimicrobial testing instrument

Plate 3.25: 45°Flammability tester

g. Flammability Test

The 45° flammability test was conducted on the selected fabric samples as per ASTM D 1230-45 on 45° Flammability Tester (Plate 3.24), Paramount, Digi-Flame Tester. The specimen size was 15cm length and 5cm wide, mounted in the sample holder and place in 45° angle. Then the sample was exposed to a standard flame 15 sec. Flame speed time was observed and recorded.

h. Sound absorbing coefficient (dB)

All the developed woven and nonwoven samples were cut on laser cutting machine (Plate 3.25) as per the sample holder's shape and size. Laser cutting technique was used to cut the samples so as to have exact shape and size, thereby sound leakage could be avoided. Sound absorption coefficient was recorded at various range of frequencies i.e. 1000-2200Hz using ToneGen software and at different distance right from 0cm i.e. sample next to the source till 80cm.

Initially an empty reading was taken i.e. without the sample and then with the samples. An average of 25 readings was generated by the software in dB and was saved

in excel sheet to calculate the sound absorption coefficient (dB). Further the average readings were plotted on the graph. The mean and standard deviation was statistically analyzed. Sound reduction (dB) was calculated using formula:

Sound reduction by fabric (dB) = $dB_{Er} - dB_{Sr}$

Where, dB_{Er} = Empty reading i.e. without the fabric sample

dB_{Sr} = Reading with the fabric sample

Plate 3.26: Laser cutting machine and process

i. Noise Reduction Coefficient (NRC)

Noise reduction coefficient at each frequency were obtained and analyzed. Hence further calculations were done using Microsoft excel for calculating per cent noise reduction coefficient (NRC) using formula given by Ghilahare. A & Pandey. M. (2017):

$$\mathbf{C} = 1 - 10^{-(dB/20)}$$

Where, C = Noise reduction coefficient of the sample

dB = Sound reduction of the fabric sample

3.3. Phase III: Sound absorbing instrument, finishes and analysis

To analyze the sound absorbing coefficient and noise reduction coefficient sound absorbing instrument was fabricated for the study. Natural resin was applied on the best resultant combination of samples to have a smooth finished product, which were further analyzed for sound reduction.

3.3.1 Fabrication of sound absorbing instrument for testing

Sound absorption testing of the fabrics constructed were tested on the fabricated Sound absorbing instrument based on ASTM E 1050 with needed modifications for the

study. Fabricated tool and instruments were installed in the Department of Clothing and Textiles, Faculty of Family and Community Sciences, The Maharaja Sayajirao University of Baroda, Vadodara.

Principle:

The instrument will test the absorption of direct sound by the fabric. Where the sound wave of required frequency moves straight towards the fabric sample and partially penetrates through it, which is sensed by the dB meter.

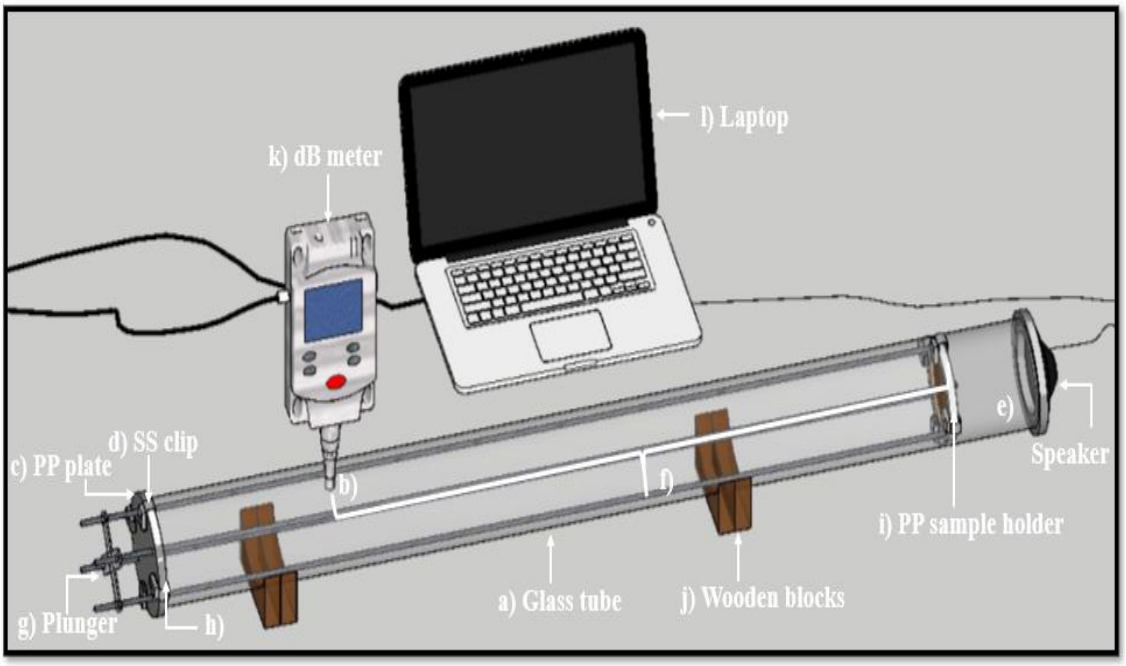
Functioning:

The sound passes from one end of the cylindrical glass tube using speaker i.e. source and on the other end it tightly closed. The mounted sample can be adjusted as per the need. The schematic diagram (Figure 3.2) of the instrument was designed using google sketchup software.

The fabricated instrument is based on the direct and constant frequency. For complete functioning of the instrument, two different software's were installed in the laptop - Tone Generator 100-15 kHz and Soundlab. Sound of a particular wavelength was passed from one end of the cylindrical glass tube though the speaker was fixed and by using the Tone Generator 100-15 kHz software. The sound in form of plain waves travels within the tube and hits the mounted sample at a particular distance where it is partially absorbed and due to scattering partially it penetrates though the sample. At the other end a detachable disc on four iron rods is used for compact closure.

The laser cut fabric sample was kept movable hence were mounted between two hollow PVC rings which were on four iron rods. The purpose of keeping the sample movable and adjustable was to measure the sound absorption at various distances as well as to increase the number of layers for maximum absorption.

Digital dB meter having sensor was placed near to the other end which detects the amount of sound passed though the sample. Further, the dB meter was attached with the laptop wherein the fluctuation of the wavelength in frequency was noted using the Soundlab software, as shown in plate 3.27.



a. ***Cylinder glass tube****: 1100mm, diameter 115mm (OD) & 105mm (ID)*

b. ***Hole for dB meter****: 15mm at 100mm distance*

c. ***PP Plate****: 132mm diameter, 10mm thickness and 4 holes for rods at 90mm distance*

d. ***SS Clip****: 132mm (dB meter side), 98mm (speaker side)*

e. ***Shaped Glass tube****: after 1000mm till end with 79mm diameter (speaker side)*

f. ***Length after db towards reducing glass tube section****: 900mm*

g. ***Plunger****: (4 MS moving rods) 1190mm Length, 6mm diameter, (2 MS strips for as plunger holder): 100mm length, 6mm diameter holes at 90mm distance*

h. ***Rubber sheet****: (dB meter side) OD 132mm & ID 30mm, (speaker side) -OD 98mm & ID 30mm (Note: Black colour rubber packing between glass tube and pp circle)*

i. ***PP sample holder:*** *OD 130mm, ID 30mm, 10mm thickness*

j. ***Wooden blocks:*** *Length – 115mm with curve at one side for support, height – 70mm, width 20mm*

k. ***dB meter***

l. ***Laptop***

Figure 3.2: Schematic diagram of sound absorbing instrument

Plate 3.27: Fabricated Sound Absorbing testing instrument

3.3.2 Selection and application of natural resin on the woven fabrics

The three woven fabrics given best absorption results were further treated with resin. Britacel Silicones Ltd., Mumbai has used their own natural resin and the application process were also conducted by them. Application of Midori EC 2.1 300GPL resin solution was done using padding mangle (Plate 3.28) having two bar mangle pressure, 65% pick up and was dried at 150 ̊C. Table 3.10 mentions about the coding of resin finished samples and its details.

Table 3.10: Coding of resin finished (selected) fabric samples

Sr. No.	Sample Code	Description
1.	SUP_R	Sisal Untreated handspun yarn Plain Weave Resin finished
2.	$RTBT_R$	Ramie Treated rove yarn Broken Twill Weave Resin finished
3.	$RS\ FUP_R$	Ramie and Sisal Fiber Untreated Plain Weave Resin finished

Another trial was also conducted by them using Cashew Nutshell Liquid (CNSL), but conversion of oil into resin and its application on the samples showed poor results and thus it was eliminated.

Plate 3.28: Resin application using padding mangle

3.3.3 Evaluation of the resin finished fabrics

The resin finished fabrics were evaluated to identify the changes on the surface of the fabric after the treatment and changes in the sound absorbing due to it.

a. Determination of surface characteristics of the fabric samples using scanning electron microscope.

SEM analysis was performed to analyze the surface morphology of the raw and resin finish woven samples. The samples were observed under scanning electron microscope and SEM images were obtained. Only the three best sound absorbing samples – Ramie Treated Broken Twill Weave (RTBT), Sisal Untreated Plain Weave (SUP) and Ramie & Sisal Fiber Untreated Plain Weave (R/S FUP) were analyzed. The weave structure, porosity and surface modifications were the main parameters for the observations. Before testing, the samples were sliced as per the need and to make it conductive, sputtering for few minutes depending on the sample thickness was done. Micrographs were recorded at different magnifications to ensure the clear images. Analysis was conducted at SEM Department, Electrical Research and Development Association (ERDA), Vadodara.

b. Sound Resistant (dB)

Three resin treated woven samples were analyzed for Sound resistant (dB) to identify the change after the application of resin onto it. The testing was carried out at 1400Hz and the procedure was same as mentioned in section 3.2.2 (h).

c. Noise Reduction Coefficient (NRC)

Noise reduction coefficient at 1400Hz frequency were obtained and analyzed of all the three resin treated samples using the formula which is mentioned in section 3.2.2 (i).