CHAPTER 3
MATERIALS AND METHODS

3.1 INTRODUCTION

This chapter discusses three different lignocellulosic fibres (banana, coir and sisal) used as reinforcement in preparing biocomposites. Recycled paper was chopped and pulped to give a density of approximately 650 kg/m$^3$ for the matrix. Methodology for the production of the biocomposites, experiment design and tests carried out for the physical, mechanical properties and sound absorption coefficient are discussed here.

3.2 MATERIALS

3.2.1 Fibre Selection

Fibre parameters that were used in this research include type of fibres, and their blend ratio with recycled paper pulp. The fibres used in the experiments include cotton, scoured cotton, jute, coir, banana and sisal. Samples were made from these fibres and tested for sound absorption. Sound absorption coefficients of these materials are listed in the Table 3.1.

From the above test results, it was concluded that coir, sisal and banana fibres have better sound absorption properties when compared to cotton, scoured cotton and jute. Hence only banana, coir and sisal fibres were chosen for further development with varied blend ratios of these fibres in further samples.
Table 3.1 Sound absorption values of Samples

<table>
<thead>
<tr>
<th>Fibre used with paper pulp</th>
<th>Sound Absorption (dB)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cotton</td>
<td>20.4</td>
</tr>
<tr>
<td>Scoured cotton</td>
<td>21.2</td>
</tr>
<tr>
<td>Jute</td>
<td>26.3</td>
</tr>
<tr>
<td>Coir</td>
<td>31.1</td>
</tr>
<tr>
<td>Banana</td>
<td>29.5</td>
</tr>
<tr>
<td>Sisal</td>
<td>30.2</td>
</tr>
</tbody>
</table>

3.2.2 Reinforcement and Matrix

The banana, coir and sisal fibres with average cut length of 1.5 cm, 2.5 cm and 3.5 cm respectively, were sourced locally (Erode – Gobichettipalayam, Tamilnadu, South India) and used for reinforcement. Recycled paper was chopped and pulped to give a density of approximately 650 kg/m$^3$ as the matrix.

3.3 PRODUCTION OF THE BIOCOMPOSITES

Banana, coir and sisal-fibre reinforced paper pulp biocomposite blocks of 30 cm$^2$ with varying thicknesses (2, 4 and 6 cm) were prepared. Samples with average cut fibre lengths (1.5 cm, 2.5 cm and 3.5 cm) and fibre volume fractions (0.15, 0.20 and 0.25) were prepared (Table 3.2 and 3.3). Box and Behnken experimental design for three variables was used as the basis for producing the samples (Debnath et al. 2000 & Douglas et al. 2001). The three levels for the chosen variables are given in Table 3.2 and the experimental combinations for producing the samples are given in Table 3.3.
Table 3.2 Actual levels of corresponding to coded levels

<table>
<thead>
<tr>
<th>Variables</th>
<th>Code levels</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>-1</td>
</tr>
<tr>
<td>Fibre Average cut length, cm (X1)</td>
<td>1.5</td>
</tr>
<tr>
<td>Composite thickness, cm (X2)</td>
<td>2</td>
</tr>
<tr>
<td>Fibre Volume fraction, V_f(X3)</td>
<td>0.15</td>
</tr>
</tbody>
</table>

Table 3.3 Box and Behnken Experimental design for three variables

<table>
<thead>
<tr>
<th>Sample. No.</th>
<th>Average cut length, cm (X1); -1</th>
<th>Composite thickness, cm (X2); 0</th>
<th>Fibre volume fraction (X3); 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>1.5</td>
<td>2</td>
<td>0.20</td>
</tr>
<tr>
<td>S2</td>
<td>1.5</td>
<td>6</td>
<td>0.20</td>
</tr>
<tr>
<td>S3</td>
<td>3.5</td>
<td>2</td>
<td>0.20</td>
</tr>
<tr>
<td>S4</td>
<td>3.5</td>
<td>6</td>
<td>0.20</td>
</tr>
<tr>
<td>S5</td>
<td>1.5</td>
<td>4</td>
<td>0.15</td>
</tr>
<tr>
<td>S6</td>
<td>1.5</td>
<td>4</td>
<td>0.25</td>
</tr>
<tr>
<td>S7</td>
<td>3.5</td>
<td>4</td>
<td>0.15</td>
</tr>
<tr>
<td>S8</td>
<td>3.5</td>
<td>4</td>
<td>0.25</td>
</tr>
<tr>
<td>S9</td>
<td>2.5</td>
<td>2</td>
<td>0.15</td>
</tr>
<tr>
<td>S10</td>
<td>2.5</td>
<td>2</td>
<td>0.25</td>
</tr>
<tr>
<td>S11</td>
<td>2.5</td>
<td>6</td>
<td>0.15</td>
</tr>
<tr>
<td>S12</td>
<td>2.5</td>
<td>6</td>
<td>0.25</td>
</tr>
<tr>
<td>S13</td>
<td>2.5</td>
<td>4</td>
<td>0.20</td>
</tr>
<tr>
<td>S14</td>
<td>2.5</td>
<td>4</td>
<td>0.20</td>
</tr>
<tr>
<td>S15</td>
<td>2.5</td>
<td>4</td>
<td>0.20</td>
</tr>
</tbody>
</table>
The recycled paper was chopped and ground using water to ease pulp formation. The excess water was drained from the pulp using fine nylon woven fabric. The fibres were cut to required length. After getting a homogeneous mix of the fibres and recycled paper pulp, the mixture was transferred to a mould and 25 kg/cm² pressure was applied to get the desired size of the biocomposite blocks (30 cm³). The specimens were removed from the mould after 24 hours and then kept at room temperature for one week to dry completely. The process flow chart for the production of above mentioned biocomposites has been given in Figure 3.1. Physical (density and porosity) (Jayaraman 2005), mechanical (ASTM D 1037-99) and sound absorption properties (Young et al. 2007) were measured according to the relevant standards. The biocomposite blocks were then conditioned at 25°C and 65% RH for 24 hours before testing.

Figure 3.1 Process of lignocellulosic fiber reinforced recycled paper pulp biocomposite specimen preparation
3.3.1 Parameter Selection for Manufacturing the Biocomposites -
Justification

The biocomposite parameters that were selected in this research include fibre volume fraction ($V_f$), fibre cut length and composite thickness. Other natural lignocellulosic fibres have been researched by scholars (Kazragis et al. 2002, Han-Seung Yang et al. 2004, Jayaraman 2005, Crocker & Arenas 2007, Hoda 2009, Jorge et al. 2010, Ismail et al. 2011, Kim et al. 2012, Sargianis et al. 2013, Noura et al. 2014 & Pickering et al. 2016), who found the optimum parameters for sound absorptive biocomposites were 0.15, 0.20 and 0.25 for fibre volume fraction; 1.5 cm, 2.5 cm and 3.5 cm for fibre cut length and 2 cm, 4 cm and 6 cm for composite thicknesses. These guidelines were used for designing further experiments.

The above selected parameters influence the modulus of rupture and internal bonding of fibre boards. Any decrease in the use of short fibre does not lead to any further improvement of bending strength. In addition, the effect of fibre length is inversely proportional to sound absorption; the longer (1-6 cm) is the fibre, the lighter the board and the higher the sound absorption. Han-Seung Yang et al. (2004) proved that test samples prepared from composite board containing 30 wt. % of random cut rice straws, without size screening have good acoustic insulating properties over a wide frequency range (125-8000 Hz), because of the larger pores (0.8 porosity). The bending strength and sound absorption is increased when the rice straw particle length and content were increased. The higher thickness (6 cm) of the porous surface has a large effect on sound absorption in terms of frequency. Generally thicker and porous surfaces provide better sound absorption in lower frequency range (250-1000 Hz).
Numerous studies (Koizumi et al. 2002, Jayaraman 2005, Hoda 2009, Young et al. 2007 & Teli et al. 2007, Jorge et al. 2010, Mohammad 2010, Mohammad et al. 2011) that dealt with sound absorption in porous materials have concluded that low frequency sound absorption has a direct relationship with thickness. The effective sound absorption of a porous absorber is achieved when the material thickness is about one tenth of the wavelength of the incident sound. Peak absorption occurs at a resonant frequency of one quarter wavelength of the incident sound. A study showed the increase of sound absorption only at low frequencies, as the material gets thicker. However, at higher frequencies thickness has insignificant effect on sound absorption. When there is air space inside and behind the material, the maximum value of the sound absorption coefficient moves from the high to the low frequency range.

The density and porosity of the fibre board depends on the three different selected parameters. The number of fibres per unit area increases when the apparent density is large (Hoda 2009, Young et al. 2007 & Teli et al. 2007). Energy loss increases as the surface friction increases, thus the sound absorption coefficient increases. Less dense and more open structure absorbs sound of low frequencies (500Hz). Denser structures perform better for frequencies more than 2000Hz. To allow sound dissipation by friction, the sound wave has to enter the porous material. This means, there should be enough pores on the surface of the material for the sound to pass through and get dampened. In designing a material to have a high sound absorption coefficient, porosity should increase along the direction of propagation of the sound wave. From the above discussion, following conclusions can be drawn:

- Higher fibre volume fraction and cut length give better sound absorbency due to more pores and higher number of fibre to fibre contact points.
• Thicker, less dense and more open structures, absorb sounds of low frequencies (500Hz). Higher density structures perform better for frequencies above 2000 Hz.

• Increase in porosity value results in better sound absorption

3.4 PHYSICAL PROPERTIES

3.4.1 Bulk density ($\rho_b$)

Bulk density ($\rho_b$) was calculated by using the following relationship:

$$\rho_b = \frac{W}{t} \text{kg/m}^3$$  \hspace{1cm} (3.1)

where, $W$ is the weight per unit area, $t$ is the thickness determined as per ASTM D 3776.

3.4.2 Average Absolute Density and Porosity

Average absolute density of the samples were calculated using the formula (Jayaraman 2005),

$$\rho_a = \frac{(P_f D_f + P_p D_p)}{(P_f + P_p)} \text{kg/m}^3$$  \hspace{1cm} (3.2)

$P_f$ and $P_p$ are the % blend proportion of fibre and recycled paper respectively; $D_f$ and $D_p$, the absolute densities of fibre (1350, 1150 & 1450 kg/m$^3$ for banana, coir and sisal fibres respectively) and used newspaper (650 kg/m$^3$) respectively. Each value represents the average of five samples.
where, $\rho_a$ is the weight average absolute density of biocomposite blocks (kg/m$^3$)

Porosity ($H$) is calculated using the following equation (Jayaraman 2005):

$$H = 1 - \left( \frac{\rho_b}{\rho_a} \right)$$

(3.3)

Average of five samples was taken for results and discussion.

### 3.5 MECHANICAL PROPERTY

After preparation of the biocomposites samples were conditioned at 25$^0$C and 65% RH for 24 hours before testing. Three-point bending strength was determined by using a Universal Testing Machine (TUE-C-1000) as per ASTM D 1037 (Fig. 3.2). A strain rate 10 mm/min was applied to the test piece to measure the maximum load ($P$) before failure. The bending strength of individual test pieces was calculated from the formula shown below (Han-Seung et al. 2004):

$$\text{Bending strength (N/mm}^2) = \frac{3P_mL}{2bt^2}$$

(3.4)

where,

- $p_m$ - Maximum load, N
- $L$ - Span length, mm
- $b$ - Width of test piece, mm
- $t$ - Thickness of test piece, mm

Average of five samples was taken for results and discussion.
3.6 SOUND ABSORPTION COEFFICIENT (SAC) TESTING (ASTM C 423)

The sound absorption coefficient (α) of a material is the fraction of incident sound energy the surface absorbs or otherwise does not reflect. This value is used to select or recommended the materials in noise control applications. Where low or very high frequencies are involved, it is usually better to compare sound absorption coefficients (Yang 2003). Tests were carried out as per ASTM C 423 (Fig. 3.3 & 3.4). In this method, a loudspeaker (Larson-Davis 900 B preamp), a microphone (omni-directional with a flat within 1/3 band, Larson-Davis 0.5 inch, model 2559), a sound pressure level meter (real time spectrum analyzer, Larson-Davis 3200 series) and reverberation chamber are used. The chamber’s air temperature and humidity are maintained at 21° C and 50 % RH respectively.
Figure 3.3 Audio processing laboratory set-ups

Figure 3.4 Photograph of the specimen made for reverberation time test.

The 1/3 octave band noise at 90 dB, was used as a test signal and was turned on long enough for the sound pressure level to stabilize. The reverberation chamber method (ASTM C 423) was used to measure sound absorption of the biocomposite boards as shown in Figure 3.4. The frequency values, low (125 Hz & 250 Hz), lower middle (500 Hz), upper middle (1000 Hz) and high (2000 to 4000 Hz) were selected which lie in the normal speech range. The sound absorption of the reverberation chamber was measured before the test specimen was installed. This measurement shall be referred to as the empty room test. Test specimen (Fig. A6) was placed directly against
the test surface of the reverberation room with the absorptive side exposed to the sound field. The perimeter of the sample was sealed to the cubical box edges with duct tape. The sound absorption test was then re-run. The absorption measurement with the specimen inside the chamber shall be referred to as the full room test.

For the empty and full room tests, ten decay measurements were conducted at each of the five microphone positions. The sound absorption test was conducted at 1/3 octave band frequencies ranging from 125 to 4000 hertz. The air temperature and relative humidity conditions were monitored and recorded during the empty and full room measurements.

The sound absorption was calculated using the following equation (ASTM C-423):

\[
\text{Sound absorption} (\alpha) = \frac{(A_2 - A_1)}{S} \tag{3.5}
\]

The Sound Absorption Coefficient (SAC) is the full room absorption \( A_2 \) in \( m^2 \) minus the empty room absorption \( A_1 \) in \( m^2 \) divided by the area of the sample \( S \) in \( m^2 \). The Sound Absorption Coefficient is dimensionless.

The Noise Reduction Coefficient (NRC) rating is the arithmetic average of the sound absorption coefficients at 125, 250, 500, 1000, 2000 and 4000 Hz. Noise Reduction Coefficient (NRC) is measured by over a frequency range of 125-4000 Hz as shown below:

\[
\text{Noise Reduction Coefficient (NRC)} = \left( \frac{a_{125} + a_{250} + a_{500} + a_{1000} + a_{2000} + a_{4000}}{6} \right) \tag{3.6}
\]

Average of five samples was taken for results and discussion.
3.6.1 Actual Testing Procedure

The sound absorption measurements were done by using reverberation method with the aid of sound level meter, computers, clamping devices and audio systems. Effect of fibre, Thickness and Blend ratio of fibres on sound absorption were studied with graphs and reported in this research.

A cubical box of dimension 25cm x 16cm x 15cm is taken (Fig 3.4). The sample to be tested is loaded such that it covers all the faces of the cube except one side. The side without sample will be facing the speakers. On the opposite side will be the sound level meter (decibel meter) to receive the sound waves after it gets absorbed by the composite sample. Before starting the experiment the ambient noise in the room is measured. Readings are taken for various frequencies from 125 Hz to 4000 Hz with and without sample. The readings are tabulated and graphs are generated for the study.

3.7 EXPERIMENTAL DESIGN AND EMPIRICAL MODEL

Design of Experiments (DOE) is a set of techniques that revolve around the study of the influence of different variables on the outcome of a controlled experiment (Douglas et al. 2001). Generally, the first step is to identify the independent variables or factors that affect the product or process, and then study their effects on a dependent variable or response.

Experimental design methodology is a very economic way for extracting the maximum amount of complex information, leading to a significant saving in experimental time, material used for analysis and costs (Kwak 2005). The objectives of this study are to establish the functional relationships between the selected variables (fibre cut length, volume fraction and composite thickness) on acoustical properties for three different lignocellulosic fibres. In the following sections, the Box–Behnken with
regression model has been used for the design of experiments and for modeling the effects of selected variables in terms of three different lignocellulosic fibres.

Box and Behnken (Debnath et al. 2000, Douglas et al. 2001, Aslan & Cebeci 2007) experimental design for three variables was used as the basis for producing the samples. The three levels for the chosen variables are given in Table 3.2 and the experimental combinations for producing the samples are given in Table 3.3 An empirical model of multiple linear regression equation was derived to predict the acoustic properties of the samples produced using Box and Behnken experimental design (Debnath et al. 2000). To correlate the effects of factors and the response, the following multiple linear regression model is considered:

\[ y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \epsilon \]  

where, \( y \) represents the responses and \( \beta_0, \beta_1, \beta_2, \beta_3 \) are the coefficients of the selected factors.

### 3.7.1 Justification of the Proposed Regression Model

In most of the real world problems, the optimum values of the parameters and the error variance will not be known, and they must be estimated from sample data. Regression analysis is a collection of statistical tools for finding estimates of the parameters in the regression model. Then the fitted regression equation or model is typically used in prediction of future observations, for estimating the mean response at a particular level of selected parameters. A regression equation is an empirical model relating a response variable to one or more regressions (Douglas et al. 2001).
The performance of many sound absorption materials is usually available in literature. In addition the number of products, which lead to a low noise level, increases daily. Products made from natural materials are already available on the market for noise control applications. The physical properties of most of them have not been entirely determined (Connelly & Hodgson 2015). The previous researchers used multiple linear regression models successfully to study the acoustical characteristics of the absorbing materials. Although the independent variables of most statistical methods for predicting sound absorption are the specific flow resistivity, porosity and density (Miki 1990, Shoshani & Yakubov 1999, Yakir & Yakov 1999, Del Rey et al. 2007), the model presented here considers alternate parameters: fibre volume fraction, cut length and composite thickness. However, there is little published literature which hypothesizes or reports the relationship between the properties of biocomposite substrates and their acoustical characteristics on the sound absorption of substrates.

The intention of this study is the characterization of biocomposite systems using a statistical model based on multiple linear regressions. The data for the statistical model were obtained through experimental tests. Besides, the predicted results were compared to the experimental values, which consider only three successive independent variables: fibre volume fraction, cut length and composite thickness. First, some materials were tested in reverberation chamber and impedance tube methods in order to obtain their acoustic characteristics. Second, statistical models based on multiple linear regressions were used to predict the sound absorption coefficient of different biocomposites. Third, the experimental values were compared with the developed statistical model and their accuracy verified.